

# The Value of Art – Studies in the Material Character of the Terracotta Figurines of the Nok Culture of Central Nigeria

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**THE VALUE OF ART**  
**Studies in the Material**  
**Character of the Terracotta**  
**Figurines of the Nok Culture**  
**of Central Nigeria**

**Christina Beck**



The job of the artist is always to deepen the mystery.

Francis Bacon (1561-1626)



# Contents

List of Figures	IX
List of Tables	XV
List of Appendices	XV
Acknowledgements	XIX

## **A Research Question and General Background**

1 Introduction	3
2 Nok Culture Research Histories: Beginnings and New Research	7
2.1 The Discovery and Early Investigation of the Nok Culture in the 20 <sup>th</sup> Century	7
2.2 New Research: The Frankfurt Project	12
3 Geology and Geochemistry of Tropical Soils	19
3.1 General Background and Environment	19
3.2 Starting Point: The Underlying Bedrock and the Overlying Parent Material	20
3.3 Chemical Weathering and the Developing Soils	24
3.3.1 Chemical Weathering	24
3.3.2 Soils	25
3.3.3 Clay Minerals and Trace Elements	27

## **B Scientific Materials Analysis – Methods**

4 Mineralogical Analysis: Materials and Results	33
4.1 The Importance of Clay and Temper for the Manufacture of Ceramic Objects	33
4.2 Materials and Method	35
4.3 Results of Microscopic Analysis	38
4.3.1 Clay Matrix	38
4.3.2 Temper	40
4.3.3 Manufacturing Technique	44
4.4 Summary	46

5	Geochemical Analysis: Methods and Materials	47
5.1	X-ray Fluorescence – Method	49
5.1.1	Operating Principle	49
5.1.2	Device Parameters	51
5.1.2.1	Sensitivity	51
5.1.2.2	Depth of Information	52
5.2	Measurement Prerequisites	53
5.2.1	Calibration and Comparison of WD-XRF and ED-XRF	53
5.2.2	Measuring Procedure	55
5.3	Multivariate Statistics: Method and Data Preparation	60
5.3.1	Principal Component Analysis – Method	61
5.3.2	Preparation of the Data prior to Analysis	64
5.4	Sample Selection	66
5.5	Side Note: Clay Procurement and Processing in Central Nigeria Today	69

## **C Results of Scientific Materials Analysis**

6	Results of Geochemical Analysis (Terracotta and Pottery) for Selected Sites	79
6.1	Ido (ID)	79
6.2	Ifana (IFA)	88
6.3	Ungwar Kura (UK)	95
6.4	Summary	100
7	Overall Results of the Geochemical Analysis (Terracotta and Pottery) and Comparison to Recent Clay Samples	103
7.1	Comparison of the Chemical Composition of the Terracotta and Pottery from 43 Sites	103
7.2	Comparison of the Chemical Composition of the Recent Clay Samples to the Terracotta and Pottery	108
7.3	Comparison of the Results of the Materials Analysis of the Terracotta and Pottery to a Forgery	115
7.4	Summary	118
8	Central or Local Production? Evidence of the Scientific Materials Analysis	119
8.1	The Geological Origin of the Modern Raw Clay Sources	119
8.2	The Use of Special Clay Sources in Manufacturing the Figurines	122

8.3 Processing of the Clay and its Effects on the Chemical Composition	125
8.4 Random or Systematic Measurement Errors	130
8.5 Summary	131
<b>D Theoretical Considerations</b>	
9 Complexity, Specialisation, Value? Theoretical Approaches and their Application for the Nok Culture	135
9.1 Complexity: Vertical versus Horizontal Hierarchies and the Concept of Ritual Power	135
9.2 Craft Specialisation without Centralisation?	139
9.3 The Value of Art	143
<b>E Summary and Outlook</b>	
10 Terracotta and Beyond: Further Prospects of the Geochemical Analysis	149
Bibliography	155
Appendices	169





## List of Figures

1. Map showing the location of archaeological sites discovered by Bernard Fagg and others during the 20<sup>th</sup> century (green dots) (based on Fagg 1990; amended by the Frankfurt project). 9
2. Map showing the distribution of all archaeological sites recorded by the Frankfurt project (2005-2013) (map by Eyub F. Eyub). 15
3. Summed probability density of the radiocarbon dates obtained by the Frankfurt project (OxCal4.2, IntCal13, 95.4% probability). 17
4. Geological map of the Kaduna plains, Kaduna State, Nigeria (WALL 1979; Text Map 5.8). 21
5. Soil profile in the Nok Valley with blue-grey clay at the bottom. Length of scale: 3 m. Photo taken in 2009. 23
6. **Soil profile near Nok village. Layers of grey clay between layers of red laterite.** Photo taken in 2009. 26
7. Map showing the archaeological sites for which thin section samples are available. The box in the centre indicates the project's key study area with the research station (blue triangle) (map by Eyub F. Eyub). 36
8. Reference chart for the estimation of the percent by volume of the particles in the thin sections (after MATTHEW ET AL. 1991: 218). 37
9. Reference chart for the estimation of the degree of sorting in the thin sections (after ORTEN ET AL. 1993: 239, Fig. A.6). 37
10. Reference chart for the estimation of the degree of roundness (shape) of the particles in the thin sections (after ORTEN ET AL. 1993: 239, Fig. A.5). 38
11. Amount of small (< 0.05 mm) quartz particles in the matrix (terracotta); red line: 3%. Samples sorted by catalogue number of sites. 39
12. Amount of small (< 0.05 mm) quartz particles in the matrix (pottery); red line: 4%. Samples sorted by catalogue number of sites. 39

13. Total amount of tempering material in the pottery; red line: 20%. Samples sorted by catalogue number of sites.	41
14. Total amount of tempering material in the terracotta; red line: 25%. Samples sorted by catalogue number of sites.	41
15. Comparison of granite (blue) and quartzite (red) in the temper of 70 thin sections of pottery (P) and terracotta (T). Samples sorted geographically from West to East.	43
16. Thin section of a pottery sample from Ungwar Kura (2007/1) with oriented pores reflecting the manufacturing technique. Plain polarised light.	45
17. Thin section of a terracotta sample from Kochio (2005/10) with traces of a fine-grained slip. Plain polarised light.	45
18. The mobile XRF-analyser of the Frankfurt project.	48
19. Operating principle of the device (HELFERT & BÖHME 2010: 14, Fig. 2).	50
20. Comparison of stationary WD-XRF and mobile ED-XRF – major elements.	54
21. Comparison of stationary WD-XRF and mobile ED-XRF – trace elements.	55
22. Comparison of place of reading (surface/breaking edge) – major elements.	59
23. Comparison of place of reading (surface/breaking edge) – trace elements.	59
24. Example of a diagram of eigenvalues with Kaiser-Guttman criterion and broken stick model.	63
25. Example of a PCA distance biplot (pottery and terracotta).	64
26. Example of a PCA correlation biplot (corresponding to the distance biplot in Fig. 25).	65

27. **Detail of the Excel sheet containing the measurement values of the major elements and further information used in the statistical analysis.** 67
28. **Map showing all sites included in the XRF analysis (n=43)** (map by Eyub F. Eyub). Most of the sites, as well as the Nok research station (blue triangle), are located in the key study area (black rectangle). Sites further away are shown in the small locator map (top left). 68
29. **Clod-formation of clayey soil on a fresh ploughed field in central Nigeria.** Photo taken in 2011. 70
30. **Mud bricks drying in the sun, in the village of Chinka.** Photo taken in 2009. 70
31. **Secondary clay source in a dried out riverbed (clay sample 112).** Length of scale: 2 m. Photo taken in 2011. 71
32. **Two potters from Taffa (clay samples 30-32) digging for primary clay under a laterite crust.** Photo taken in 2009. 72
33. **Raw clay lying in the sun to dry, in the village of Kwagiri (clay sample 59).** Photo taken in 2009. 74
34. **Clay in a mortar, ready for pounding, in the village of Chinka (clay sample 27).** Photo taken in 2009. 74
35. **Typical reddish pots, manufactured by the potter from Ankoro (clay samples 108-113).** Photo taken in 2011. 75
36. **Pots with black colour made by the potter from Fadan Attakar (clay sample 88).** Photo taken in 2011. 75
37. **Diagram of the eigenvalues of the PCA of the pottery and terracotta of Ido with broken stick and Kaiser-Guttman criterion.** 81
38. **Distance biplot of the PCA of the pottery and terracotta of Ido (axes 1 and 2).** Terracotta figures are given in blue, potsherds in red. 82
39. **Distance biplot of the PCA of the pottery and terracotta of Ido (axes 2 and 3).** 84

40. Distance biplot of the PCA of the pottery and terracotta of Ido (axes 1 and 3).	85
41. 3D-plot of the first three axes of the PCA of Ido. Left: terracotta; bottom right: Post-Nok pottery; top right: Nok pottery.	85
42. Correlation biplot of the PCA of Ido, axes 1 and 2.	86
43. Correlation biplot of the PCA of Ido, axes 2 and 3.	87
44. The terracotta deposition from IFA 1 during excavation in 2011.	89
45. Diagram of eigenvalues of the PCA of the pottery and terracotta of Ifana with broken stick and Kaiser-Guttman criterion.	90
46. Distance biplot of the PCA of Ifana, axes 1 and 2.	91
47. Correlation biplot of the PCA of Ifana, axes 1 and 2.	92
48. Distance biplot of the PCA of Ifana, axes 1 and 3.	93
49. Correlation biplot of the PCA of Ifana, axes 1 and 3.	94
50. 3D-plot of the first three axes of the PCA of Ifana. Left: terracotta, right: pottery.	94
51. Diagram of eigenvalues of the PCA of the pottery and terracotta of Ungwar Kura with broken stick and Kaiser-Guttman criterion.	96
52. Distance biplot of the PCA of Ungwar Kura, axes 1 and 2.	97
53. Correlation biplot of the PCA of Ungwar Kura, axes 1 and 2.	98
54. Distance biplot of the PCA of Ungwar Kura, axes 2 and 3.	99
55. Correlation biplot of the PCA of Ungwar Kura, axes 2 and 3.	100
56. 3D-plot of the first three axes of the PCA of Ungwar Kura. Left: terracotta; top right: Late Nok pottery; front right: Middle Nok pottery.	101

57. Diagram of eigenvalues of the PCA of the pottery and terracotta of 43 sites with broken stick and Kaiser-Guttman criterion.	104
58. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2). The difference between the chemical compositions of the figurines in contrast to the potsherds is clearly visible.	105
59. Correlation biplot of the PCA of pottery and terracotta of 43 sites.	106
60. Diagram of eigenvalues of the PCA of the pottery and terracotta of 43 sites with broken stick and Kaiser-Guttman criterion – trace elements only.	108
61. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2) – trace elements only.	109
62. Correlation biplot of the PCA of pottery and terracotta of 43 sites – trace elements only.	110
63. Diagram of eigenvalues of the PCA of the pottery and terracotta of 43 sites and the recent clay samples with broken stick and Kaiser-Guttman criterion.	111
64. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2) and the recent clay samples.	112
65. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2) and the recent clay samples and recent pottery – trace elements only.	113
66. Artist Audu Washi with the newly made, still unfired, forgery of a Nok terracotta.	115
67. Thin section of a forgery (top) in comparison to an original Nok terracotta (bottom). The dark granules in the forgery's matrix are biotite-chlorite intergrowth, absent in Nok material.	116
68. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2), the recent clay samples, recent pottery, and the six forgeries – trace elements only.	117

69. **Geological map with the distribution of the recorded present-day clay samples.** Red dot: research station of the Frankfurt project. Geological units 15g, 18d, 21c, 22c, 24b: undifferentiated Basement Complex; 15e: Nupe Sandstones; 15f: Newer Basalts over Basement Complex; 18c: Colluvial deposit over granitic material. Source of geological map: Soil Survey Division, Federal Department of Agricultural Land Resources (FDALR), Kaduna, Nigeria, 1990. 120
70. **Geological map with the distribution of recorded present-day clay samples.** Blue dots: clay samples that show the same chemical composition as the terracotta figurines. Red dot: research station of the Frankfurt project. Geological units 15g, 18d, 21c, 22c, 24b: undifferentiated Basement Complex; 15e: Nupe Sandstones; 15f: Newer Basalts over Basement Complex; 18c: Colluvial deposit over granitic material. Source of geological map: Soil Survey Division, Federal Department of Agricultural Land Resources (FDALR), Kaduna, Nigeria, 1990. 121
71. **Dried-out sink where clay has accumulated (clay sample 135).** Photo taken during the dry season 2009. 124
72. **Unfired recent clay samples from central Nigeria.** 127
73. **Thin section of a secondary clay source (clay sample 124).** Cross polarised light. 128
74. **Thin section of a primary clay source (clay sample 133).** Cross polarised light. 129
75. **Thin section of a recent pottery sample manufactured with secondary clay (sample number 59).** Cross polarised light. 129
76. **Thin section of a recent pottery sample manufactured with primary clay (sample number 13).** Cross polarised light. 129

## List of Tables

1. Limit of Detection of the device for the different elements (HELFERT & BÖHME 2010: 16). Because of the measurement in a SiO <sub>2</sub> matrix, no value for Si could be given.	52
2. Repeated measurements of a terracotta fragment (sample UK7-677), statistical key figures; measurement values in ppm.	57
3. Repeated measurements of a piece of domestic pottery (sample UK7-548), statistical key figures; measurement values in ppm.	58
4. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of Ido.	81
5. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of Ifana.	91
6. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of Ungwar Kura.	96
7. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of 43 sites.	104
8. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of 43 sites – trace elements only.	107
9. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of 43 sites and the clay samples.	111

## List of Appendices

Appendix 1 Calibration Curves	171
Appendix 2 Statistical key figures of the raw data of all chemical elements	181
Terracotta	182
Pottery	187
Clay Samples	192





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**A**

**Research Question and General Background**



# 1 Introduction

This doctoral thesis was written within the frame of the first application period (04/2010-03/2013) of the Research Training Group “Value and Equivalence” funded by the *Deutsche Forschungsgemeinschaft* (DFG). The interdisciplinary approach of the Research Training Group combines the disciplines of archaeological sciences, cultural anthropology, and sinology. By focusing on the concepts of “Value” and “Equivalence”, the research programme aims at the study of material things in different societies and different time periods on five continents. This study of the material composition of the terracotta figurines of the central Nigerian Nok Culture belongs to the Research Training Group’s research area (A) that is concerned with the generation of values and the modes of circulation within a society.<sup>1</sup>

The joint research project of the Goethe University in Frankfurt/Main, Germany,<sup>2</sup> and the National Commission for Museums and Monuments (NCMM) in Abuja as well as the Universities of Jos and Zaria, all located in Nigeria, provides the second framework for this thesis.<sup>3</sup> The Frankfurt project explores different cultural aspects of the Nok Culture of central Nigeria with a special emphasis on the topics of chronology, settlement structures, and regional distribution. The doctoral thesis presented here deals with the questions of production and social value of the famous Nok Culture terracotta<sup>4</sup> figurines. By means of scientific analysis of the raw materials used in the production, a new hypothesis regarding production modes will be presented and the role of the terracotta figures as objects of social and symbolic value and their implications for the social organisation of the Nok Culture will be discussed.

Since their discovery at the beginning of the 20<sup>th</sup> century, Nok terracotta sculptures have been a mystery to scholars. They soon became widely known to archaeologists, but also aroused the interest of art collectors all over the world, leading to illegal diggings and the extensive destruction of Nok sites in central

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<sup>1</sup> Further research areas are: (B) the transformation of value through trade and transnational mobility; and (C) goods and traders (added in the second application period).

<sup>2</sup> In the following, this project will be referred to as “Frankfurt project”.

<sup>3</sup> This doctoral thesis was completed in June 2015 and successfully defended in November 2015. It therefore reflects the state of research at that time. Since then, research on the Nok Culture has continued resulting in newly discovered and excavated sites, new <sup>14</sup>C dates *etc.* As no new results affecting the topic of this thesis have been achieved, these new findings have not been incorporated in this text.

<sup>4</sup> The term “terracotta” (or “terra-cotta”) literally means “baked earth” and therefore initially describes the material only (SCHNEIDER 1989). In this study it will be used for the figurines of the Nok Culture as has been common practice since their discovery by Bernard Fagg in the 1940s.

Nigeria (RUPP 2010: 68). Analysis of Nok sculptures – whose exact function is still unknown – has so far been almost entirely restricted to their stylistic features as seen within a European conception of art (e.g. BOULLIER 2001; CHESI & MERZEDER 2006; DE GRUNNE 1999; FAGG 1990; SHAW 1981; WILLET 2002). These investigations showed that the figurines follow a highly standardised style, which makes it easy to recognise them at a glance. Nok terracotta sculptures vary considerably in size: their height ranges from nearly life-size to very small miniatures of only few centimetres. Nevertheless, all human and animal depictions alike have triangular or crescent-shaped eyes incised deeply into the clay. The highly bent eyebrows make the eye look more or less circular. The iris is mostly rendered as a cut-out hole, sometimes only dented in the case of smaller figurines. The typical “Nok eye” can also be found incised in vessels or on body parts of terracotta sculptures. All figures feature an elaborated hair dress or headgear and are decorated with many forms of bracelets, necklaces, foot jewellery, and other kinds of ornamentation. Nevertheless, every Nok sculpture is unique in its appearance. Various gestures, ornaments, forms of headgear, hair styles, and postures have been documented; these are combined to form an individual figure, so that no two terracotta figurines look exactly the same.<sup>5</sup>

This strict stylistic similarity was maintained for probably more than 500 years in the first millennium BCE throughout the whole distribution area of the Nok Culture. Clearly, the effort expended by the Nok people in manufacturing the terracotta sculptures in such a uniform style was immense. It is possible to maintain stylistic similarities over such a long time in a large area – assuming a codex of ritual regulations – but it almost certainly involves the work of specialists to ensure compliance with these rules. In combination with the fact that all figurines excavated so far are broken and cannot be pieced together into complete statues as parts are always missing<sup>6</sup>, the maintained style suggests a specific, presumably ritual, function and a high symbolic value for the sculptures. Such symbolic meanings are an integral part of a society and also find their expression in the technological range of a culture. Therefore, it is likely that the particular importance of the figurines can also be traced in the material composition of both components contained in all ceramic objects: clay and temper. If suggesting that the terracotta sculptures had a ritual function and were produced by specialists, a difference in the material composition of the clay and temper used in their production should become visible – when compared to the domestic pottery that presumably was produced on a household level in the villages.

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<sup>5</sup> For a complete description of the stylistic features, see MÄNNEL & BREUNIG 2014.

<sup>6</sup> Only one figurine excavated by the Frankfurt project at the site of Janjala is (although broken into three pieces) more or less complete.

Scientific investigations of the material composition of the two ceramic categories (terracotta and pottery) and comparisons between them will allow insight into the differences of the raw materials used, thereby providing information on the manufacturing process and the (social) organisation behind the production of these highly artistic terracotta figurines.

This doctoral thesis is divided in five major sections (A to E). In Section A general background information on the Nok Culture and its distribution area will be given. While Chapter 2 summarises the research history in the 20<sup>th</sup> and 21<sup>st</sup> century, the geology and geochemistry of the soils in the Nok Culture area – as an important raw material source – are addressed in Chapter 3. Here, a special focus is laid on the chemical behaviour of trace elements in relation to clay minerals in tropical soils.

Section B deals with the methods used in the scientific materials analysis. Mineralogical thin section analysis (Chapter 4) concentrates on the tempering material and reveals first differences in the manufacturing process between terracotta and pottery. In addition, it allows some valuable inferences concerning the nature of the clay that was used for the figurines. The clay itself was analysed by a geochemical approach, using a portable X-ray fluorescence (XRF) analyser (Chapter 5). After a description of the XRF method and the measuring procedure applied, the multivariate statistics used for the detailed analysis of the large amount of chemical compositional data is explained. An explanation of sample selection and a side note on clay procurement and clay processing in central Nigeria today conclude Chapter 5.

The results of the scientific materials analysis – the centrepiece of this thesis – are presented in Section C, beginning with the comparison of the geochemical composition of pottery and terracotta at the Nok sites of Ido, Ifana, and Ungwar Kura (Chapter 6). At all three sites, the clay used in the production of terracotta and pottery exemplifies the differences clearly visible in the composition of the trace elements contained in the clay matrix. Additionally, the analysis of the terracotta figures from the site of Ifana provides the possibility to study the potential differences between figurines that were deliberately deposited (Ifana 1) and those found in refuse pits (Ifana 2). In Ungwar Kura, figurines from different occupation episodes in the first millennium BCE and therefore different periods of the Nok Culture (Middle and Late Nok (FRANKE 2015)) may be investigated. The detailed inspection of the pottery from Ido and Ungwar Kura can reveal differences in the chemical composition of the pottery within the Nok Culture and also to younger time periods. The results presented in Chapter 6 are tested and confirmed in Chapter 7, in which the overall geochemical analysis of ceramic material from 43 sites included in this study is discussed. To gain further evidence concerning the mode and organisation of the figurine production, the results of the geochemical

analysis of Nok terracotta and pottery are compared to modern clay samples collected from raw clay deposits still in use today in central Nigeria. The comparison of the material composition of a Nok terracotta forgery to that of the excavated archaeological material demonstrates the potential of these analyses and further supports the interpretation of the geochemical results achieved. Chapter 8 discusses the combined results of the geochemical and mineralogical materials analysis with regard to the causes of the differences in clay and temper used in the production of terracotta figures and pottery. Three possible scenarios are suggested: 1) the use of a special clay source for the terracotta production; 2) a special kind of clay processing procedure for the terracotta figures; and 3) any kind of systematic or random error in the analysis. The results of the scientific materials analysis support the first scenario, leading to the hypothesis that both terracotta figures and pottery were produced locally. However, special clay sources were used exclusively for the production of terracotta while the clay for the pottery was taken from different sources. A critical assessment of this hypothesis with regard to the organisation of the terracotta production and the presumed involvement of specialists in this process concludes Section C.

Section D is dedicated to cultural theories on complexity, specialisation, and value. Even if the artistic terracotta figures which seem to follow stylistic regulations over a long time period and a large area suggest a complex structure of Nok society, the evidence from the archaeological excavations of the Frankfurt project has not revealed any such traces. How, then, is it possible that – without any form of archaeologically visible central organisation – the terracotta figurines were manufactured by specialists that obviously followed a stringent stylistic and material norm? Which conclusions can be drawn concerning the concept of value, its generation, and the modes of circulation displayed in the figurines? These questions are discussed on a general theoretical basis as well as in relation to the Nok Culture (Chapter 9).

This doctoral thesis concludes in Section E with a summary and an outlook on further research topics (Chapter 10). The geochemical analysis has proven the high potential for the interpretation not only of the terracotta figures but also of the domestic pottery, although the latter was initially only intended for use as comparative material. Given that the political situation in Nigeria stabilises in the next few years, the results can be transferred to other regions within the Nok Culture area and – if material is available – to other Nigerian terracotta traditions.



## 2 Nok Culture Research Histories: Beginnings and New Research

Compared with other archaeological find complexes like Bronze or Iron Age cultures in Central Europe, the Nok Culture research history is relatively short. It can be divided into two phases of investigation: 1) the definition of the Nok Culture and early studies in the 20<sup>th</sup> century, led primarily by British archaeologist Bernard Fagg, his daughter Angela Fagg-Rackham<sup>7</sup>, and Nigerian archaeologist Joseph Jemkur; and 2) the more recent large-scale investigations by the Frankfurt project. As detailed accounts of the two research phases have recently been published (BREUNIG 2014a; A. FAGG 2014; JEMKUR 2014), only a brief outline of the early research will be given here followed by a more detailed description of the investigations of the Frankfurt project.

### 2.1 The Discovery and Early Investigation of the Nok Culture in the 20<sup>th</sup> Century

Often, the discovery of those archaeological objects which lead to the definition of a new culture is due to a series of lucky coincidences. Such was the case with the Nok Culture as well: in the early 20<sup>th</sup> century, tin-mining activities in an area of approx. 26,000 km<sup>2</sup> on and west of the central Nigerian Jos Plateau (Fig. 1) unearthed archaeological artefacts reaching from the Middle Stone Age to the Iron Age as well as various geological finds (FAGG 1969: 44). As such accompanying finds were not uncommon in the open-cast mines, the first Chief Inspector of Mines, Mr. E. A. Langslow Cock, started in the 1920s a collection of these objects in the headquarters of the Department of Mines in Jos. In 1943, this collection drew the attention of the young British administrative officer Bernard Fagg, who had studied archaeology in Cambridge prior to his posting to Nigeria. Fagg and his wife Catherine used their free time to examine and catalogue the artefacts, among them pottery, stone tools, iron objects, and a small terracotta monkey head found 1928. In 1944, Fagg was told of a second terracotta head found by a mining engineer near Jemaa (SHAW 1981: 45, 47). The stylistic similarities between the two pieces led Fagg to coin the term “Nok Culture”, after the village where the first find – the monkey head – was made. Fagg stated in 1956:

*It was evident at the first sight that these two heads were made by people whose traditions were so similar (and so distinct from any art style surviving to-day)*

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<sup>7</sup> Angela Rackham, née Fagg, is the daughter of Bernard and Catherine Fagg. As she publishes under her maiden name, she will be referred to as A. Fagg.

*that they could be regarded as the product of the same art tradition ... we began to call it the Nok Culture after the site where it was first found.*<sup>8</sup> (FAGG 1956a: 1083)

The publication of his findings in 1946 and 1947 (FAGG 1946; 1947) caused Fagg to be reassigned from his post in colonial administration to become the first Government Archaeologist and Assistant Surveyor of Antiquities for Nigeria in 1947. At the 3<sup>rd</sup> International West African Conference in Ibadan, Nigeria, he and British geologist Geoffrey Bond presented the first detailed geological sequence of the Nok Valley and the Stone Age stratigraphy of the Plateau Minesfield (BOND 1956; FAGG 1956b). Within that sequence, one layer occurred in which pottery and figurine fragments had been found. Bond and Fagg thus came to a first age estimation for the finds of the Nok Culture:

*To cut the channels in which the figurine deposits rest would need something more than a minor fluctuation [in rainfall] ... The figurine culture is found below a layer of blue clay ... Clays of this kind must accumulate slowly, and this consideration suggests that the figurine culture flourished not long after the passing of the rainfall maximum during which the channelling occurred. An age of approximately 2000 years may seem excessive ... the date must be regarded as tentative.* (BOND 1956: 200)

Subsequently, research on the Nok Culture progressed rapidly: Bernard Fagg ordered that all future archaeological finds be collected and brought to him, resulting in a staggering increase in finds discovered not only during mining but also during road construction or farming activities. Whenever their free time allowed, Fagg and his family continued their work on the Nok material. Fagg even managed to secure some 4 ha in the minefields around Nok exclusively for archaeological research. In the 1950s, the collection grew considerably: in 1952, Fagg initiated the building of the Jos Museum to store, preserve, and analyse the growing archaeological collections; by 1956 he was installed as Director of the Nigerian Federal Department of Antiquities. Fagg did not miss the chance to take advantage of the newly developed possibilities of radiocarbon dating and had a number of samples analysed.<sup>9</sup> The results

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<sup>8</sup> Bernard Fagg chose the term “culture” to describe the Nok Valley finds, and thus initiated the use of the term “Nok Culture”. In doing so, he conflated the terms “art” and “culture” – in the archaeological sense, however, a culture is a combination of different elements (not necessarily all preserved over time or reconstructible by archaeological methods) such as house construction, funeral rites, tools, pottery *etc.* which provide concrete evidence for social traditions (for further discussion of this topic, see CHILDE 1929: v-vi; FAGG 1990: 24; and SHAW 1981: 56). Nevertheless, the term will be retained here, as its use has become common practice among researchers.

<sup>9</sup> For a complete list of all early radiocarbon datings of Nok Culture material see JEMKUR 2014: 97.

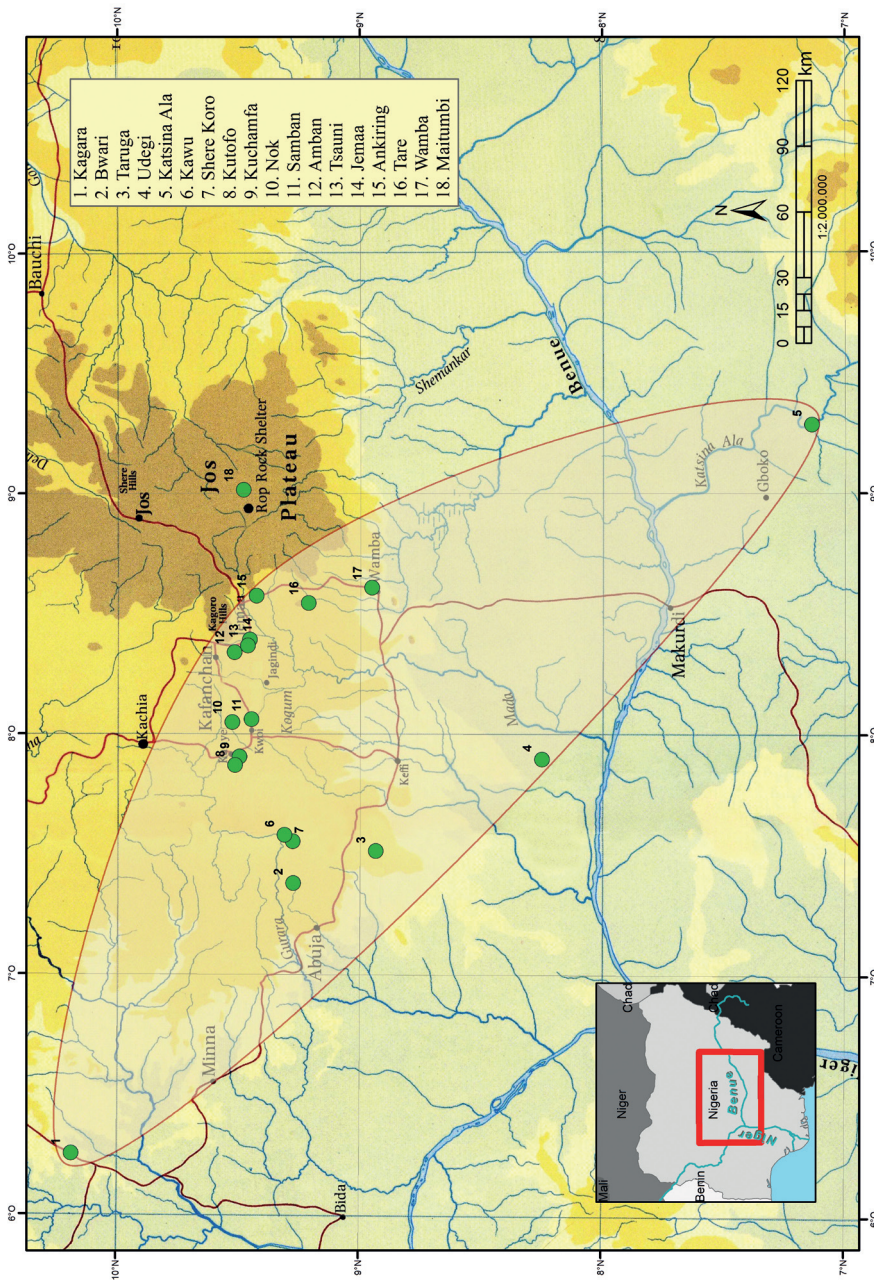


Fig. 1. Map showing the location of archaeological sites discovered by Bernard Fagg and others during the 20<sup>th</sup> century (green dots) (based on Fagg 1990; amended by the Frankfurt project). The presumed distribution area of the Nok Culture is shown in the red-rimmed ellipse. The finds from „Maitumbi“ (No. 18) were relocated to Jemaa by A. Fagg in 2013 (see footnote 16).

were published in 1957 (BARENDSEN ET AL. 1957) and reinforced his earlier age estimate based on the geological sequence, placing the Nok Culture between 500 BC and 200 AD.<sup>10</sup> An earlier date pointing to the early 10<sup>th</sup> century BC (BARENDSEN ET AL. 1957: 916) was rejected by Fagg because it was considered too early (FAGG 1959: 291-292; 1962: 445).<sup>11</sup> Fagg's early publications also drew the attention of art dealers all over the world, resulting in the start of illegal diggings for the figurines in the wider surroundings of the village of Nok.

Up to that time, all finds had derived from alluvial contexts. The first finds from a non-alluvial context – a terracotta head and, in 1954 and 1956, more figurine fragments – were made in 1951 in Katsina Ala (about 150 km south of Nok, south of the Benue River (Fig. 1)) during construction of a new hockey pitch on the grounds of Katsina Ala Middle School. An archaeological investigation of the site was not possible until 1963, when Robert Soper, then archaeologist with the Nigerian Department of Antiquities, was sent there to assess the potential and extent of the site. He conducted a small rescue excavation and discovered figurine fragments, potsherds, stone tools, tuyère fragments, and some iron slags (JEMKUR 1992: 2-3).<sup>12</sup> Although discovered in 1960, after the Katsina Ala site, the first excavation of Nok material in a non-alluvial context was conducted by Bernard Fagg (and in 1967/68 also by Angela Fagg) in Taruga. In three field seasons (1960/61, 1965/66, and 1967/68), he excavated several trenches which yielded a large number of *in situ* finds: figurine fragments, decorated pottery, stone tools, iron objects, iron-reduction furnaces, and charcoal for radiocarbon dating. In 1965/66, a proton magnetometer was used to trace geomagnetic anomalies in the soil. This contributed significantly to the discovery of thirteen iron-reduction furnaces (of which 10 were excavated) and several slag heaps (FAGG 1968; 1969): the first direct evidence of iron working in the Nok Culture and some of the earliest traces of iron metallurgy in sub-Saharan Africa, with dates in the middle of the first millennium BCE (FAGG 1965; TYLECOTE 1975).

Much like her father Bernard, Angela Fagg (who also had studied archaeology and worked for the Nigerian Federal Department of Antiquities) was interested in the exploration of the Nok Culture. She assisted her father, and, after his return to England in 1963, also conducted an excavation of her own in Samun Dukiya (field season 1969/70). The find spectrum there was

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<sup>10</sup> Despite the knowledge that <sup>14</sup>C dates could not be directly transferred to calendar dates because of fluctuations in the <sup>14</sup>C content in the atmosphere, the early dates were not calibrated (SHAW 1968: 456). Thus, all dates obtained by Fagg that are quoted here were uncalibrated bp-years, using 1950 as base year for the conversion into calendar years.

<sup>11</sup> For a complete review of the early radiocarbon and thermoluminescence datings, see FRANKE 2015; FRANKE & BREUNIG 2014.

<sup>12</sup> So far, no details of Soper's excavation have been published.



analogous to those from the alluvial deposits and from Taruga, though Samun Dukiya yielded no traces of iron reduction (but iron tools). Therefore, Angela Fagg assumed that chronological and/or regional differences occurred during the duration of the Nok Culture (A. Fagg 1972).

In 1973, Joseph Jemkur joined Angela Fagg in her work. The two consolidated the first radiocarbon age determinations by using a newly developed method for the direct dating of fired clay artefacts: thermoluminescence (TL) dating (AITKIN ET AL. 1964: 1032-1033). The 23 dates obtained<sup>13</sup>, however, did not always match the available radiocarbon dates. Especially the TL dates from Katsina Ala, Ankiring and Chado date largely after the turn of the Common Era. In Katsina Ala, a date of 1610±23 CE for a potsherd may reflect the presence of a later occupation level (CALVOCRESSI & DAVID 1979: 10; JEMKUR 1992: 67), while in Ankiring the dates (255±140 CE; 395±140 CE) were taken from associated find material but not from terracotta finds directly. Furthermore, the figurines show features that might place them outside of a Nok context (Fagg 1990: 139; JEMKUR 1992: 69; WILLETT 1986: 88). Thus, the dates may be correct but not necessarily connected to the Nok Culture. The results for Chado all date after the turn of the Common Era. Although the dated figurines show clear Nok attributes (Fagg 1990: 124), their find context remains unclear with some stemming from mining activities and others recovered by JEMKUR (1992: 39-40, 86). Maybe the TL dates in this case do not reflect the time of production but are altered by external conditions like bush fires. These relatively young TL dates therefore are not sufficient to extent the duration of the Nok Culture after the turn of the Common Era.<sup>14</sup>

After Angela Fagg had returned to Great Britain, Jemkur remained the sole researcher investigating the Nok Culture. Much of his work focussed on educating the local public in order to stop or limit illegal digging. He devoted particular time and attention to the region around Jemaa, outside the Nok Valley. Between 1974 and 1983, and then again in 1999, he led several field surveys in the area and discovered various Nok Culture sites, including Chado, Old Zankan<sup>15</sup> and Janjala (JEMKUR 1986; 1992; 2014).

In summary, from the discovery of the Nok Culture in the early 20<sup>th</sup> century to the beginning of the 21<sup>st</sup> century, finds were made in an area in central Nigeria stretching approx. 500 km from Katsina Ala in the south-east to

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<sup>13</sup> For a complete list of all early thermoluminescence datings of Nok Culture material see JEMKUR 2014: 98.

<sup>14</sup> For a complete review of all dates (TL and radiocarbon) also see FRANKE 2015, Chapters 5 and 6.

<sup>15</sup> In Old Zankan, terracotta figurines were secondarily used as burial goods. Thus, the site itself does not belong to the Nok Culture, which is also confirmed by the found pottery assemblage (JEMKUR 1992: 45-49).

Kagara in the north-west and approx. 170 km from Taruga in the south-west to Ankering in the east (Fig. 1). The Nok Culture is therefore distributed west and southwest of the Jos Plateau.<sup>16</sup> Most sites lay close to Nok and Jemaa, presumably because of the intensive mining activities in that region (JEMKUR 1992: 4; SHAW 1981: 51). The typical find spectrum consisted of figurine fragments, potsherds, stone tools, iron objects, and iron-reduction furnaces (in the case of Taruga). Radiocarbon and thermoluminescence datings pointed to an age between 500 BCE and 200 CE; an earlier date had been discarded (FAGG 1959) because of the Nok Culture's connection with iron production, which was at the time suggested to have been imported from Carthage or Meroe where it was developed in the 7<sup>th</sup> century BCE (EGGERT 2014: 55). Other (mostly later) dates are not securely related with the Nok Culture because of unclear stratigraphies or their origin from alluvial deposits. In recent years, the Frankfurt project's research has modified this picture – especially with regard to the dating of the Nok Culture.

## 2.2 New Research: The Frankfurt Project

After nearly 20 years of archaeological work in West Africa, especially in the Chad Basin of Nigeria, a team of archaeologists around Prof. Peter Breunig from Frankfurt's Goethe University shifted its focus to central Nigeria. The goal was to trace the same developments which had already been observed in northern Nigeria's Chad Basin around 500 BCE: the emergence of large, sometimes fortified settlements, specialised craftsmanship, large-scale food storage and first traces of iron technology (BREUNIG 2009a; 2010; BREUNIG & NEUMANN 2002). What was known of the Nok Culture at the beginning of the 21<sup>st</sup> century – its beginnings around 500 BCE, the production of iron, and impressive terracotta sculptures – suggested that the Nok Culture would be a suitable candidate for examining the aforementioned phenomenon. In 2005, initial research to test the potential of the Nok Culture began within the scope of the Research Unit 510 "*Ecological and Cultural Change in West and Central Africa*" (2004-2009), funded by the *Deutsche Forschungsgemeinschaft* (DFG).<sup>17</sup>

On the ground in Nigeria, the team cooperated closely with Nigeria's National Commission for Museums and Monuments (NCMM) and the Universities of Jos and Zaria. Research on the Nok Culture has laid idle since the work of Joseph Jemkur, so one objection was to assess how much was

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<sup>16</sup> So far, no finds from the Jos Plateau itself have been recorded. Initially, Nok finds were reported from Maitumbi on the Jos Plateau. This information was corrected and the finds relocated to Jemaa by A. Fagg in June 2013 (pers. comm.).

<sup>17</sup> This was the precursor to the Frankfurt project.

still left of Nok sites that had fallen prey to looting especially since the 1990s when prices for figurines on the international art market grew (HAMBOLU 2014). The first investigations concentrated on an area between Abuja and the Jos Plateau, mostly near Nok and Jemaa, where Joseph Jemkur had already worked some years earlier. Preliminary studies were very promising: with the help of local informants, it was possible to locate a large number of sites which proved – after early test excavations, and even when looted – their archaeological potential in the undisturbed areas (RUPP 2010; RUPP, AMEJE & BREUNIG 2005). Especially the high find densities and the apparently differing contexts in which the figurines were found (in pits with other settlement waste, as in Ungwar Kura, but also in clear depositions, as in Utak Kamuan Garaje Kagoro (RUPP 2014a; b)), seemed to suggest the possibility of unravelling the enigma behind this largely unknown culture with its terracotta statues, on which the early research had focussed (BREUNIG 2009b; RUPP, BREUNIG & KAHLHEBER 2008).

In 2009, the DFG agreed to fund a long-term project (named “*Development of complex societies in sub-Saharan Africa: The Nigerian Nok Culture*”<sup>18</sup>, and referred to in this text as the Frankfurt project). The aim of the project, which can continue until 2020, is to intensify investigations so as to provide a more comprehensive picture of the Nok Culture.

Besides the figurines, a focus is laid on the investigation of the material culture (pottery, stone artefacts, iron objects, remains of iron reduction<sup>19</sup>), the economic system and reconstruction of the environment (based on archaeobotanical research), the social organisation (which is the prerequisite for the production of special goods like iron or terracotta figurines), the structure of sites, settlement patterns, and also ritual aspects concerning the terracotta finds (BREUNIG 2009b; HÖHN & NEUMANN 2014; NEUMANN & HÖHN 2014; RUPP 2014a; b; c).

Given the large expansion of the Nok Culture area as known from 20<sup>th</sup> century investigations, the Frankfurt project decided to focus on one area surveyed during the preliminary investigations between 2005 and 2008. In this area, a large number of sites had already been documented, and

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<sup>18</sup> The term „complex society“ is to be regarded with care concerning the Nok Culture. So far, the excavations have not revealed any evidence for social hierarchy or complexity although the skilfully made terracotta figurines and the early iron production (and therewith specialised craftsmanship) promised to provide an opportunity to trace the same developments as already observed in the Chad Basin during the same period of time. How a presumed specialisation for example in the terracotta production could be organised without a complex social organisation is discussed in Chapter 9.

<sup>19</sup> Since 2013 a dissertation project has been concerned with the analysis of the iron-reduction furnaces and the slags. First results have recently been published (JUNIOUS 2016).

the local people were aware of the archaeological work and its significance and concerns. Generously, the District Head of Janjala provided a plot of land where a research station was built in 2009. The work discussed in the following is concentrated in an area of about 20 × 15 km surrounding this station, defined as key study area (Fig. 2). Whenever necessary and possible, sites outside this key research area were considered as well (BREUNIG 2014a). In total, 315 archaeological sites have been recorded by the Frankfurt project between 2005 and 2013, mainly inside the key area of research but also in the wider surroundings. 261 were classified as Nok Culture sites (of which 165 are located within the key study area) according to absolute dates and/or the analyses of the finds, whereas 54 belong to earlier or later complexes (FRANKE 2015: 24).

The project is divided into four phases of three years each (BREUNIG 2009b; 2014a; BREUNIG & RUPP 2010):

The **first phase (2009-2011)** focussed on the chronology of the Nok Culture, including the absolute dating and the analysis of domestic potsherds (FRANKE 2015; FRANKE & BREUNIG 2014). The early <sup>14</sup>C and thermoluminescence datings for the Nok Culture by Bernard and Angela Fagg as well as Joseph Jemkur (Chapter 2.1) had produced dates between 500 BCE and 200 CE. At the end of the 20<sup>th</sup> century, French art historian Claire Boullier radiocarbon dated charcoal material from the infill of a number of figurines from different museums and private collections within the scope of her dissertation project (BOULLIER 2001; BOULLIER ET AL. 2002/2003). As the origins of these figurines are not clearly documented, these dates should be regarded with caution; nonetheless, the obtained dates point to a somewhat earlier beginning of the Nok Culture between 900 and 800 BCE, and a somehow earlier end around the second century BCE (BOULLIER ET AL. 2002/2003: 27).

In contrast, the dates obtained by the Frankfurt project (Fig. 3) derive from scientifically documented contexts. Since the beginning of the project, 174 <sup>14</sup>C (mostly from annual plants) and 27 TL samples have been dated (FRANKE 2015: 41). The analysis of these datings has led to the division of the Nok Culture into three phases: the “Early Nok” phase ranges from 1500 to 900 BCE. Even though terracotta figurines are absent in excavations dating to this period, it is assumed to belong to the Nok Culture. The “Middle Nok” phase, in turn, ranges from 900 to 400 BCE. It is in this phase that the figurines and also the iron production find their beginnings. It is also the best-documented period, with the highest density of sites. Around 400 BCE, the number of sites and therefore the possible datings become rarer and seem to disappear around the turn of the Common Era. The period from 400 BCE to the turn of the Common Era is therefore considered the “Late Nok” phase. Sites after the turn of the Common Era do not belong to the Nok Culture, as there are no terracotta finds



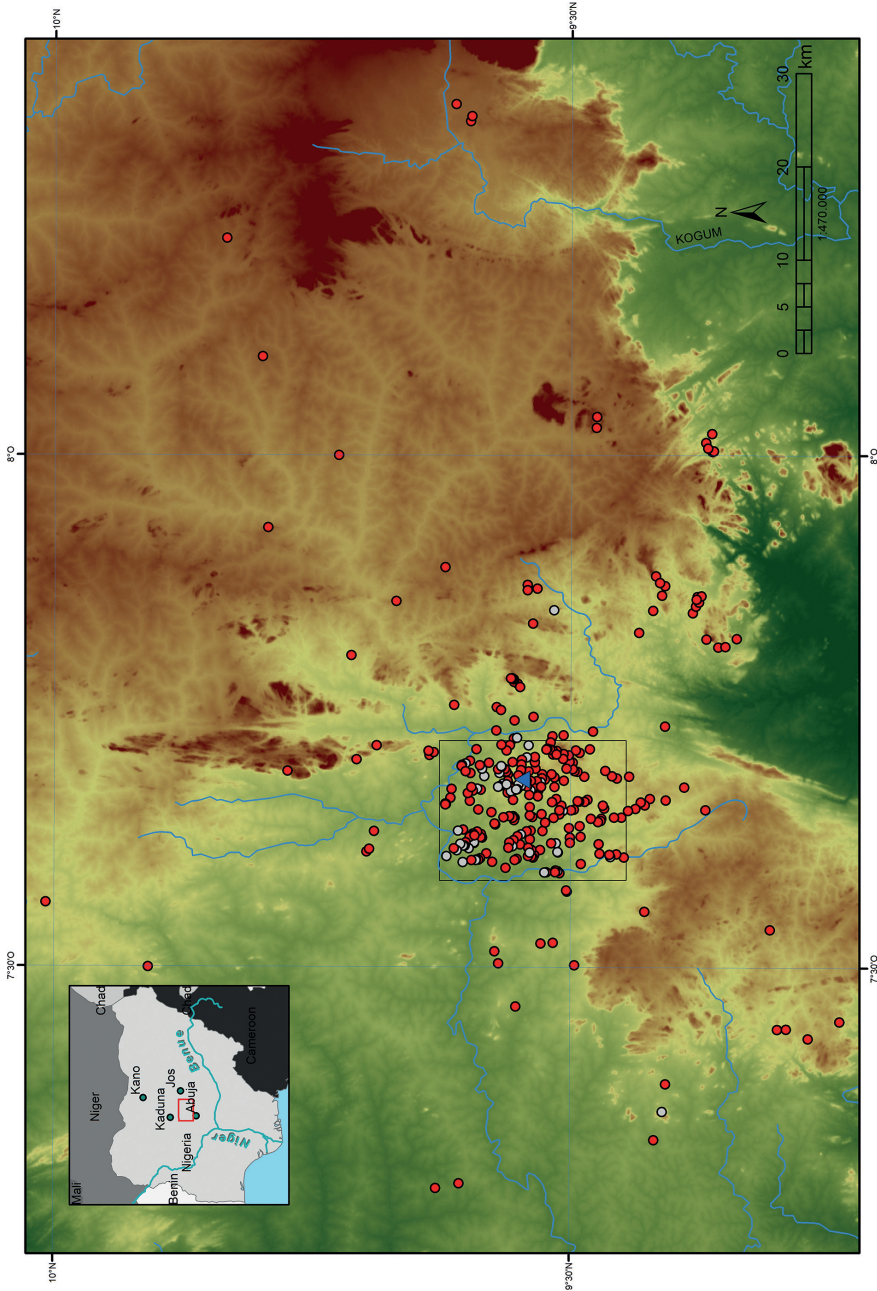


Fig. 2. Map showing the distribution of all archaeological sites recorded by the Frankfurt project (2005-2013) (map by Eyub F. Eyub). Nok sites are marked by red dots (n=261), sites not belonging to the Nok Culture by grey dots (n=54). The box in the centre indicates the project's key study area with the research station (blue triangle).

and the pottery decoration shows no link to previous Nok phases. The period is therefore termed “Post-Nok”<sup>20</sup>. This chronological subdivision of the Nok Culture has been the subject of a doctoral thesis (FRANKE 2015; 2016).

Thus, the duration of the Nok Culture has been amended and expanded from Fagg’s initial estimate of 700 years (500 BCE to 200 CE) to approx. 1500 years (1500 BCE to the turn of the Common Era) (FRANKE 2014; 2015; FRANKE & BREUNIG 2014). Fagg’s earlier date, discarded then as too early for the Nok Culture, was most likely connected with it.

The **second project phase (2012-2014)** comprised the functional and structural analysis of excavated sites as to three factors: 1) site structure, 2) site interpretation and classification, and 3) occupation patterns.

Despite many excavations and a large number of documented sites inside and outside the key area of research, the structure and classification of a Nok site remains difficult. Features are rare and, if present, consist mainly of amorphous pits filled with all kinds of cultural material or of stone settings – obvious features are missing. So far, it remains impossible to classify the sites as “burial”, “settlement”, “ritual” site, *etc.* (RUPP 2010; 2014a; b).<sup>21</sup> Attempts were also made to solve the classification problem by using several technical methodologies such as three-dimensional recording of all finds, magnetic prospection (BREUNIG & RUPP 2010; RUPP 2014b), or element mapping of the soil in order to find activity zones (NAGEL 2014). Concerning occupation patterns and spatial distribution of Nok sites, all available data (geographical position, access to water, orientation of the site, climatic conditions, *etc.*) were collected and are currently being analysed using Geographical Information Systems (GIS). Work on the functional and structural aspects of the project is ongoing.

Despite the increasingly difficult political situation in Nigeria beginning in late 2011/early 2012 a large-scale excavation at the site of Pangwari was conducted during the winters of 2012/2013 and 2013/2014. Aim was to learn more about the internal structure of a Nok site. An area of *ca.* 2,700 m<sup>2</sup> was excavated in ten units. Due to single find recording, the find distribution can

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<sup>20</sup> The term “Post-Nok” was coined to separate all younger sites without any Nok terracotta figurines and/or pottery with decorations different from those found on sites of the Nok Culture. It is a collective term for various, not necessarily connected complexes that cannot be differentiated for the time being due to the lack of archaeological research on this time period in the key study area.

<sup>21</sup> A few exceptions to this finding exist: for example, the site of Utak Kamuan Garaje Kagoro is classified as a ritual site due to the most likely deposition context of broken terracotta parts (RUPP 2010; 2014a), whereas the circular stone setting at Puntun Dutse is interpreted as the remains of a house classifying Puntun Dutse as a settlement site (RUPP 2014b).

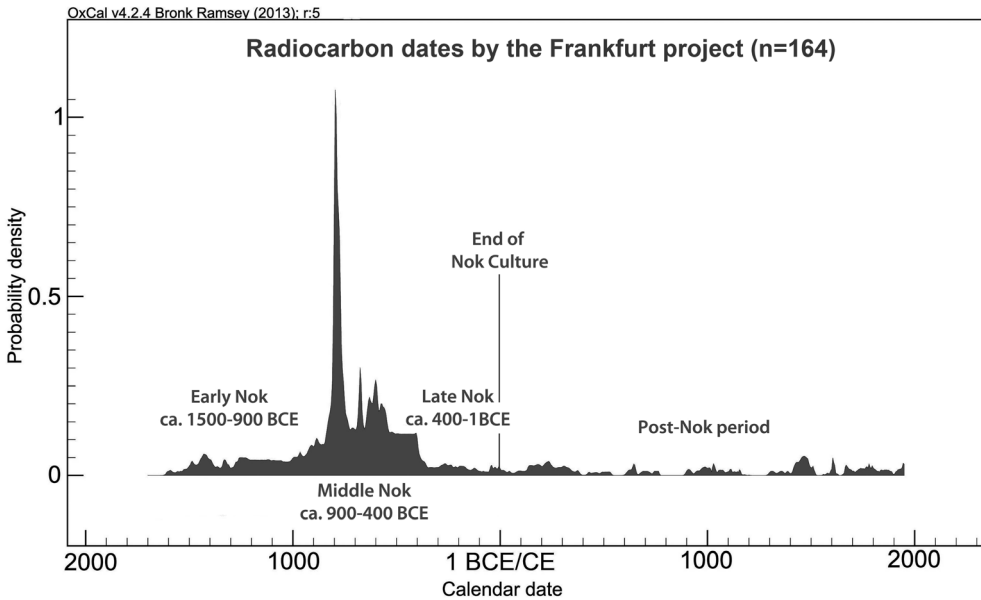


Fig. 3. Summed probability density of the radiocarbon dates obtained by the Frankfurt project (OxCal4.2, IntCal13, 95.4% probability). The programme produces the sum of the  $^{14}\text{C}$  dates' individual probability distributions for each year. The high peak around 800 BCE is caused by the large number of  $^{14}\text{C}$  dates that have their highest probability there, at the beginning of the calibration curve plateau (graphic courtesy of G. Franke, November 2016).

be reconstructed and the features and their finds were analysed in the scope of a Master Thesis finished in 2014 (SCHMIDT 2014).

Further large-scale excavations could not be conducted as planned since the political situation has become more and more unstable; fieldwork was reduced to a minimum in 2013 and stopped after the completion of the excavation in Pangwari in early 2014. Therefore, the exhibition of the results of the Frankfurt project, initially planned for the fourth project phase (2018-2020), was antedated to 2013. Between October 30, 2013 and March 23, 2014 the highlights of the excavated terracotta figurines and the archaeological methods and results were presented to the public at the *Liebieghaus Skulpturensammlung* in Frankfurt/Main. A companion volume with the results of the Frankfurt project was published.<sup>22</sup>

<sup>22</sup> The title of the exhibition was "Nok. Ein Ursprung afrikanischer Skulptur". The German version of the catalogue was published in 2013 (BREUNIG 2013) and in 2014 translated into English (BREUNIG 2014b).



### 3 Geology and Geochemistry of Tropical Soils

The tropical climate in the area under consideration has led to severe chemical weathering of the parent bedrock (mainly granites, gneisses, and metasediments), setting free the primary rock-forming minerals Si, Al, Fe, Na, K, Ca, and Mg. This leads to the formation of several soil types typical of the northern Guinea savanna of Nigeria (Lixisols, Acrisols, Plinthosols, and Fluvisols). All of these contain a high concentration of clay minerals (which in turn consist of these primary rock-forming minerals and H<sub>2</sub>O), mainly kaolinite, goethite, and illite, derived from the weathering of aluminium silicate minerals like feldspars and biotite (HARTMANN 2013: 42-43). These clays were used in the Nok Culture to produce domestic pottery and terracotta sculptures.

Clay minerals are phyllosilicates, which means their crystalline structure consists of parallel sheets (layers). Between these layers, other minerals or chemical elements (respectively their cations or anions) and especially trace elements are incorporated (KABATA-PENDIAS & PENDIAS 1992). Thus, clay minerals act as a trace element sink, whose composition in turn can be used as a “fingerprint” to characterise and distinguish different clay deposits. Section C of this thesis deals with the analysis of the clays (and especially their trace element composition) used for the Nok Culture’s domestic potsherds and terracotta figurines. The clay has proven to be the most valuable source of information in distinguishing these two archaeological find categories.

In the following, a brief description of the environmental factors at play in the Nok Culture area will be given, followed by a more detailed account of the geology of the wider surrounding of the research area of the Frankfurt project and the geochemistry of tropical soils in general. A special focus is laid on the behaviour of trace elements during the weathering process and in the soil as they have proven to be the best distinguishing characteristic between the pottery and the figurines. The Jos Plateau will be included in this account because, as a prominent geological feature providing some of the parent material for the weathering process, it plays an important role in the formation of soils and clay-bearing layers of the area. No Nok Culture finds have yet been made on the Jos Plateau itself, however.

#### 3.1 General Background and Environment

Most Nok Culture finds are distributed in an area between 9° to 10° N and 7° to 9° E in central Nigeria (Fig. 1). The region is part of Nigeria’s northern highlands, which form the hilly foreland of the Jos Plateau adjacent in the East. The undulating landscape is characterised by hills and granite outcrops

(inselbergs) with elevations between 300 and 900 m and up to 1800 m on the Jos Plateau (BOND 1956: 189). The area is criss-crossed with numerous smaller and larger rivers, the vast majority of which carry water only in the wet season between April and October. Nearly all rivers, among them the largest tributaries of the Niger River (Kaduna and Sokoto River) and the Benue River (Gongola River), rise on the Jos Plateau (AKINTOLA 1982: 12; NELSON ET AL. 1972: 29). The region lies in the northern part of the Southern Guinea vegetation zone (KEAY 1953). In the hilly foreland, it displays tropical climate conditions with a mean annual rainfall of 1800 mm and mean monthly temperatures of 25°C (ABAJE ET AL. 2010), while mean annual rainfall and mean monthly temperatures on the Jos Plateau itself are 1300 mm and 23°C respectively (BOND 1956: 187-188). These tropical climate conditions in particular influence the weathering of the underlying bedrock and the resultant soil formation process, which are described in more detail below.

### **3.2 Starting Point: The Underlying Bedrock and the Overlying Parent Material**

Approximately 40% of Nigeria's geology belongs to the Precambrian crystalline basement which constitutes the underlying bedrock (Fig. 4; RAHAMAN & MALOMO 1983: 19; SCHLÜTER 2008: 19). In central Nigeria it is composed primarily of three different rock types: 1) Older Granites, consisting of porphyritic granites, 2) a migmatite-gneiss complex comprised of migmatitic gneisses and a series of basic and ultrabasic metamorphosed rocks, and 3) metasediments which are typically composed of semi-pelitic biotite-muscovite schists. Additionally, and as a fourth rock type, Younger Granite suites, which are composed of granitic/volcanic ring complexes, intruded into the basement complex in the Jurassic age, forming – together with the Older Granites – among others, the Jos Plateau. The granites and gneisses occur mainly as inselbergs or undifferentiated plains, while the metamorphosed rocks of the migmatite-gneiss complex occur mainly in linear ridges (WALL 1979: 13). In varying proportions, all these rocks contain quartz ( $\text{SiO}_2$ ), K-feldspars ( $\text{KAlSi}_2\text{O}_8$ ), plagioclases ( $\text{NaAlSi}_3\text{O}_{10}$ – $\text{CaAl}_2\text{Si}_2\text{O}_8$ ), biotite ( $\text{K}(\text{MgFe})_3(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$ ), and muscovite ( $\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{F,OH})_2$ ) (CAEN-VACHETTE & EKWUEME 1988; EPHRAIM 2012; OBAJE 2009; RAHAMAN & MALOMO 1983; TURNER 1989).

Due to the tropical climate and the bedrock's age, much of the rocks have been chemically weathered into saprolite (DUROTOYE 1983: 12-13; TRESCASES



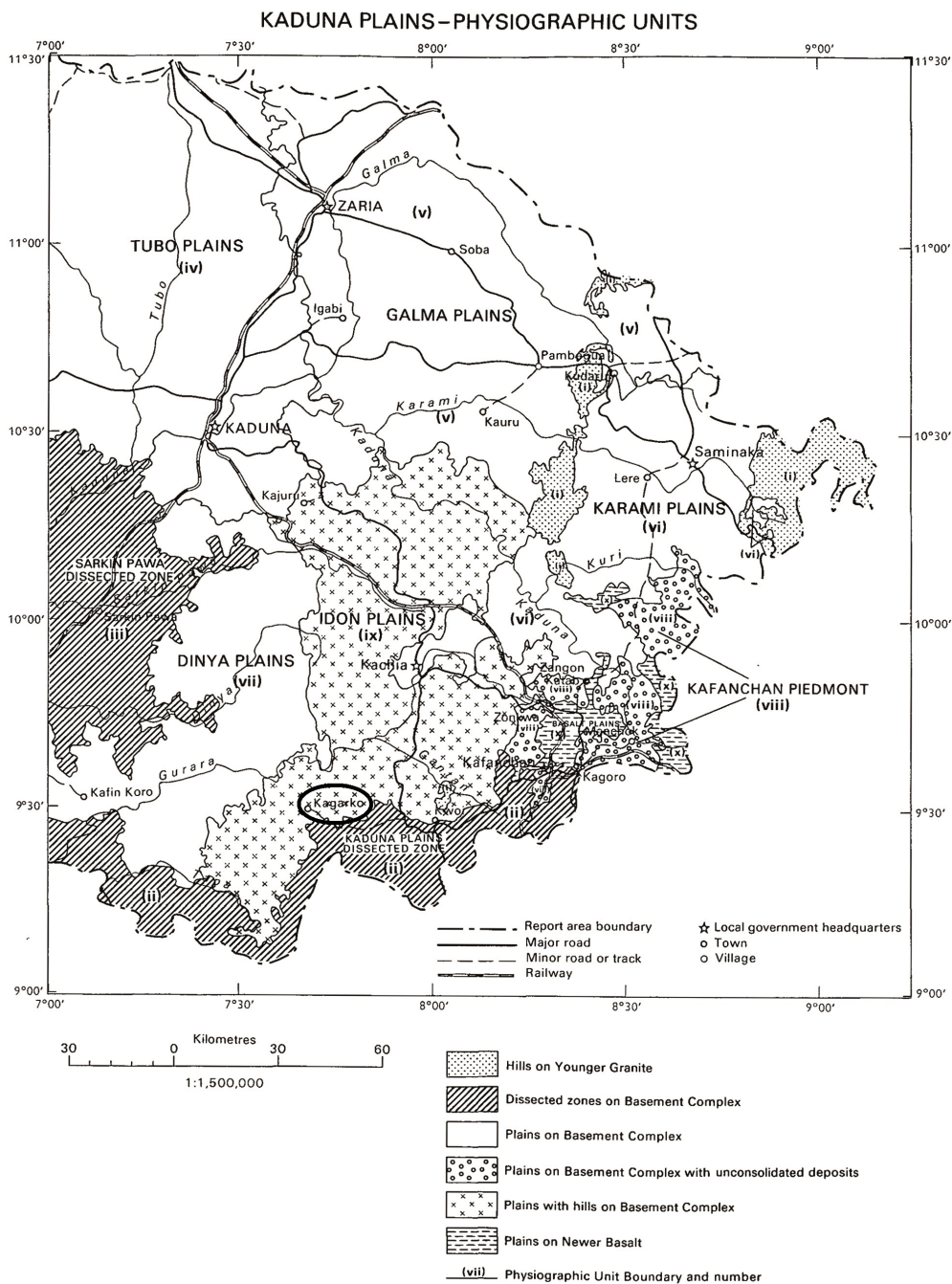


Fig. 4. Geological map of the Kaduna plains, Kaduna State, Nigeria (WALL 1979; Text Map 5.8). The key research area of the Frankfurt project is situated in the south, near Kagarko (black ellipse). Not displayed: the Jos Plateau which would be adjacent in the East.

1992: 27-28). These rocks are overlaid with parent materials<sup>23</sup> which are the actual substrates of the Holocene soil formation and therefore have the most significant influence on soil chemistry. Three different types may be distinguished in the area under consideration: slope, river, and aeolian deposits.

Slope deposits derive from upslopes through erosion. They are composed largely of angular, coarse-grained granite grus which is highly chemically weathered. River deposits are transported by water and redeposited in layered structures. These may be sand- or clay-dominated, depending on the exact position of their deposition (marked banks *vs.* flattish areas in floodplains<sup>24</sup>). Since nearly all rivers in this area rise on the Jos Plateau, the geochemistry of the river deposits is most likely analogous to that of the underlying bedrock and parent material types (BENNETT ET AL. 1977; HARTMANN 2013). BOND (1956: 193-194) describes the alluvial deposits of the Nok River and its tributaries in the Nok Valley as a succession of sands and clays – intermixed with gravels in the lower part of the profile, and bearing signs of erosion-related events – at a depth of approximately 22 m (71.5 feet).<sup>25</sup> Figure 5 shows a similar succession of sands and clays from the Nok Valley (although only about 4 m (13 feet) deep) with a blue-grey clay at the basis.<sup>26</sup> The aeolian deposits consist of silty material, mainly from Harmattan dust, brought in from the North during the dry season. Depending on its geological source, the season of accumulation and the transport history, the dust's chemical characteristics can vary widely (MORENO ET AL. 2006: 261). In the northern and central part of Nigeria, the Harmattan dust contains 25% clay, 57% silt, and 18% sand; the mineralogical composition is dominated by (in descending order of proportion) quartz, K-feldspars, mica, and Ca-Na-feldspars (MØBERG ET AL. 1991: 76-77).

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<sup>23</sup> Parent material is the transition zone between the underlying bedrock and the fully developed soil. In this zone, the weathering and conversion from bedrock to soil occurs (HARTMANN 2013).

<sup>24</sup> These floodplains will be important when discussing a central *versus* local production of the figurines (Chapter 8).

<sup>25</sup> Embedded in this sequence, below a layer of blue clay, at a depth of about 5 m (18 feet), Bernard Fagg discovered artefacts of the Nok Culture (FAGG 1956b).

<sup>26</sup> The blue-grey clay at the basis of this soil profile is not the same as the one described by BOND (1956) and FAGG (1956b). No figurines or other archaeological materials were found here.





Fig. 5. Soil profile in the Nok Valley with blue-grey clay at the bottom. Length of scale: 3 m. Photo taken in 2009.

### 3.3 Chemical Weathering and the Developing Soils

The underlying bedrock and the overlying parent material provide the starting point for the soil formation process. They contain the primary mineral constituents eventually found in the soils. These minerals (Si, Al, Fe, Na, K, Ca, and Mg) differ in their resistance to the weathering processes, so that they can be differently affected at varying depths of the profile. In addition, they – after further extensive weathering – make up the clay minerals that incorporate the trace elements (TRESCASES 1992: 36). Often, several of these weathering processes occur simultaneously or even interact, affecting the mineral constituents (and therefore the geochemical properties) of the soils and the developing clays. Though the trace elements found in these soils and clays are by definition present only in small concentrations (KABATA-PENDIAS & PENDIAS 1992: 23), their presence and concentration can be used, as will be shown in Section C, to gain insights into the manufacturing process of the Nok terracotta sculptures.

#### 3.3.1 Chemical Weathering

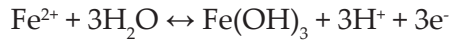
The weathering process can be chemically described as the hydrolysis, oxidation, reduction, hydration, dissolution, and carbonation of the primary minerals.<sup>27</sup> These processes lead to the formation of relatively stable minerals and chemical components through the dissolution or the reaction of the minerals with chemical agents. As the bedrock in the study area consists largely of granites and metamorphic rocks, carbonation plays only a minor role. The following remarks are a summary of the accounts by KABATA-PENDIAS & PENDIAS (1992), MALOMO (1983) and TRESCASES (1992):

- **Hydrolysis** is the chemical reaction of minerals, primarily silicates and carbonates, with water. It is the initial reaction in the transformation of silicate minerals (such as feldspars and mica) into clay minerals (such as kaolinite or illite). Under tropical climatic conditions, hydrolysis is the most prevalent reaction, with the secondary minerals kaolinite and Fe-Al oxyhydroxides forming laterites. This process is intensified in low pH-value milieus.
- **Oxidation** involves the loss of electrons from the atoms of a mineral. Oxygen is often incorporated into the chemical components or increases the element

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<sup>27</sup> Aside from the chemical alteration of the rocks and minerals, physical and biological weathering also takes place. These last two factors will not be considered here, however, since in tropical climate, chemical weathering has the most significant effect on parent material.

potential. For example, ferrous iron, which is a major rock constituent, is oxidised to ferric iron:



During the weathering process, ferric iron rapidly becomes the only stable form, forming goethite ( $\text{FeO}(\text{OH})$ ) and/or haematite ( $\text{Fe}_2\text{O}_3$ ), which are responsible for the reddish colour in most tropical soils.

- **Reduction** is the reverse process of oxidation. It involves the addition of electrons to the atoms of a mineral undergoing weathering.
- **Hydration** can be characterised as the increase of water content in the minerals. Several minerals – especially iron oxides and copper sulphates – incorporate water and change to their hydrated form. It is by this process, for example, that magnetite ( $\text{Fe}_3\text{O}_4$ ) changes to haematite ( $\text{Fe}_2\text{O}_3$ ).
- **Dissolution** is the simplest weathering mechanism and affects only a limited number of minerals – essentially those with a high solubility product (such as salts, sulphates, and carbonates). It results in the creation of new ions and/or insoluble components.
- **Carbonation** is the alteration of compounds into carbonates by the incorporation of  $\text{CO}_2$ . It occurs primarily in rocks containing calcium carbonate (*e.g.* limestone, chalk), forming calcium bicarbonate.

### 3.3.2 Soils

In general, the soils in the area under consideration are red, reddish, yellowish brown or dark brown in colour, deeply weathered, well drained, and fine to medium textured. Laterite that has developed through hydrolysis occurs in many areas (Fig. 6). The soils in the study area show textures typical of soils developed over granites and gneisses under these geographic and climatic conditions: sandy topsoils, clayey subsoils, relatively high silt concentrations (brought in by the Harmattan winds), and only a thin organic surface layer. In areas where the metasediments dominate, extremely high contents of clay can be found (BENNETT ET AL. 1977).

In the Nok Culture area, four different soil types can be distinguished: Lixisols, Acrisols, Plinthosols, and Fluvisols. All four are typical of the savanna zone (HARTMANN 2013).<sup>28</sup> Likewise common to all four soil types is

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<sup>28</sup> As the exact soil type is not of cardinal importance to the study presented here, only a brief general description is given. For a more detailed account of these soil types with special reference to the study area, see HARTMANN 2013.



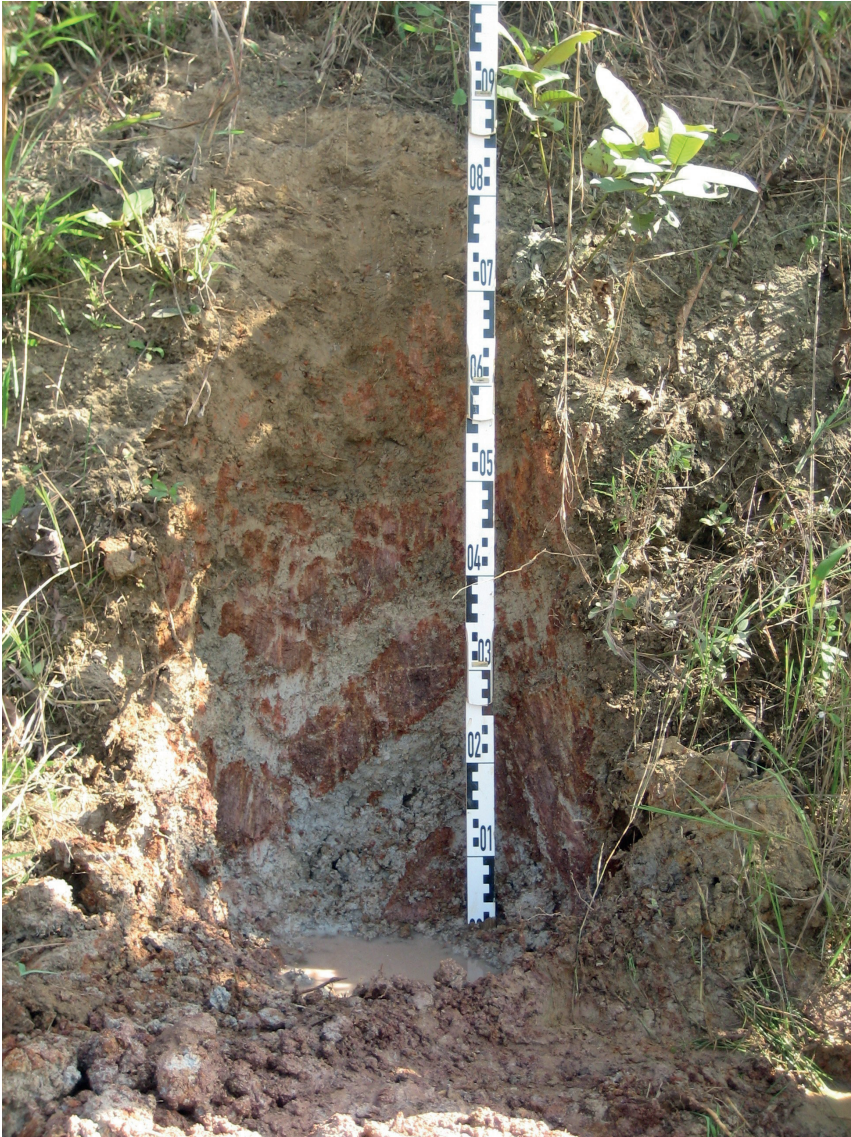


Fig. 6. Soil profile near Nok village. Layers of grey clay between layers of red laterite. Photo taken in 2009.

the relatively high concentration of clay, increasing with depth. Typically, Lixisols and Acrisols have developed on upper and lower pediment positions respectively. Both are underlain by slope deposits. Plinthosols are the dominant soil type in the upper plains with – again – slope deposits as parent material; while Fluvisols are the typical soil type in valleys, with river deposits as parent material (WALL 1979).

### 3.3.3 Clay Minerals and Trace Elements

The most common clay mineral in tropical soils is generally kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$ ), followed by illite ( $(\text{K},\text{H}_3\text{O})(\text{Al},\text{Mg},\text{Fe})_2(\text{Si},\text{Al})_4\text{O}_{10}[(\text{OH})_2, (\text{H}_2\text{O})]$ ). Both are produced by the chemical weathering of aluminium silicate minerals such as feldspars and biotite. Additionally, goethite ( $\text{FeO}(\text{OH})$ ) can be found in the clay fraction. Although it is not a “classical” (*i.e.*, silicate) clay mineral but rather an iron-bearing oxide mineral, it is often counted among the clay minerals because its grain size is similar to that of clay minerals. In the area under consideration, kaolinite dominates at 50-70% by volume, with goethite representing approximately 20% and illite only approximately 5% in the clay fraction. This composition is typical of those northern Guinea savanna areas underlain by granites. The clay fraction brought in by the Harmattan dust – typically the clay mineral vermiculite ( $(\text{Mg},\text{Fe},\text{Al})_3(\text{Al},\text{Si})_4\text{O}_{10}(\text{OH})_2 \cdot 4\text{H}_2\text{O}$ ), chlorite ( $(\text{Mg},\text{Fe})_3(\text{Si},\text{Al})_4\text{O}_{10}$ ) and Ca-Na-feldspars ( $(\text{Ca},\text{Na})\text{Si}_2\text{O}_8$ ) – is negligible (HARTMANN 2013: 42-45; KABATA-PENDIAS & PENDIAS 1992: 45-48).

Aside from the major rock-forming minerals, a number of trace elements occur in the soil substrate; the elements considered in this study are Ba, Cr, Nb, Rb, Sr, V, Y, Zn and Zr.<sup>29</sup> They are subsumed under the term “trace elements” because they often exist only in quantitatively negligible amounts. Trace elements are inherited primarily from parent material, and their reactions during the weathering process differ greatly depending on the stability of the host mineral. The trace elements can occur as cations (*e.g.*  $\text{Rb}^+$ ) or anions (often in chemical compounds with oxygen, *e.g.*  $\text{CrO}_4^{2-}$ ). Depending on the amount of charge, therefore, they can be relatively reactive. Thus, trace element ions often attach themselves to several minerals replacing other ions. Since clay minerals have a high sorption capacity, they act as an important trace element sink.

Briefly, the chemical properties, weathering resistance, and mobility in the soil of the trace elements considered in this study are as follows (KABATA-PENDIAS & PENDIAS 1992):

- **Barium (Ba)** belongs to the alkaline earths and occurs as  $\text{Ba}^{2+}$ , with an oxidation state of +2. In geochemical processes, it is mostly associated with  $\text{K}^+$  and therefore closely linked to the occurrence of alkali feldspars and biotite. In the soils, it is not very mobile and strongly absorbed by clays. As Ba is relatively resistant to weathering, its concentration in soils is similar to its concentration in the soils’ parent rocks.

<sup>29</sup> These particular trace elements were selected based on the technical capabilities of the Frankfurt project’s mobile XRF-analyser (for technical details, see Chapter 5.1).

- **Chromium (Cr)** varies highly in its oxidation states ( $\text{Cr}^{2+}$  to  $\text{Cr}^{6+}$ ) and is also known from complex anionic and cationic ions such as  $\text{Cr}(\text{OH})^{2+}$  or  $\text{CrO}_3^{3-}$ . Most of the Cr in the soil derives from chromite ( $\text{FeCr}_2\text{O}_4$ ), the most common Cr mineral and relatively resistant to weathering. With progressive oxidation, however, it forms chromate ions ( $\text{CrO}_4^{2-}$ ) which are highly mobile and can easily be absorbed by clays or hydrous oxides. Other Cr ions are less mobile but can be dissolved in highly acidic environments.
- **Niobium (Nb)** is most stable in the state of  $\text{Nb}^{5+}$ , but its chemical behaviour in weathering strongly depends on the host mineral. Thus, Nb may be released from biotite or amphibolite, *e.g.*, and then redeposited in silicates, whereas it remains more resistant in minerals such as sphene (titanite) or zircon. It is most abundant in acidic, magmatic rocks and less abundant in granites or gneisses.
- **Rubidium (Rb)** belongs to the alkali trace elements with a single electron in the outermost energy level ( $\text{Rb}^+$ ), making it highly reactive. It is often linked to K (Potassium) but has even stronger bonds to silicates such as feldspars, mica, and clay minerals. Its presence in the soils is largely due to the parent rocks, with the highest concentrations in soils over granites and gneisses and in alluvial soils. Rb has a relatively low resistance to weathering and is consequently mobile in the soils until it is incorporated into a silicate.
- **Strontium (Sr)** belongs to the alkaline earths and forms double positive cations ( $\text{Sr}^{2+}$ ). It is relatively common in the earth's crust and is often found in intermediate magmatic rocks where it is frequently associated with Mg (Magnesium). The soil's Sr content is controlled largely by the Sr content of parent rocks. Much like Rb, Sr is easily mobilised in the weathering process, especially in oxidising acid environments. Nevertheless, it is subsequently strongly fixed in clay minerals, but could be highly leached down the profile in acid soils.
- **Vanadium (V)** is a rare metal and is concentrated primarily in mafic rocks but is also abundant in soils derived from granitic bedrock. The geochemical characteristics of V strongly depend on its oxidation state – which varies between +2 ( $\text{V}^{2+}$ ) and +5 ( $\text{V}^{5+}$ ) – and the acidity of the surrounding soil. Mostly, V replaces other metals in the crystal structures, such as Fe (Iron), Ti (Titan), or Al (Aluminium). The mobility of V during weathering depends, as with Nb, on the host mineral, but when dissolved, it is often absorbed and incorporated into clay minerals or iron oxides. In general, it is distributed in the soil profile uniformly, and the variations in V content are inherited from the parent rock.
- **Yttrium (Y)** is quite common in the earth's crust and does not show any great differences in its abundance across different rock types. It is incorporated

(primarily as  $Y^{3+}$ ) into several minerals, most frequently into silicates, phosphates, and oxides. Yttrium is not very mobile in the soil and relatively resistant to weathering.

- **Zinc (Zn)** is a trace element of the alkaline earths. Most commonly, it occurs as the highly mobile  $Zn^{2+}$ , which is strongly bound in clay minerals, especially in oxidising environments. In environments with low pH values ( $< 7$ ), Zn is often leached out, resulting in a relatively low concentration.
- **Zirconium (Zr)** is a trace element belonging to the rare earth elements commonly found in rocks. The most stable and most frequently occurring form is  $Zr^{4+}$ , which most often bonds with oxygen in silicates. Its low solubility in water makes it highly resistant to weathering and therefore only slightly mobile in soils. A soil's Zr content is generally inherited from the parent rocks.

These trace elements and the primary rock-forming minerals Si, Al, Fe, Na, K, Ca, and Mg were analysed by XRF within the scope of this thesis (see Chapter 5 for an explanation of this method). They occur in different proportions in the clay – depending on the bedrock from which they derived and on the way of transportation through the landscape the clay has taken prior to its deposition. Their individual composition constitutes the chemical fingerprint of the clay source. Thus, they can be used as diagnostic elements when analysing the differences between domestic potsherds and the terracotta sculptures (Section C).





# **B**

## **Scientific Materials Analysis – Methods**



## 4 Mineralogical Analysis: Materials and Results

Ceramic objects consist basically of clay and temper – any form of non-plastic inclusions like minerals, rock fragments, organic particles like crushed shells or bones, and so on. The composition of the latter (*i.e.* type, amount, size, sorting, and angularity of the individual grains) can be analysed in thin sections using a polarising microscope to determine the optical properties of the non-plastic inclusions. The clay minerals, in contrast, merge during the firing process and therefore cannot be analysed in the thin sections directly. But they form the clay matrix, which in turn contains very small (< 0.05 mm) particles (mainly minerals like quartz, feldspars, mica, and heavy minerals), that are – because of their size – naturally occurring in the clay. These particles, together with the general impression of the clay matrix, may deliver some valuable information on the nature and origin of the clay. The analysis of the composition of the temper used in the terracotta sculptures and the pottery by means of thin sections was the subject of my Master's thesis (BECK 2008) and has already been discussed elsewhere.<sup>30</sup> Therefore, only a short summary of the results will be given here.

The investigations attempted to answer the following three questions:

- 1) Is the stylistic uniformity of the terracotta sculptures also traceable in their material composition in comparison to the domestic pottery?
- 2) Does the mineralogical composition of the terracotta sculptures differ according to their find site?
- 3) Which conclusions about the raw materials can be drawn from the answers to 1) and 2)?

### 4.1 The Importance of Clay and Temper for the Manufacture of Ceramic Objects

“Clay” in its pure form does not exist in nature – especially as “clay” rather describes a grain size (all particles < 0.002 mm (AG BODEN 1996: 134))<sup>31</sup> than a material. What is, however, generally referred to when using the term “clay” is a sediment that mainly consists of a mixture of different clay minerals, but as well of other particles of different grain sizes (silt, sand, gravel, stones) and organic material like roots or leaves. The amount, size, and nature of these

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<sup>30</sup> For a detailed account of the mineralogy see BECK 2012.

<sup>31</sup> The subsequent grain sizes are: silt (0.002 to 0.063 mm), sand (0.063 to 2 mm), gravel (2 to 63 mm), and stones (> 63 mm) (AG BODEN 1996: 134).

non-plastic inclusions – collectively subsumed under the term “temper” – determine the properties of the clay during the manufacture of ceramic materials (GIBSON & WOODS 1997: 257). Thus, potters have to take care that their amount matches the requirements of the future object. A cooking pot must have other characteristics (*e.g.*, a high thermal shock resistance) than a water jug (which must be open-pored, so that the clay can absorb the water and cool down its content via evaporation). Most of the times, it is necessary to alter amount or size of the naturally occurring temper particles: potters may remove some non-plastic inclusions and add others (organic like for example plant remains or crushed shells or bones; or inorganic like for example rock fragments or sand) to achieve the desired condition of the clay (RICE 1987: 52). Thus, ceramic objects always consist of two main components: clay and temper. The choice of both materials is a conscious decision by the potter; it does not occur accidentally.

Aside from determining the properties of the finished product, the proportions and composition of the temper are particularly important during the manufacturing process. Pure clay is too “fat” – it lacks the necessary strength to hold up the unfired walls of a vessel or figurine. Without the tempering material, the walls would collapse during manufacturing. During drying, coarse temper particles also ensure the opening of the clay body and thus a quicker and more uniform drying, which prevents cracks (ARNOLD 1989: 21-22). Another important purpose of temper has to do with firing: during initial heating, the chemically bound water evaporates and must escape from the clay body. Due to the porous structure caused by the temper, the water vapour can leak easily, otherwise the steam pressure would lead to cracks or spalling. The selection of the ideal mixture of clay and temper is particularly important when using open firing techniques, as temperatures in open firing rise quickly and cannot be controlled in the same way as in a kiln (GIBSON & WOODS 1997: 276).

Even if prehistoric potters were not aware of the precise physical and chemical significance of temper, they nevertheless knew which temper to use in what proportions for which object – experience and tradition taught them. The choice of the composition of the material was not accidental and often closely related to the culture in which it occurred: the precise selection of the different components can be used to separate out not only different manufacturing processes but also different cultures (GIBSON & WOODS 1997: 30-31). Temper is thus a good criterion for an initial assessment of unknown pottery material, not least because it is comparatively simple and inexpensive to analyse via thin sections. As noted above, the clay itself cannot be investigated by this method because the clay minerals melt during the firing process. But the resulting clay matrix can yield some valuable information about the nature and provenance

of the clay (RICE 1987: 52). To study the clay directly, other methods like X-ray fluorescence analysis are needed (Chapter 5).

## 4.2 Materials and Method

To answer the questions posed above, 70 thin sections (36 terracotta and 34 pottery samples serving as comparison material for the figurines) from 15 sites (Fig. 7) were analysed.<sup>32</sup> When choosing the samples, care was taken to cover as big an area as possible to gain a broad overview of the mineralogical composition of the material and any variations in it. Finds from excavations or at least smaller test pits were favoured over surface finds. Care also was taken to select representative samples (*i.e.* not being unusual in their macroscopic appearance in comparison to the whole excavated material) from the respective sites to avoid sampling bias. In some cases, however, visibly unusual sherds (*e.g.*, unusually coarse or fine tempered pottery) were selected in addition to answer specific questions (BECK 2008: 21-22).

Thin sections consisted of covered sections with the standard thickness of 30  $\mu\text{m}$  (0.03 mm). They were analysed using a polarising microscope with plane- and cross-polarised transmitted light. The inclusions were identified by their optical properties (colour, pleochroism, extinction angles, and birefringence) as well as shape and cleavage. For each individual component, the percent by volume as well as sorting and the degree of rounding were determined and recorded using printed reference charts (Fig. 8-10). All particles smaller than 0.05 mm were taken as belonging to the clay matrix and were recorded separately. Additionally, the amount, sorting, and shape of the pores were recorded. All information was collected in Excel sheets and used for exploratory data analysis (BECK 2008: 29, 31-37; BECK 2012: 254).

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<sup>32</sup> For the Master thesis 33 thin sections of pottery and terracotta from seven sites were analysed. To verify those results, additional 37 thin sections of both archaeological materials from eight sites belonging to the Nok Culture were investigated within the scope of the doctoral thesis. In the following, all thin sections will be discussed together.

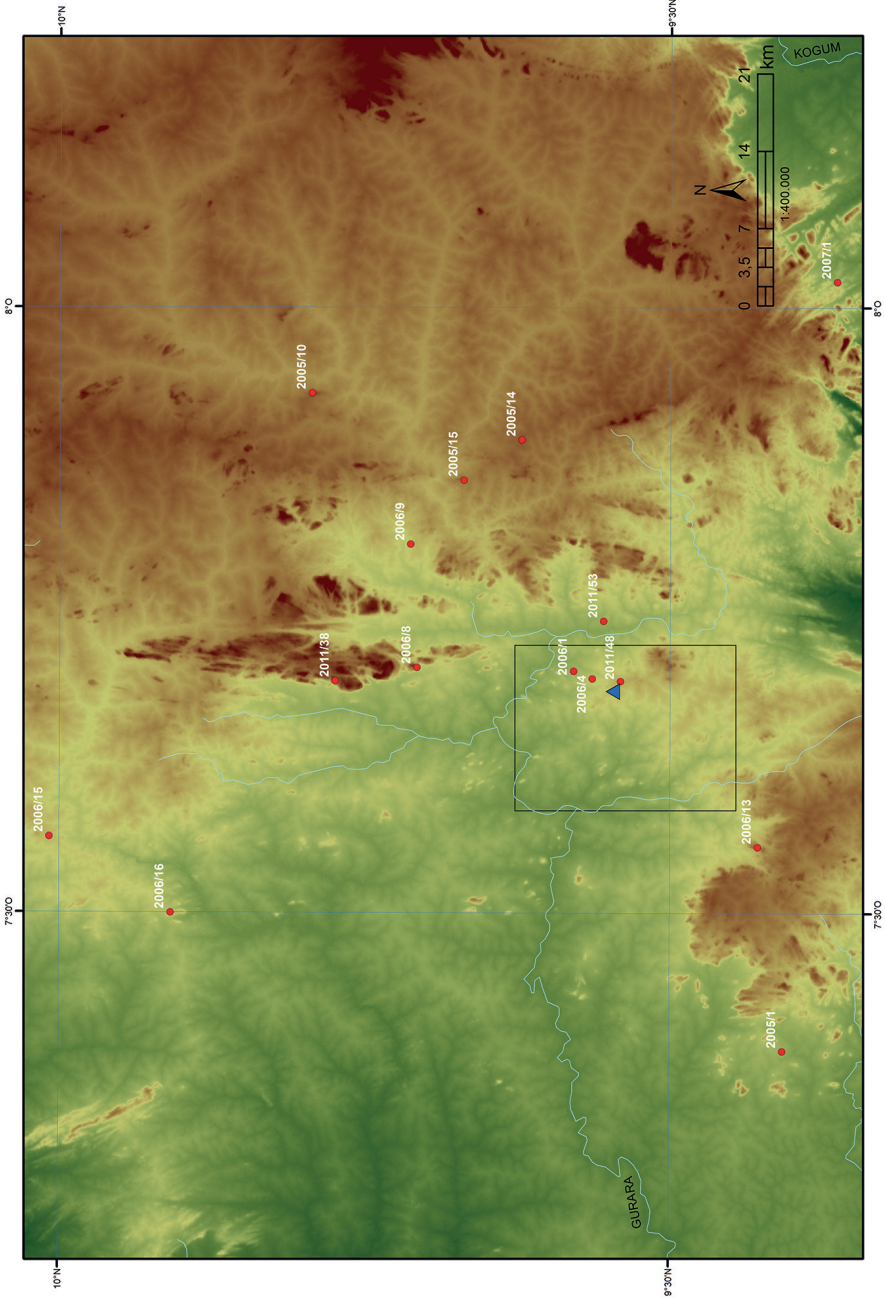


Fig. 7. Map showing the archaeological sites for which thin section samples are available. The box in the centre indicates the project's key study area with the research station (blue triangle) (map by Eyub F. Eyub).



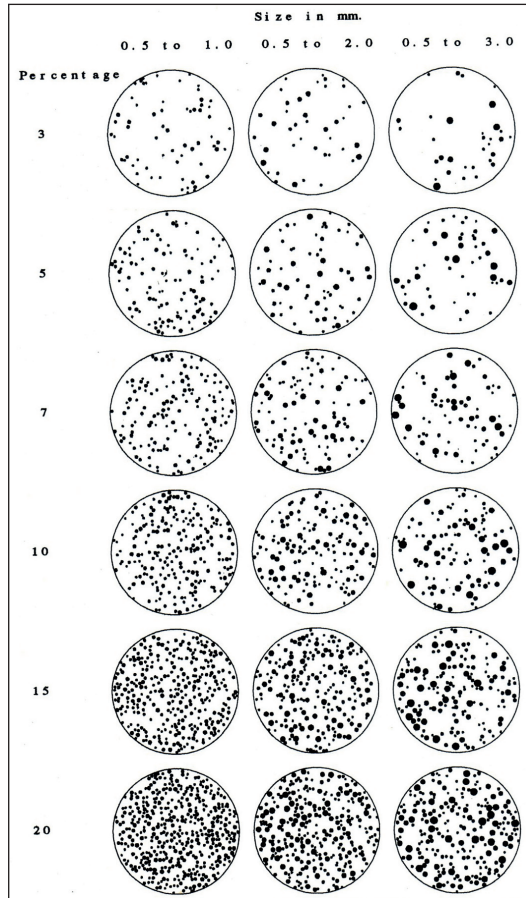


Fig. 8. Reference chart for the estimation of the percent by volume of the particles in the thin sections (after MATTHEW ET AL. 1991: 218).

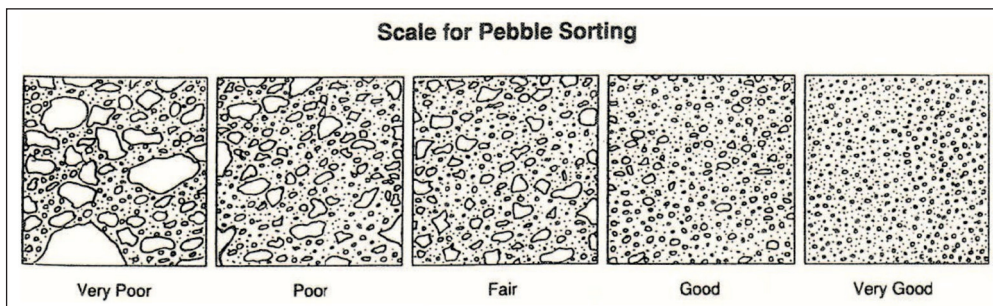


Fig. 9. Reference chart for the estimation of the degree of sorting in the thin sections (after ORTEN ET AL. 1993: 239, Fig. A.6).













Class	1	2	3	4	5	6
	Very-Angular	Angular	Sub-Angular	Sub-Rounded	Rounded	Well Rounded
High Sphericity						
Low Sphericity						

Fig. 10. Reference chart for the estimation of the degree of roundness (shape) of the particles in the thin sections (after ORTEN ET AL. 1993: 239, Fig. A.5).

## 4.3 Results of Microscopic Analysis

### 4.3.1 Clay Matrix

Unexpectedly, the most valuable source of information for distinguishing between the figurines and the domestic pottery proved to be very small quartz grains (< 0.05 mm) in the matrix. Grains of this size can be classified as coarse silt (grain sizes between 0.02 and 0.063 mm), and thus have a size comparable to flour (AG BODEN 1996: 139). The matrix of the terracotta figures is characterised by an average quartz content of approximately 3% (Fig. 11), while the concentration in the matrix of the pottery fluctuates more strongly, around approximately 4% (Fig. 12).

These grains are so small that they cannot be seen macroscopically in the clay; it is therefore highly unlikely that they were added deliberately by the potter. However, it is possible that these small quartz fragments derive from the homogenisation of the clay (*e.g.* pounding in a mortar or grinding of the clay on a grinding stone to crush the larger non-plastic inclusions).<sup>33</sup> In this way they could have been added, albeit unknowingly, by the potter. This

<sup>33</sup> See Chapter 5.5 for a description of clay processing in central Nigeria today.



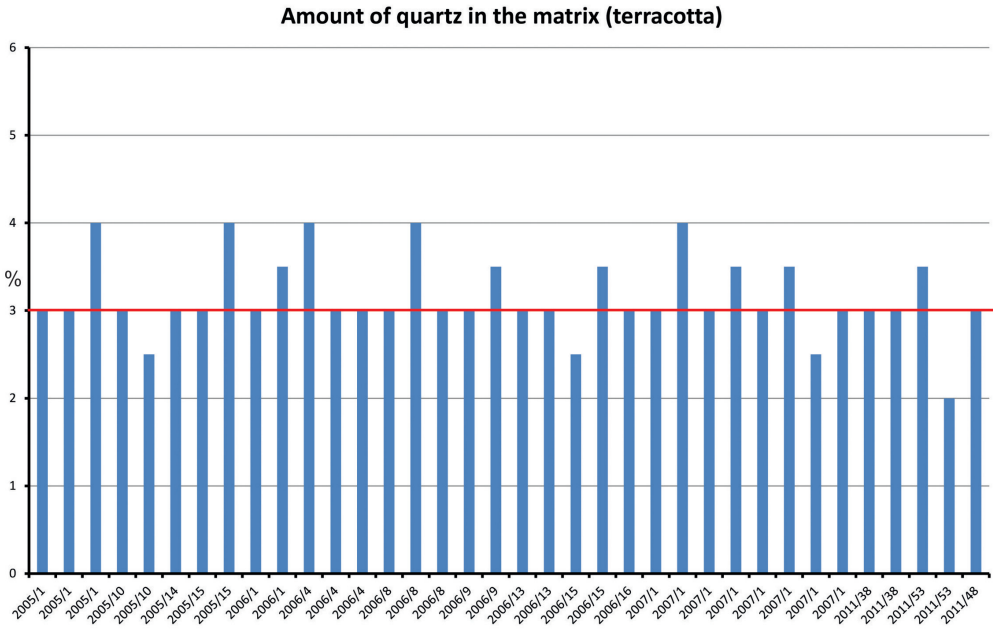


Fig. 11. Amount of small (< 0.05 mm) quartz particles in the matrix (terracotta); red line: 3%. Samples sorted by catalogue number of sites.

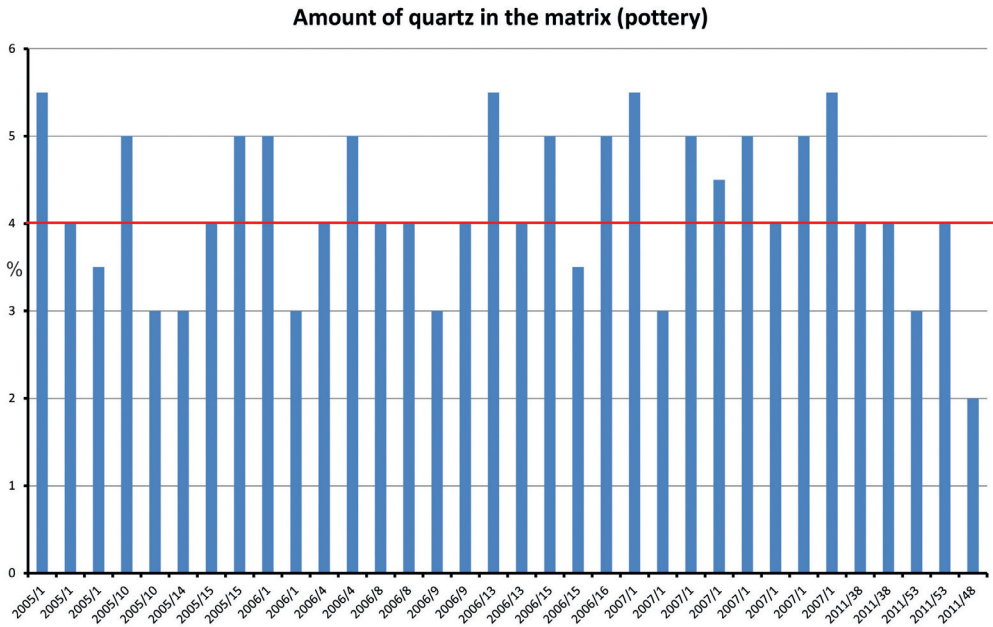


Fig. 12. Amount of small (< 0.05 mm) quartz particles in the matrix (pottery); red line: 4%. Samples sorted by catalogue number of sites.

might explain the varying amounts of these small quartz grains in the matrix of the pottery – here, different proportions of the fine powder (*i.e.* the small quartz grains) that for example accumulates at the bottom of the mortar could have been randomly inserted in the clay. But this does not explain how the potters of the Nok Culture managed to achieve a constant amount of 3% of small quartz grains in the clay used for the figurines. Instead, it is most likely that they occur naturally in the clay and are therefore a material property that characterises different clay sources.

Therefore, this finding suggests the use of special clay sources for the manufacture of the figurines. It is simply possible to test clay for such small grains in the matrix by rubbing it between the fingers. Potters usually choose their clay based on features that influence its characteristics during forming, drying, and firing: plasticity, workability, thermal shock resistance, colour development during firing, or drying behaviour (RICE 1987: 52-53). These are not influenced by the small quartz particles, so the particles' different proportions in the pottery and the terracotta do not have manufacturing reasons. Evidently potters chose this special clay for other unknown, possibly ritual causes (see Chapter 9 for a theoretical discussion).

#### **4.3.2 Temper**

Both terracotta and pottery were tempered with the same inorganic particles: quartz, alkali feldspar (orthoclase), soda-lime feldspar (plagioclase), black mica (biotite), common mica (muscovite), pyroxene, amphibole, tourmaline, myrmekite, heavy minerals, rock fragments of quartzite and granite, as well as grog (crushed ceramic material). Grain sizes vary between 0.05 and almost 5 mm. Organic temper is completely absent (BECK 2008: 38, 42). The macroscopic impression that the terracotta figures contain slightly more temper than the potsherds was confirmed. While the pottery contains an average of approximately 20% temper materials (Fig. 13), the terracotta temper contains around 25% (Fig. 14). In addition, the comparison of particle size shows that the temper of the figurines is slightly coarser (BECK 2008: 42-47).

This difference may have technological reasons. A higher amount of non-plastic inclusions ensures that the large figurines do not break during forming, drying, and firing. The ratio of 25% temper within the clay represents the perfect composition for an open pit-firing (ARNOLD 1989: 22), which is the most probable method used for the ceramic material of the Nok Culture.<sup>34</sup> The amount of temper materials in the pottery fluctuates more strongly but

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<sup>34</sup> See Chapter 4.3.3 for a discussion of the firing technique.

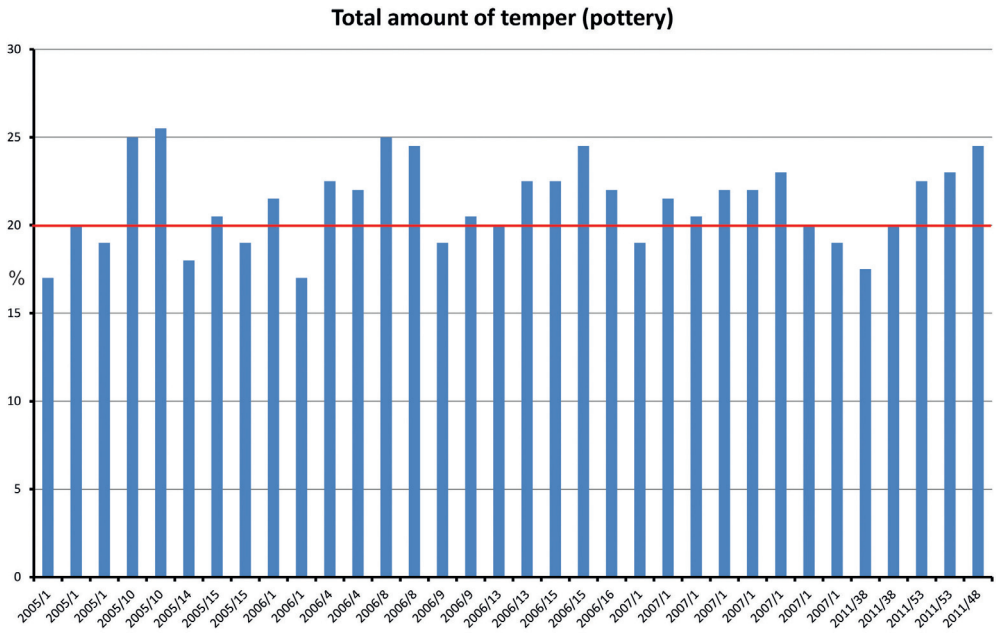


Fig. 13. Total amount of tempering material in the pottery; red line: 20%. Samples sorted by catalogue number of sites.

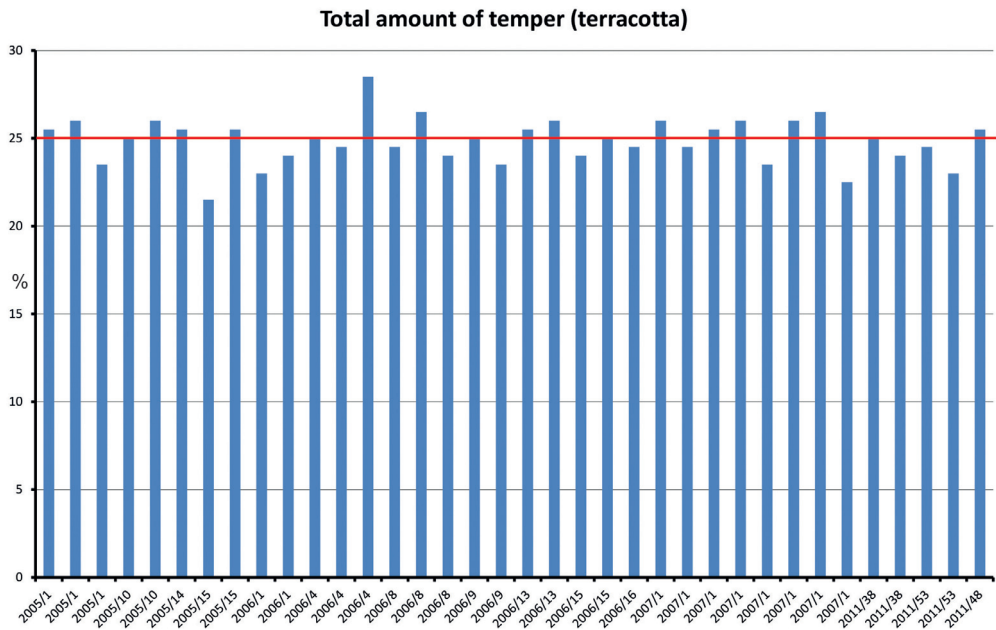


Fig. 14. Total amount of tempering material in the terracotta; red line: 25%. Samples sorted by catalogue number of sites.

also lies within the optimum range between 20 and 25%. Presumably, it was not necessary to use the precise optimum mixture to manufacture the pottery because the vessels are much smaller and less complicated in form. Although the same components were used for both ceramic materials, they obviously underwent two different manufacturing sequences.

Besides tracing the differences between terracotta and pottery in the mineralogical composition, the temper can provide useful information about the provenance of a clay object if the geological variation within a given area is sufficiently large. According to ethnographic research, non-plastic inclusions that are added deliberately by the potter are not transported over greater distances (GIBSON & WOODS 1997: 265; PEACOCK 1970: 379). Thus, the different temper particles and their concentrations (if added by the potter) can be used to determine or at least narrow down the area where the clay object was manufactured.

This determination, however, which particles occur naturally in the clay and which were added, is not always possible. All minerals found in the Nok material are products of the weathering of granites and gneisses – the main rock types in the area under consideration. Thus, their occurrence in the clay may be partly natural and partly due to the potter. This problem can be solved by analysing other properties like sorting or grain shape. Clearly rounded and well-sorted grains are often considered as coming from a natural source, their rounded form being the result of a long transport prior to their sedimentation in the clay. Highly angular and poorly sorted grains, in contrast, are often considered deliberate additions by the potter, deriving from crushing of stones. An argument against this assumption is that, on the one hand, well-sorted sand, which often was transported over longer distances by wind or water and therefore is highly rounded, is often intentionally used as temper; on the other hand, coarse, poorly sorted grains may also be the result of weathering processes (GIBSON & WOODS 1997: 257-261). Thus, the decision as to which non-plastic inclusions can be used for looking for the potential place of manufacture of the terracotta and the pottery is not simple. Only three ingredients are most probably deliberate additions: the rock fragments of quartzite and granite<sup>35</sup> as well as grog (*i.e.* crushed potsherds). The latter, however, is not very helpful in tracing differences between terracotta and pottery, as it occurs in the same concentration in both materials.

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<sup>35</sup> These rock fragments may also (at least partly) occur naturally in the clay and be crushed during the homogenisation process like for example pounding the clay in a mortar or grinding it on grinding stones, as can be observed in Nigeria today (see Chapter 5.5). Nevertheless, their presence in the clay reflects an intentional decision of the potter as they were not removed from the clay prior to the homogenisation.

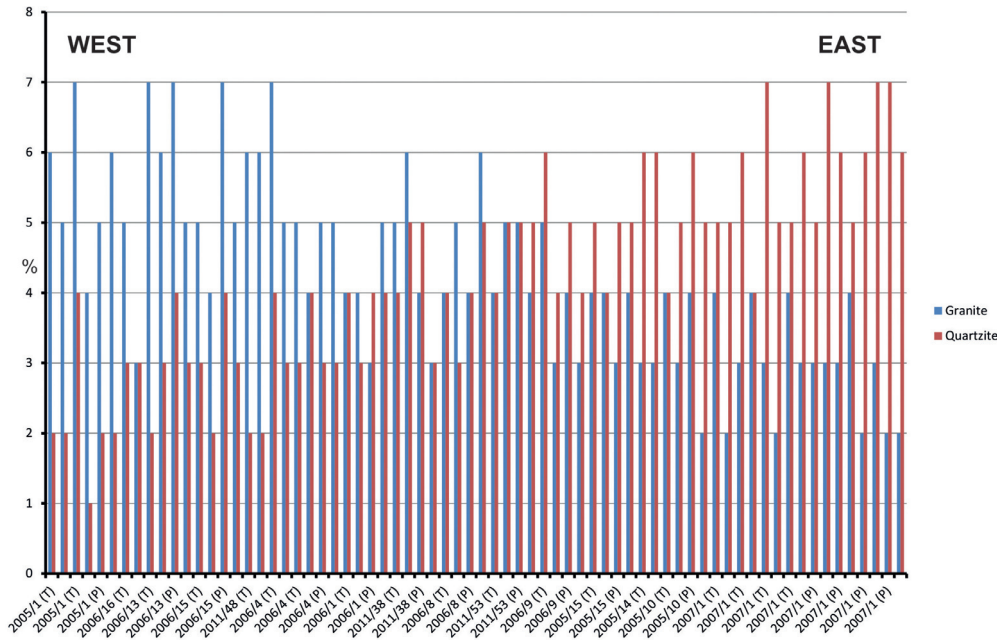


Fig. 15. Comparison of granite (blue) and quartzite (red) in the temper of 70 thin sections of pottery (P) and terracotta (T). Samples sorted geographically from West to East.

A closer look at the quartzite and granite reveals no significant difference between the terracotta figurines and the pottery, but does reveal differences between the sampled sites. The ones lying in the western part of the research area are tempered primarily with granite, while finds from the eastern sites are tempered mainly with quartzite (Fig. 15).

By taking the other tempering materials (quartz, feldspars, mica, amphiboles, and pyroxenes) into account, this observation was verified using statistical cluster analysis (BECK 2008: 49-57). This regional pattern is probably due to the use of local rock deposits. Quartzite is easier to crush than the clearly harder granite, and quartzite flakes are often found on Nok sites. As quartzite is less common in the research area than granite, it seems to have been preferred where available, which is more often the case in the eastern regions near the Jos Plateau. The use of local raw materials for temper is not surprising, especially in case of heavy rock fragments that are readily available. ARNOLD (1989) has surveyed ethnographic literature from all over the world to quantify the size of the areas potters use to acquire their raw materials. The average distance potters travel to reach the source of temper materials (especially stone) is approximately 4 km. The average distance travelled to the nearest clay source is only slightly greater at 6 km (ARNOLD 1989: 50). Unfortunately, the geological

situation in central Nigeria is too undifferentiated and the two rock types too common to draw any detailed conclusion about the potential provenance of the terracotta and pottery. These results nevertheless clearly speak for the use of – at least concerning the tempering materials – local raw material deposits for the manufacture of these two archaeological find categories.

### 4.3.3 Manufacturing Technique

In addition to the analysis of the composition of the tempering materials, thin sections can be used for reconstructing manufacturing techniques of both pottery and terracotta. During forming, elongated clay additions (*e.g.*, plant remains or some minerals like mica) and the pores in the clay matrix orient perpendicular to the direction of the exerted force (GIBSON & WOODS 1997: 217-219). This orientation could be observed in most of the samples (Fig. 16). Where it was not found, either the force was not high enough to affect the pores and particles, or the selected orientation of the individual thin section was so unfavourable that the phenomenon was not visible. As the sherds used for thin sections were not always diagnostic (*e.g.*, a part of the vessel wall or of the body of a terracotta figure), the exact orientation of the sample – and therefore of the pores and elongated ingredients in the thin sections – could not be determined for each sample. In some cases, however, it was possible. In the case of the pottery, the orientation is perpendicular to the rim. The same could be observed in the terracotta figures, where “rim” is taken to be the head (or the hands/feet in arms or legs, respectively). This suggests the typical forming techniques that are still in use today in Nigeria: a combination of coiling and pinching, which pulls and draws the clay upwards from the bottom to thin out the walls.<sup>36</sup>

The thin sections also reveal that today’s coarse appearance of the figurines and potsherds caused by the large temper particles does not correspond to their original appearance at the time of the manufacture. All samples, regardless of whether terracotta or domestic pottery, were coated with a – mostly red – slip or engobe. Being buried in the acidic soil for over two millennia caused the erosion of most of the original surface, and the smooth top coating can today be seen macroscopically only in exceptional cases. Under the microscope, however, traces of the slip are still visible – as much finer-grained material on the surface of the sample (Fig. 17).

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<sup>36</sup> For a more detailed description of the pottery manufacturing technique, see FRANKE 2014: 170. The making of a forgery of a Nok terracotta with similar techniques is described by BREUNIG & AMEJE 2006.



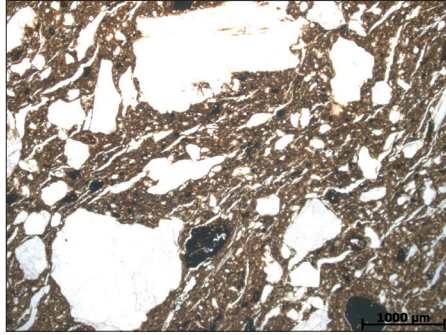


Fig. 16. Thin section of a pottery sample from Ungwar Kura (2007/1) with oriented pores reflecting the manufacturing technique. Plain polarised light.

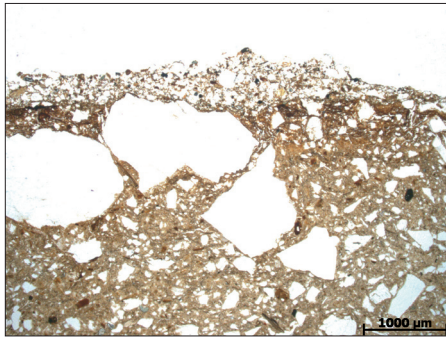


Fig. 17. Thin section of a terracotta sample from Kochio (2005/10) with traces of a fine-grained slip. Plain polarised light.

With the help of the thin sections, it was also possible to reconstruct the firing process. Some samples show darker (brown and black) and lighter (red) “stripes” caused by the different phases of burning of carbonaceous material or by the different oxidation states of the iron oxides in an uneven firing atmosphere. Such stripes are typical of the varying temperature conditions of open pit-firing (RICE 1987: 335), a technique that is still used in Nigeria and must be assumed for the pottery and terracotta of the Nok Culture. Another argument for open pit-firing is the unaltered appearance of the quartz particles of the temper. Open pit fires reach temperatures of approximately 700°C, while kilns reach 1000°C and more. There, additionally, the temperature can be kept constant over longer periods of time (RICE 1987: 156-158). When exposed to extreme heat (900°C and higher), the crystalline structure of quartz starts to “weather”, resulting in cracks and a change in colour from bright white to dirty grey (pers. comm. A. Röpke, January 2015). Such an alteration of the quartz could not be observed in the quartz particles of the pottery and

terracotta, indicating that they were never exposed to such heat and thus not fired in a kiln.

## 4.4 Summary

The mineralogical investigations provide some answers to the questions posed above:

1) The potters who made the terracotta figurines followed not only a stylistic but also a material-related norm in manufacturing the terracotta figurines. In comparison to the domestic potsherds, the figurines are tempered with more and coarser non-plastic inclusions than pottery, probably to match the technological requirements of the manufacturing process. Surprisingly, the clay matrix proved to be the best attribute to trace the material difference between these two archaeological object groups. The clay matrix of the statues contains a homogeneous proportion of small quartz grains, while this proportion fluctuates more strongly in the matrix of the pottery. Therefore, clay with a very distinctive material property was used for the terracotta sculptures. Pottery and terracotta probably derive from two different manufacturing sequences.

2) A detailed look at the two most likely deliberately added non-plastic inclusions, quartzite and granite, shows that local tempering materials were used. Samples (equally pottery and terracotta) from sites lying in the western part of the researched area mainly contain granite, while the ones in the east mainly contain quartzite.

As the clay matrix of the terracottas suggests the use of clays with a very distinctive material property (in the thin sections displayed by the small quartz grains that occur naturally in the clay) for the manufacture of the figurines, the focus was shifted to the further analysis of the clay. Thin sections are not helpful in this respect, as the clay minerals melt during the firing process and lose their optical characteristics, which prevents the clay itself to be analysed by this method. But the firing process does not affect the chemical properties of the clay. Thus, X-ray fluorescence (XRF) analysis (see Chapter 5 for a description of the method) has been chosen to determine the chemical composition of the clay of the terracottas, and again of the domestic potsherds as comparison material. By this, the hypothesis of the use of special clay sources for the terracotta figurines – in combination with a controlled production of the statues as objects of high social value by specialists – can be tested and discussed further. The implications of these results for the social organisation of the Nok Culture will be presented in Chapter 9.



## 5 Geochemical Analysis: Methods and Materials

As shown in Chapter 4, the clay has proven to be the best distinguishing criterion between the terracotta figures and the pottery. The potters' conscious choice of a clay with a distinctive – non-technical – material property for manufacturing the figurines shows that a (invisible) material norm existed in combination with the visible stylistic norm. The material composition thus can be used to trace the figurines' cultural value and symbolic function.

Clay sources feature a unique chemical composition (comparable to a fingerprint), deriving from the bedrock from which the clay minerals developed during weathering. Additionally, the clay is influenced by the geology of the area through which the minerals were transported prior to their deposition. The chemical composition can thus be used to trace different clay sources, assuming that the differences within a source are smaller than differences between sources (RICE 1987: 413-414).

There are several geochemical methods for determining the chemical composition of materials. The different methods are based on the physical or chemical properties – mainly deriving from the structure of the atom – of the material under analysis. Methods like Optical Emission Spectroscopy (OES), Atomic Absorption Spectroscopy (AAS), or Inductively Coupled Plasma Mass Spectrometry (ICP-MS) are based on visible light, whereas techniques like X-ray Fluorescence Spectrometry (XRF), Analytical Electron Microscopy (AEM), or Proton-Induced X-ray Emission Spectrometry (PIXE) use X-rays. Other techniques include Neutron Activation Analysis (NAA) and mass spectrometric and chromatographic techniques. Especially XRF, OES, and NAA have traditionally been used to group potsherds according to their chemical composition.<sup>37</sup> The choice of the appropriate method depends on the precise research question and not least on the availability and affordability of different technologies, since most techniques are very costly. Therefore, only small sample series usually were analysed, as archaeometric investigations required expensive and costly preparations which would have resulted in at least partial destruction of the archaeological material. In recent decades, however, geochemical techniques have advanced considerably and the quality of results has increased, resulting in lower detection limits and smaller sample sizes. Yet, limiting factors continued to be high cost and the destruction of sample material (TITE 2008). A new development has helped lessen this

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<sup>37</sup> Both, case studies and comparisons of the different methods, are numerous and published in the respective journals (*e.g.*, *Archaeometry* or *Journal of Archaeological Science*). A complete review is beyond the scope of this study, but comprehensive overviews are given in MOMMSEN 1986, NEFF 1992, POLLARD & HERON 2008, SCHNEIDER 1989, and WAGNER 2007.

problem: mobile, handheld XRF-analysers have enabled researchers to cost-effectively study a large number of samples in a relatively short time. Their biggest advantage, however, is that samples no longer have to be prepared and measured in laboratories but can be analysed directly and non-destructively on site. Thanks to a generous donation of the William Buller Fagg Charitable Trust, the Frankfurt project was able to purchase such a mobile XRF-analyser: Thermo Fisher Scientific Niton's model Xl3t 900SHe+<sup>38</sup> (Fig. 18). With this device, a large number of non-destructive measurements of the Nok ceramic material have become possible.



Fig. 18. The mobile XRF-analyser of the Frankfurt project.

In the following, a brief description of the XRF operating principle and the significant parameters of the mobile device will be given, followed by a description of the measuring procedure itself, the multivariate statistics necessary for the interpretation of the measurement results, and the sample selection. The chapter is concluded by a side note on today's clay procurement and processing in the area under consideration.

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<sup>38</sup> In the following, the mobile XRF-analyser of the Frankfurt project will be called "device".

## 5.1 X-Ray Fluorescence – Method

XRF is a fast method of analysing the elemental composition of nearly all materials. The latest development of small, mobile devices has led to a wider usage of the method, especially as now non-destructive measurements become possible. Initially developed for use in metal working to sort different metal alloys, the handheld analysers also found a quick reception in archaeology and are used there to analyse substances like pottery, metal, glass, stone, or even soils (HELFERT & BÖHME 2010: 15).

### 5.1.1 Operating Principle

As the name suggests, XRF directs X-rays, generated by an X-ray tube, at a sample. Within the electromagnetic spectrum, X-rays have wavelengths between  $10^{-8}$  and  $10^{-11}$  m and therefore lie within the range of the atomic distances of the crystalline structure. This means that their energy is high enough to “knock” an electron out of an atom’s innermost orbitals or atomic shell. This creates a “vacancy” or “hole” in the electronic structure – a condition that is extremely unstable and that is therefore compensated by another electron from a higher energy level “dropping down” to the vacant position. As the electron now is bounded to a lower energy level, it emits surplus energy in the form of (secondary) X-rays. This emitted radiation – which is characteristic of the different atoms and thus the different chemical elements – is measured by the device and reveals a complete elemental spectrum of the sample (Fig. 19).

In general, there are two ways of inferring the chemical element from the measured radiation: by wavelength (known as wavelength-dispersive XRF or WD-XRF) or by energy (known as energy-dispersive XRF or ED-XRF).<sup>39</sup> The former technique makes use of the fact that the X-ray coming from the sample has a wavelength that is characteristic of the atom from which it derives. The different incoming wavelengths are dispersed through an appropriate prism (a crystal with a suitable spacing in its crystal structure) to separate them into the different components. The beam is then detected and the elements counted according to their wavelength. Devices operating on the basis of energy dispersion measure the energy of the X-ray photons. This is achieved by using a single crystal of silicon, doped with lithium to reduce electronic impurities in the crystal. The beam strikes the crystal, which absorbs the energy, leading to the creation of a large number of electron-hole pairs. In a silicon/lithium crystal, each electron-hole pair requires an energy of 3.8 eV – thus the number

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<sup>39</sup> The device of the Frankfurt project works with ED-XRF.

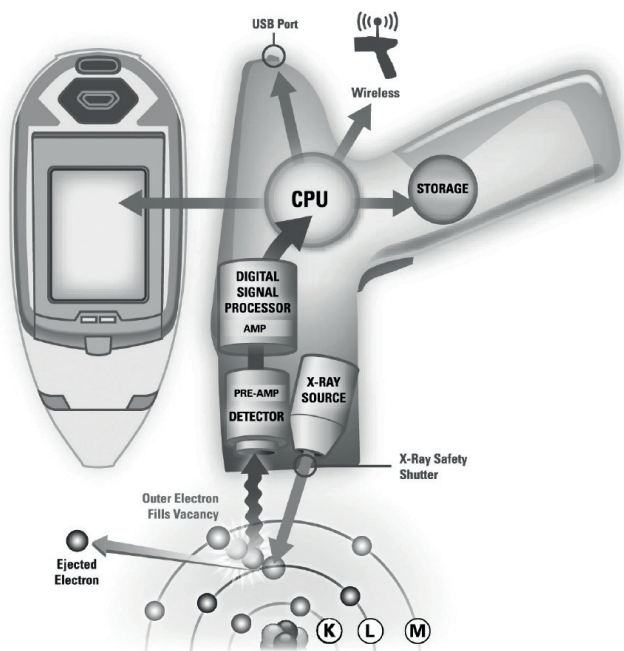


Fig. 19. Operating principle of the device (HELFFERT & BÖHME 2010: 14, Fig. 2).

of pairs created is proportional to the energy of the incoming beam divided by 3.8. To measure the energy, a voltage is applied to the crystal and the freed electrons move towards the anode. This creates an electric current with a magnitude proportional to the energy carried by the incoming photons. This current is measured, and the energy derived translated to a spectrum of elements. Thus, all elements in the sample can be recorded at the same time (POLLARD 2008: 33-43).

Analytically, there is virtually no difference between ED-XRF and WD-XRF. It has generally been assumed that WD-XRF has a lower (*i.e.*, better) detection limit and greater precision, especially concerning the lighter elements; detailed comparisons (*e.g.*, POTTS ET AL. 1985) have shown that this is not necessarily the case. Furthermore, devices operating with WD-XRF are much more costly and require laboratory conditions. In addition, they are relatively slow in measuring, and the detecting crystal has to be changed according to the elements the researcher is trying to measure. Consequently, this technique has seldom been used in archaeology apart from a few ceramic studies. In contrast, ED-XRF is much more cost-effective and faster to operate, with similar detection limits and precision and is therefore widespread in archaeology (POLLARD 2008: 44).

Recently, a new type of XRF has been developed for special problems in archaeological use. Micro-XRF ( $\mu$ -XRF) in combination with ICP-MS is used especially for the detection of individual trace elements (*e.g.*, rare earth elements) and to analyse a wider element spectrum with a lower detection limit. However,  $\mu$ -XRF is a stationary technique which requires relatively complex sample preparation; it is therefore a destructive method. A comparison of  $\mu$ -XRF with portable ED-XRF analysis has revealed that the two methods are compatible with one another and systematic deviations can be adjusted (HELFFERT ET AL. 2011).

### 5.1.2 Device Parameters

The Frankfurt project's mobile XRF-analyser uses ED-XRF technology. The device is equipped with a Silicon-Drift-Detector (SDD) that was specifically developed for small, handheld instruments and is termed GOLDD-Technology (Geometrically Optimized Large Area Drift Detector) by Thermo Fisher Scientific. In this way, any element with an atomic number greater than 12 (Magnesium) can be measured. No vacuum is needed, although it is possible to use helium purging to improve the measurement of the light elements. Just like stationary laboratory instruments, it is equipped with an X-ray tube with an Ag anode (50 kV/2 W/100  $\mu$ A max.). Measuring times are short, ranging from a few seconds to several minutes, depending on the task. The small-spot focus and the integrated camera allow exact control of the analysed point even on small samples. They also make it possible to answer concrete questions (including photo documentation of the specimen) regarding *e.g.* measurements along a weld seam, or different alloys or decors of potsherds. With these features, the handheld device is in no way inferior to "conventional", stationary laboratory equipment (HELFFERT & BÖHME 2010: 14-15).

#### 5.1.2.1 Sensitivity

The limit of detection (LOD) in a SiO<sub>2</sub> matrix for the Xl3t 900SHe+ with GOLDD detector is specified by Thermo Fisher Scientific as stated in Tab. 1. The LOD was calculated according to the IUPAC (International Union of Pure and Applied Chemistry) convention as the  $3\sigma$  value for a measurement duration of 60 seconds per filter.<sup>40</sup> It should be noted that these values are strongly

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<sup>40</sup> The device is equipped with four filters: main, low, high, and light filter for the different energy ranges of the X-ray spectrum. Each filter focusses on different elements. See Chapter 5.2.2 for further explanation.

dependent on the sample's geometry (the homogeneity of the matrix). This is especially true for the elements Mg, Al, Si, P, and S (HELFERT & BÖHME 2010: 15).

Limit of detection (LOD), 3σ, ppm, (60s/filter)		Limit of detection (LOD), 3σ, ppm, (60s/filter)	
Element	XI3t 900SHe+ GOLDD	Element	XI3t 900SHe+ GOLDD
<b>Ba</b>	50	<b>Cu</b>	12
<b>Sb</b>	15	<b>Ni</b>	22
<b>Sn</b>	16	<b>Co</b>	15
<b>Cd</b>	8	<b>Fe</b>	25
<b>Mo</b>	3	<b>Mn</b>	30
<b>Nb</b>	3	<b>Cr</b>	25
<b>Zr</b>	3	<b>V</b>	12
<b>Sr</b>	8	<b>Ti</b>	6
<b>Rb</b>	6	<b>Ca</b>	70
<b>Bi</b>	3	<b>K</b>	250
<b>As</b>	5	<b>Cl</b>	150
<b>Se</b>	4	<b>S</b>	150
<b>Au</b>	15	<b>P</b>	600
<b>Pb</b>	4	<b>Si</b>	-/-
<b>W</b>	50	<b>Al</b>	2000
<b>Zn</b>	6	<b>Mg</b>	2.5%

Tab. 1. Limit of Detection of the device for the different elements (HELFERT & BÖHME 2010: 16). Because of the measurement in a SiO<sub>2</sub> matrix, no value for Si could be given.

### 5.1.2.2 Depth of Information

The depth of information (the distance below the sample's surface on which information is available and/or can be processed) is a particularly important measure for the handheld devices, as they are used non-destructively most of the time. The crucial factor in determining XRF depth of information is not the depth of penetration of the X-rays but the depth from which the emitted X-ray photons reach the detector. On their way out of the sample, the secondary X-rays are partly absorbed by the surrounding material and thus lose energy. Therefore, the higher the energy at the point of emission, the greater the distance the photon can cover until its energy is too low to be detected by the device. Thus, every element displays a different depth of information that is additionally dependent on the density of the matrix. The



depth of information can vary considerably and be quite difficult to determine – especially in potsherds with non-plastic inclusions (temper) which vary in size and composition. A model calculation (HELFFERT & BÖHME 2010: 17) yielded a depth of information in the range of only  $\mu\text{m}$  for the (light) elements Mg, Al, Si, P, S, and Cl; of several dozen  $\mu\text{m}$  for K, Ca, and Ti; of several hundred  $\mu\text{m}$  for Cr, Mn, Fe, Cu, Zn, As, Au, Hg, and Pb; and within the millimetre range for the (high energetic) elements Rb, Sr, Y, Zr, Nb, Ag, Cd, and Ba. Heterogeneous samples like ceramics should therefore, especially when looking at the lighter elements, be measured several times in different places to rule out possible matrix effects. For certain specific questions, it may after all be necessary to prepare (and therefore destroy) a sample (HELFFERT & BÖHME 2010: 16-18).

## 5.2 Measurement Prerequisites

Several steps, including establishing a replicable measuring protocol, were necessary before the analysis of Nok material could begin. First, the samples were washed with clear water and dried completely. To ensure comparability, raw clay samples (Chapter 5.4) were formed into small bricks and fired in an open pit to match the firing conditions of the pottery and terracotta sculptures. Subsequently, basic technical requirements had to be established and several tests had to be conducted to define the measuring procedure.

### 5.2.1 Calibration and Comparison of WD-XRF and ED-XRF

The device supports different measuring modes that may be chosen depending on the nature of the sample. The mode “mining / minerals with Cu/Zn” is used for ceramics, because it is specific to silicate matrices such as clay objects. The basic calibration of these modes is conducted by means of fundamental parameters on pure element standards (HELFFERT ET AL. 2011: 6). Nevertheless, all measuring modes need to be calibrated on the material intended to be measured prior to their application. This calibration work can be used to directly compare the precision of the two XRF methods (stationary WD-XRF *vs.* mobile ED-XRF). To adjust the device parameters to the requirements of the relatively porous clay matrix, 20 samples from Nok terracotta sculptures and domestic pottery (10 samples each) had previously been analysed by M. Daszkiewicz in the “ARCHEA – archaeometric analysis and research” laboratory in Warsaw, Poland, using stationary WD-XRF. ARCHEA’s element concentrations were used as reference for all subsequent calibration steps. The



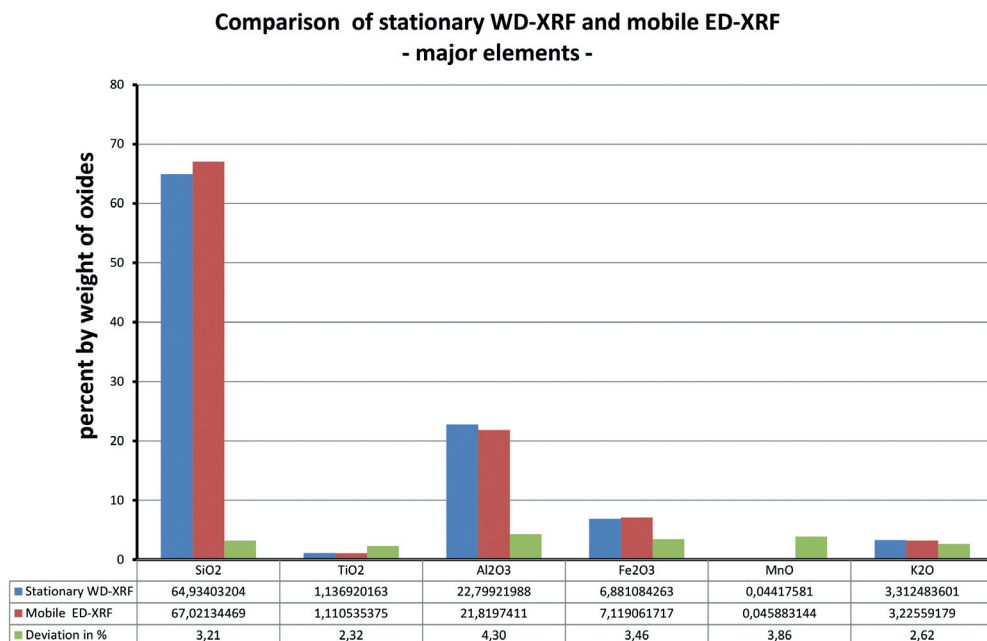


Fig. 20. Comparison of stationary WD-XRF and mobile ED-XRF – major elements.

result of the comparison of the stationary and mobile XRF measurements is shown in Figures 20 and 21.<sup>41</sup>

The median values for stationary WD-XRF for each element are given in blue, the values for mobile ED-XRF in red. The percentile deviation between the two methods, which lies between 1.17% (Ba) and 4.87% (Cr), is displayed in green.<sup>42</sup> Such low deviation values that are caused by random error and device parameters clearly show the comparability of the precision of the two methods. Portable ED-XRF is evidently in no way inferior to standard stationary WD-XRF facilities.

The calibration was fine-tuned by the company “AnalytiCON-Instruments” (Rosbach, Germany), using the same 20 samples already analysed by stationary WD-XRF in Poland. To ensure even greater reliability and accuracy, the calibration was additionally adjusted by myself. Several measuring cycles

<sup>41</sup> The elements Ni, Cu, Ca, P, and S are excluded from the comparison and from the analysis. Due to low concentrations in the samples they could not be calibrated properly (see also below).

<sup>42</sup> The concentration of the major elements is given in percent by weight of oxides, as is customary in large laboratories. The handheld device displays all measured values in ppm (parts per million), which can be easily converted to percent by weight of oxides (and *vice versa*) if necessary.

**Comparison of stationary WD-XRF and mobile ED-XRF  
- trace elements -**

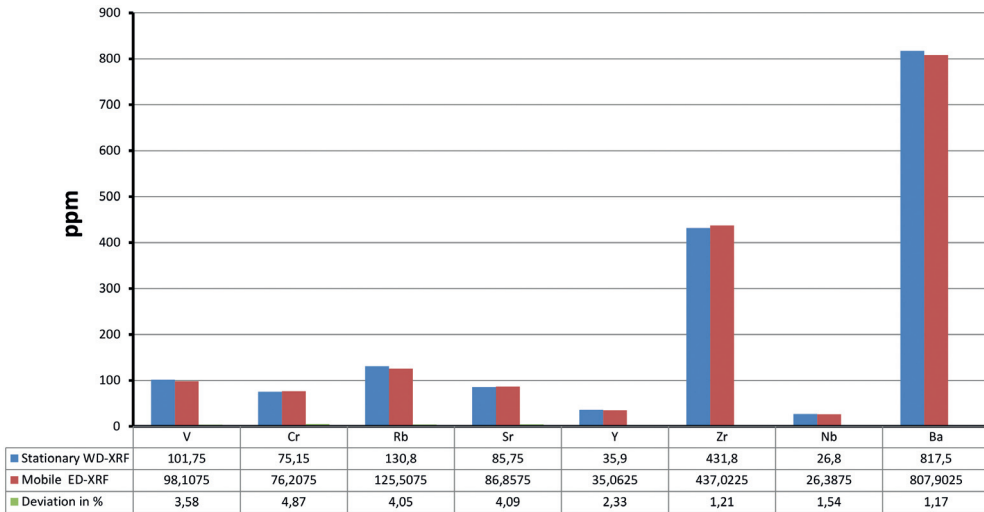


Fig. 21. Comparison of stationary WD-XRF and mobile ED-XRF – trace elements.

were executed, the measured values compared, and the calibration adjusted until the differences of the measurement values of the ED- and WD-XRF lay below the 2 sigma standard deviation.<sup>43</sup> Only two elements could not be calibrated properly: the Ni and Cu quantities often lie below the device's limit of detection, which makes a correct adjustment of the calibration curve impossible. These two elements were therefore excluded from the analysis. The same is true of Ca, P, and S: their content in the samples was so low that they were excluded from the analysis before final calibration. Thus six major elements (Si, Ti, Al, Fe, Mn, and K) and nine trace elements (V, Cr, Zn, Rb, Sr, Y, Zr, Nb, and Ba) remained in the analysis.

### 5.2.2 Measuring Procedure

Prior to the first analysis, a measurement protocol had to be established. This includes the questions of 1) the duration of the measurements and the combination of the different filters; 2) the necessary number of readings to achieve a high replicability, and 3) the place of the readings (surface or fresh breaking edge).

<sup>43</sup> See Appendix 1 for the final calibration curves for each element.

### 1) *Duration of Measurements*

The device is equipped with four filters (or ranges) which focus on different elements. The main filter focusses on the elements Cd, Ag, Nb, Zr, Y, Sr, Rb, Th, Bi, As, Se, Pb, Hg, Zn, Cu, Re, Ta, Hf, Ni, Co, Fe, Mn, Cr, V, and Ti; the low filter on Cr, V, Ti, Ca, and K; the high filter on Ce, La, Ba, Cd, and Ag; and the light filter on the elements Al, P, Si, Cl, S, and Mg.<sup>44</sup> These filters were adjusted as follows: main filter: 60 seconds; low filter: 90 seconds; high filter: 30 seconds; light filter: 120 seconds. The measurement times of the low and light filters were increased to allow a more accurate evaluation of the lighter elements. In contrast, the measurement time of the high filter was reduced because the elements analysed there were not included in the analysis, with the exception of Ba. This leads to a total time of 300 seconds per measurement. This combination of the filter times provides the best balance between the most exact measurements and the shortest reading time.

### 2) *Number of Readings*

Within this study, tests concerning the replicability of the measurements were also conducted. Test measurements with WD-XRF on Roman potsherds have shown that the replicability and accuracy – especially of the lighter elements (e.g. Al, Mg, or Si) – may be limited when analysing ceramic material via mobile XRF, due to the missing sample preparation (SCHNEIDER & DASZKIEWICZ 2010). In order to find the optimum balance between accuracy and number of readings, two samples (one terracotta, one potsherd) were measured 20 consecutive times at different spots. Afterwards, the mean and median values, standard deviation, and the coefficient of variation (in %) were calculated for each element. The latter serves as a measure of random error. Results are shown in Tables 2 and 3.

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<sup>44</sup> The appearance of an element in two filters is due to the fact that the device takes different energy levels for the same element into account. The energy of the secondary X-rays emitted by the atom depends on the orbital from which the electron drops down to fill the vacancy in the innermost orbital.

Element	Mean value	Median	Standard deviation	Coefficient of variation (%)
<b>Si</b>	283675.71	283798.92	2334.14	0.82
<b>Ti</b>	10325.51	10344.73	75.39	0.73
<b>Al</b>	90844.49	90339.56	1186.69	1.31
<b>Fe</b>	51552.35	51528.32	288.80	0.56
<b>Mn</b>	451.33	450.63	31.14	6.90
<b>K</b>	27533.80	27606.2	266.96	0.97
<b>V</b>	104.22	104.84	2.48	2.38
<b>Cr</b>	140.86	140.19	4.41	3.13
<b>Ni</b>	100.97	103.47	12.65	12.53
<b>Cu</b>	43.38	40.34	15.46	35.64
<b>Zn</b>	93.02	92.66	3.36	3.61
<b>Rb</b>	136.67	136.42	0.94	0.69
<b>Sr</b>	40.42	40.26	0.78	1.94
<b>Y</b>	17.79	17.75	0.51	2.88
<b>Zr</b>	344.44	344.07	3.08	0.89
<b>Nb</b>	35.05	34.90	0.73	2.08
<b>Ba</b>	664.82	669.44	31.58	4.75

Tab. 2. Repeated measurements of a terracotta fragment (sample UK7-677), statistical key figures; measurement values in ppm.

In 14 cases (Si, Ti, Al, Fe, K, V, Cr, Zn, Rb, Sr, Y, Zr, Nb, and Ba), the coefficient of variation is below 5%; in six of these cases it is below even 1%. For Mn, it lies a little higher at close to 7%. This may be due to relatively low Mn concentrations of approximately 450 ppm and occasional values below the limit of detection. Only for Cu and Ni are the coefficients of variation outside this range, which is due to the incorrect calibration of these two elements (see Chapter 5.2.1 and Appendix 1). As Ni and Cu are excluded from the analysis, this poor quality of the measurements can be neglected.

The results clearly show that the measurements are highly replicable. At the same time, they can be used to determine the minimum number of readings necessary. After four measurements at different spots, robust values are achieved, and further readings do not contribute significantly to a lower coefficient of variation. Thus, all samples were analysed four times in four different spots, taking care not to place the measuring window of 8 mm over an area with noticeable contamination or visible temper particles. This was controlled using the integrated camera. Based on the measurement duration of 300 seconds, this procedure resulted in a total time of 20 minutes spent on each sample.

<b>Element</b>	<b>Mean value</b>	<b>Median</b>	<b>Standard deviation</b>	<b>Coefficient of variation (%)</b>
<b>Si</b>	243608.72	243458.75	2335.21	0.96
<b>Ti</b>	10129.17	10121.57	68.45	0.68
<b>Al</b>	101320.70	101028.46	1038.88	1.03
<b>Fe</b>	69426.06	69377.51	277.80	0.40
<b>Mn</b>	450.68	448.90	33.12	7.35
<b>K</b>	29666.94	29803.57	290.61	0.98
<b>V</b>	145.15	144.65	5.19	3.58
<b>Cr</b>	231.30	234.38	9.42	4.07
<b>Ni</b>	113.68	117.11	13.58	11.95
<b>Cu</b>	57.02	46.65	18.62	32.65
<b>Zn</b>	115.49	115.64	4.91	4.25
<b>Rb</b>	160.86	160.82	1.40	0.87
<b>Sr</b>	31.93	31.89	0.42	1.31
<b>Y</b>	20.22	20.21	0.53	2.63
<b>Zr</b>	303.45	302.99	2.90	0.96
<b>Nb</b>	41.39	41.39	1.17	2.83
<b>Ba</b>	668.12	659.27	33.40	5.00

Tab. 3. Repeated measurements of a piece of domestic pottery (sample UK7-548), statistical key figures; measurement values in ppm.

### 3) *Place of Readings*

Especially important in the analysis of the terracotta figurines is the question if measurements can be conducted on the surface of the sample or if fresh breaking edges are required, which would result in a – at least partly – destruction of the sample. Thus, tests were conducted to determine whether different measurement spots on the fragments yielded different results. Eighteen samples (9 potsherds, 9 terracotta parts) were measured four times each on the surface and on a fresh breaking edge. The results of this comparison are shown in Figures 22 and 23.

The deviation of the measured element values at the two different places (surface and breaking edge) always remained below 5% and can thus be neglected. This result is likely due to the poor surface preservation of the ceramic material. If the slip had preserved, it would have affected the measurements, as the device yields only a small depth of information – between several  $\mu\text{m}$  and several mm, depending on the element (MEYER, HEGEWISCH & SCHNEIDER 2012: 388-389). In the case of Nok samples, however, the mobile XRF can be truly and reliably used as a non-destructive methodology.

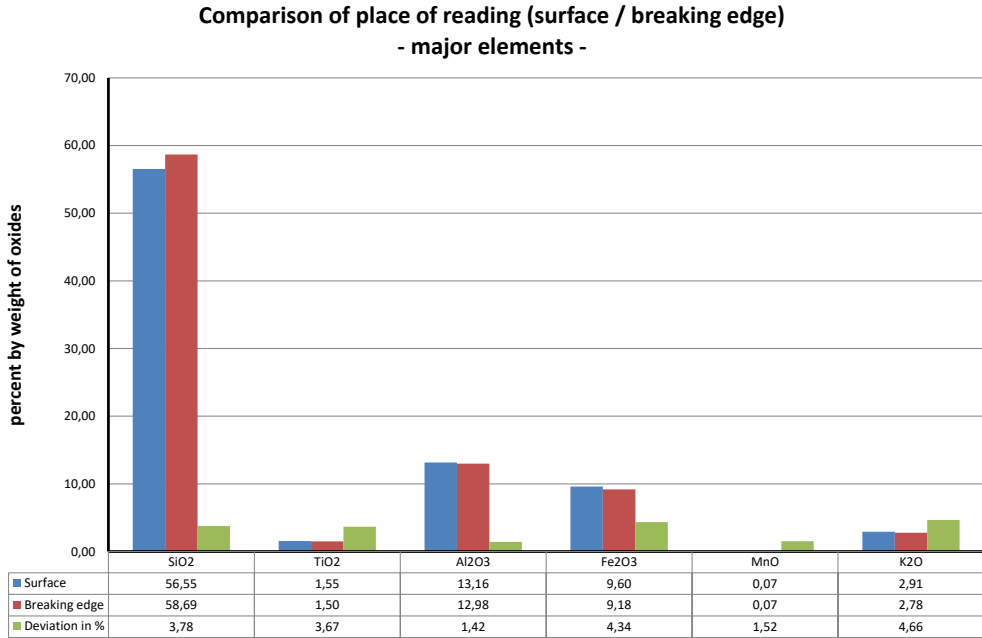


Fig. 22. Comparison of place of reading (surface/breaking edge) – major elements.

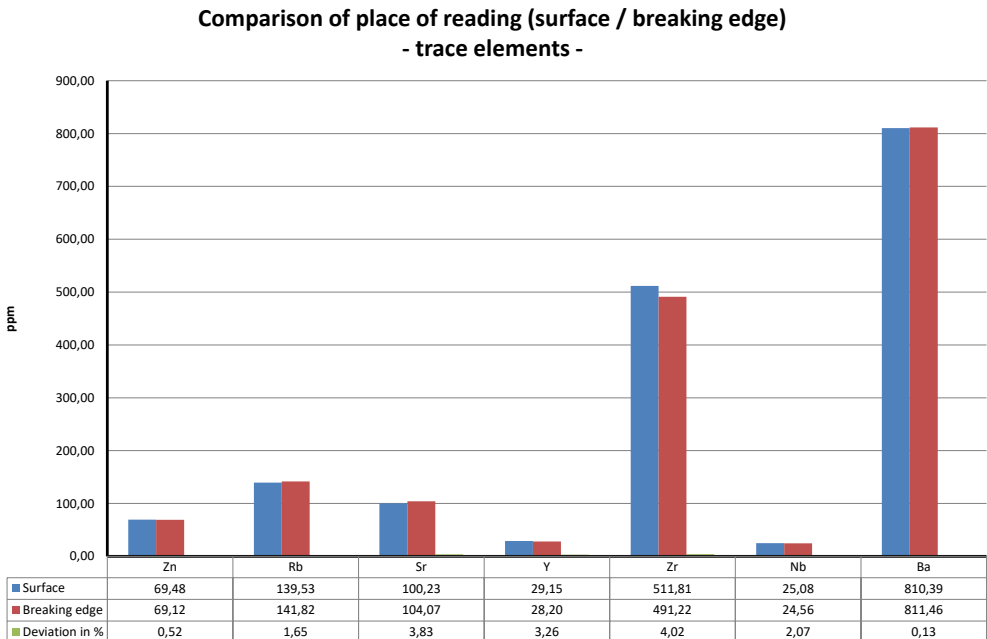


Fig. 23. Comparison of place of reading (surface/breaking edge) – trace elements.

After determining these basic requirements the analysis of the Nok material started. The measurements were conducted at Frankfurt's Goethe University at room temperatures of 20-25°C. Each day, a reference standard (GBW 7411) was analysed at the beginning and the end of each measurement period to control the correct functioning of the device.

### 5.3 Multivariate Statistics: Method and Data Preparation

3051 pottery and terracotta samples and 163 clay "reference" samples were measured to obtain information about the qualitative and quantitative concentration of 15 chemical elements. Each sample was measured four times to minimise systematic errors; with a given time of 300 seconds per measurement and the extra time to change the measurement spot and restart the instrument, each sample's complete measurement took *ca.* 30 minutes. This resulted in approximately 1600 hours or 40 weeks (8 hours/5 days) of total measurement time. The database created comprised accordingly 3214 data sets with 15 different element values; such a large amount of information cannot be evaluated manually. Thus, the statistics programme "R"<sup>45</sup> was used for analysis, as it is freeware which runs on nearly all platforms and supplies all required features. The database consists of multivariate data sets of chemical elemental compositions; such a database is particularly suited to principal component analysis (PCA), as its basic principle is the reduction of the dataset's dimensions without losing general information or the relationships between the variables. Hidden structures can thus be uncovered and displayed in a low-dimensional space (MÜLLER-SCHEESSEL 2008: 235). In the following, a brief description of this method is given, followed by the description of data preparation prior to statistical analysis.

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<sup>45</sup> The software can be downloaded from the website [www.r-project.org](http://www.r-project.org) (©The R Foundation for Statistical Computing). Version 3.1.1 (2014-7-10) was used for all calculations.

For the PCA, extension package *vegan* was used (OKSANEN ET AL. 2015; <http://cran.r-project.org/web/packages/vegan/vegan.pdf>; 16.06.2015). The 3D-plots were produced with extension package *rgl* (ADLER ET AL. 2015; <http://cran.r-project.org/web/packages/rgl/rgl.pdf>; 16.06.2015).

A methodological introduction into the R software was provided by Dr. Georg Roth, University of Cologne. He supplied the necessary commands to calculate the PCA, and graphically display the results. I am equally grateful for the discussion on the interpretation of the results.



### 5.3.1 Principal Component Analysis – Method

Principal component analysis (PCA) is one of the oldest ordination methods and was originally described by PEARSON (1901) and later – independently – proposed by HOTELLING (1933) (LEGENDRE & LEGENDRE 2012: 430). Mathematically, it is based on the same calculation parameters as the correspondence analysis (CA) method widely used in biology, sociology, ecology, and archaeology today. The main difference between these two methods is that CA uses nominally-scaled variables (*e.g.*, recorded types of pottery decorations – thus “constructed” or assigned numbers), while PCA uses metrically-scaled variables (“true” numbers such as chemical compositional data) (MÜLLER-SCHESSEL 2008: 235). PCA requires that metrically-scaled data set variables be normally distributed and symmetric. Especially with chemical compositional data, however, this is not always the case. The most common way of achieving this distribution with such non-normal, asymmetric data is to transform the data using natural logarithms; the distance preserved is the Euclidean distance and the relationships become linear (BORCARD, GILLET & LEGENDRE 2011: 117).

The PCA algorithm is based on a matrix, in which the degree of correlation (a measure of the similarity of two variables) for each pair of variables is displayed. This matrix is called covariance (or dispersion) matrix. This matrix may be distorted if the variables are not in the same physical unit or if the measured values are distributed across a large range (*e.g.*, several orders of magnitude, as is often the case with chemical composition data: major elements often have values of 200,000 ppm or more, while trace elements may occur in amounts of less than 100 ppm). In such cases, the variables will be weighted differently, and the matrix has to be standardised by subtracting the mean and dividing it by the standard deviation (also known as “zero mean, unit variance”). This transformation results in the disappearance of any dimensional units and creates a range of correlations between -1 and +1. The standard deviation for all variables is 1; they are thus considered unweighted in the analysis. This transformed matrix is known as the correlation matrix (LEGENDRE & LEGENDRE 2012: 445-446) and is the one used in this study.

Essentially, PCA isolates the primary trends within the data as continuous axes. It thus serves as an instrument for reducing the dimensions of the dataset, as it represents the data along a reduced number of orthogonal axes constructed in such a way that they represent the main trends of the data. A data set in the form of  $n \times p$  contains  $n$  objects (in this case the 3214 analysed samples) and  $p$  variables (the 15 measured chemical elements). The analysed objects can thus be represented as a cluster of  $n$  points in a  $p$ -dimensional space. This cluster of points is not generally spheroid but elongated or flattened in one or

more of the  $p$  directions. In order to facilitate the interpretation of this cloud of points, a new axis is computed that follows the furthest extent of the ellipsoid (*i.e.*, the direction in which it is most elongated), describing its multinormal distribution. It thus lies along the direction of the largest variance of the cluster. The second axis describes the second most important dimension of the cluster of points and lies orthogonal (linearly dependant, uncorrelated) to the first axis. This process continues until all axes – the principal components, or PCs – are computed. One can obtain a maximum of  $p$  principal components in a data set with  $p$  variables. In other words, the PCA carries out a rotation of the original system of axes defined by the variables, such that the successive new axes (PCs) are orthogonal to each other and correspond to the successive dimensions of maximum variance of the scatter of points (BORCARD, GILLET & LEGENDRE 2011: 116).

The proportion of the dispersion that is displayed on an axis is called an *eigenvalue*. The first PC therefore has the highest eigenvalue, the second the second highest, and so on. The eigenvalue is thus a measure of the “importance” (*i.e.*, degree of variance) of the axis and can be used to determine how many axes are needed to interpret the dataset; axes of higher order show largely redundant information. Thus, the PCA is a powerful tool to summarise – in a smaller number of dimensions than the dataset had originally – most of the variability of a large number of variables (BORCARD, GILLET & LEGENDRE 2011: 115-117; LEYER & WESCHE 2008: 111). In case of the analysis of the Nok material, two or at most three axes are needed to display the information contained in the combination of the 15 analysed chemical elements (Chapters 6 and 7).

As mentioned above, eigenvalues can be used as a heuristic for how many axes should be interpreted to record the range of the variance. There are several methods of choosing an appropriate number of axes to represent the variabilities of interest. One is arbitrary selection: picking that number of axes that are needed to represent, say, 75% of the variance. Another procedure relies on the “Kaiser-Guttman criterion”; here, the mean of all eigenvalues is computed, and only those axes whose eigenvalues are larger than that mean are interpreted.<sup>46</sup> A third method is the “broken stick model”, illustrated in Figure 24. In this method, a theoretical stick is randomly divided into the same number of pieces as there are PCA axes. These pieces are then put in order of decreasing length and compared to the eigenvalues; only the axes whose eigenvalues are larger than the length of the corresponding piece of stick are interpreted. A combination of these methods should be used to decide how many axes are needed for the interpretation (BORCARD, GILLET & LEGENDRE 2011: 121-122). Therefore, the decision of how many axes of the PCA

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<sup>46</sup> In a PCA based on a correlation matrix, the mean of eigenvalues is 1.

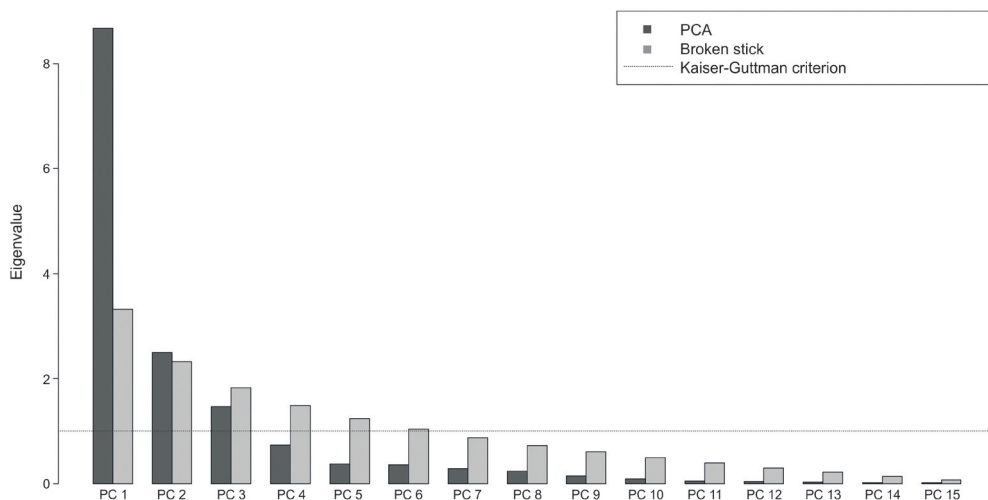


Fig. 24. Example of a diagram of eigenvalues with Kaiser-Guttman criterion and broken stick model.

are interpreted in this study relied on the Kaiser-Guttman criterion and the broken stick model.

Depending on the properties, which are the focus of the study, the results of a PCA can be plotted based on two different parameters: either the objects (shown as points in a distance biplot, Fig. 25) or the variables (shown as arrows/vectors in a correlation biplot, Fig. 26). There is no way of displaying and interpreting objects and variables together in a PCA biplot, as each of the two scalings has differing properties which must be kept in mind for proper interpretation. In the distance biplot, the distances between the objects (points) correspond to their Euclidean distances in multidimensional space, while the angles among the vectors are meaningless. The distance biplot is useful primarily for interpreting the distribution of objects in the multivariate space. In contrast, in the correlation biplot of the variables, the angles between the arrows reflect their correlation, while the distances among the objects are meaningless. Thus, vectors pointing in the same direction correlate positively, vectors with opposite directions correlate negatively, and perpendicular vectors show no correlation. Long vectors have higher correlations than shorter ones (LEGENDRE & LEGENDRE 2012: 447-448; MÜLLER-SCHIESSL 2008: 236).

In this study, both plots are used: distance biplots for the interpretation of the differences or similarities of the chemical composition of the samples, and correlation biplots to deduce which chemical elements are responsible for the pattern seen in the distance biplots.

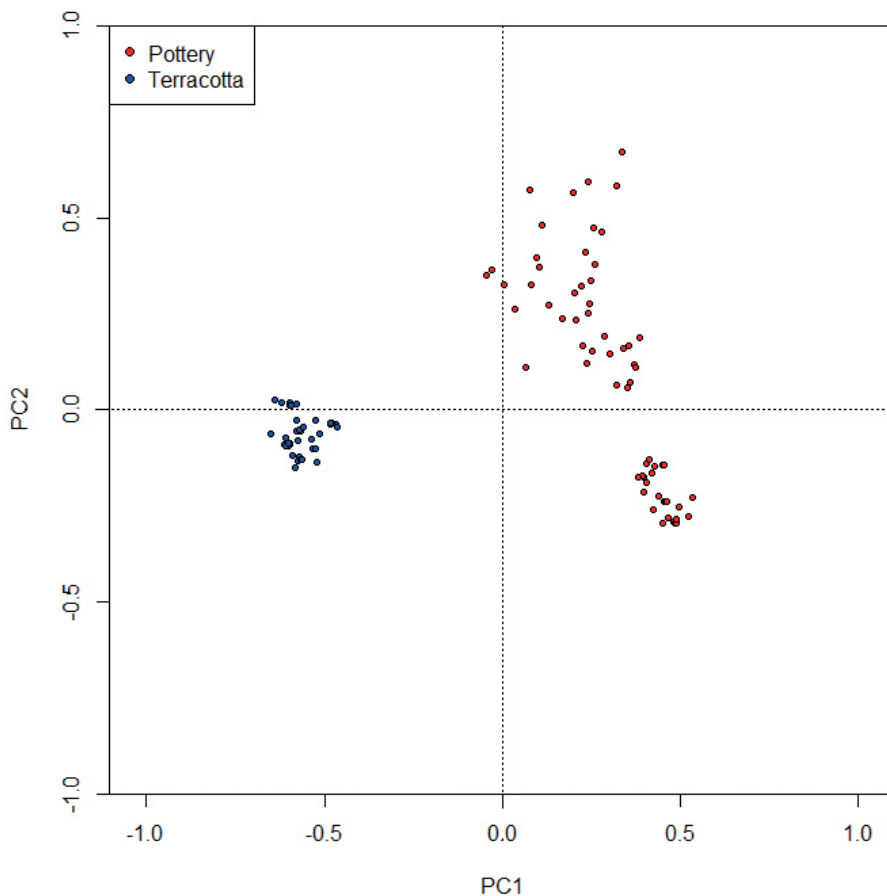


Fig. 25. Example of a PCA distance biplot (pottery and terracotta).

### 5.3.2 Preparation of the Data prior to Analysis

Sample measurements were recorded in an Excel sheet. The variables comprised of the quantitative concentrations of the 15 selected chemical elements; where available, any other information, such as excavation trench, individual find number of the sample, dating, and GPS coordinates of the site were also recorded (Fig. 27). Each chemical element (over all samples, *i.e.* one column in the Excel sheet) was tested on symmetry and normal distribution. The results of these tests – including statistical key figures such as standard

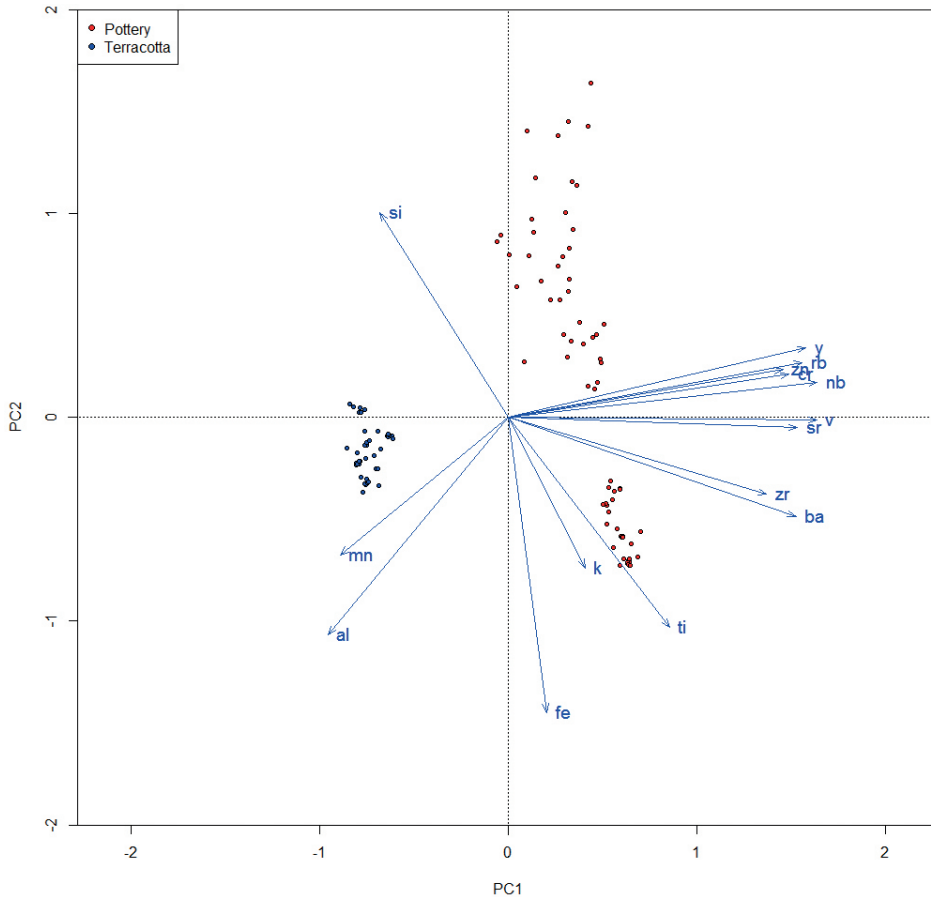


Fig. 26. Example of a PCA correlation biplot (corresponding to the distance biplot in Fig. 25).

deviation, mean value, median value, skewness, and kurtosis<sup>47</sup> – are given in detail in Appendix 2. Extreme outliers likely caused by measurement errors were then eliminated from the database as they would have had too great of an influence on the calculations. Because all elements proved non-symmetrical, the natural logarithm of the data set was taken. Initially, the three material groups (terracotta, pottery, and recent clay samples) were considered

<sup>47</sup> Skewness and kurtosis are measures of the asymmetry of datasets. The skewness value can be positive or negative. A positive skewness means that the median is higher than the arithmetic mean, whereas a negative skewness describes a median smaller than the arithmetic mean. The kurtosis is a descriptor of the shape of a probability distribution (SCHÖNWIESE 2000: 47). For examples of kurtosis and a positive or negative skewness see Appendix 2.

separately; later on, depending on the intended analysis, they were combined into one database to facilitate calculations.

## 5.4 Sample Selection

Terracotta and pottery samples from 43 sites of the Nok Culture were analysed (Fig. 28). Sites with large-scale excavations and single find recording were preferred over test pits where possible. To cover as large an area as possible, samples collected on the surface and samples taken from the vicinity of illegal diggings were also selected. Attempts were made to choose a statistically productive number of potsherds and terracotta fragments alike per site. This, however, was not always possible: from the Pangwari site, for example, only the terracotta finds were exported to Germany for the Nok exhibition in 2013/2014,<sup>48</sup> so that potsherds from this site could not be analysed. Surface collections, especially from the early project years (2005-2008), often contained only terracotta fragments but no pottery. Some excavations and test pits, on the other hand, revealed only potsherds and/or non-diagnostic ceramic fragments which could not be definitively classed as terracotta or pottery. Altogether, 3051 samples, of which 1639 were potsherds and 1412 were terracotta parts, were analysed and formed the database for the investigation of the geochemical difference between the two find groups. This is the first comprehensive database of material with undoubted provenance and therefore definitely genuine terracotta figurines.

In order to draw conclusions concerning the raw material sources for the figurines and therefore their production sites, reference groups are needed. These reference groups are ideally established by analysing the chemical composition of misfired pottery found in refuse pits near former pottery workshops. This method very clearly links workshops and raw materials. Finds with unknown provenance can then be assigned to the production site by comparing their chemical compositions with those of the misfired pieces (POLLARD & HERON 2008: 100; WILSON & POLLARD 2001: 507ff). Unfortunately, geochemical information of this type for Nigeria – or indeed West Africa – is lacking. Furthermore, no pottery workshop with misfired pots has been excavated so far; neither for the Nok Culture nor for any neighbouring region. Archaeological material in West Africa has generally been analysed geochemically only in exceptional cases (*e.g.*, IGE ET AL. 2009; TANDOH ET AL. 2009; A. USMAN ET AL. 2005); a database containing any reference material

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<sup>48</sup> *Nok. Ein Ursprung afrikanischer Skulptur*. Liebieghaus Skulpturensammlung, Frankfurt, October 30, 2013 – March 23, 2014.

	A	B	C	D	E	F	G	H	I	J	K	L	M
1	Si	Ti	Al	Fe	Mn	K	Site	Trench	Find number	Material	Date ( 1 Sigma)	Deviation	BC/AD
1644	219218,66	10378,14	103993,45	72127,32	720,01	32432,63	AK		1	4 Terracotta		450	49 BC
1645	226026	11456,95	123767,53	74533,4	863,8	33854,44	AK		1	5 Terracotta		450	49 BC
1646	230423,065	11104,71	139184,71	72619,945	1132,16	29538,955	AK		1	6 Terracotta		450	49 BC
1647	211219,22	7094,265	94032	64105,82	526,54	24396,63	AK		1	7 Terracotta		450	49 BC
1648	262853,13	9264,655	134697,4	64099,96	799,785	31405,92	AK		1	8 Terracotta		450	49 BC
1649	242180,02	11145,07	148812,36	76417,16	1137,97	30406,68	AK		1	9 Terracotta		450	49 BC
1650	227629,97	9835,25	94729,1	66085,865	740,335	30113,475	AK		1	10 Terracotta		450	49 BC
1651	229910,99	8614,54	106241,905	64294,05	927,97	28485,785	AK		1	11 Terracotta		450	49 BC
1652	230740,78	11568,025	122928,85	76958,47	1160,995	35088,345	AK		1	12 Terracotta		450	49 BC
1653	243866,65	10749,055	158784,345	78174,03	1041,825	25388,81	AK		1	13 Terracotta		450	49 BC
1654	214982,61	10287,025	112192,22	74764,845	753,22	26829,84	AK		1	14 Terracotta		450	49 BC
1655	243565,82	8929,59	146811,46	66880,915	702,23	22890,105	AK		1	15 Terracotta		450	49 BC
1656	222391,665	11350,66	126465,93	76772,08	937,705	33326,915	AK		1	16 Terracotta		450	49 BC
1657	227356,955	11643,605	161246,845	73886,58	1019,535	26497,17	AK		1	17 Terracotta		450	49 BC
1658	221528,07	8854,76	137879,15	65995,12	757,87	21863,685	AK		1	18 Terracotta		450	49 BC
1659	246335,61	10219,68	145264,71	70670,18	1235,415	31453,495	AK		1	19 Terracotta		450	49 BC

Fig. 27. Detail of the Excel sheet containing the measurement values of the major elements and further information used in the statistical analysis.

does not exist. Therefore, the search for formerly used clay sources or pottery workshops is difficult. To fill this gap, another approach is needed.

Even today, domestic pottery is still made in the villages located in the Nok Culture area, although it is a dying craft maintained only by old women who learned it from their mothers and grandmothers. The easiest way to locate clay sources used by traditional potters is thus interviewing the local people. To create a database of today's clay sources, intensive fieldwork was carried out in 2009 and 2011. An area measuring a total of  $150 \times 70$  km was investigated and 163 samples from clay sources still in use today and from 139 villages were collected systematically.<sup>49</sup> In six cases recent pottery samples manufactured with the corresponding clays were available and collected as well. For each site, the raw clay samples – and if available any information on raw material acquisition, preparation, and temper – were recorded (Chapter 5.5). The clay samples were exported to Germany for further analysis.

Of course, it seems extremely unlikely to be able to directly relate a recent clay source to clay sources used during Nok times. But as the sampled region overlaps with the key study area of the Frankfurt project, it may at least be possible to draw conclusions concerning the type of formerly used clay (primary or secondary clay); it may also be possible to delimit the area in which the terracotta workshops might have been.

<sup>49</sup> The difference between the number of villages (139) and the number of clay sources (163) derives from the fact that in some villages more than one clay source or different clay layers in one source were used. In such cases, each clay sample was counted separately but as belonging to one site.



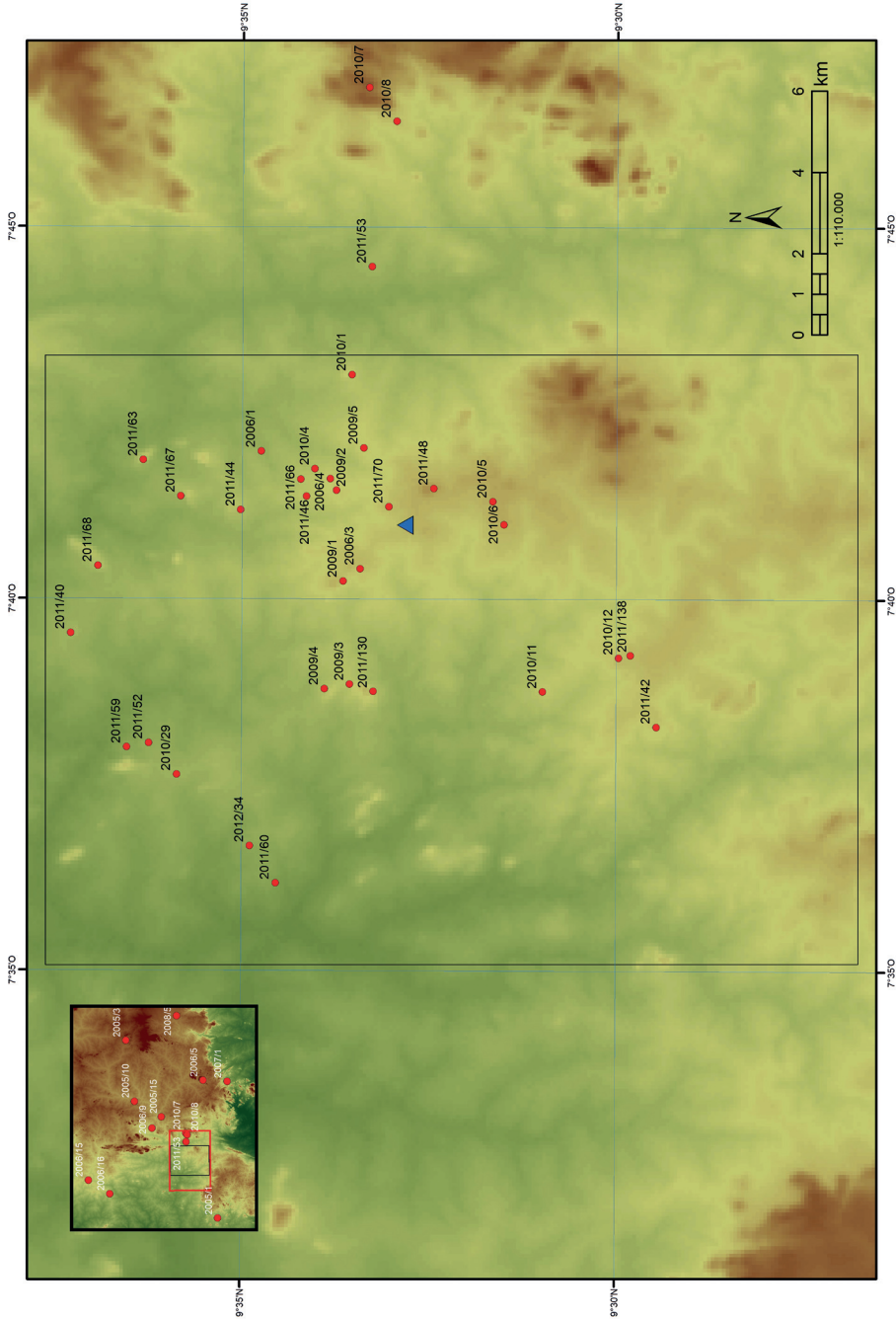


Fig. 28. Map showing all sites included in the XRF analysis (n=43) (map by Eyub F. Eyub). Most of the sites, as well as the Nok research station (blue triangle), are located in the key study area (black rectangle). Sites further away are shown in the small locator map (top left).

## 5.5 Side Note: Clay Procurement and Processing in Central Nigeria Today

The production of pottery is a dying craft in present-day Nigeria. The old tradition of producing pots is being increasingly supplanted by imported vessels made of metal or plastic – mainly from Asian countries. Until some decades ago, each village had its own potter. Today, ceramic pots are still bought on the markets, but they derive almost exclusively from a few larger villages that have specialised in a large-scale production solely for sale. Four such centres exist in the research area: Taffa (samples 30-32) in the west; Kachechere (sample 55) and Tachira (sample 89) in the east; and Kachia (sample 82) in the north.<sup>50</sup> In the villages between these centres, pots are produced by elderly women (indeed, pottery is a women-only craft in this region), often only by request and on a very small scale – if at all: the region in the middle and east of the researched area, between Nok and Kafanchan, remains void of samples since not a single potter or clay source could be found. Where there is still a potter, frequently there is no successor who will take over her craft. Even more often, the old potter had already died, but the people in the village at least remembered from where she had taken the clay. They could not, however, provide any information on the exact processing procedure. Only at 15 of the 139 villages visited (including the 4 centres of intensive production) was there still a potter present who was able to answer all questions concerning the procurement and further processing of the clay up to the manufacture of the pots and the subsequent firing. The methods applied more or less resemble each other, with small differences in the details. Thus, a summarising overview of the techniques will be given here.

The clay sources were generally no further away from the villages than 20 to 30 minutes by foot, most of the time markedly closer. This corresponds to ethnographical investigations by Arnold from all over the world, according to which the average distance that is covered to reach the next clay source is 6 km (ARNOLD 1989: 50). Clay sources that were passed on the way but not exploited were refused because they were “too weak” (*i.e.*, they contained too little clay and too many non-plastic inclusions that had to be removed in a time-consuming process) or simply “not suitable”. To the inexperienced eye, these clays often did not differ at all from the ones that were considered appropriate.

The whole region is streaked with small rivers and old, dried-out river beds, making clays readily available everywhere. The typical clod-formation

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<sup>50</sup> For the geographical location of the clay sources see Fig. 69 (Chapter 8).

of clayey soils could be observed for example on fresh ploughed fields (Fig. 29). But these clays were never used for the production of pottery, according to the information of the potters – at most, they were used to make sun-dried mud bricks for the construction of houses (Fig. 30).

The clays that were considered suitable for pottery production stem from two different contexts: most are derived from actual or dried-out river beds (secondary clays) (Fig. 31); others were primary clays that derived from *in situ*



Fig. 29. Clod-formation of clayey soil on a fresh ploughed field in central Nigeria. Photo taken in 2011.



Fig. 30. Mud bricks drying in the sun, in the village of Chinka. Photo taken in 2009.





Fig. 31. Secondary clay source in a dried out riverbed (clay sample 112). Length of scale: 2 m. Photo taken in 2011.

weathering and have been preserved because they were covered by laterite caps (MACLEOD ET AL. 1971: 11).

These primary clays are extremely fine-grained with almost no non-plastic inclusions (GIBSON & WOODS 1997: 237, 245) and more difficult to reach, as access to the clay layers first has to be dug through the extremely hard laterite crust. This makes use of primary clays relatively rare, and if the effort is made, the clay layers are generally exploited horizontally underground, forming tunnels and corridors until the covering soils collapse (Fig. 32). The exploitation of primary clays is thus a dangerous task. Secondary clays, in contrast, are widely distributed and formed by fluvial processes. Even if they dry up in the dry season, the numerous rivers and lakes carry water as soon as the rainy season starts, resulting in an immense increase in the water level in a relatively short time. This leads to flooding of parts of the landscape and partly to the relocation of the initial riverbed. Clay is then deposited especially in flooded depressions and cut-off meanders, but also on the slopes of the riverbanks (GUMNIOR 2006: 67-68, 177). Thus, secondary clays are easily available and can be dug up without too much effort. However, they contain a higher number of non-plastic inclusions and often a great deal of organic material. They are generally not as fine-grained as primary clays (GIBSON & WOODS 1997: 237, 245).



Fig. 32. Two potters from Taffa (clay samples 30-32) digging for primary clay under a laterite crust. Photo taken in 2009.



Only the amount of clay needed for the intended number of pots is taken from the clay source and brought back to the village. The potters do not keep larger amounts of clay in stock, but prefer to collect it fresh. The reason given for this was that 'this is simply the way it is done and has always been done before'. Back in the village, the clay is dried in the sun, sometimes for several days (Fig. 33). The subsequent processing of the clay differs according to its origin. Very fine-grained and pure primary clays are generally mixed with other, coarser secondary clays or, more often, with sand from the riverbeds. Sometimes differences in the proportions of the mixture are made, depending on the size of the future pot. This corresponds to the different amount of temper contained in Nok pottery and figurines, the latter tempered more strongly to match the material requirements of the large, complicated statues (Chapter 4). In no case are organic tempering materials used – another parallel to the Nok samples. In contrast, secondary clays are only rarely tempered with sand. Instead, they are either vigorously pounded in a mortar or ground on grinding stones to crush the smaller stones and other impurities already contained in the clay. These particles, once crushed, remain in the clay as temper. Both clays are then soaked in water (a procedure that might take a whole day until the dried clay is completely wet) and are pounded until they become soft and smooth (Fig. 34). Some potters now put the finished clay in a pot with a little bit more water and leave it to draw through in a cool place until the next day.

The processing of the clay is now completed. After that, pots are shaped, dried, and fired in an open pit.<sup>51</sup> The resulting pots are all very fine-grained and hard-fired, and mostly reddish to brown (Fig. 35) in colour due to an oxidising firing atmosphere. The rare black or grey pots (Fig. 36) are sometimes produced by a second firing, in which the ceramics are purposely covered with grasses to reach a reductive firing atmosphere. A potter from Fadan Attakar (clay sample 88) especially referred to such practice.

As this short account of the processes of clay procurement and processing in modern Nigeria shows, two types of clay are used and treated differently according to their properties. Clays from secondary contexts are preferred, as they are easier to acquire. The more extensive preparation in mortars or on grinding stones is not outweighed by the hard and dangerous work the potter has to undertake when digging for primary clays under laterite crusts. All in all, the processing methods of both types differ in some ways, as especially the primary clays are additionally tempered with sand.

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<sup>51</sup> See FRANKE 2014: 170 for more information on these last steps of pottery production, given by a potter from Taka Lafiya (clay sample 105) in 2011.



Fig. 33. Raw clay lying in the sun to dry, in the village of Kwagiri (clay sample 59). Photo taken in 2009.



Fig. 34. Clay in a mortar, ready for pounding, in the village of Chinka (clay sample 27). Photo taken in 2009.





Fig. 35. Typical reddish pots, manufactured by the potter from Ankoro (clay samples 108-113). Photo taken in 2011.



Fig. 36. Pots with black colour made by the potter from Fadan Attakar (clay sample 88). Photo taken in 2011.



**C**

**Results of Scientific Materials Analysis**



## 6 Results of Geochemical Analysis (Terracotta and Pottery) for Selected Sites

In Chapter 4, the hypothesis has been established that centralised production of the terracotta figures made use of special clay sources, whereas domestic pottery was manufactured locally. This hypothesis is now tested by analysing the chemical composition of the clay, determined by XRF. In a first step, the clay compositions of pottery and terracotta will be compared on a site level.<sup>52</sup> Nok sites often show few distinctive features, if any. In addition, they lack a clear vertical or horizontal stratigraphy which makes it difficult to structure them spatially. Detailed spatial analyses of the find inventory of some sites and a critical assessment of the absolute dates have shown that many Nok sites were occupied (or at least visited) at several different points in time (FRANKE 2015: 205-218). Aside from the question as to the material difference between the pottery and the figurines, a detailed analysis of the chemical fingerprint can thus offer valuable results for the internal structure of Nok sites, if these multiple occupation episodes can also be traced in the chemical composition of the pottery, as it was locally produced from varying clay sources.

Three sites have been chosen as examples to demonstrate the potential of a site-level analysis: Ido, Ifana, and Ungwar Kura. At all three sites, the pottery and terracotta clearly show a distinct difference in the chemical composition – they were thus manufactured using different clays. Besides the informational content that can be drawn from this comparison, the combination of the geochemical analysis with the chronological evidence and the three-dimensional distribution of finds is striking. The chronological subdivision of the Nok Culture based on the analysis of pottery decoration and form in combination with absolute dating (FRANKE 2015) can be supported by a chronology of the use of different raw clay sources.

### 6.1 Ido (ID)

Ido is a site in the centre of the key research area, about 4 km north-west of the research station (Fig. 2). It was chosen because the pottery excavated there appears to belong to several utilisation phases – from the time of the Nok Culture to the Post-Nok period. Five radiocarbon dates taken from annual

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<sup>52</sup> The comparison of the two material groups (*i.e.*, terracotta sculpture vs. domestic pottery) across the entire data set – no longer on a site level – is presented in Chapter 7. The results of this can then be used to trace the differences between the material groups and draw conclusions concerning the provenance of the clay used for the terracotta figurines.

plants and charcoal range from *ca.* 1400-1200 BCE to *ca.* 700-400 BCE, with three dates clustering between *ca.* 1000 and 800 BCE. A thermoluminescence date on a potsherd with carved roulette decoration, which does not occur in Nok pottery, resulted in an age of 1238 to 1388 CE. Thus, Ido shows three occupation phases during the Nok Culture (Early, early Middle, and later Middle Nok) and several uses in Post-Nok times. These results are supported by a detailed pottery analysis (FRANKE 2015: 211-215).

One trench (10 × 13 m), excavated in 2009, was laid out between looting holes on the fringe of a hilltop. The find spectrum comprises pottery, terracotta, charcoal, stone artefacts, burnt clay, tuyère fragments, metal objects, slag, and bone fragments. The last four were only found close to the surface in the southern half of the excavation unit and probably belong to the Post-Nok occupation phase. Spatial analysis of the find distribution (FRANKE 2015, Fig. 95-97) shows that finds from the Nok Culture are found mainly in the northern half of the excavation area, which contains several pit features consisting of stone settings. It is in these features that the Nok pottery was found; in contrast to the Post-Nok pottery, which is distributed in the surface layers of the southern half of the excavation area. Terracotta finds comprise only few truly diagnostic fragments (*i.e.*, identifiable figurine parts), concentrated in the features. However, smaller terracotta pieces are distributed over the whole area in the uppermost layers, probably as spoil from illegal diggings. One feature in the northern section is especially remarkable: a setting of grinding stones with two complete, very finely worked pots. Stone beads that were arranged as if on a string were found next to the pots. This setting may be interpreted as a burial, with the bones not preserved due to the acidic soil conditions (NAGEL 2014: 154).

For the geochemical analysis, 44 terracotta and 75 pottery fragments were selected. The number of investigated figurine parts is smaller than the one of the pottery, because terracotta fragments were less strongly represented in the excavated material. Nevertheless, it is sufficiently high for a representative statistical analysis. The potsherd samples were chosen from the complete excavated inventory, considering Nok and Post-Nok sherds alike. The two complete pots from the possible burial were also taken into account.

The diagram of the PCA eigenvalues of the pottery and terracotta of Ido suggests the interpretation of the first three axes (Fig. 37). The Kaiser-Guttman criterion (only the principle components with eigenvalues that are larger than the mean should be considered; see Chapter 5.3)<sup>53</sup> clearly supports this solution. The “broken stick” model (only the axes whose eigenvalues are larger than the

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<sup>53</sup> As the PCA was computed with the correlation matrix, the mean eigenvalue is 1.

length of the corresponding “piece of stick” should be interpreted; see Chapter 5.3) even suggests the interpretation of only the first two axes. However, the latter criterion is based on random subdivision and should therefore be used only as another hint in the case of indistinct findings.

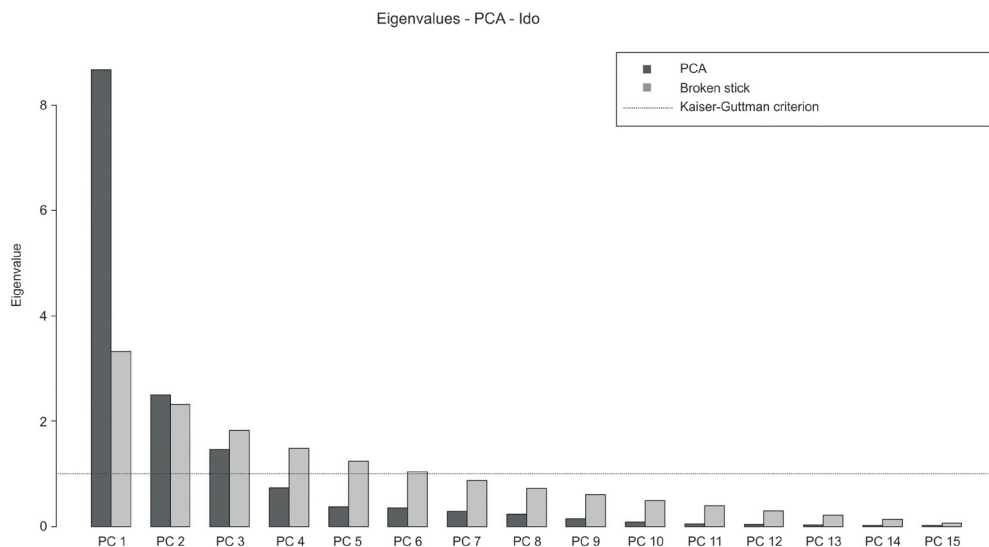


Fig. 37. Diagram of the eigenvalues of the PCA of the pottery and terracotta of Ido with broken stick and Kaiser-Guttman criterion.

A look at the cumulative eigenvalues (Tab. 4) further supports the decision to interpret the first three axes, as they represent 84% of the variance (information) of the data set. When comparing the increase of displayed variance from axis 2 to 3 (10%) with the one from axis three to four (5%), disregarding the third axis would lead to the loss of a significant portion of information. All following axes explain only relatively small portions of the variance and therefore just represent noise in the dataset.

PC1: 57.80%	PC2: 74.49%	PC3: 84.27%	PC4: 89.16%	PC5: 91.63%
PC6: 94.01%	PC7: 95.93%	PC8: 97.49%	PC9: 98.49%	PC10: 99.08%
PC11: 99.37%	PC12: 99.62%	PC13: 99.80%	PC14: 99.91%	PC15: 100%

Tab. 4. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of Ido.



Subsequently, distance biplots of each axis combination (1/2, 1/3, 2/3) as well as a 3D-plot of all three axes together (Fig. 38-43) were generated, with the goal of interpreting the differences in the chemical composition of the samples.<sup>54</sup> Axes 1 and 2 (Fig. 38) represent the most relevant information from the data: the chemical difference between the figurines and the domestic pottery.

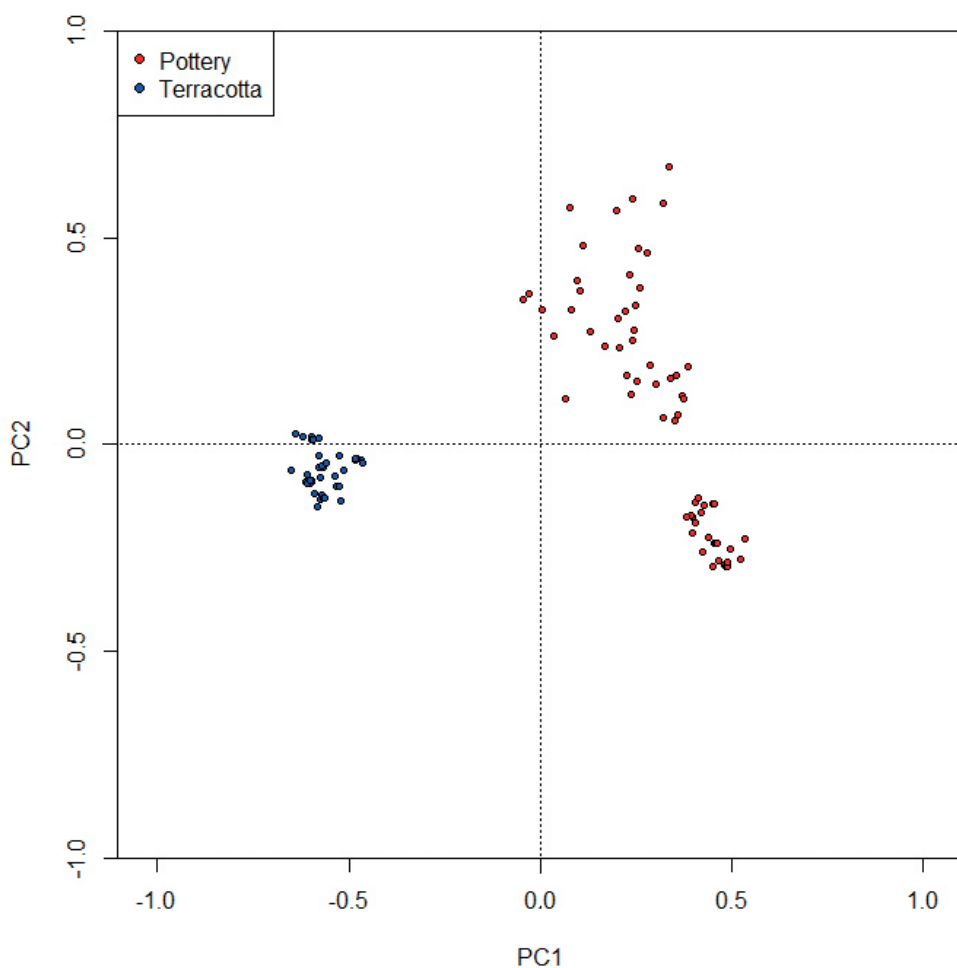


Fig. 38. Distance biplot of the PCA of the pottery and terracotta of Ido (axes 1 and 2). Terracotta figures are given in blue, potsherds in red.

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<sup>54</sup> In the 3D-plot, it was not possible to distinguish the two material groups with different colours. However, the terracotta samples form a clearly separated and dense cluster of points, so that both ceramic materials are nevertheless clearly distinguishable.

While the terracotta samples (shown in blue) form a clearly separated, dense cluster of points, the potsherds (shown in red) vary much more, forming two distinct groups. The chemical composition of the clay used for the figurines is thus very homogeneous and differs clearly from clay used for the pottery. The lack of any internal structure in the terracotta samples suggests the use of one clay source for all tested figurines of this site. The small variance in the terracotta clay can be attributed to the natural heterogeneity in a single clay deposit that is never fully uniform in its composition. The variance in the pottery samples, however, is visibly greater and cannot be explained by the natural inhomogeneities of a raw clay deposit. Instead it is likely that this dispersal in the biplot stems from the different clay sources used during the different occupations at the site (all distinct from the clay source used for the figurines). This is particularly evident when looking at the smaller group of potsherds clustered in the lower part of the plot. Based on the pottery decoration analysis, these samples belong to the Post-Nok phase of Ido's occupation history, since they are decorated with different carved roulettes, a technique not used in the key study area until after the Nok Culture (FRANKE 2015). The biplot demonstrates that the occupants of the site after the time of the Nok Culture exploited completely different clay sources. Interestingly, in Post-Nok times, the clay sources used for pottery are more consistent in their chemical composition than the clay sources used for pottery during the Nok Culture. This can perhaps be attributed to the use of locally limited clay sources. Thus, the change in decoration techniques from incised patterns to rouletting matches the change in the use of raw material deposits. To verify this finding, two other sites (Doguwa and Kurmin Uwa 2) with Nok and Post-Nok inventories were analysed in the same way; the results were confirmed. There too, the Post-Nok sherds clearly differ from Nok sherds in their chemical composition. In the future, attempts will be made also to differentiate the different Nok phases with the help of the chemical fingerprints of the domestic pottery. Initial results of this line of inquiry are promising.<sup>55</sup>

The consideration of axes 2 and 3 (Fig. 39) shifts the viewing angle such that the pottery and terracotta samples are lying on top of one another. However, given that the potsherds to the left portion of the diagram belong to Post-Nok times, the two groups can still be distinguished in the biplot. Thus, the figurines still show a grouping distinct from the more or less contemporary potsherds, albeit not as clearly as in the plot of the first two axes. The third axis

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<sup>55</sup> A detailed assessment of the results is being prepared for publication. As this finding is a by-product of the analysis, and as the pottery serves merely as comparison material for the terracotta, the finding will not be discussed here any further.

obviously reveals some structure to the terracotta samples, as the cluster of points now shows more variation, but still no separate grouping of only some figurine samples is detectable. The factors that are responsible for this finding will be discussed with the correlation biplots further below. The forming of the small group of pottery samples on the bottom right cannot currently be explained: the sherds cannot be assigned to a special decoration type, nor are chronological or spatial patterns evident. The two finely worked pots from the presumed burial feature do not stand out in any of the statistical analyses.

The comparison of the PCA of axes 1 and 3 does not reveal anything substantially different than the comparison of axes 1 and 2 (Fig. 40). The

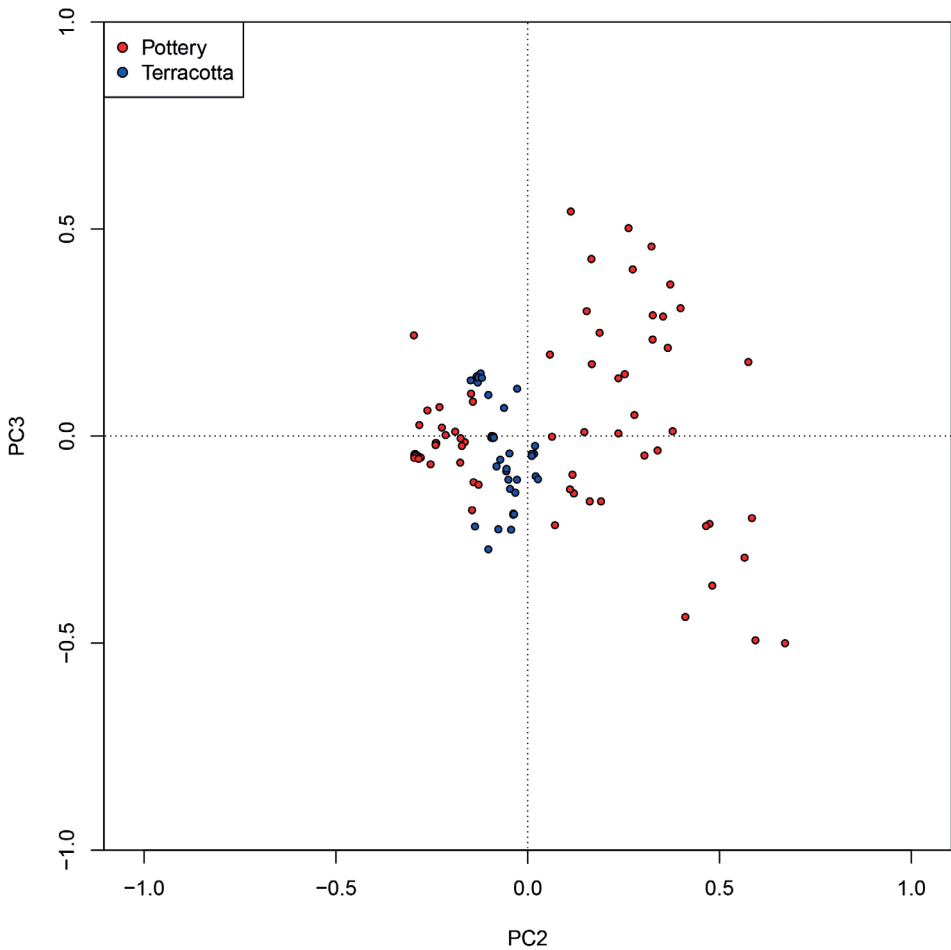


Fig. 39. Distance biplot of the PCA of the pottery and terracotta of Ido (axes 2 and 3).

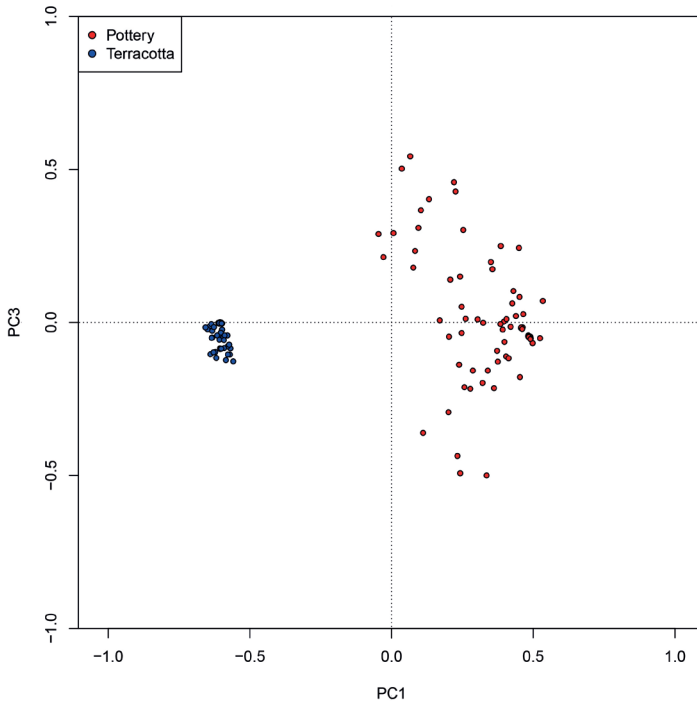


Fig. 40. Distance biplot of the PCA of the pottery and terracotta of Ido (axes 1 and 3).

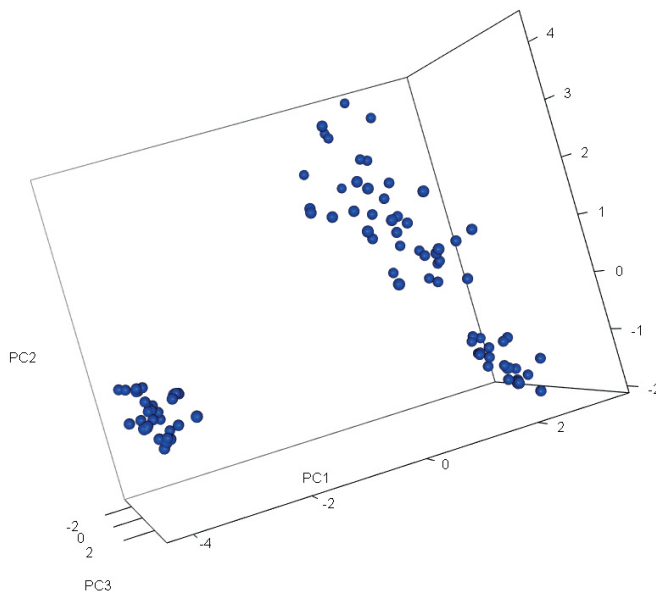


Fig. 41. 3D-plot of the first three axes of the PCA of Ido. Left: terracotta; bottom right: Post-Nok pottery; top right: Nok pottery.

terracotta samples still form a homogeneous scatter of points with no visible internal structure; due to the shift in viewing angle, the differences in the pottery inventory are no longer visible. The 3D-plot of all three axes together summarises the findings from the two-dimensional biplots (Fig. 41).

The question of which variables (here: which chemical elements) are responsible for the formation of the patterns described above can be answered using the correlation biplots. As only the two distance biplots of axes 1/2 and 2/3 revealed any interesting structures in the dataset, only the two corresponding

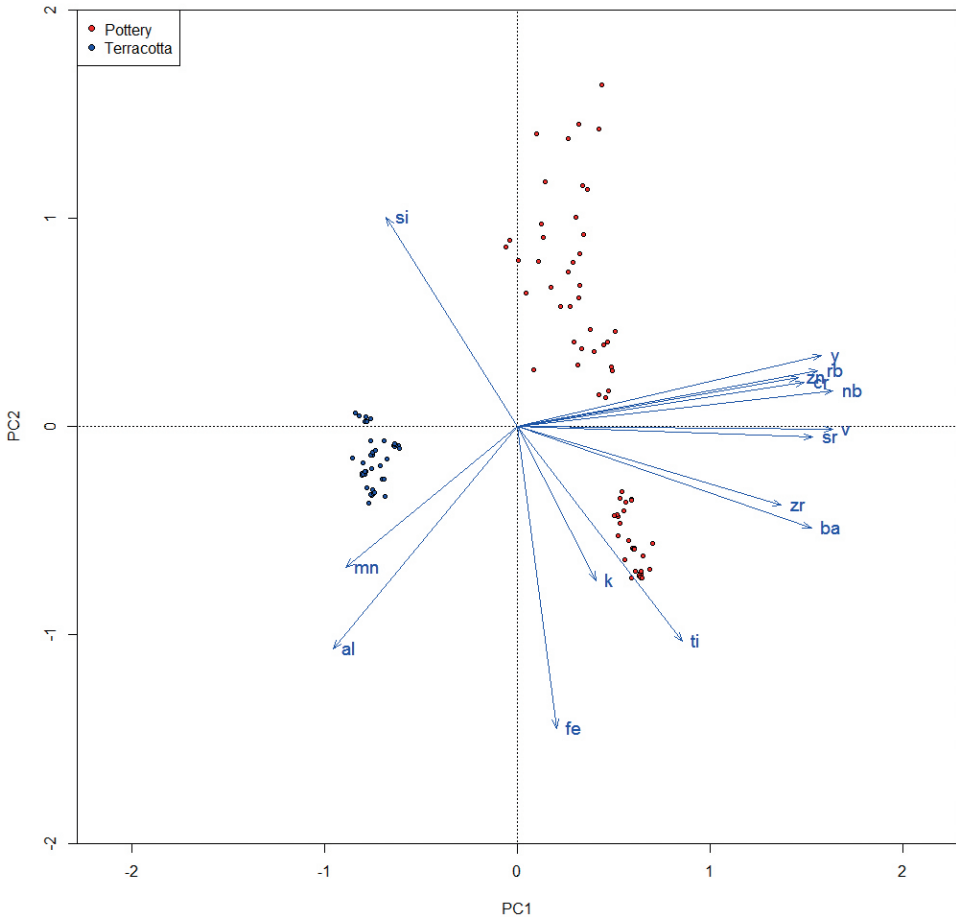


Fig. 42. Correlation biplot of the PCA of Ido, axes 1 and 2.

correlation biplots will be discussed here. In this form of representation, only the arrows (vectors) are interpretable while the points are only inserted for better orientation. The angles between the arrows reflect the correlation of the variables.

As can be seen in the correlation plot of axes 1 and 2 (Fig. 42) the variables (chemical elements) are arranged into four groups. The biggest group, consisting of all trace elements (Ba, Cr, Nb, Rb, Sr, V, Y, Zn, and Zr), shows similar behaviour and is negatively correlated with the elements Al and Mn – meaning that high amounts of trace elements go together with low amounts

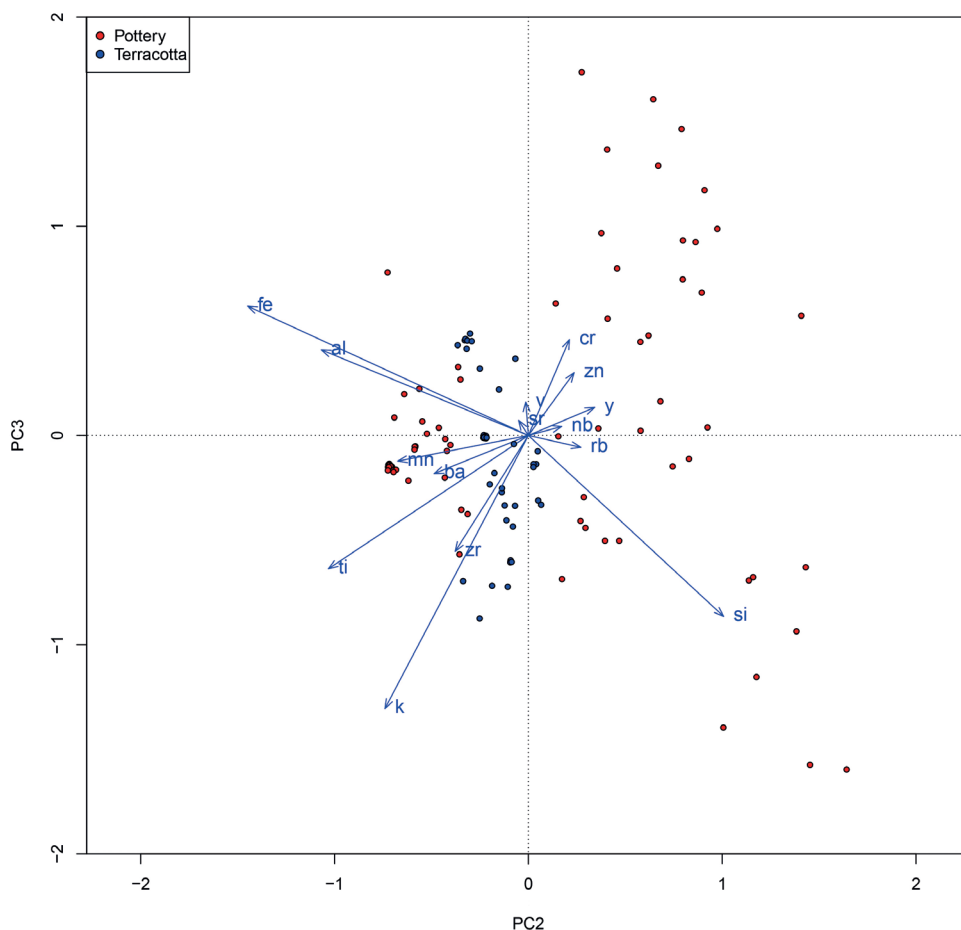


Fig. 43. Correlation biplot of the PCA of Ido, axes 2 and 3.

of Al and Mn and *vice versa*. The same is true for the other two groups: Si has a negative correlation with the group comprising Fe, K, and Ti. The trace elements (and their negative correlation with Al and Mn) account for the difference between the pottery and terracotta: they – metaphorically speaking – “draw” the pottery away from the figurines. The separation of the Post-Nok sherds in a distinct group is the result of differing concentrations of Fe, K, and Ti on the one hand and Si on the other, while the concentrations of the other elements seem to have no influence on this pattern.

The correlation biplot of axes 2 and 3 (Fig. 43) helps explain the structure in the chemical composition of the terracotta figurines revealed by axis 3. While the major elements mainly keep their association to each other, the trace elements are now split up in two groups with a negative correlation (Ba and Zr *vs.* Cr, Nb, Sr, V, Y, and Zn). This fact is responsible for the slight differences in the figurine samples. The clay source used for the terracotta was apparently not completely homogeneous in the distribution of the trace elements. As trace element concentrations derive from the weathering of the bedrock and are additionally altered during transportation and deposition of the clay through the environment, this finding is not remarkable. A completely homogeneous clay source in even the finest concentrations of trace elements does not exist in nature. Slightly different constellations of trace elements are thus not unusual. The fact that this pattern shows up only on the third axis demonstrates the low significance of this variance for the dataset and the high uniformity of the clay used for the figurines.

## 6.2 Ifana (IFA)

Ifana, like Ido, lies in the centre of the key research area of the Frankfurt project (Fig. 2). Two trenches with a total size of 89 m<sup>2</sup> were excavated in 2011. Old looting holes confirm earlier illegal diggings in the close proximity. Trench IFA 1 contained a feature with large fragments of six terracotta figurines (Fig. 44). Trench IFA 2 contained two pit-like features. The absolute datings of this site (all on *Pennisetum* grains) are very consistent and point to the Middle Nok period: The date for IFA 1, taken from beneath the terracotta deposition, lies between 893-772 BCE (2-sigma range) and two dates for IFA 2 fall between 801-555 BCE (feature in the south-west corner of the trench) and 811-762 BCE (feature in the north-east corner of the trench). The occupation period of Ifana is therefore dated to the first half of the first millennium BCE, within in early Middle Nok phase, most probably between the 9<sup>th</sup> and 8<sup>th</sup> century BCE (FRANKE 2015).





Fig. 44. The terracotta deposition from IFA 1 during excavation in 2011.

Finds consisted of the “typical” Nok assemblage of pottery, terracotta, charcoal, burnt clay, and stone artefacts. The pottery assemblage is – compared to the size of the excavation and with other sites – relatively small and poorly preserved. The analysis of the decoration patterns shows that the inventory is a mixture of different styles, although all belong to Middle Nok – meaning that if the site was occupied or used several times, then within a short period of time. No differences between IFA 1 and IFA 2 were apparent (FRANKE 2015). Aside from the six large figurines, larger and smaller terracotta fragments were distributed over the whole area of IFA 1, most of them diagnostic. In IFA 2, several fragments of at least two larger figurines were found, but reconstruction was impossible because of the large amounts of missing parts. In addition, some smaller fragments were distributed throughout the unit. In IFA 2, the number of figurine pieces in general is relatively small.

Ifana was chosen for site-level analysis because of the terracotta deposition – a feature that can be found at some, though not many Nok sites (e.g. in Utak Kamuan Garaje Kagoro or in Daji Gwana) – and because of the sufficient

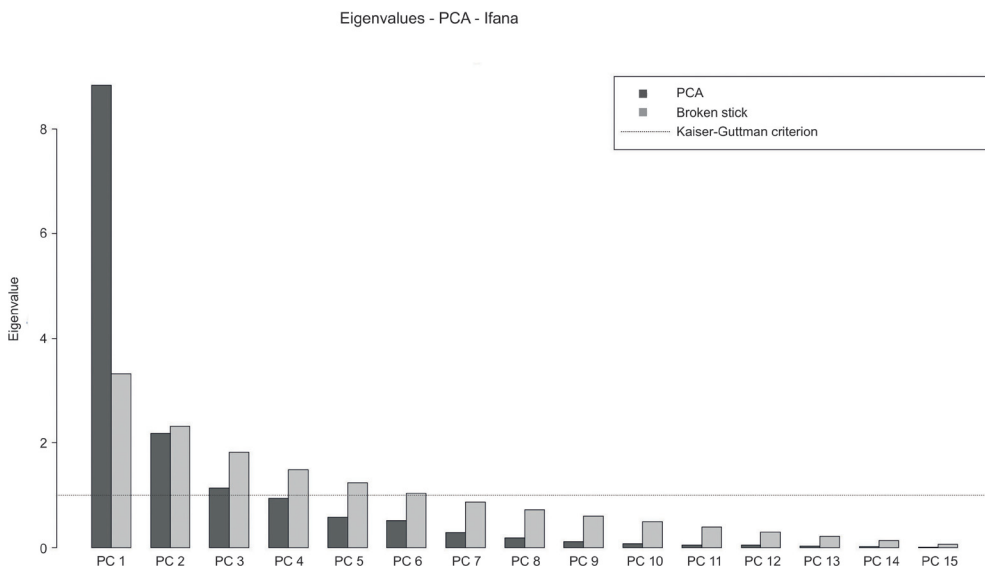


Fig. 45. Diagram of eigenvalues of the PCA of the pottery and terracotta of Ifana with broken stick and Kaiser-Guttman criterion.

amount of ceramic material (other terracotta parts and pottery) for statistical analysis. These factors, along with the fact that the site can be reliably dated to the Middle Nok period, make Ifana a good candidate to investigate possible differences between the distinct find contexts of the figurines (deposition of largely complete figurines in IFA 1 *vs.* various pieces in refuse pits mixed with other cultural material in IFA 2). For statistical analysis, 93 pottery samples (48 from IFA 1 and 45 from IFA 2) and 157 terracotta samples (IFA 1: 135, IFA 2: 22) were selected.

The diagram of the eigenvalues of the Ifana samples is shown in Figure 45. The Kaiser-Guttman criterion suggests, as in Ido, the interpretation of the first three axes. The broken stick model, however, suggests only the first, or maybe also the second; but a look at the cumulated eigenvalues of the PCA (Tab. 5) justifies the inclusion of the first three axes as they explain *ca.* 81% of the variance.

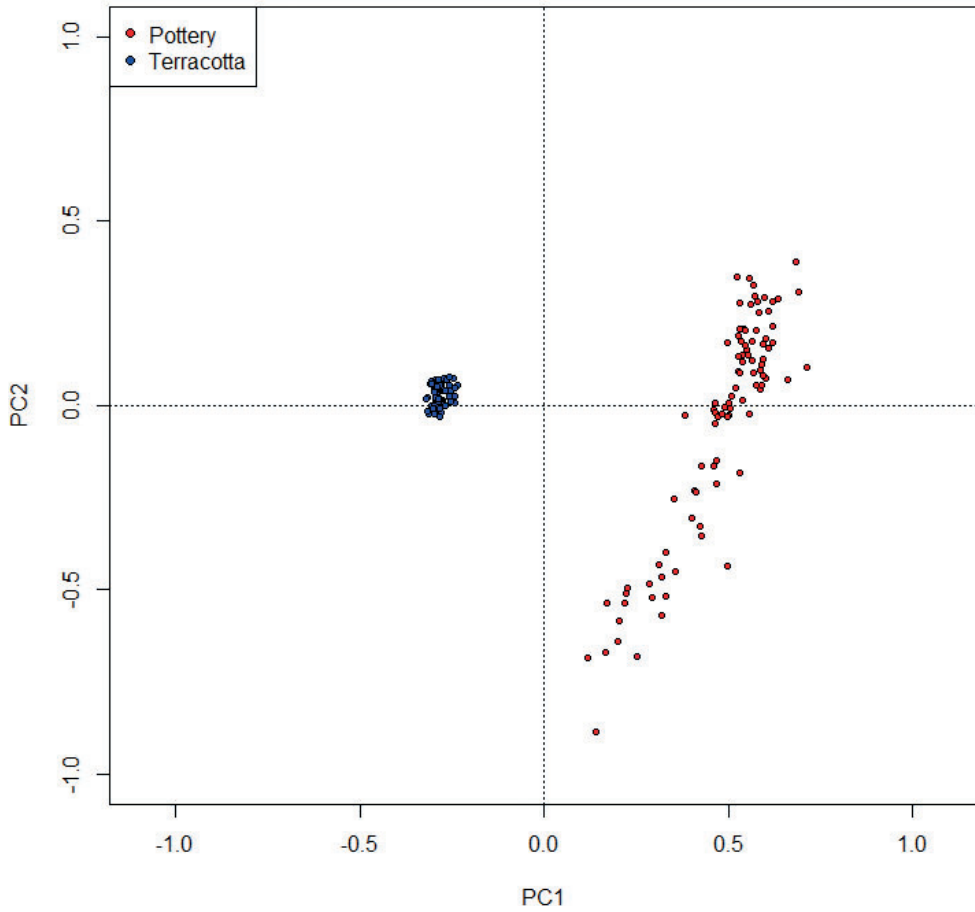


Fig. 46. Distance biplot of the PCA of Ifana, axes 1 and 2.

PC1: 58.87%	PC2: 73.41%	PC3: 81.02%	PC4: 87.28%	PC5: 91.17%
PC6: 94.61%	PC7: 96.52%	PC8: 97.75%	PC9: 98.54%	PC10: 99.06%
PC11: 99.37%	PC12: 99.65%	PC13: 99.83%	PC14: 99.93%	PC15: 100%

Tab. 5. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of Ifana.

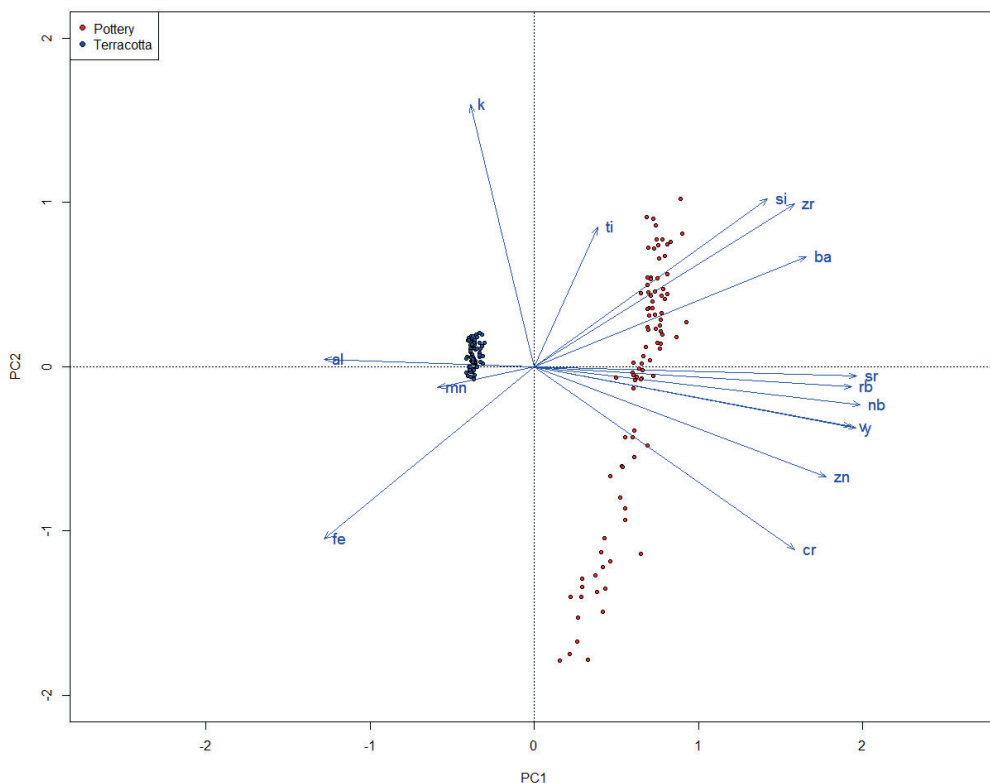


Fig. 47. Correlation biplot of the PCA of Ifana, axes 1 and 2.

The distance biplot of axes 1 and 2 (Fig. 46) displays the same picture as in Ido: the terracotta samples (blue) form a distinct cluster of points in the left part of the diagram, whereas the pottery samples show a less homogeneous chemical composition and scatter broadly. The pottery group set off slightly to the bottom right cannot currently be explained, as it consists of a mixture of potsherds from IFA 1 and 2 alike, for which, due to poor preservation, no similarities in decoration can be found. With regard to the terracotta samples, no structure or differences between the deposited large figurines from IFA 1 and the smaller fragments from the refuse pits of IFA 2 are visible.

The corresponding correlation biplot (Fig. 47) reveals the reason for the separation of the two material groups: as in Ido, the trace elements are again responsible for the distinct building of the two clusters, while the major elements are responsible for the internal pattern of the pottery (K, Si, and Ti in negative correlation with Al, Fe, and Mn).

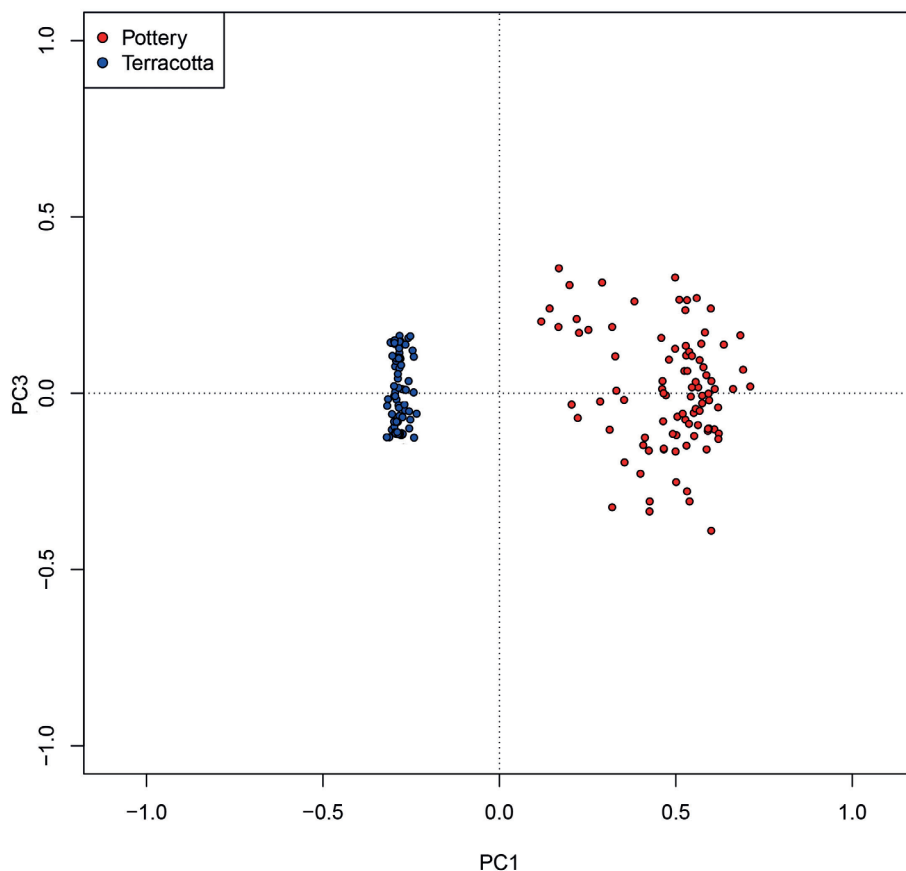


Fig. 48. Distance biplot of the PCA of Ifana, axes 1 and 3.

A little bit more structure is visible when looking at the distance biplot of axes 1 and 3 (Fig. 48). Axis 3 shows a slight difference in the composition of the terracotta, whereas the scattering of the pottery remains more or less the same, with no explainable structure in either of the two material groups. The correlation biplot (Fig. 49) of the same axes reveals that the variation within the terracotta samples is due to the negative correlation of the major element K with Al, Fe, Mn, and Ti. The trace elements (together with Si) are, again, responsible for the separation of the pottery and terracotta.

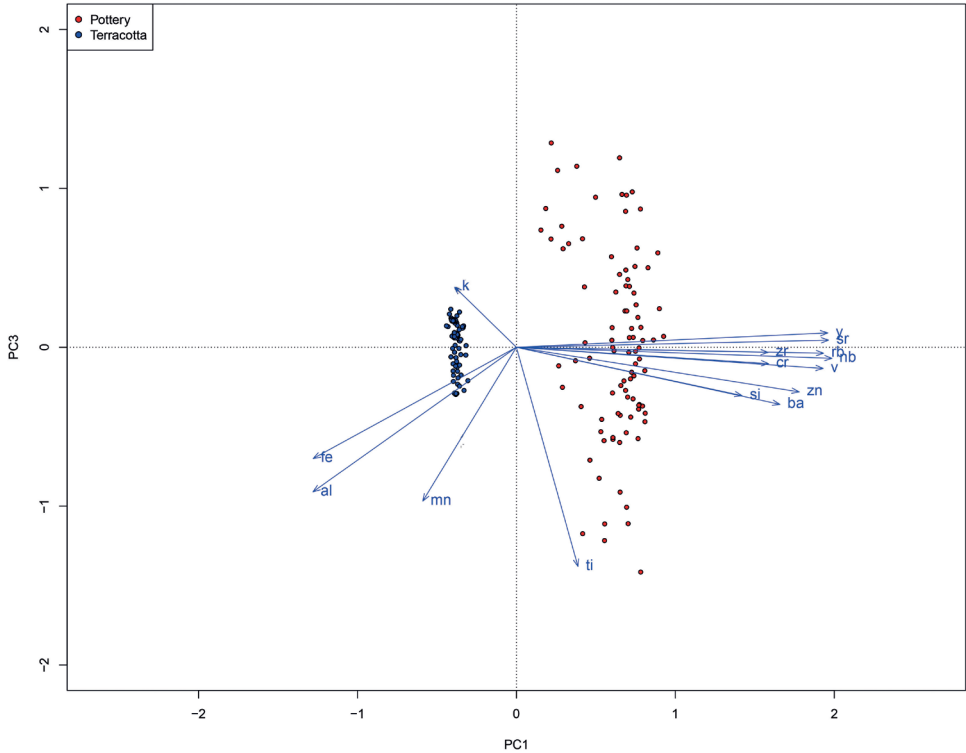


Fig. 49. Correlation biplot of the PCA of Ifana, axes 1 and 3.

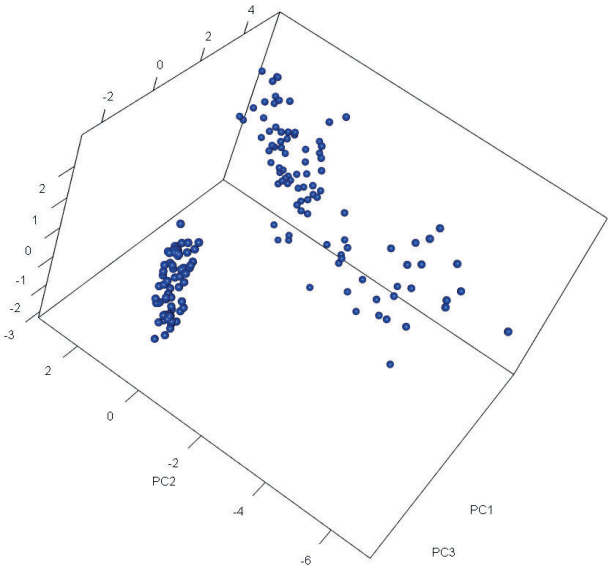


Fig. 50. 3D-plot of the first three axes of the PCA of Ifana. Left: terracotta, right: pottery.

The consideration of axes 2 and 3 yields no new insight into the dataset and is therefore not presented here. The 3D-plot of Ifana (Fig. 50) summarises the results of the observations from the individual reviews of the axes, again showing the separation of the densely clustered terracotta samples and the more widely spread potsherds.

### 6.3 Ungwar Kura (UK)

The site of Ungwar Kura was excavated in 2007 as one of the Frankfurt project's first major excavations. Located on a big plateau and a smaller, directly adjacent hilltop, the excavation consists of 16 trenches with a total size of 325 m<sup>2</sup>. The 17 absolute datings of the different trenches of Ungwar Kura have a wide range between *ca.* 800 and 200 BCE. The pottery analysis supports the absolute datings falling mostly into the Middle Nok phase, and a detailed spatial analysis is planned for the future. Especially the potsherds from UK 9, 12, 14, and 16 show decoration patterns different from the rest of the research area (FRANKE 2015), as well as some characteristics resembling the finds Fagg made in Taruga (FAGG 1967). Some of the absolute dates for the trenches, along with these uncommon decorations and forms, suggest a Late Nok occupation phase. The site thus may feature a complex occupation history, with a peak of activity in the latter half of the first millennium BCE (FRANKE 2015).

Several pit-like structures containing most of the finds were found in the trenches. The find spectrum at Ungwar Kura includes pottery, terracotta, burnt clay, charcoal, stone artefacts, iron objects, and slag. As already pointed out, the decoration differences in the ubiquitously distributed pottery may indicate different occupation episodes. The terracotta finds are likewise abundant. Several nearly complete figurines and larger parts were found (*e.g.*, a double-headed lizard, a male figure with hat, and a nearly life-size male head, all from UK 9, as well as some smaller complete heads from UK 1 and 6), all in refuse pits mixed with other cultural material – not deposited as in Ifana 1, for example.

Ungwar Kura was chosen primarily because of its location outside the key research area of the Frankfurt project, approximately 45 km south-east of the research station in Janjala. It therefore provides the opportunity to test whether the findings made in the key research area can be transferred to the wider surroundings – even if not to the entire distribution area of the Nok Culture. In addition, the different occupation phases visible in the absolute dates and the pottery analysis can be used to test whether the different chronological phases of the Nok Culture are reflected in the chemical composition of the pottery (here: Middle *vs.* Late Nok).



A total of 88 pottery samples (22 from UK 6, 29 from UK 7, 28 from UK 9, and 9 from UK 14) were selected for analysis. The selection of the terracotta samples consisted of 140 samples (43 from UK 6, 25 from UK 7, 44 from UK 9, and 28 from UK 14).

According to the Kaiser-Guttman criterion in the diagram of Ungwar Kura eigenvalues (Fig. 51), the first three axes of the PCA can be interpreted, although – as in Ifana – the broken stick model suggests a two-dimensional solution. Here again, the cumulated eigenvalues support the recommendation of the Kaiser-Guttman criterion (Tab. 6).

PC1: 58.97%	PC2: 74.98%	PC3: 81.89%	PC4: 87.47%	PC5: 91.34%
PC6: 94.51%	PC7: 96.58%	PC8: 97.76%	PC9: 98.48%	PC10: 98.97%
PC11: 99.33%	PC12: 99.65%	PC13: 99.80%	PC14: 99.92%	PC15: 100%

Tab. 6. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of Ungwar Kura.

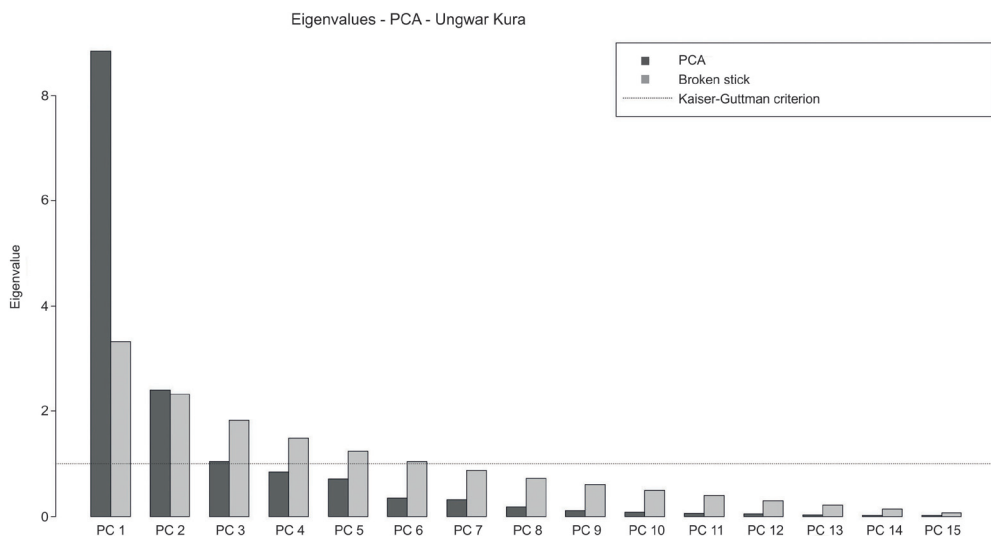


Fig. 51. Diagram of eigenvalues of the PCA of the pottery and terracotta of Ungwar Kura with broken stick and Kaiser-Guttman criterion.

The distance biplot of axes 1 and 2 (Fig. 52) shows nearly the same picture as the two previously presented biplots of Ido and Ifana: while the figurines form a dense cluster of points, the potsherds scatter considerably. A smaller group of pottery tends toward the lower edge of the diagram, although its separation from the main group is not very clear. A close examination of the dataset revealed that this smaller group consists of potsherds from UK 9 and 14, possibly belonging to the Late Nok phase.

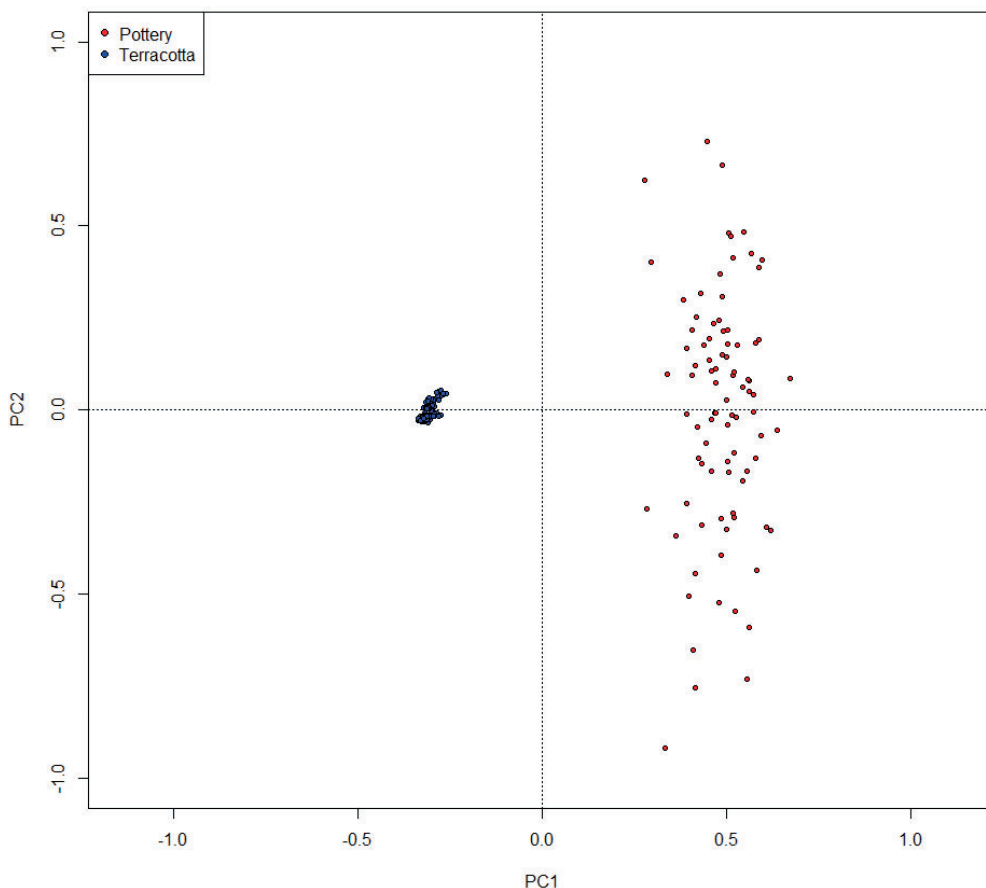


Fig. 52. Distance biplot of the PCA of Ungwar Kura, axes 1 and 2.

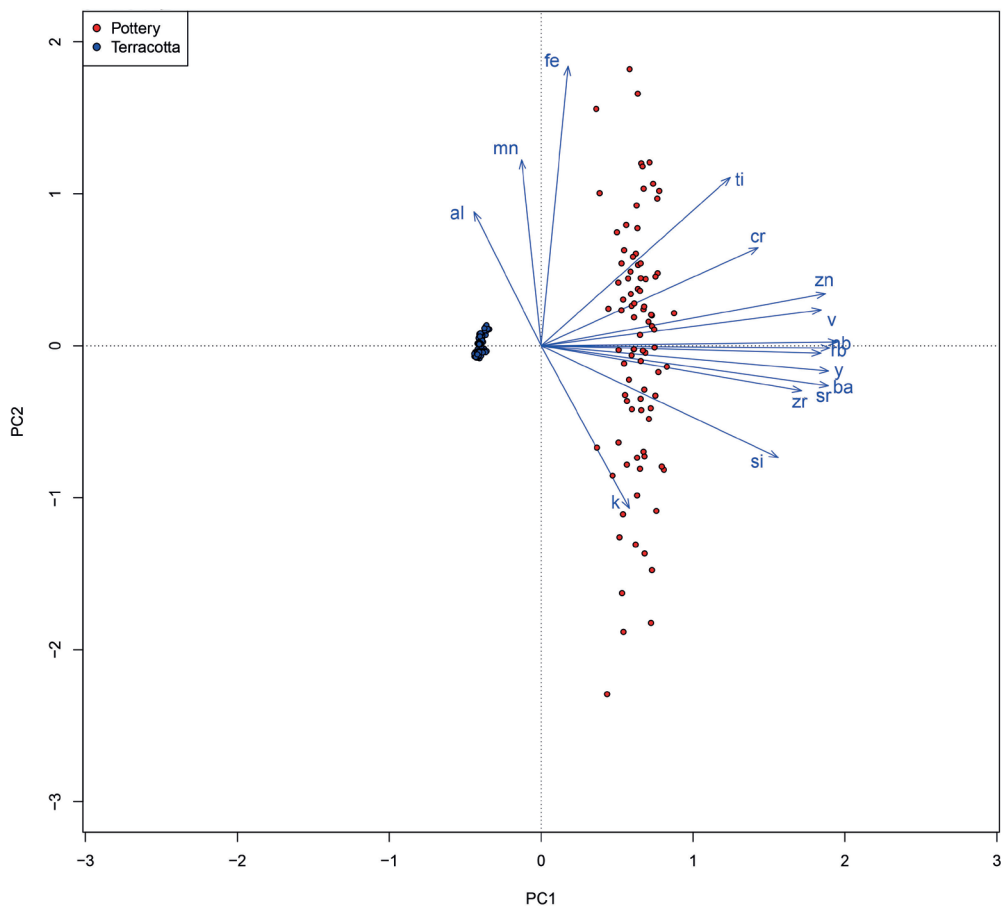


Fig. 53. Correlation biplot of the PCA of Ungwar Kura, axes 1 and 2.

The chronological depth of the occupation of Ungwar Kura, traceable in decoration and form of the pottery, can thus also be seen (though not as clearly as the general pottery/terracotta distinction) in the chemical composition, *i.e.*, the use of raw material sources. The example of Ido showed that it is possible to separate Nok and Post-Nok pottery material by this method; the result from UK now suggests that at least tendencies within the Nok Culture are also recognizable.

The corresponding correlation biplot (Fig. 53) illustrates the reason for this picture: as before, the trace elements are responsible for the separation of pottery and terracotta. Also, K and Si correlate negatively with Al, Fe, and Mn, accounting for the variance in the pottery samples as a whole and the separation of the Middle and Late Nok material.

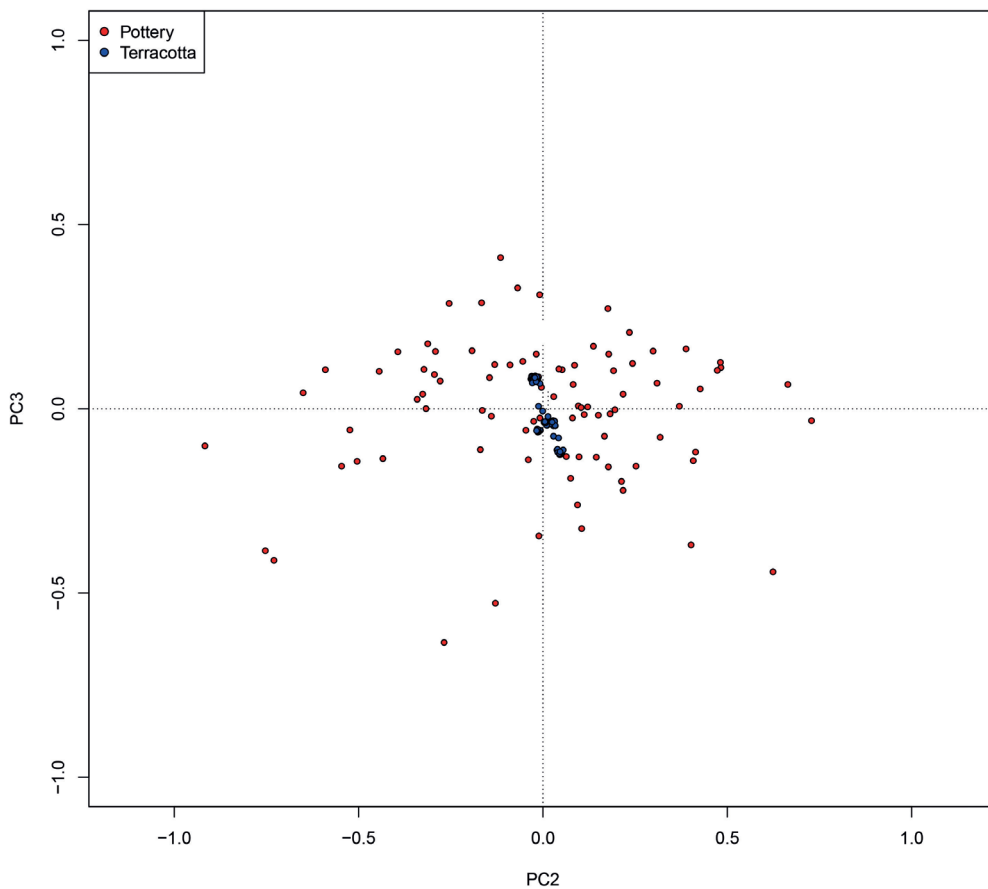


Fig. 54. Distance biplot of the PCA of Ungwar Kura, axes 2 and 3.

A distance biplot of axes 2 and 3 (Fig. 54) shifts the viewing angle and leads to the overlapping of pottery and terracotta samples. Nevertheless, axis 3 again reveals some internal pattern within the figurine group. According to the corresponding correlation biplot (Fig. 55), this is caused by negative correlations of the trace elements Ba, Nb, Rb, Y, and Zr, and the major elements K and Si with the trace element Cr and major element Ti. All other elements account primarily for the variation between the pottery samples.

The 3D-plot of all three axes (Fig. 56) shows that no great variation occurs between axes 1 and 3. Therefore, no detailed description will be presented here.

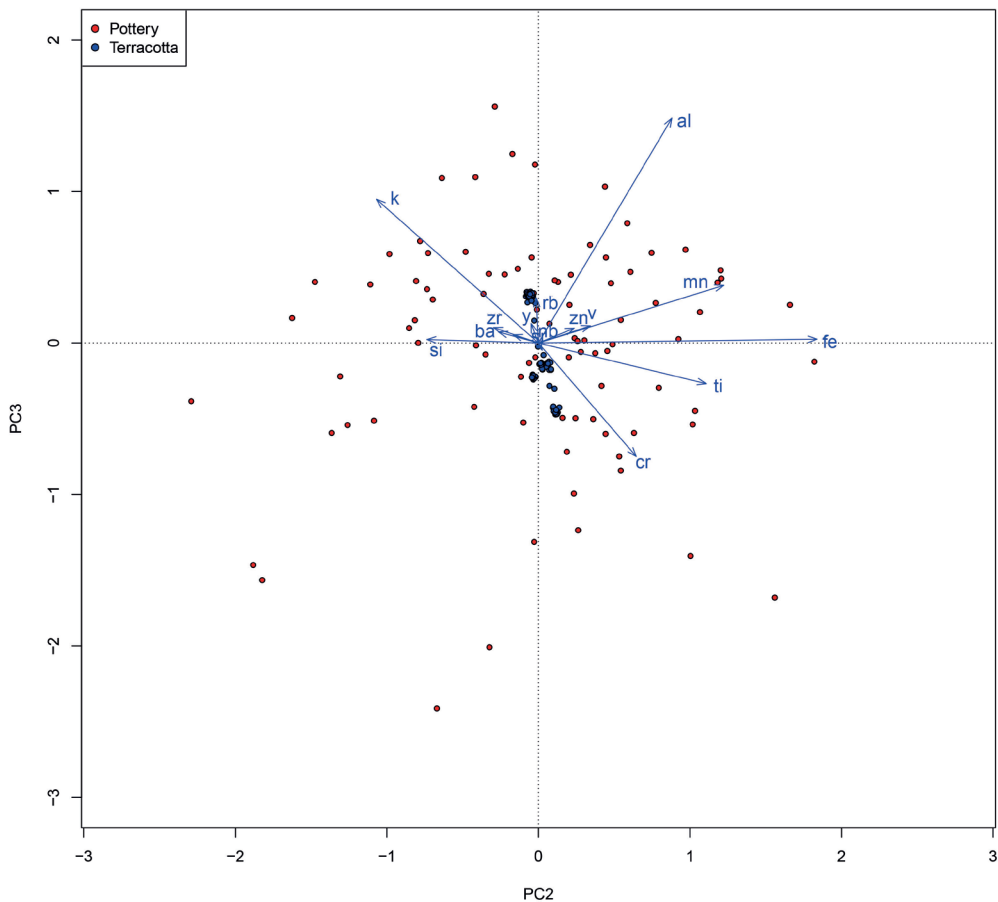


Fig. 55. Correlation biplot of the PCA of Ungwar Kura, axes 2 and 3.

## 6.4 Summary

The statistical analysis of the chemical composition of terracotta and pottery on a site level impressively demonstrates the potential of geochemical analysis in archaeology in general and for the Nok Culture in particular. The use of clay with distinct properties – as suggested by the thin sections (Chapter 4) – was successfully confirmed by the chemical analysis. In all three site-level PCAs, the figurines cluster together in a narrow group with no clear internal differentiation. **Only on the third axis of the PCA (which not all different statistical criteria suggest for consideration) do slight patterns emerge that are related to minimal variances in the composition of the clay.** Naturally occurring clay sources are never completely homogeneous in their composition, especially not with regard to trace elements, as these derive from

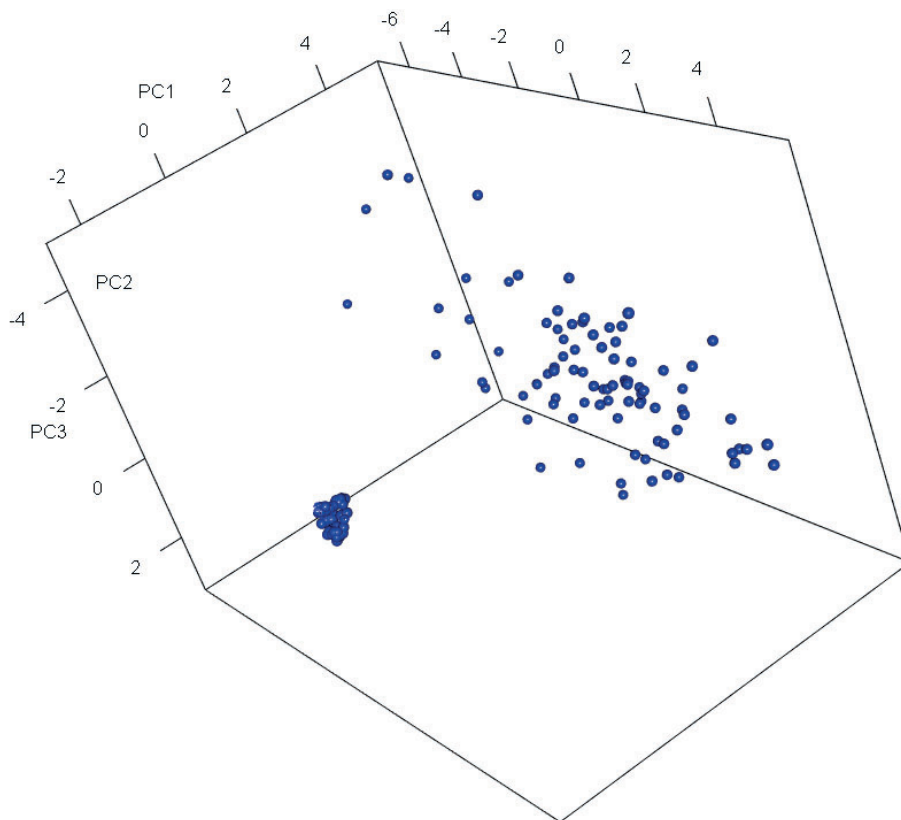


Fig. 56. 3D-plot of the first three axes of the PCA of Ungwar Kura. Left: terracotta; top right: Late Nok pottery; front right: Middle Nok pottery.

the weathering of the parent bedrock and the transportation and depositional history of the clay minerals. The fact that nevertheless the variances in the clay used for the figurines are so small indicates the use of a very distinctive, clearly defined clay source.

In Ifana, the analysis also focussed on the question of whether different find contexts of the figurines (in distinct depositions or as part of refuse pits) – and the corresponding possible difference in the use or purpose of the figurines – can also be traced in a different chemical composition. This is not the case; the different find contexts (and possible uses or purposes) of the terracotta sculptures are evidently independent of the manufacturing process. The same seems to be true for the chronological depth of the terracotta production. In Ungwar Kura at least, where the absolute dates and the pottery chronology prove a (not necessarily continuous) occupation history of *ca.* 600 years, no chemical difference between the statues could be detected. Furthermore,

Ungwar Kura proves that the uniformity of the chemical property of the clay is no locally restricted phenomenon of the key research area, as the same pattern was found outside the key research area. The question of how such a consistency in a material property that could not be seen or tested at that time can be maintained is discussed in Chapter 9.

In contrast with the terracotta, the pottery displays significant variations in chemical composition across all reviewed sites. This may be attributed to the use of various local raw clay deposits for domestic pottery production. The terracotta sculptures and the pottery, then, definitely do not stem from the same manufacturing process, as completely different clays were used. However, the example of Ido shows that no discrimination concerning the different intended purposes of the pottery was apparently made: the two visually unusual (because very finely made and decorated) pots whose find context suggests a function as grave goods do not stand out in the chemical analysis. They were thus produced with the same clays as the “normal” pottery.

The analysis of the pottery has proven to be especially interesting when looking at the chronological depth. In Ido, a clear separation of Nok and Post-Nok sherds is possible. As Ungwar Kura shows, such differences in the use of raw material sources in the different chronological phases can be traced even within the Nok Culture: the clay sources used to manufacture pottery evidently changed during the almost 1500 years of duration of the Nok Culture. XRF analysis may thus help – together with the analysis of pottery decoration and form – structure especially the Middle Nok period, where exact absolute datings are difficult because of the Hallstatt Plateau of the radiocarbon calibration curve. In future, chemical analysis can also help safely place Early Nok sites of the late second millennium BCE (where no terracotta figurines appear) in a Nok Culture context. Some preliminary analyses not presented here suggest that the recognisable development and continuity of the decoration styles is accompanied by a continuity of the raw materials used.

This result opens many new possibilities for establishing chronological systems in archaeology. In the case of the Nok Culture, XRF analysis provides the chance to combine the chronology established by absolute datings and pottery analysis with a chronology of raw materials used.



## **7 Overall Results of the Geochemical Analysis (Terracotta and Pottery) and Comparison to Recent Clay Samples**

The statistical analysis of the chemical composition of terracotta and pottery at the three Nok sites of Ido, Ifana, and Ungwar Kura (Chapter 6) has revealed distinct differences between the two find groups. The stylistic uniformity that makes the figurines immediately recognisable as Nok artefacts is apparently linked to a similar uniformity of material. This material uniformity, while not macroscopically visible, is at least as consistent as the similarities in outward appearance, if not more so. It is maintained throughout different find contexts (apparently purposeful deposition *vs.* “waste disposal” in pits) and different age groupings (Middle and Late Nok), as shown at Ifana and Ungwar Kura, respectively. In addition, the pottery at Ido and Ungwar Kura yielded valuable information on the chronological differences of raw materials used. This supports the chronology of the Nok Culture and aids distinguishing later cultural complexes (see FRANKE 2015).

In order to rule out the possibility of a coincidence or statistical error brought on by a selection of specific sites or specific samples, this finding will now be tested against the complete dataset of analysed terracotta and pottery (3051 samples – 1412 terracotta fragments and 1639 pottery samples – from 43 sites; Fig. 28). By this method, the analysis can cover a large geographical area and the whole duration of the Nok Culture, supplemented by some pottery samples belonging to the Post-Nok phase. In a second step, the results will be compared to the composition of present-day clay samples from central Nigeria (see Chapter 5.4. for a description of sample selection). From this comparison, it may be possible to extrapolate information about the nature and perhaps even the provenance of the clay used for the figurines, despite the long time that passed between the terracotta production and the present.

A comprehensive discussion of the results of the statistical analyses presented in Chapters 6 and 7 is given in Chapter 8.

### **7.1 Comparison of the Chemical Composition of the Terracotta and Pottery from 43 Sites**

This statistical analysis focuses on the terracotta and pottery from all 43 sampled sites. The diagram of eigenvalues showing the “broken stick” criterion and the Kaiser-Guttman criterion (Fig. 57) suggests interpretation of at most the first two axes – according to the “broken stick” model, no more than even the first.

The table of cumulated eigenvalues (Tab. 7) supports using the first two axes, as they explain approx. 77% of the variance in the data. The large increase in the number of analysed samples as compared to the site-level analyses clearly reinforces the inherent pattern in the dataset (*i.e.*, a definite difference between pottery and terracotta), such that the greatest proportion of the variance is explained in the two-dimensional space spanned by the first two PCA axes. In fact, 10 PCA axes are sufficient to describe all the variance, even though 15 variables (the measured elements) are present in the dataset, which could result in a possible maximum of 15 dimensions.

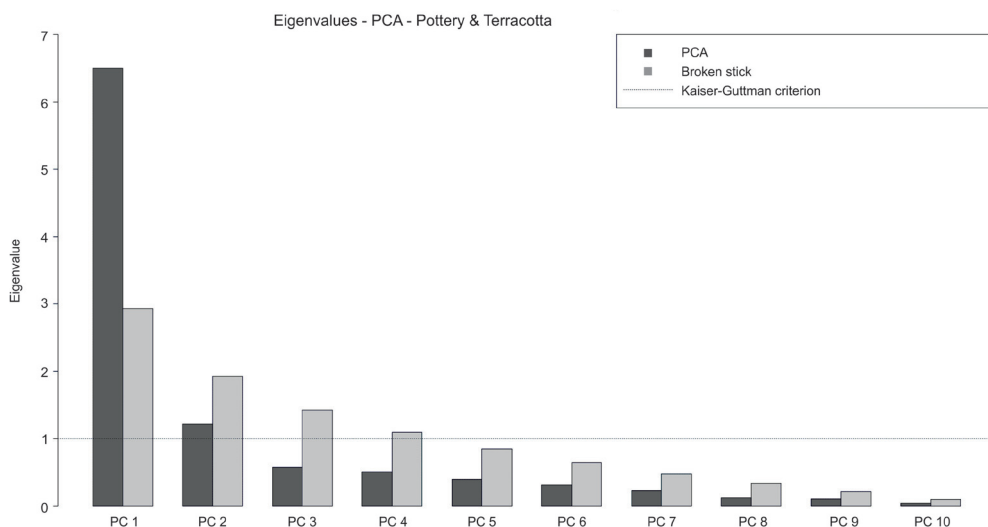


Fig. 57. Diagram of eigenvalues of the PCA of the pottery and terracotta of 43 sites with broken stick and Kaiser-Guttman criterion.

PC1: 65.99%	PC2: 77.15%	PC3: 82.94%	PC4: 87.97%	PC5: 91.96%
PC6: 95.06%	PC7: 97.33%	PC8: 98.53%	PC9: 99.56%	PC10: 100%

Tab. 7. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of 43 sites.

The distance biplot of axes 1 and 2 is shown in Figure 58. The result mirrors the findings already made at the three sites Ido, Ifana and Ungwar Kura: while the terracotta samples constitute a dense cluster of points, the pottery points scatter and show at least some internal pattern. Although a slight internal grouping of the pottery samples is recognisable, some specimens do appear to orient to the upper or lower end of the main scatter of the pottery samples. The lower group can be explained: they represent

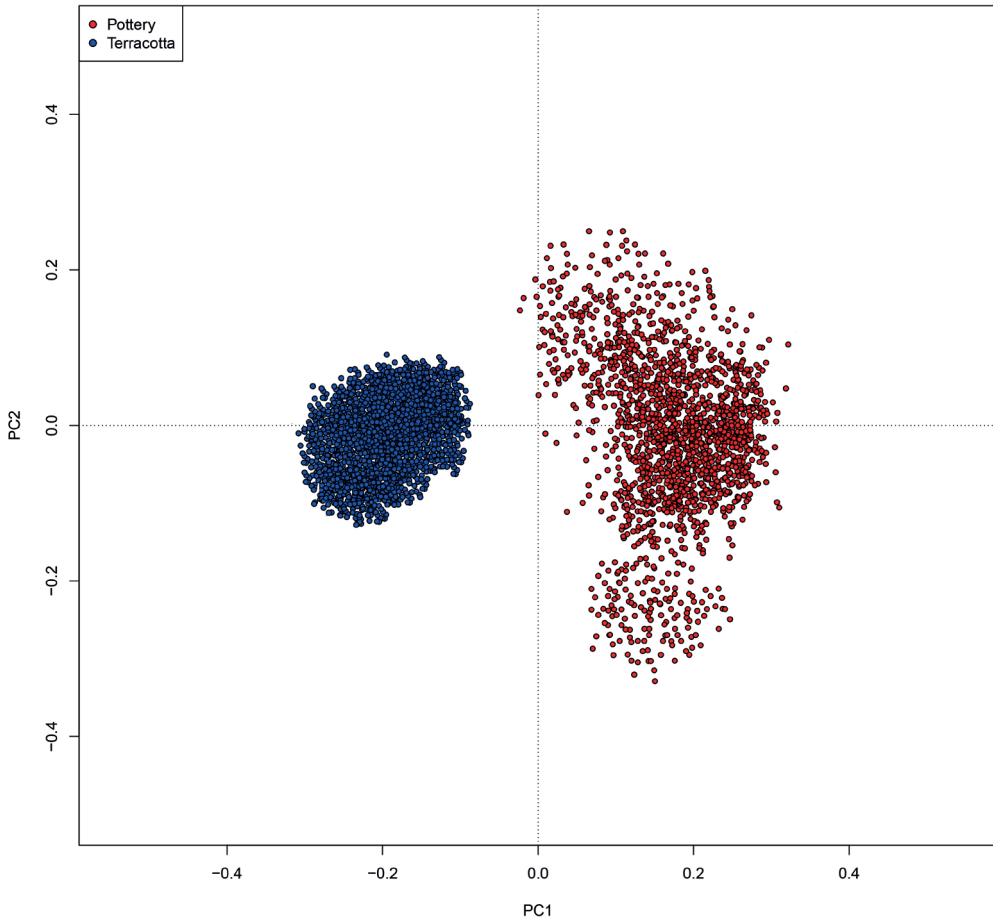


Fig. 58. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2). The difference between the chemical compositions of the figurines in contrast to the potsherds is clearly visible.

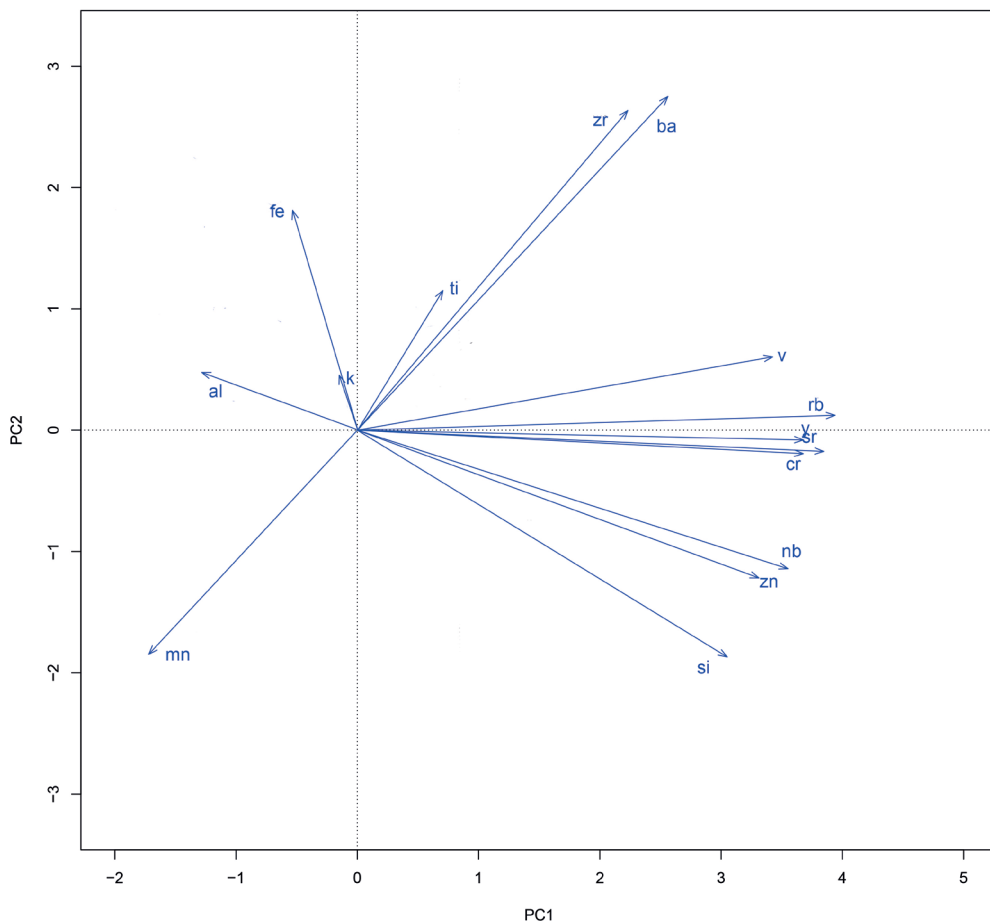


Fig. 59. Correlation biplot of the PCA of pottery and terracotta of 43 sites.

samples not belonging to the Nok Culture.<sup>56</sup> The result already observed in Ido – that potsherds from the Post-Nok period feature a different chemical composition than Nok potsherds – is thus confirmed. The distinction between the different Nok phases which is evident in the analysis of the Ungwar Kura samples (see Chapter 6.3) cannot be seen when looking at all 43 sites. The variances between the different Nok phases are so small that they are dwarfed by the high number of samples. Such small intra-group differences require a site-level analysis.

<sup>56</sup> Along with sites where Nok material and Post-Nok material are intermixed (such as Ido), this group also contains sites which, according to absolute dating and pottery analysis, belong exclusively to the Post-Nok period (such as Gimba – approx. 1400 CE – and Mashikin Dandoka – approx. 900 CE) (FRANKE 2015).

The corresponding correlation biplot (Fig. 59)<sup>57</sup> also confirms the result of the site-related study: the reason for the separation of the pottery and terracotta into two groups lies in the different proportions of trace elements. Vectors associated with trace elements have more or less the same length and are positively correlated. Only Ba and Zr do not contribute as strongly to this separating effect. In contrast, the major elements follow two sets of negatively correlated elements: Si (following the same direction as the trace elements) correlates negatively with Al, Fe and K; Ti (together with Ba and Zr) is negatively correlated with Mn. The correlations of the major elements have less influence on the overall displayed pattern, as the arrows are much shorter (Si is an exception here) and are not concentrated in one direction, thus generating smaller variances in the dataset.

Consequently, another PCA was computed with a subset of the data only containing the nine trace elements Ba, Cr, Nb, Rb, Sr, V, Y, Zn, and Zr – thus eliminating the minor variances produced by the major elements. The diagram of eigenvalues (Fig. 60) confirms this approach: now, only five (of potentially nine) PCA axes are needed to display the variance of the entire dataset. Of these axes, only the first is clearly interpretable as it already contains more than 80% of the variance. Axis 2 contributes only approx. 8% additional information (Tab. 8). The structure in the dataset is so clear that the dimensions could be reduced extremely – theoretically, one axis would be sufficient for the analysis. However, the second axis is included, as the Kaiser-Guttman criterion allows the interpretation of the first two axes, and the second dimension provides a more accurate plot.

PC1: 80.40%	PC2: 88.86%	PC3: 96.04%	PC4: 98.84%	PC5: 100%
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Tab. 8. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of 43 sites – trace elements only.

<sup>57</sup> The terracotta and pottery points are not displayed in this chapter's correlation biplots to achieve a better visibility of the arrows which would otherwise be superimposed by the dense clusters. While the points can serve for a better orientation, they bear no information context in correlation biplots in which the distances between the objects (points) are meaningless. For interpreting the objects and their relation to each other, distance biplots are needed (see also Chapter 5.3).

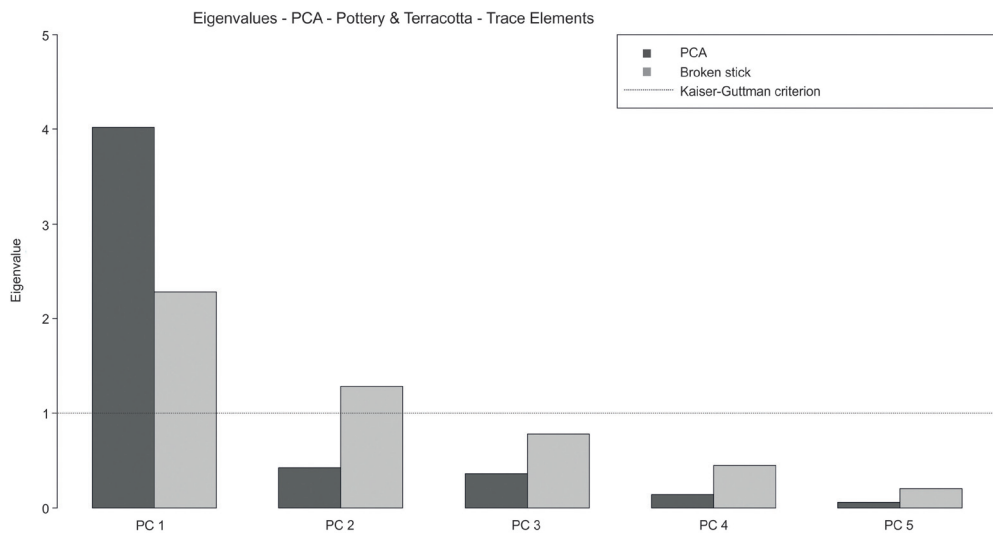


Fig. 60. Diagram of eigenvalues of the PCA of the pottery and terracotta of 43 sites with broken stick and Kaiser-Guttman criterion – trace elements only.

Accordingly, the distance biplot (Fig. 61) reveals – as expected – the same picture: the terracotta cluster points are even denser than before, while the pottery points still fluctuate considerably, with a small group separating at the lower end of the scatter. While these Post-Nok sherds now form a clearly distinct group, no internal pattern is visible among the Nok potsherds. The sample number is too large and the chemical compositions overlap too much. Although it is obvious that this picture is created by the difference in trace elements, and that according to the eigenvalues no additional information can be gained from the proportions of the trace elements, the correlation biplot was computed (Fig. 62). As expected, it reveals that all nine trace elements are positively correlated. The similar length of the vectors indicates that all variables have more or less the same influence on the pattern – only Zr seems to play a minor role.

## 7.2 Comparison of the Chemical Composition of the Recent Clay Samples to the Terracotta and Pottery

Clearly, the proportions of the trace elements in the clay are characteristic of each clay source used, while the major elements contribute as minor factors to the variance of the samples. The clay that was used for the terracotta figurines features an extremely homogeneous composition, while the pottery's clay shows some patterns of difference. On a larger scale, these may be related

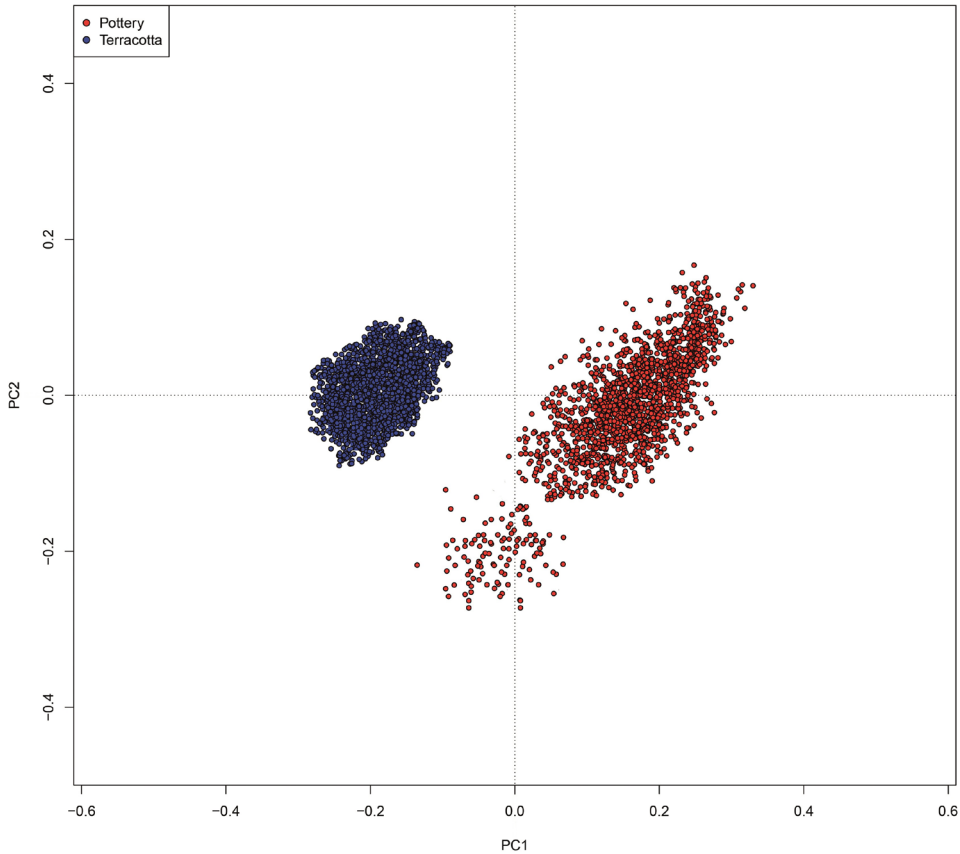


Fig. 61. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2) – trace elements only.

to different archaeological complexes (as visible in the difference between Nok and Post-Nok pottery); on closer inspection on the level of individual sites, however, they may be related to the different chronological phases of the Nok Culture. How could such a pattern be created? And where were the homogeneous sources of the terracotta's clay located? Put another way, what else characterises the clay sources used for the terracotta sculptures, and can they be linked to today's clay sources? For the moment, the only possibility of answering these questions are the clay samples collected in present-day Nigeria (Chapter 5.4). Therefore, these were included in a new PCA of the pottery and terracotta samples.

The distribution area of the clay samples overlaps widely with the key research area of the Frankfurt project and therefore with the area from which the pottery and terracotta samples were taken. The clay samples all



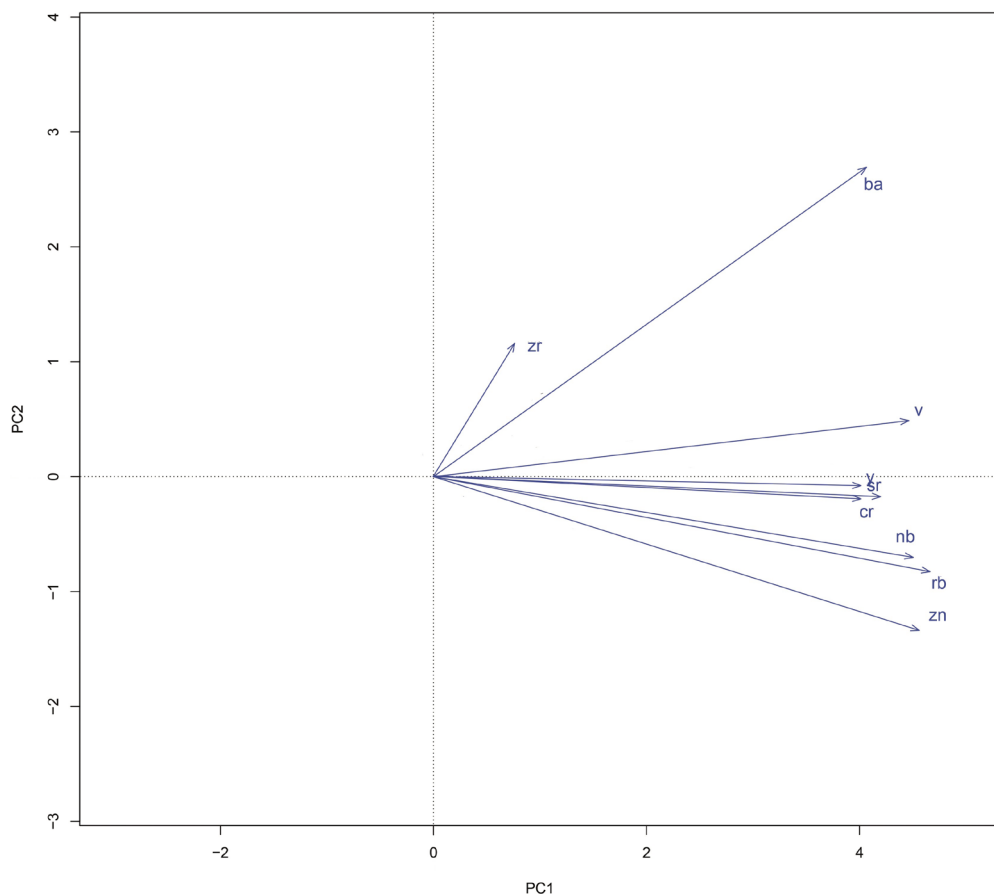


Fig. 62. Correlation biplot of the PCA of pottery and terracotta of 43 sites – trace elements only.

come from deposits that were still in use (or at least have been in the last decades) for the production of domestic pottery in 2009 and 2011. The samples also come from different geological contexts – as variable as the geological contexts can be in central Nigeria (Chapter 3). Where possible, the raw clay samples were supplemented by sherds of pots manufactured with these clays.<sup>58</sup> This selection enables a direct comparison of the raw material and the finished product, and provides information on the influence which the clay's preparation and the addition of temper have on the chemical composition in the XRF measurements.

<sup>58</sup> See the side note on clay procurement, processing, and manufacturing of pots in Nigeria today in Chapter 5.5.

Again, the first PCA was conducted with all chemical elements; a second calculation was performed with a reduced dataset limited to the trace elements to reach comparability to the previous analysis.

As was to be expected, the overall structure of the dataset did not change considerably by adding the 163 present-day samples to the original 3051 samples. The diagram of the eigenvalues (Fig. 63) still suggests the interpretation of the first two axes; the second again with caution. The cumulated eigenvalues (Tab. 9) show that five axes are sufficient to record all information in the dataset and that the first two axes explain more than 83% of the variance.

PC1: 62.94%	PC2: 83.44%	PC3: 91.93%	PC4: 97.45%	PC5: 100%
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Tab. 9. Cumulated eigenvalues of the PCA (in %) of the pottery and terracotta of 43 sites and the clay samples.

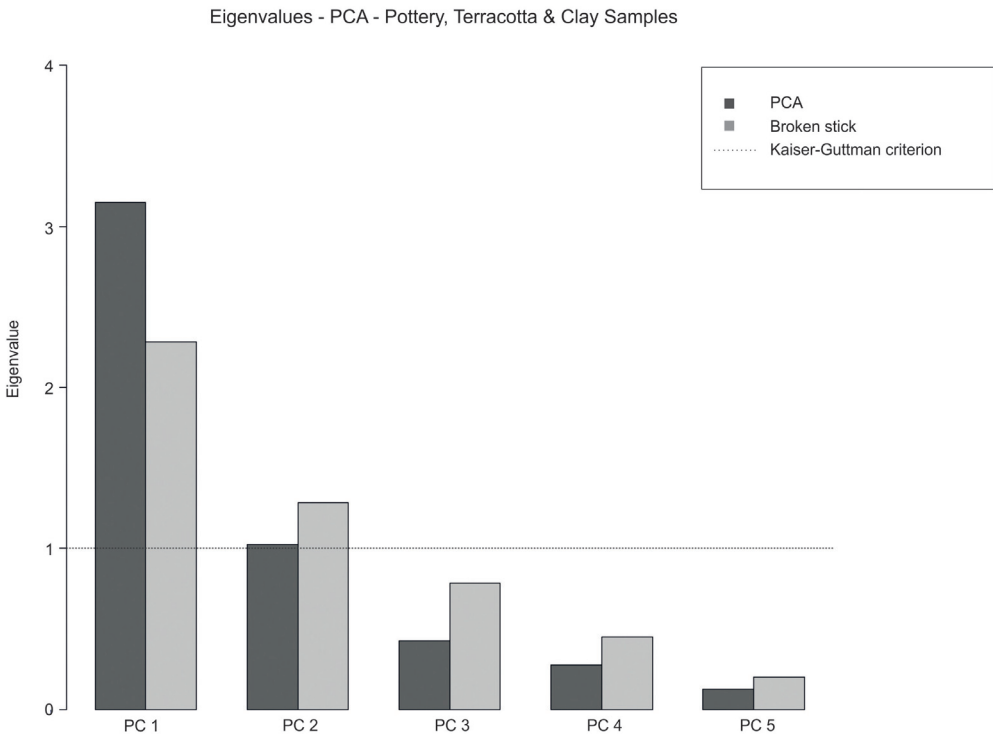


Fig. 63. Diagram of eigenvalues of the PCA of the pottery and terracotta of 43 sites and the recent clay samples with broken stick and Kaiser-Guttman criterion.

In the distance biplot (Fig. 64)<sup>59</sup>, the distribution of the pottery and terracotta remains unchanged compared to the biplot without the present-day clay samples. These recent samples, however, add a completely new pattern: they scatter over both the figurines and the domestic potsherds, with some clay samples coinciding with each find group. To verify this picture a PCA was conducted solely with the trace elements. The additional inclusion of six recent pottery samples (see Chapter 5.4) shows that their chemical composition is

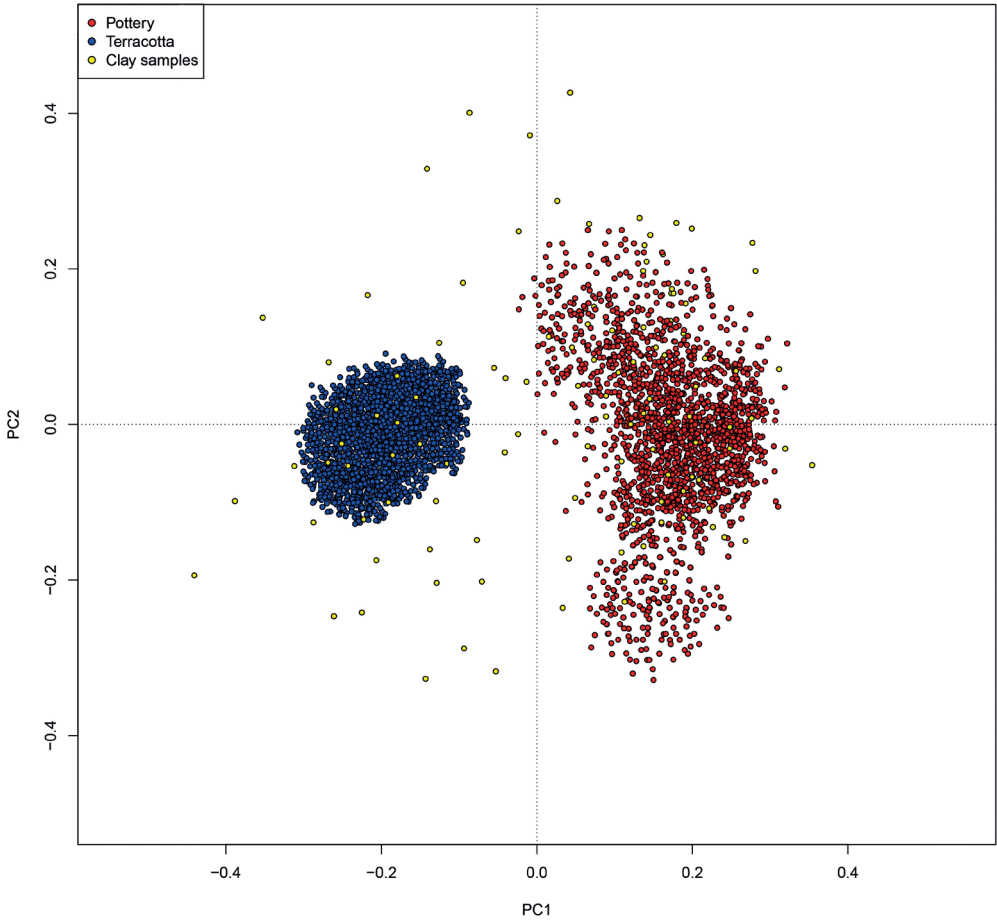


Fig. 64. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2) and the recent clay samples.

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<sup>59</sup> As neither of the following PCA changed the overall structure of the dataset from what was presented in Chapter 7.1, only distance biplots are discussed here – correlation biplots offered no new information.

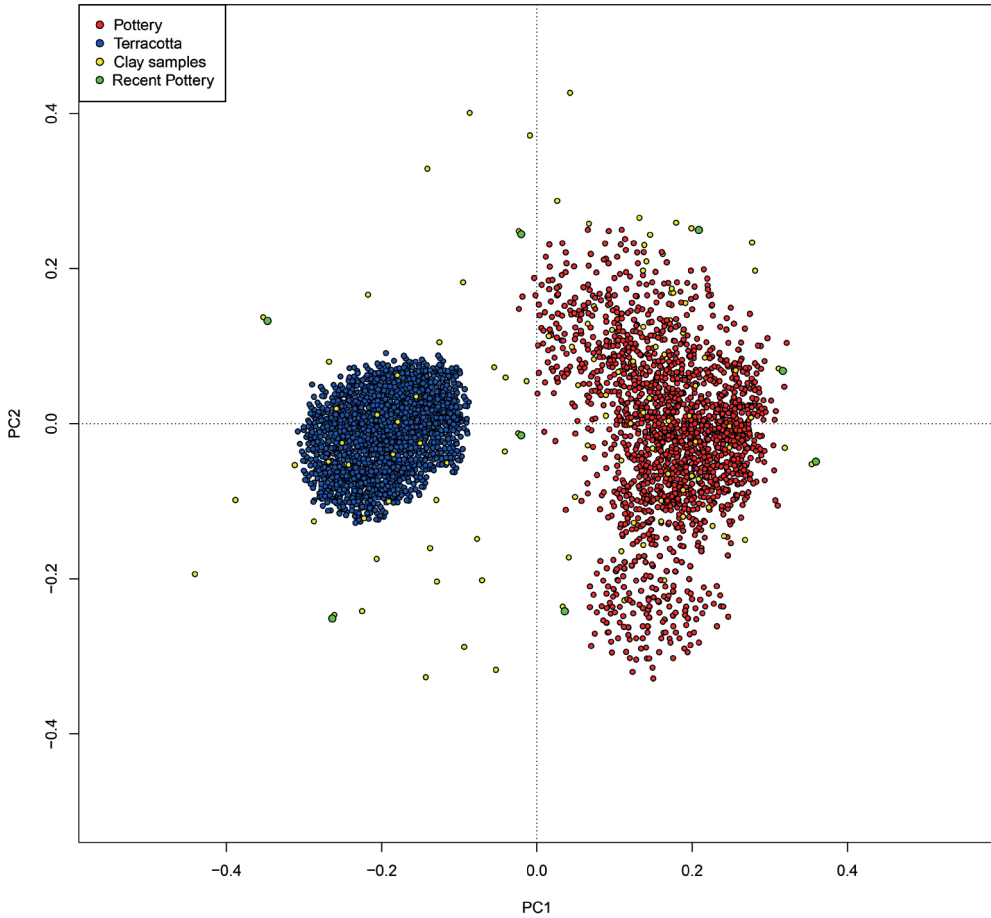


Fig. 65. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2) and the recent clay samples and recent pottery – trace elements only.

very similar to that of their corresponding clay (Fig. 65) – ceramic material can therefore be linked with the raw clay source material from which it was made.<sup>60</sup> Unfortunately, none of the recent pottery samples shows the same chemical composition as the two analysed archaeological materials.

<sup>60</sup> This important finding – although based on only a very small number of samples – will be further discussed in Chapter 8.

However, the distribution of the recent clay samples in the PCA is striking in two ways:

1) Despite the relatively uniform geology of the area under consideration, the variance of the recent clay samples is extremely high when compared with the archaeological material. 39 of the 163 clay samples show a completely different chemical composition, and clays with such properties clearly were not used to manufacture the archaeological material analysed here – no matter at what time or in which cultural complex.

2) Even more striking is the fact that the greatest proportion of the clay samples (124 out of 163) overlap in their chemical composition with the analysed archaeological material. Thirteen clays are identical/very similar in their properties to the clays used for the terracotta figurines. Although it seems unlikely – given the timespan between the Nok Culture and the present day – that any one of these clay deposits itself was used to manufacture the terracotta sculptures, the deposits show a chemical fingerprint that is extremely similar to that of the statues. This means that clay sources with the same chemical properties as the ones used during Nok times still exist and are in use in Nigeria today. Detailed archaeological investigations in the areas where clay samples with these properties occur may lead to the localisation of Nok figurine workshops (see Chapter 8 for discussion). In comparison, the finding that the remaining 111 clay samples more or less coincide (chemically speaking) with the Nok and Post-Nok pottery is no less interesting. Here again, a detailed analysis of the location of these raw clay deposits may contribute to the understanding of the dynamics of the settlement history in the area under consideration, especially as regards the structuring of sites with multiple occupation phases. For this, it is useful that the chemical analysis has been able to begin distinguishing different Nok phases (Chapter 6.3). The explicit analysis of the relationship between the recent clay samples and the Nok and Post-Nok pottery is beyond the scope of this thesis, but remains an interesting and promising task for future research.

### 7.3 Comparison of the Results of the Materials Analysis of the Terracotta and Pottery to a Forgery

A unique possibility of examining the material composition of a forged Nok terracotta sculpture emerged when the Frankfurt project (deliberately) purchased six forged terracotta statues (Fig. 66). They were brought to Germany for educational purposes. The manufacture of one was documented in great detail (BREUNIG & AMEJE 2006). Macroscopically, it was impossible to distinguish them easily from authentic Nok figures – except for the fact that they are complete and perfectly preserved, with no parts missing. They simply appear “new”, a feature that has never been observed in an excavated figurine of unquestionable origins.<sup>61</sup> The Nigerian artist who manufactured the figurines stated that he uses a mixture of different clays from the area around the village of Nok. He mixes this with crushed terracotta pieces to give the



Fig. 66. Artist Audu Washi with the newly made, still unfired, forgery of a Nok terracotta.

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<sup>61</sup> This appearance is another reason why many of the Nok sculptures in museums and private collections should be viewed sceptically: they are, in most cases, complete or nearly complete and seem generally perfectly preserved. Such a state is only very rarely observed in archaeologically excavated material.



figurines the correct “shine”. He could not name the exact origin of the clays, nor whether they were processed in some way, as he himself bought them from somebody else who in turn does not speak about the clay sources. But the artist insisted that this was the “correct” clay to manufacture a terracotta (BREUNIG & AMEJE 2006: 93).

Macroscopically he was right, but the materials analysis reveals a different picture. One of the forgeries broke on its way to Germany, and the chance was taken to prepare a thin section from one of the pieces. In addition, all six statues were measured with XRF and compared to the original figurines. Thin section analysis revealed that the materials used were completely different: the modern temper contained chlorite-biotite intergrowth never observed in the archaeological material. Furthermore, the whole texture of the matrix is completely different – suggesting that the clay used was not the same as the one used for the Nok terracotta (Fig. 67).

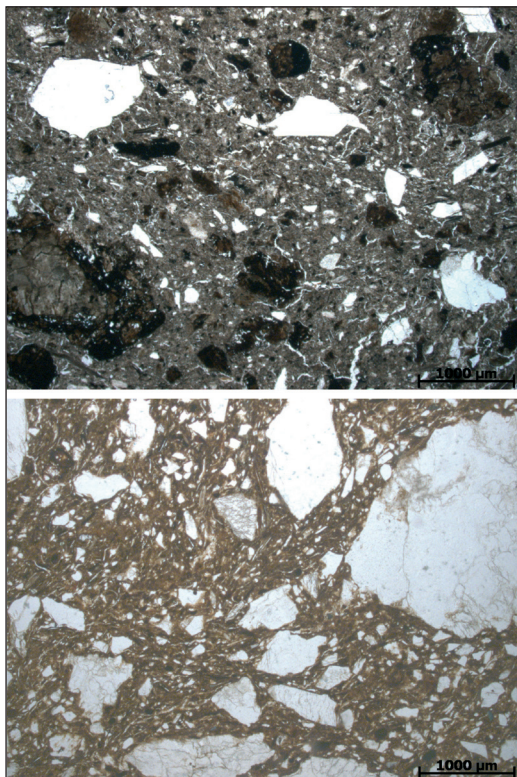


Fig. 67. Thin section of a forgery (top) in comparison to an original Nok terracotta (bottom). The dark granules in the forgery’s matrix are biotite-chlorite intergrowth, absent in Nok material.



This was confirmed by XRF analysis: the six forgeries show a unified chemical composition of the trace elements that clearly differs from the one seen in the excavated material (Fig. 68). Thus, as perfect as the forgeries may look and as much effort goes into making them look “real”, the material composition clearly unmask them as fakes. As only figurines from one artist were analysed, it cannot be ruled out that other forgeries from other artists are made with materials that more closely resemble those of original Nok terracotta.

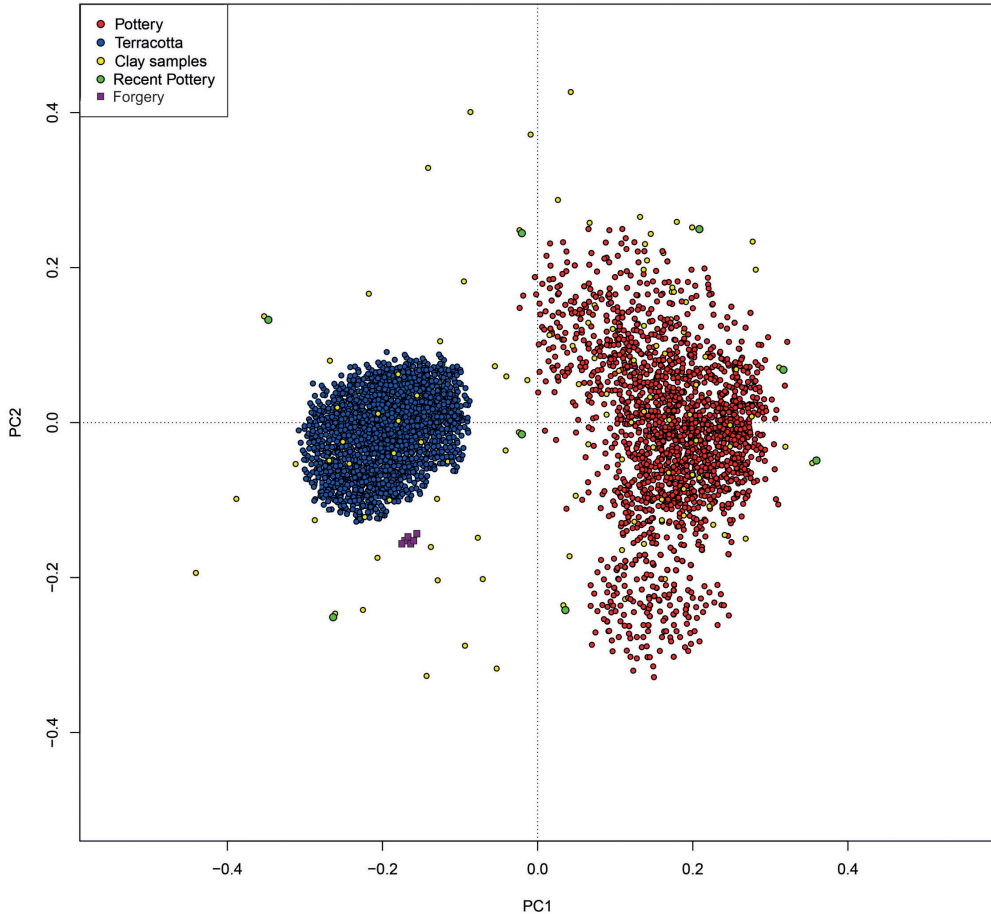


Fig. 68. PCA distance biplot of the pottery and terracotta of 43 sites (axes 1 and 2), the recent clay samples, recent pottery, and the six forgeries – trace elements only.

Materials analysis, however, offers the possibility to identify forgeries that may have found their way into private collections or museums.<sup>62</sup>

## 7.4 Summary

Statistical PCA analysis of the chemical composition of Nok terracotta figurines and potsherds has revealed a distinct chemical difference between the two archaeological materials: primarily with regard to the differing proportions of trace elements. Furthermore, a comparison with present-day clay samples shows that at least some clay sources that are still in use in Nigeria today have a chemical composition corresponding exactly to that of the terracotta sculptures. An even larger group of recent clay samples resembles the domestic pottery from Nok and Post-Nok times. The comparison of the geochemical composition of the archaeological material to that of the six modern forgeries demonstrates that the raw materials used nowadays by (at least this specific) forger may produce macroscopically perfect figurines, but that the material composition clearly exposes their fictitiousness.

Possible evidence concerning the localisation of the figurine workshops and implications for the organisation of terracotta manufacturing are discussed in the following chapter.

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<sup>62</sup> Work on this topic was not intensified any further as the focus of the Frankfurt project and this thesis clearly lies on the excavated Nok material from unquestionably authentic contexts. In future, however, this authentication method may be taken up again, especially with regard to XRF analysis.

## 8 Central or Local Production? Evidence of the Scientific Materials Analysis

Terracotta sculptures not only follow a stylistic norm, their uniformity can also be traced in their material composition: as shown in Chapters 6 and 7 their consistent style is mirrored in the chemical fingerprint of the clay used for their manufacture. Especially the proportions of trace elements separate the Nok terracotta figures clearly from contemporaneous and later domestic pottery. How is such a picture created? Were different clay sources used in the production of terracotta and pottery? Were terracotta figures made in one or more production centres while pottery was made on a village-level? Or did differences exist in the processing of the clay? And how can present-day clay samples help answer the question of the figurines' place of manufacture?

There are three factors that may be responsible for the distinct chemical composition of the terracotta figurines in comparison with the pottery: 1) the **use of special clay sources** for the figurines; 2) the **processing of the clay**; and/or 3) **random or systematic measurement errors** – for example because of sample selection or external influences. The present-day clay samples may offer valuable arguments for the acceptance or rejection of these factors; they are the only reference when trying to locate and distinguish the clay sources used in Nok times. First, the geological origin of the collected modern samples will be presented and comparisons will be made with analysed Nok samples. Then, it will be shown that they support the first two factors while the third one can be ruled out. Based on the evidence collected, the most likely scenario is the use of chemically similar clay sources for the terracotta production that not necessarily had to be concentrated in centres but could have occurred in a more local context. The question of the involvement of specialists and of the organisation of the terracotta production is the subject of Chapter 9.

### 8.1 The Geological Origin of the Modern Raw Clay Sources

Today, both primary and secondary clays are used in pottery production; it is likely that the same was the case in the time of the Nok Culture. Knowing from which geological context the raw clay samples originated may therefore help determine not only the processing method that was applied to the raw clay but also help finding similarities between clay sources used today and in Nok times. The distribution of the 163 recorded present-day clay sources on a geological map (Fig. 69) shows that the geology of the sampled area is not very diverse.

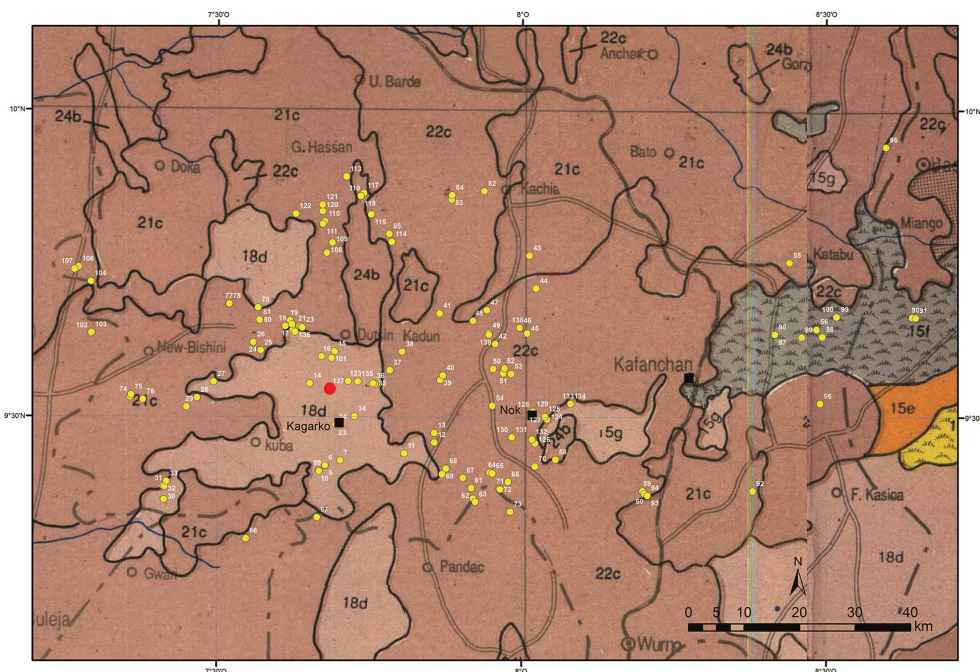


Fig. 69. Geological map with the distribution of the recorded present-day clay samples. Red dot: research station of the Frankfurt project. Geological units 15g, 18d, 21c, 22c, 24b: undifferentiated Basement Complex; 15e: Nupe Sandstones; 15f: Newer Basalts over Basement Complex; 18c: Colluvial deposit over granitic material. Source of geological map: Soil Survey Division, Federal Department of Agricultural Land Resources (FDALR), Kaduna, Nigeria, 1990.

The area mainly consists of rocks belonging to the undifferentiated Basement Complex (15g, 18d, 21c, 22c, and 24b in the map) with small areas of Nupe sandstones (15e) and Newer Basalts over Basement Complex (15f) as well as colluvial deposits over granitic material (18c) in the east. The raw clay sources scatter across the geological units, except for 15e and 18c. Four large pottery centres in the area today lie in different geological areas, suggesting that all clays are equally suitable for pottery production (Taffa: samples 30-32, geology: 18d & 21c; Kachechere: sample 55, geology: 21c; Tachira: sample 89, geology: 15f; Kachia: sample 82, geology: 22c). But the clay sources used for small-scale production on a household level likewise scatter across geological units, not concentrating in any particular pattern.

As the geochemical analysis in Chapter 7 has shown, thirteen raw clay samples have the same chemical composition as the clay used for the figurines.



These are samples no. 10, 24, 35, 44, 48, 70, 76, 79, 86, 96, 107, 111, and 116.<sup>63</sup> Their distribution on the geological map (Fig. 70) shows that they are dispersed across the whole investigated area and that they all lie within the geological formations of the undifferentiated Basement Complex (numbers 15g, 18d, 21c, 22c, and 24b) – no conformity was found with the other geological units (15e: Nupe sandstones; 15f: Newer Basalts over Basement Complex; 18c: colluvial deposits over granitic material). The 111 raw clays that feature the same chemical composition as the domestic pottery analysed by XRF are just as widely distributed over the whole area and present in all geological units, both those with and those without terracotta-like clay sources.

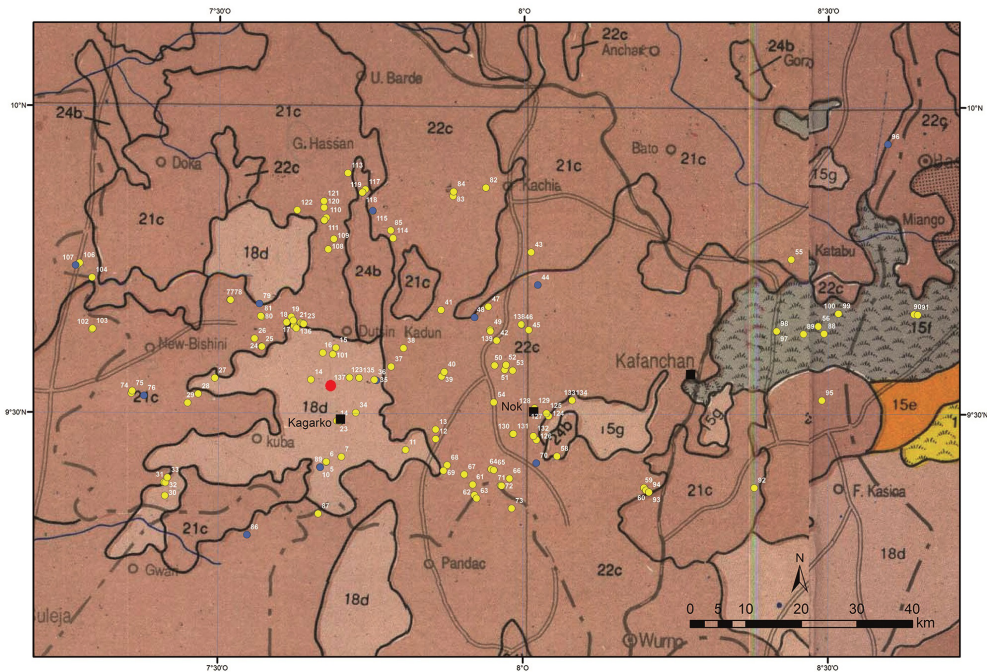


Fig. 70. Geological map with the distribution of recorded present-day clay samples. Blue dots: clay samples that show the same chemical composition as the terracotta figurines. Red dot: research station of the Frankfurt project. Geological units 15g, 18d, 21c, 22c, 24b: undifferentiated Basement Complex; 15e: Nupe Sandstones; 15f: Newer Basalts over Basement Complex; 18c: Colluvial deposit over granitic material. Source of geological map: Soil Survey Division, Federal Department of Agricultural Land Resources (FDALR), Kaduna, Nigeria, 1990.

<sup>63</sup> The clay samples that overlap with the analysed pottery will not be listed here, as the main focus of the analysis is laid on the figurines. The clays potentially used for the pottery will be discussed as a whole without referring to different time periods or sites. A detailed analysis with reference to the potsherd is beyond the scope of this work but will be conducted in future research (see Chapter 10).

The rarity, age, and low resolution of geological maps of central Nigeria, however, make it difficult to determine details and differences. Here, observations made in the field while walking to the clay sources are often much more helpful in determining the nature of the clays that were used during Nok times – all modern clay samples that have the same chemical composition as the figurines were collected in or near riverbeds, therefore deriving from secondary contexts. Concerning the pottery, mostly secondary clays are used, but also exceptionally primary clays were exploited. It seems as if during the time of the Nok Culture (and during later times, at least as far as can be determined from the relatively small number of Post-Nok samples) clays from primary contexts were used only in exceptional cases. Admittedly, secondary clays are more widely distributed in the sampled area and easier to find and exploit, so that this result may not be very surprising; it becomes important, however, when considering the factors that may have influenced the differences in chemical composition of terracotta and pottery: either the use of different clay sources (Chapter 8.2) or different procedures in clay processing (Chapter 8.3).

Looking at the spatial spread of those clay samples that are geologically similar and show the same chemical composition as some of the analysed terracotta samples makes one thing quite clear: The idea of one single “special terracotta clay source” from which the clay for all terracotta figures came, in a distinct region of the research area or a distinct geological unit, can be discarded.

## **8.2 The Use of Special Clay Sources in Manufacturing the Figurines**

As seen above, the clay sources that have the same chemical composition as the terracotta are distributed over the whole sampled area. This makes the use of one single clay source and a resulting centralised production of the figurines unlikely at first glance. In addition, a single special clay source that was used for the manufacture of thousands if not hundreds of thousands of terracotta sculptures for half a millennium of figurine production (BREUNIG 2014c: 272) would have to have been unimaginably large. Even assuming not one but several centres of terracotta production, the resulting clay pits would have had dimensions readily visible on satellite pictures – always keeping in mind that these clay pits would have had to have the same chemical composition which, once again, seems implausible. Furthermore, the thin section analysis has shown that both terracotta and pottery were tempered with local raw materials (Chapter 4). It is highly unlikely and against many worldwide ethnographical records (*e.g.*, ARNOLD 1989) that the specialists

who were supposedly responsible for manufacturing the figurines travelled – maybe over several hundreds of kilometres (considering the Nok Culture distribution area) – to a special source, collected the raw clay, and then returned to their villages, where they tempered the clay with local materials to make the figurines.

Thus, the questions are: How is it possible that obviously special clays with very similar chemical compositions, which originated from the weathering of bedrock and the transportation through the landscape prior to deposition, were used for half a millennium without some centralised clay source (or several centralised clay sources)? And how can such special clays be found unfailingly by the people who manufactured the Nok terracotta sculptures without having the possibility to test their chemical composition?

Nearly all clays that were used for the archaeological material derive from a fluvial, secondary context – if considering the similarities with the modern clay samples. Thus, a closer look at the river systems in the area and the fluvial clay sedimentation processes may help answer these questions. The whole sampled area is very well drained, with numerous larger and smaller rivers that mostly dry up between November and March. Nearly all rivers rise on the Jos Plateau, and most of the area drains to the Niger River (via the Kaduna and Gurara Rivers); only small areas in the south are drained by Benue River tributaries (AKINTOLA 1982: 12; NELSON ET AL. 1972: 29; WALL 1979: 13). The smaller rivers tend to meander in large river loops, often with a riverbed that is frequently redirected – especially with the onset of heavy rainfall during the rainy season which causes a fast rise of the water levels during the peak rain months of late August and September (WALL 1979: 10). This cuts off old meanders, which then dry out slowly, and leads to partial flooding of a wide area on both sides of the riverbed. The flooding is so severe that it can reach areas of several square kilometres in extreme cases (REINECK & SINGH 1975: 231). In the floodplains, the current velocity is relatively low, which results in a high sedimentation rate for clay as these fine particles sink to the ground only slowly (NANSON & CROKE 1992: 460). Large quantities of clay can also be found where the water is cut off from the current river system: in old meanders or in sinks/small ponds that are filled with water by the rivers during the rainy season and slowly dry out afterwards (Fig. 71; ENGELHARDT ET AL. 1973: 142; GUMNIOR 2005: 169).

Thus rivers – and therefore clay – are abundant in the entire research area, which corresponds to the observations when driving or walking through the landscape and during the work with the local potters. The clay sources can reach a considerably size – although of course not a size that would be sufficient for all Nok terracotta figures as a whole. The fact that nearly all rivers rise on the Jos Plateau and are part of more or less the same connected





Fig. 71. Dried-out sink where clay has accumulated (clay sample 135). Photo taken during the dry season 2009.

drainage system in an area with a quite undifferentiated geology (Chapter 3) may explain the origins of the clays used for the figurines. Because of the similar geology, the chemical composition of the clays is not very diverse compared to other regions where more different rock types exist. As the XRF analysis has shown, secondary (fluvial) clays were the only ones used for Nok terracottas. These clays were sedimented by river systems flowing through this geologically very uniform area. The chemical composition of the clays transported and deposited during flooding and sedimentation is therefore not likely to be very diverse (pers. comm. A. Röpke, January 2015).

It is thus theoretically possible that the specialists who were responsible for the manufacture of the figurines did not use one or a few clay sources but simply special clays of the same origin and similar deposition processes that could be found at different places along the river systems of the analysed area. This would allow for a local production of the terracotta sculptures but – in contrast to the pottery – with an exactly defined type of clay source. In this hypothesis, the temper seems of minor importance, which would support the

attested use of local non-plastic material. The clays used for the pottery may have derived from the same river systems, but from different sedimentation layers. The flooding events that lead to the sedimentation of clay occur annually, resulting in several subsequent, stratified clay layers along the river beds. Not all of these clay layers may be suitable for potting (today's potters regularly refuse to use some of them), but often more than one clay layer from a riverbed is exploited. Therefore, it is possible to use (chemically) different clay sources for pottery and terracotta that nevertheless derive from the same river system. The "special" clay for the figurines may have been defined according to its position in the stratigraphy (*e.g.* "always take the one under the yellow sand deposit" or "use the clay overlain by the blue-grey clay").

How such a non-centralised production of the terracotta figures by specialists with special clays might have been organised and what implications can be drawn from this result for the social organisation of the Nok Culture will be discussed on a more theoretical level in Chapter 9.

### **8.3 Processing of the Clay and its Effects on the Chemical Composition**

As stated in Chapter 4, raw clay is rarely used in its natural form for the manufacture of ceramic materials. It is nearly always processed in some way. This may include homogenisation in order to remove impurities, or mixing with other clays to reach the required material properties, or finally and crucially, the addition of any kind of temper (ARNOLD 1989: 21-22; BECK 2014: 247; RICE 1987: 52). The exact procedure varies according to the distinct intended purpose of the finished product and often also differs from culture to culture, each following its own pattern. Most of the time, reconstructing the preparation method of the raw clay is only partly possible, especially as regards the removal or addition of non-plastic material. In this case, the closest thing we have to an account of the clay processing methods (the acquisition of the raw clay, its preparation, and the manufacture of a vessel) used by the Nok Culture are the accounts which today's potters in the Frankfurt project's key research area provided in 2009 and 2011 (see Chapter 5.5). Unfortunately, none of the present-day clay samples that match the terracotta were those where a potter was still alive to provide any information about the way these clays were processed.

The comparison of recent clay samples and pottery with the archaeological material in the thin sections and the XRF analysis provides the possibility of drawing conclusions on the effect that clay processing has on the results of the chemical analysis. If any effect that might influence the geochemical analysis

is present, then it must have affected the analysed material in the same way. The direct comparison of the (unprocessed) clay samples with the processed recent pottery clearly shows that processing does not change the chemical composition of the clay, no matter whether the clay comes from a primary or secondary context and how it was processed. Thus, it can be clearly ruled out that the chemical differences between terracotta and pottery, which were revealed in XRF analysis, were created by a different handling of the raw materials.

In the following, only the 124 clay samples whose chemical compositions overlap with the archaeological material will be discussed. As described in Chapter 5.5, the processing of the clay varies according to its geological origin – and of course according to the intended purpose of the finished object. As great care was obviously taken in the manufacture of the figurines to comply with a stylistic norm and evidently in the use of distinct raw materials, a difference in processing between the clays used for the terracotta figures and those used for the pottery appears likely. From a technical point of view, at least, the latter demand a less precise composition of the raw materials because it is much smaller and less complex in form. The analysis of the tempering materials (Chapter 4) has shown that terracotta and pottery, albeit using the same non-plastic inclusions, do not stem from the same manufacturing processes. The exact composition of the temper, its size, and its angularity differ between the two archaeological materials. The possibility that, in addition to a stylistic norm, there were also regulations concerning the processing of the raw materials used for the figurines – and that this resulted in an alteration of the chemical composition of the clays in a way that led to the picture seen in the XRF analysis – must therefore be considered.

The precise processing steps conducted prior to the forming of a terracotta sculpture cannot be reliably determined. However, the thin section analysis of Nok material (Chapter 4) has shown that at least the temper (exclusively inorganic) is similar to present-day temper. Furthermore, at least some clay sources with the same chemical properties (and thus originating from the same geological context) as those utilised for the figurines are still in use. The present-day clay samples were taken directly from the raw material deposits and were not processed in any way. The only “processing” they were subject to prior to the XRF analysis in Germany was soaking with clear water, the forming of small bricks (Fig. 72), and the subsequent firing in an open pit fire to achieve comparability with the Nok material for the chemical analysis. Thus, no process which could affect their chemical composition was included (or excluded). The clays therefore show how untempered raw clays with naturally occurring non-plastic inclusions appear in thin sections and geochemical analysis. The recent (processed) pottery samples can then





Fig. 72. Unfired recent clay samples from central Nigeria.

allow a direct comparison between the chemical composition of the processed clay and the corresponding raw material, making it possible to determine whether the processing of the clays alters the chemical composition. The recent clays that feature the same or similar chemical composition as the archaeological material are nearly exclusively secondary clays which today are rarely additionally tempered and only contain the crushed minerals and rock fragments that naturally occur in them. As it remains unclear to what

extent the terracotta and the pottery were additionally tempered and which inclusions occurred naturally in the clay (Chapter 4) a comparison to the raw clays seems a reasonable first approach.

The unprocessed clay samples and their (processed) corresponding pottery show nearly the same chemical composition in the XRF analysis (Fig. 65 in Chapter 7). Considering that the present-day potters' alterations of the clay are not very extensive, we can conclude that processing does not generally change the chemical composition of the clay. Thus, processing, no matter how, is not responsible for the clear separation of the terracotta figurines from the pottery. This result is supported by the thin sections: the naturally occurring inclusions in the secondary clays that resemble the archaeological material are at least partly similar to the particles contained in the Nok material. The similarity includes the type of inclusion and the amount and shape of the grains: mainly angular particles of crushed granite and quartzite and the minerals quartz, feldspar, mica, and minor fractions of amphiboles or pyroxenes (Fig. 73).<sup>64</sup> In contrast, thin sections of the primary clay sources contain considerably less non-plastic inclusions, as they are added in form of temper (sand) later (Fig. 74).

The same could be observed in the thin sections of present-day pottery: the pieces manufactured with secondary clays resemble the archaeological material (Fig. 75), whereas the ones made with primary clays show distinct tempering with rounded quartz grains (sand) (Fig. 76). As a side effect, in the thin sections, the clay samples give an impression on the clay matrix and

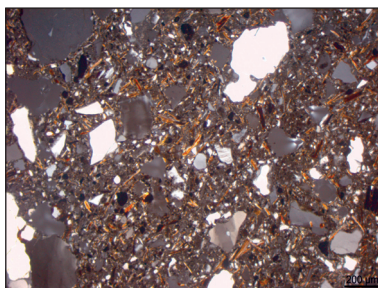


Fig. 73. Thin section of a secondary clay source (clay sample 124). Cross polarised light.

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<sup>64</sup> In the beginning, during the early thin section analysis (Chapter 4), the crushed granite and quartzite in the pottery and terracotta were considered deliberate inclusions by the potter. However, after the results of the most recent analysis and the observation of the clay processing procedures in modern Nigeria, it seems likely that these particles occurred naturally in the clay and that their coarse nature and angular shapes resulted from the homogenisation of the clay in a mortar or on grinding stones. This would leave only the grog (crushed potsherds or terracotta fragments) as definite deliberate additions by the potter.

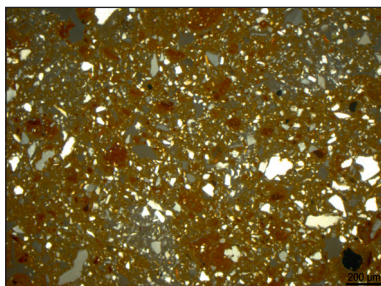


Fig. 74. Thin section of a primary clay source (clay sample 133). Cross polarised light.

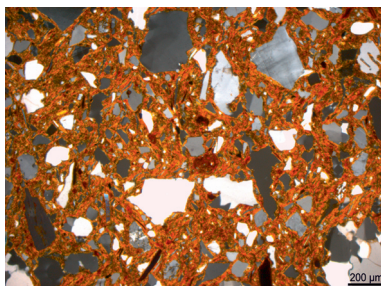


Fig. 75. Thin section of a recent pottery sample manufactured with secondary clay (sample number 59). Cross polarised light.

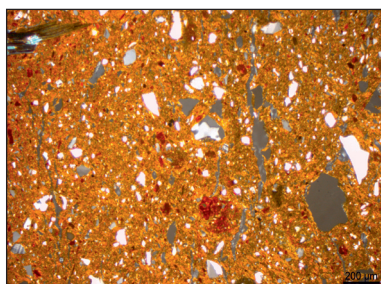


Fig. 76. Thin section of a recent pottery sample manufactured with primary clay (sample number 13). Cross polarised light.

its naturally occurring inclusions despite the melting of the clay minerals in the firing process. The small quartz particles in the matrix proved to yield valuable information for the distinction of pottery and terracotta. The clays



that have the same chemical composition as the figurines feature a similar texture of the matrix.

Overall, this comparison of modern unprocessed and processed material has shown that processing is not responsible for the differences seen in the chemical composition of Nok terracotta and pottery.

## 8.4 Random or Systematic Measurement Errors

Whenever scientific methods are used, no matter in which field of research, errors influencing the measurements have to be considered (SCHÖNWIESE 2000: 105). Two types of errors exist. Although it is highly unlikely that they were responsible for the chemical difference of pottery and terracotta, they will be briefly discussed here.

The first are **random errors** caused by unknown and unpredictable changes in the measurements, for example because of alterations of the experimental set-up or changes in environmental conditions. These cannot be influenced but are relatively easy to handle: they tend to be easily visible in the dataset because of obvious divergences in the measurement values, and can thus be removed manually. The amount of random errors can be estimated by looking at the precision of the measurements. It can be reduced by conducting several measurements of the same sample and using the median or mean value for analysis (VOGEL 1999: 5-7). In the study presented here, the experimental set-up was not changed during the measurements; the same device was used and the environmental conditions remained more or less the same. Additionally, all samples were measured four times, using the median in the analysis (Chapter 5.2). Furthermore, the precision of the mobile XRF-analyser used is very high and absolutely comparable to that of stationary laboratory equipment. Thus, while random error is definitely present in the samples, it is negligible.

**Systematic errors** are caused by the device itself (malfunction) or any kind of mistakes in the preparation or the selection of samples (SCHÖNWIESE 2000: 106). In this case, laboratory standards were measured at the beginning and the end of each measuring day to rule out device malfunction. By this procedure, the (relative) accuracy of the analytical results was ensured. The preparation of the samples prior to measurement consisted of washing with clear water and drying at room temperature (Chapter 5). Even if this procedure did change the chemical composition of ceramic material, it would have affected both the terracotta and the pottery alike. The same applies to any kind of leaching or incorporation of elements in the material during their storage in Nigerian soil. Influences which might have interacted with the material during transport from Nigeria to Germany (*e.g.*, X-ray machines at the airport; cosmic radiation



during the flight), or during the storage of the samples in plastic bags, may be excluded for the same reason.

An error caused by mistakes in sample selection is also improbable: a total sample size of more than 3000 fulfils all requirements for a statistically sufficient number of samples. The site-level analyses in Chapter 6, where the sample sizes were much smaller, reveal the same patterns, indicating that even here sample selection did not skew the results.

The only remaining possibility of a systematic error at a scale that could have influenced the measurement results is the limited extent of the considered area. When choosing the samples for the analysis, care was taken to consider a geographical region as large as possible. Due to the increasing political instability from 2012 onwards, the Frankfurt project was not able to expand the research area further beyond the key area of research. Initially, material from other regions was to be included in the analysis. This is definitely a shortcoming of the analysis presented here, making the results reliable only for the relatively small region, but not for the whole Nok Culture distribution area. Nevertheless, the samples stem from an area large enough to yield solid results.

Thus, random and systematic errors are surely present in the dataset, but care was taken to minimise them to a level that does not affect the inherent content of information in the material. Several factors cannot be excluded completely, but as they affect all material groups alike, they may have an absolute influence on the analysis while the relative results (for example when comparing the terracotta and the pottery) remain untouched.

## 8.5 Summary

Since random or systematic errors can be ruled out, the reason for the apparent differences between the pottery clay and terracotta clay must lie in true differences in the materials' composition. Today, primary and secondary clays are used to manufacture pottery; during Nok and Post-Nok times, however, mostly secondary clays deposited along the riverbeds or in old, dried-out meanders were used – according to the results of the geochemical analysis. It is impossible to say with any certainty how these clays were processed prior to the manufacture of the figurines, but thin section analysis suggests that processing methods are similar to those used today. XRF analysis of present-day untempered raw clay sources and the corresponding pottery shows that, no matter whether the clay came from a primary or secondary context, the different processing procedures do not have a significant influence on its

chemical composition. Thus, the Nok processing methods, whatever they may have been, had no influence on the picture seen in XRF analysis.

The only valid possibility, then, to explain the chemical difference between the figurines and the potsherds is the use of special clay sources. Present-day clay sources sharing their chemical composition with the terracotta are distributed throughout the entire sampled area. The use of one or even a few clay sources to manufacture the figurines is unlikely and technically not realisable – such large clay sources for the production of vast amounts of figurines do not exist. Even if they did, the resulting holes in the landscape would have reached gigantic dimensions and should still be visible today. What explanation remains (and what is geologically possible) is not the use of one (or a few) raw material deposits in one special place, but the use of a special clay that could be found at different places in a large area along the wide-ranging river systems. This clay would have originated on the Jos Plateau and had being transported by severe flooding and sedimentation events, which would explain its very homogenous chemical composition. The temper used, to the contrary, seems less important and may have been chosen simply for its technical properties. The pottery, however, is manufactured using several different clay sources, which fits well with the assumption of a local pottery production on a household level.

The results of the materials analysis, then, lead to a new hypothesis of how the terracotta production was conducted and organised – results that so far have not been possible with the analysis of stylistic elements: Because of the very stringent stylistic norm – a norm maintained for half a millennium in a large cultural area – the figurines were surely manufactured by specialists. These artists, however, seem not to have worked in large, centralised workshops and later distributed the figurines in the whole area of the Nok Culture. Instead, they travelled around and produced the terracotta sculptures locally using a special clay (however it may have been defined and constituted) available in several places along the river systems in the Nok Culture's expansion area. Thus, a deliberate stringent stylistic rule was combined with a material norm that happens to be reflected in the chemical composition of the clay. How such a special but widely distributed clay deposit may have been defined; and how it could have been found repeatedly over time; and how a non-local, controlled production by (full-time or part-time?) specialists may have been organised will be discussed in Chapter 9, with the aim of illuminating the social organisation of the Nok Culture on a more theoretical level.

# **D**

## **Theoretical Considerations**



## **9 Complexity, Specialisation, Value? Theoretical Approaches and their Application for the Nok Culture**

As the materials analysis presented in this thesis has shown, the Nok terracotta figures were most likely made on a rather localised level (rather than in one or a few production centres) – based on the chemical similarities in the clay used. This clay would have come from special clay deposits in river sediments that were wide spread in the Nok Culture expansion area and that were not used in pottery production. This chapter will provide some theoretical approaches for how these material findings can be applied to get a deeper insight into the structure of Nok society, into how it created and circulated value.

The findings would support the model of a horizontally organised society with central authority resting in the ritual sphere and relying on ritual power, in which the terracotta figures link the people living in dispersed settlements as a symbol of identity. Production was in the hand of (male or female) specialists that may have travelled from village to village, producing figurines on demand for the local community according to the stylistic and material norms established by the society. The terracotta figures' main value would have been in their symbolic and social meaning – integrating the wide-spread villages or homesteads, influencing and regulating social behaviour, and holding a society together for more than half a millennium by providing an integral internal structure.

### **9.1 Complexity: Vertical versus Horizontal Hierarchies and the Concept of Ritual Power**

Initially, the Frankfurt project's aim was to investigate whether the prehistoric developments discovered in the Nigerian Chad Basin in the 1990s (BREUNIG 2005) were traceable in other parts of Nigeria as well (Chapter 2). In northern Nigeria, the Gajiganna Culture of the second and first millennium BCE transitioned from hunting and gathering to sedentary farming communities, followed by a significant reduction in settlement activities and a return to increased mobility in the early first millennium BCE. Around the middle of the first millennium BCE, the settlement pattern again shifted towards large, often fortified settlements. Large-scale food storage indicates a successful agricultural system, and the inhabitants now also specialised in craftsmanship. Additionally, the first traces of iron technology occurred (BREUNIG 2009a).

Such comprehensive changes must surely have affected the organisation of the society.

The Nok Culture showed similar features: craft specialisation in the form of terracotta production and early dates for iron technology around the middle of the first millennium BCE. It was thus the perfect candidate to test whether the findings in the Chad Basin were exceptional or part of a widespread phenomenon. If they were part of a pattern: did these developments result in the emergence of complex societies, which appeared in northern and southern Nigeria from the late first millennium CE onwards (CONNAH 1981: 220-221; 1987: 142-144; SHAW 1978: 98-99, 188-189)? The title of the Frankfurt project was designed to reflect this question: *“Development of complex societies in sub-Saharan Africa: The Nigerian Nok Culture”*. Early results indicated that the aim of illuminating these questions was not unrealistic. A high settlement density and the supposed simultaneous emergence of the highly artistic terracotta figurines and iron production suggested comprehensive social changes during the time of the Nok Culture (BREUNIG 2009b). These changes could have led to the emergence of early complex societies in Nigeria in the first millennium BCE. This picture, however, has changed during the almost ten years of research.

Traditionally, social complexity is closely related to social stratification and linked with a number of other attributes as well: monumental art and architecture, rich grave goods, an elaborate ceremonial and political life, increasing populations, agricultural intensification, evidence of control over vast territories (and/or people and wealth), and long-distance exchanges. Generally, complex societies are hierarchically organised, with a centralised authority based on coercion and control (McINTOSH 1999: 4).

Of these attributes, the Nok Culture – considered generously – features only two: elaborate (though not truly monumental) pieces of art and – in connection with the figurines – surely some sort of ceremonial (ritual) life (BREUNIG 2014c). Any kind of architecture is missing, except for the stone circle of Puntun Dutse (RUPP 2014b) – but these remains of a house are far from being monumental.<sup>65</sup> In contrast, the settlement pattern of the Nok Culture tends to feature smaller settlements without any apparent internal structure, widely distributed in the landscape and sometimes at considerable distance to each other (RUPP 2014b). Likewise, clear indications of grave sites are missing. Only at four sites (Ido, Janruwa A, Kurmin Uwa 2B and Pangwari)

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<sup>65</sup> The massive “walls” cut from the underlying granite and a second area walled with granite slabs of “megalithic” dimension at the site of Kochio (RUPP, AMEJE & BREUNIG 2005: 287) were a misinterpretation of geological formations. No hints of any form of fortification or megalithic buildings have so far been found at Nok sites (BREUNIG 2014a: 107-108).



can stone settings in combination with one or two complete pots (in Ido and Janruwa A also with an arrangement of stone beads) be interpreted as graves. This interpretation is strongly supported by geochemical soil analysis. In all cases, bones are missing due to the acidic tropical soil (NAGEL 2014). But even without human remains, elite burials should be clearly recognisable because of elaborate and valuable grave goods.

Concerning subsistence strategies, the Nok Culture also does not provide much evidence of any agricultural intensification. So far, pearl millet (*Pennisetum glaucum*) and cowpea (*Vigna unguiculata*) seem to have been the only cultivated crops. These were apparently supplemented by the collection of wild plants and fruits. Only in the Post-Nok period was Fonio (*Digitaria exilis*) cultivated – in fact, it became the dominant crop. The oil palm (*Elais*) is likewise present in archaeobotanical material only after the end of the Nok Culture (KAHLHEBER 2010; KAHLHEBER ET AL. 2009; HÖHN & NEUMANN 2014; HÖHN & NEUMANN 2016). Evidence of long-distance trade or of control over people or wealth as well as any signs of hierarchical organisation are missing completely. Thus, the term “complex society” does not seem appropriately applied to the Nok Culture.

However, a closer look at Susan McIntosh’s archaeological record of sub-Saharan Africa (MCINTOSH 1999) reveals that this dilemma is not limited to the Nok Culture, but can be found nearly everywhere in sub-Saharan Africa. In fact, only few precolonial sites show the features mentioned above. She argues that the evolutionary notions of the 19<sup>th</sup> century – *i.e.*, the theory that complexity is created by the emergence of political hierarchies with the attributes mentioned above – cannot be transferred to Africa south of the Sahara. Instead, many African societies may be found in which

*...central authority, often of a ritual nature, is paired with a power structure that is diffuse, segmentary, and heterarchical, as well as societies in which considerable complexity is achieved through horizontal differentiation and consensus-based decision making. The distribution of power among several corporate entities (e.g. lineages, secret societies, cults, age grades) can be regarded as a strategy that has successfully resisted in a variety of ways the consolidation of power by individuals. (MCINTOSH 1999: 4)*

Thus, instead of a vertically organised society with clear hierarchies and a centralised authority, administrative machinery and judicial institutions, Africa provides several examples of horizontally organised, non-centralised societies where the structure of order was provided by kinship with horizontal, cross-cutting relationships (secret societies, cult groups, *etc.*) that were important in creating complex political structures as described by FORTES and EVANS-PRITCHARD (1940). In addition, two other, more complex forms of organisation

of stateless societies were described by HORTON (1971): firstly the dispersed, territorially defined community consisting of a local confederation of lineages of mixed origin, integrated by cult organisations; and secondly large, compact villages in which a substantial population aggregation is horizontally integrated by a variety of associations, cults, and secret societies. In both cases, considerable complexity may emerge in the absence of any centralised authority (McINTOSH 1999: 9).

In these systems, ritual plays an important role in increasing complexity. Ritual is used to reduce social stress and has – through a combination of fear, supernatural sanctions, and fines that accompany ritual – the potential to secure compliant behaviour and resolve disputes. Furthermore, ritual ceremonies can serve as decision-making and integrating functions in large groups. Thus, cult associations and secret societies can be seen as ritual corporations that own property and control socially recognised resources. Through their control of ritual technology and ritual knowledge they have potent political roles and may mobilise labour (McINTOSH 1999: 12).

In conjunction with the alternative model of horizontally rather than vertically organised societies in sub-Saharan Africa, McIntosh also advocates the use of a different conception of “power” – not in the Western terms of individualistic, rational, and secular, but rather as a model of power relations that involve categories of age, gender, descent, and association, often simultaneously. Such power systems are fuelled more by social than by material wealth. As noted by HERBERT (1993: 237), power requires an understanding of the supernatural forces that affect outcomes, and sufficient knowledge to influence them – but that such a cosmology cannot be created by force or coercion. Indeed, ritual power and appropriate specialist knowledge are key elements of the capacity for effective action in many African societies. This means the existence of knowledge-based (rather than wealth-based) political economies, and power strategies that involve collective (rather than individual) action (McINTOSH 1999: 17).

Therefore, in Africa, horizontal, flexible, often multiply overlapping hierarchies with alternative modes of complex organisation should be considered a counterbalance to “classical” vertical hierarchies. As McIntosh discusses, several examples show that societies evidently found diverse ways to achieve complexity – especially ones that rely on acquiring ritual authority rather than economic or political power. The central authority thus often rested in the ritual sphere or was even divided among corporate identities. McIntosh makes a point of emphasising heterarchy rather than hierarchy. Unfortunately, such diverse, horizontally organised power structures are more difficult for archaeologists to recognise than vertically controlled hierarchies, which are more conspicuous in the archaeological record (McINTOSH 1999: 22-23).

This makes it difficult to apply the theoretical discussion to the Nok Culture. Finds and features reveal – as pointed out by McIntosh – no clear attributes that enable further examination of the exact way the society was organised – and whether it was complex in any sense at all. The terracotta figurines, however, fit very well into the model of a horizontally organised society with central authority resting in the ritual sphere and probably relying on ritual power. Their stringent style and standardised repertoire of gestures and topics, as well as the fact that they were always destroyed and at least in some cases deliberately deposited, allows no other conclusion (BREUNIG 2014c; RUPP 2010; RUPP 2014a). To the people living in dispersed settlements, they may have served as a collective item and a symbol of identity suitable for uniting the members of the society through a ritual belief system – such systems do have the potential to provide supralocal organisation.

## 9.2 Craft Specialisation without Centralisation?

The fact that the Nok Culture was clearly not centralised in any way poses another question: how was the production of the figurines organised? Since they were most likely ritual objects, their manufacture would surely have been the task of specialists. Otherwise the strict adherence to stylistic norms would not have been possible. Based on the scientific materials analysis presented in Section C, the figurines were not produced centrally. Such centres do not appear to exist, and are also not necessary in a model of a horizontally organised society. Instead, production seems to have occurred on a local level in different villages, using a special clay found along the river systems. The definition of this special clay source could have been achieved by the same ritual regulation that was responsible for the stylistic features. This would also provide an explanation why the pottery – likewise produced locally in the villages – shows a completely distinct chemical composition. The special “terracotta clay source” was clearly not used for domestic pots, though it must have been readily available in the area.

The emergence of craft specialisation is discussed just as extensively as the definition of social complexity – and both are often related to each other. Concerning the development of specialised ceramic production, there are some models which – despite the fact that they all concern domestic pottery and not figurines – can be used to evolve a theory of the emergence and organisation of the Nok terracotta production. A common model for explaining the “*evolution of craft*” (ARNOLD 1989: 225), described by many authors for societies from all over the world, is the following: pottery production is at first exclusively located on the household level and, via the stages ‘household industry on a part-time level’ and ‘full-time workshop industry’, develops into a large-scale

industry. The exact number of stages and their names and descriptions strongly depend on the author (see for example ARNOLD 1989; PEACOCK 1981; VAN DER LEEUW 1976). All these accounts, however, emphasise that such development is caused by external influences like population pressure, resulting in a decrease of the per capita returns from subsistence strategies (mostly agriculture). If this is the case, pottery production is used as a supplementary income and is – with increasing pressure – further intensified until the subsistence strategy is fully shifted to specialised, full-time pottery production.

Transferring this model to the Nok Culture, the manufacture of the domestic pottery was conducted on the household, part-time level, according to the results of the materials analysis. In contrast, the terracotta figures – because they were presumably the work of specialists – would fall into the categories of full-time workshop or even large-scale industry. This does not match the results of the geochemical investigations, nor the finding that no centralisation existed during the Nok Culture. Instead, the figurines were produced locally, with local (though special) raw materials. The ethnographic literature from all over the world provides some examples how such a controlled but not centralised production by specialists can be realised without the existence of large-scale workshops or industries. STARK & HEIDKE (1998), for example, describe the specialisation of the pottery craft in the early Classic Period (around 1150-1350 CE) in Arizona. In the Tonto Basin, different types of ceramics existed, most of them produced locally. Some wares, however, were manufactured by specialists, with every district or settlement having “its own”, but produced only for local use (STARK & HEIDKE 1998: 497).

But African examples also exist: LAVIOLETTE (2000) describes different crafts (among them potters) and their organisation in Jenné, Mali, in the early 1980s. Aside from pottery production on a part-time, household level, the potters expanded their markets in three ways:

- 1) they produced a surplus of pots throughout the year and, when water levels of the rivers rose during the rainy season, headed for the hinterland in small boats, selling their pots in the villages along the rivers;
- 2) the potters themselves moved to the hinterland, without a stock of ready-made pots, only equipped with their tools, staying in the villages for about a week and producing pottery on demand with locally available raw materials;
- 3) the potters (exclusively women) moved around, accompanying their husbands, who are mostly blacksmiths. While the men were producing iron objects on demand, the women offered their skill in pottery-making (LAVIOLETTE 2000: 67-68).

Some of these models may be transferred to the production modes of the figurines. The possibility presented by Stark and Heidke would involve full-time specialists with permanent workshops. The Nok Culture finds and features excavated thus far do not allow a reconstruction of any kind of workshop – but as construction elements (except for the single house in Puntun Dutse and frequent finds of burnt daub (RUPP 2014b: 149)) are generally missing from the archaeological record in any case, the existence of such workshops is possible. The compliance with stylistic and material norms is also conceivable via transmission or learning networks based on ritual regulations. Of the modes of production found in Jenné, the second – and/or the third, if the figurines were connected with iron metallurgy – would match the information gleaned about the manufacture of the Nok terracotta sculptures. Such part-time (or even full-time) travelling specialists, staying for some time in each of the different villages, producing with local raw materials, would be completely invisible in the archaeological record, but make the compliance of any kind of stylistic or material norms easy to realise – regardless of whether they were of a ritual character or not.

The two examples of craft specialisation presented here also show that no matter how specialised the manufacturing of pottery is, it is exclusively in the hands of women. This is contrary to theories of the ‘evolution of craft’ presented above. A typical feature of these theories – emphasised by numerous authors – is the rising engagement of males in the manufacturing process, up to the complete takeover in the later stages, whereas in earlier stages pottery production is exclusively in the hands of the women. Thus, pottery manufacture on a household, part-time level is mostly female, whereas in societies where potters are predominantly male, production occurs on a non-household, specialised level (ORTEN ET AL. 1993; RICE 1984; SKIBO & SCHIFFER 1995). ARNOLD (1989) argues that the reason for this supposedly worldwide pattern is that pottery making is compatible with other household activities as it requires dry and warm weather like many subsistence activities that take men away from home (ARNOLD 1989: 100-103).

In Africa, as nearly everywhere in the world, pottery is traditionally the domain of women (BERNS 1993; DROST 1968; GOSSELAIN 1999; 2008; INSOLL 2011; STÖSSEL 1984). But as soon as it becomes “high” art (in comparison to the “low” craft of making a pot), it is generally assumed that it becomes the domain of men. To make it short: women make domestic pots, whereas men make art (such as figurines) and everything related to the ritual sphere. BERNS (1993) gives some examples of Nigerian archaeological material for this assumption; WILLET (1967: 70-78) emphasises that the figurative art of Ife is built up in the same way as the pots, made by women. But because the interior walls of the sculptures are not finished smoothly, as are domestic pots, they

were probably the work of men. W. Fagg, meanwhile, describes the making of a terracotta figure by a female potter as a “*very rare instance of pottery sculpture produced by a woman*” (W. FAGG 1963: Fig. 133). In the book “*African Crafts and Craftsmen*”, Gardi even says that although pottery is reserved to women in Africa, they

*seem to have little talent as creative artists. In comparison with the accomplishments of their men [...] the shy decorations that one finds on the earthenware are rather ungainly* (GARDI 1969: 112).

In contrast, BERNS (1993) quotes, again for Nigeria, several examples where women explicitly are the ones who manufacture not only a wide range of vessels for domestic but also for ritual purposes, as well as figurative art used in ceremonial context. Four of the eight examples discussed by Berns will be given here. According to THOMPSON (1969: 151), women were responsible for producing ceramic vessels with figurative lids for cults among the Yoruba. In Benin, figurative ritual pots were made primarily by the wives of brasscasters (BEN-AMOS 1986). North-west of the Nok Culture area, grave sculptures are produced by Dakarkari women. These tomb ceramics may be modelled in the form of humans or animals (BASSING 1973; Y. USMAN 2014). And finally, the anthropomorphic and zoomorphic vessels made by Longuda men and women of the lower Gongola Valley in north-eastern Nigeria are used in various healing cults (BERNS 1993: 136-137).

This short overview of theories on the emergence and organisation of craft specialisation is surely not complete, but concerning the production of the Nok terracotta figures, three conclusions can nevertheless be drawn:

- 1) Craft specialisation does not require any form of centralisation. The potters may have travelled around the villages and produced the figurines locally on demand; alternatively, each larger area had “its own” specialist producing on a regional level.
- 2) Specialists do not necessarily have to work full-time. Pottery is manufactured mainly in the dry season, when certain subsistence tasks in agriculture do not need to be performed. Thus, part-time manufacturing of figurines is possible and does not conflict with the figurines’ status as ritual objects (BERNS 1993).
- 3) Although such is commonly assumed in the literature, the specialists that made terracotta figurines were not necessarily male. Instead, making pottery is traditionally a female domain and ethnographic studies show that both sexes may have been involved in the manufacturing process.



### 9.3 The Value of Art

The title of this dissertation project is challenging in two ways: firstly, its use of the term “art” – which follows from the focus on the terracotta figurines – refers to the notion the statues commonly have as sought-after objects in private collections and museums. They were, however, certainly not considered art during the Nok Culture, as the concept of “art” as such simply does not exist in prehistoric Africa (BREUNIG 2014c). Objects – not only clay objects – that are today considered “art” were formerly an integral component of ritual life. Additionally challenging, and deriving from the first point, is its use of the term “value”. This term does not of course refer to the aesthetic and economic value of the figurines today – as demonstrated by their price on the international art market and measureable in the amount of money one has to pay for a figurine. Instead, it refers to the importance and meaning the terracotta sculptures had as part of the material culture of Nok society.

In general, material culture is a forming and communicative medium, closely involved in social practice. As such, it can be used for transforming, storing, or preserving social information. It thus forms a symbolic medium for social practice and is always a social product. In this way, it can help the archaeologist discover the meaning of the past and reveal underlying principles (TILLEY 1989: 189). Therefore, material culture may be assigned a wide variety of values and functions. According to BINFORD (1962) and SCHIFFER & SKIBO (1987) four main types of values exist that may be combined partly or all at once in one object:

- 1) **Aesthetic value:** this refers, as already noted, mainly to a contemporary view of certain objects. In addition, it is difficult to prove that artefacts were valued (solely or primarily) for their aesthetic qualities in former times.
- 2) **Utilitarian value** (also termed “technomic” or “techno-functions”): for ceramic materials, this value is reflected in the technological attributes of the pottery. It may be demonstrated by the manipulation of clay, temper, or surface treatment for utilitarian purposes.
- 3) **Social value** (also termed “socio-technic”): these are objects that have their primary function in the social system. They may connect individuals to each other into cohesive groups capable of effectively maintaining themselves.
- 4) **Symbolic value** (also termed “ideational” or “ideo-technic”): its primary function lies in the ideological component of the social system. Such items thus signify and symbolise the ideological foundations of the social system and further provide the symbolic background of the society.



Anthropomorphic figurines occur as part of material cultures the world over. They can be found on nearly all continents from Mesoamerica, Europe and the Near East, to India and Japan. Their possible interpretations and purposes are equally diverse and range from funerary objects, teaching aids, and religious objects, to signs of social or economic relations, art, toys and more (LEASURE 2011: 19, 62). Except for “art” – which is most often an interpretation that derives from an outside view, but not from the culture itself – these possible usages could be subsumed under social or symbolic value. It is noticeable that figurines exclusively arise, as far as can be determined, in sedentary (in Africa also pastoral) societies. The emergence of a sedentary lifestyle enables the accumulation of personal belongings – which in turn leads to the new concept that material objects can have a value that exceeds their sheer utilitarian (technomic) value (RENFREW 2008: 135-136). Such a fundamental restructuring of a society produces social stress that has to be resolved in some way. Material culture has this potential: it can express social conflicts and at the same time resolve them, often by combining the four values detailed above. It is further a means of communication between individuals and groups (HODDER 1986: 122-124).

It is not unusual for these values to be traceable in the material composition. Investigations by H. Lechtman on different craft technologies in the Andes have shown correlations between cosmological concerns and technological practice, leading to the concept of the cultural dimension of techniques. According to this view, crucial aspects of society are embodied in the techniques used: technological choices follow a social logic (LECHTMAN 1977) and consequently, technology cannot be divorced from its social and symbolic context (HODDER 1982).

Ceramics play a significant role in conceptualising materiality and technology. According to several observations (*e.g.*, HERBERT 1993: 207-209; GOSSELAIN & LIVINGSTONE SMITH 2005: 40), African potters believe in the inherent power of clay. Pots are frequently linked to human beings, with pottery techniques relating to transitory states, fertility, death, and so on. The making of pottery, for example, is associated with the human gestation – the pot as the foetus conceived (by mixing the female clay with the male water and temper) and born with the help of the potter. Consequently, the breaking of a vessel can be related to death (GOSSELAIN 2011: 247). But the whole manufacturing process involves control over the properties of the clay and the firing technique and thus offers a serious risk of failure. Pottery is therefore often intensely ritual, with a number of taboos and regulations to assure success and minimise risks. These regulations involve all steps of the manufacturing process, from clay procurement to the firing of the finished object. Examples of such taboos may be that the access to a clay source is

strictly forbidden except for some initiated persons, that menstruating women are forbidden to dig for clay or make pots, or that unfired vessels may not be counted or removed from the village.<sup>66</sup> Similar taboos and proscriptions may be observed in other crafts, especially iron-smelting or blacksmithing, the manufacture of stone tools, and even cooking (GOSSELAIN 2011: 248-249). The reconstruction of the manufacturing process can thus truly open a window for the understanding of the social and symbolic context and value of a society. The maker's attempt to situate oneself within the society and at the same time the society within the wider world has left material traces.

The value of the terracotta figures for the Nok Culture surely lies in their symbolic and social meaning. As ritual objects, they combine all of the described values, but with the social and symbolic value clearly dominating. They would certainly have influenced social behaviour and helped integrate the people living in dispersed settlements. They may have functioned as collective items and symbols of identity, holding together the society, minimising social stress, and providing some form and internal structure – features that are especially important in horizontally organised societies. This social meaning cannot only be traced in the strong similarities of the terracottas' stylistic features but also in the raw materials used, as these were not only selected for technological reasons but were another way of expressing the identity of the Nok people.

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<sup>66</sup> For a comprehensive account of taboos related to pottery manufacture see HERBERT 1993: 206-209.



# **E**

## **Summary and Outlook**



## 10 Terracotta and Beyond: Further Prospects of the Geochemical Analysis

Since 2009 has the central Nigerian Nok Culture – until then primarily known for its highly artistic terracotta figurines and early evidence of iron working in the first millennium BCE – been the focus of a joint research project by the Goethe University Frankfurt/Main, Germany, the National Commission for Museums and Monuments, Abuja, and the Universities of Jos and Zaria, all located in Nigeria. The analysis of Nok sculptures has so far been almost entirely restricted to their stylistic features which show such great similarities that one hypothesis of the Frankfurt project has been the possible central production of these artfully crafted figurines.

This doctoral thesis – written within the frame of the Research Training Group “Value and Equivalence” funded by the *Deutsche Forschungsgemeinschaft* (DFG) – investigates the material aspects of terracotta figurines by means of scientific materials analyses (mineralogical and X-ray fluorescence analysis). It aims to provide new insights into the production mode of Nok terracotta and to answer the question of a centralised production. In accordance with the subject of the Research Training Group – the generation of value and its modes of circulation – the terracottas’ role as objects of social and symbolic value and consequent implications on the social organisation of the Nok Culture are evaluated. The materials analysis of domestic pottery is used for comparison and differentiation.

For the mineralogical analysis (Chapter 4), 70 thin sections (both Nok terracotta and pottery) from 15 archaeological sites as well as 46 thin sections of recent clay samples and potsherds were analysed for differences in non-plastic inclusions and clay matrix. The geochemical analysis of the clay was conducted with a mobile XRF-analyser (Thermo Fisher Scientific Niton), and comprised 3214 samples (1639 potsherds, 1412 terracotta fragments, 163 recent clay samples, and 6 recent potsherds). These analyses have created – for the first time in West Africa – a unique and extensive database of mineralogical and geochemical information on ceramic objects.

The mineralogical analysis has revealed an unexpected, but nevertheless important source of information for distinguishing the figurines from the domestic pottery: the clay matrix, merged during the firing process, contains very small quartz grains (<0.05 mm) which occur naturally in the clay and thus reflect a genuine material property. The matrix of the figurines is characterised by an average content of these small grains of approximately 3%, while the concentration in the matrix of the pottery fluctuates stronger around approximately 4-5%. Obviously, a clay with a very distinct material property



was used in the manufacture of the terracotta, but not in the production of domestic pottery. This leads to the hypothesis that the figurines were – as objects with social and ritual meaning – produced exclusively with special clays. Other results of the mineralogical analysis concern the use of local tempering materials in the production of both terracotta and pottery, and evidence on the manufacturing processes.

The geochemical analysis has focussed on the clay itself (not the temper) and the associated question of the existence and possible location of central terracotta production centres. In a first step, the geochemical compositions of terracotta and pottery were compared on a site-level for three Nok sites (Ido, Ifana, and Ungwar Kura; Chapter 6). The result is unambiguous: the terracotta samples show a completely different chemical composition than the potsherds, especially in the composition of trace elements. No chemical differences could be observed in the internal structure of the terracotta samples – whether they came from different find contexts (deposition *vs.* refuse pit) or from different phases of the Nok Culture period. The pottery samples, in contrast, reveal some internal structure: Nok pottery differs in its chemical clay composition from the one of later pottery traditions; and even within the Nok Culture period, some slight differences are present.

In a second step, these results were transferred to the complete dataset of 3051 archaeological samples from 43 excavated or sampled sites (Chapter 7). The geochemical analysis of the overall database confirms the picture: the figurines show a very homogenous chemical composition with no noticeable internal structure while the chemical composition of the pottery samples fluctuates stronger and shows differences especially to later non-Nok pottery. Thus, the hypothesis of a use of distinct clay sources with special material properties for the manufacture of the terracotta figurines was confirmed. Finally, the analysis of the recent clay samples was included with the aim to gain some information concerning the nature and provenance of these special clay sources. Thirteen of the 163 recent clay samples have the same chemical composition than the clay used for the figurines. While it is unlikely that the same clay deposits have been used during Nok times, considering a period of more than 2000 years lying in between, this result shows clearly that clay sources with the same chemical properties than the ones used during Nok times still exist today. An even larger group of 111 clay samples resembles the pottery from Nok and Post-Nok times. Only 39 recent clays feature a completely different chemical composition. Field observations and geological maps were used to determine that the matching recent clay samples primarily derive from secondary clay deposits, while primary deposits are only used seldom for the manufacture of pots, but never for the figurines; secondary clay deposits can be found alongside rivers in all of the sampled area.

Combining the results of the mineralogical and geochemical analyses and the geographic and geological observations, a model for the organisation and procedure of the manufacture of the famous Nok terracottas can be suggested: They were – as the potsherds – manufactured with locally available raw materials (clay and temper) but in different manufacturing sequences with regard to temper and clay composition. The clay used in terracotta production was not used for domestic pottery – here, other secondary clay deposits, also found along rivers, were used. The terracottas' clay was obviously reserved for their production only, demonstrating – aside from stylistic similarities – the value these figurines had in Nok society. The special clay did not come from one single clay source (*i.e.* one large clay pit) but from chemically very homogenous clay deposits found alongside a widely ramified river system. No difference was made in the temper material; both terracotta and pottery were tempered with locally available inorganic material, albeit in changing volumes.

The conclusion that can be drawn from the materials analysis for the organisation and procedure of the manufacture of the figurines is far-reaching and leads to new insights into the social and symbolic value system of Nok society based on archaeological material evidence. The very stringent stylistic and material norms, which were possibly maintained for more than half a millennium (around the middle of the first millennium BCE) in the large distribution area of the Nok Culture, suggest that the figures were certainly manufactured by specialists. Yet, no signs of social complexity are found in the archaeological evidence from the excavations (Chapter 9). Most likely, the specialists did not work in centralised workshops with a central distribution system, but produced the terracotta figurines locally – possibly by travelling between settlements – yet observing the stylistic and material regulations. Such stringent regulations do not require a vertical hierarchy and a complex social system – especially in Africa horizontally organised societies feature diverse forms of social, cultural, or ritual complexity, relying on ritual authority rather than on economic or political power. In this framework, the terracotta sculptures of the Nok Culture may be regarded as collective items, functioning as symbols of ritual and social identity, uniting the members of the society, which live in dispersed settlements, through a central authority resting in the ritual sphere and relying on ritual power.

## Outlook

The results presented in this study have shown the great potential of scientific materials analyses for the interpretation of the Nok Culture's social organisation. But they have also opened new perspectives for future research: the domestic pottery that was initially only considered as comparison material for the figurines has proven to yield valuable information for the distinction of the Nok Culture to subsequent traditions as not only the stylistic elements changed over time but also the use of clay sources. Additionally, first results show that even the chronological phases of the Nok Culture themselves can be differentiated by the chemical composition of the pottery. Thus, scientific materials analysis can provide an additional argument for linking the Early Nok phase to the Nok Culture despite the obvious absence of terracotta figurines: the tradition in pottery decoration and form is underlined by continuity in the use of clay sources. Equally interesting, but not yet addressed, is the question if it is possible to further structure the approximately 600 years of the Middle Nok phase, in which absolute datings are imprecise because of a plateau in the calibration curve, on the basis of the chemical composition of the pottery.

Another ceramic material frequently found at Nok sites is burned clay that was presumably used for the construction of houses (RUPP 2014b). First geochemical analysis has shown that the composition of the burnt clay resembles the domestic potsherds. Future analysis may contribute to a better understanding of the different occupation phases of Nok sites.

Further prospects exist with regard to the terracotta figurines as well. The material analysed in this study was limited to a relatively small part of the Nok Culture distribution area. So far, the political situation on Nigeria has not allowed the Frankfurt project – as desirable as it would be – to shift its focus to neighbouring areas and test the results achieved on the chemical composition of the figurines in the key area of research. The same is true for adjacent, partly contemporaneous, partly younger terracotta traditions in Nigeria like in Yelwa or the Katsina and Sokoto states, subsumed under the term “North-western Terracotta Traditions” (Y. USMAN 2014). In Ile-Ife – besides the famous bronze heads – also statues made of clay occur that show similar stylistic features than the terracottas of the Nok Culture (WILLET 1967). Yet, available information on these complexes is sparse, as they are mostly not well dated and published. For the time being, however, these prospects have to wait until fieldwork can be continued and new material for geochemical analysis can be obtained.

In the meantime, a possibility to extend and test the results of this thesis will be given in the next months by Angela Fagg-Rackham who has offered the opportunity to analyse some pottery and terracotta material from her

father's excavation in Taruga that is still awaiting proper analysis.<sup>67</sup> Thus, the investigated area may be extended to the western fringes of the Nok Culture distribution area and the early beginnings of the exploration of the Nok Culture may be united with the newest research by the Frankfurt project.

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<sup>67</sup> Since the completion of this dissertation, the ceramic material from Taruga has been analysed. The terracotta and pottery samples follow the same pattern than the ones from the sites within the key area of research of the Frankfurt project. While the figurines from Taruga fall within the large cluster of points of all other statues (albeit lying at the lower edge of the cluster), the pottery samples show a slightly different chemical composition than the ones from the area researched in this study – which corresponds to the idea of a local production on a household level. Again, sherds from Taruga belonging to the Post-Nok phase clearly separate from the ones belonging to the Nok Culture. A publication of the material excavated by Fagg, linking it to the new research by the Frankfurt project, is currently being prepared.



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# Appendices



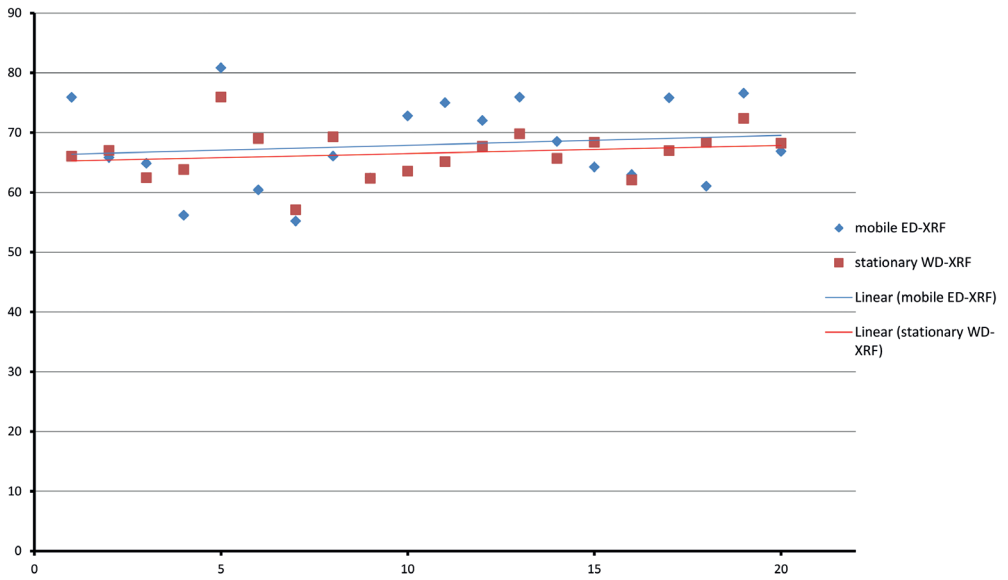


# Appendix 1

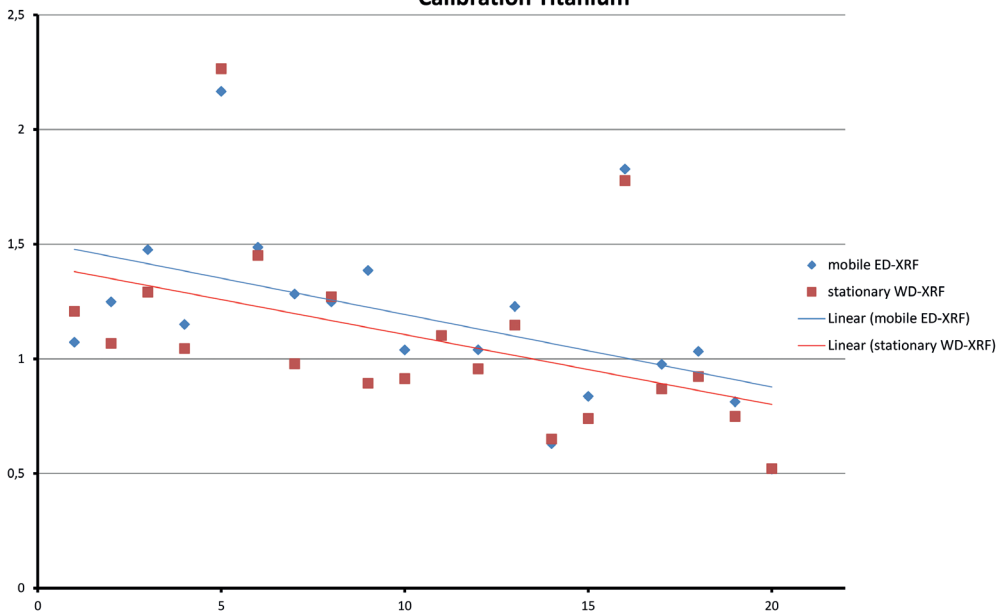
## Calibration Curves

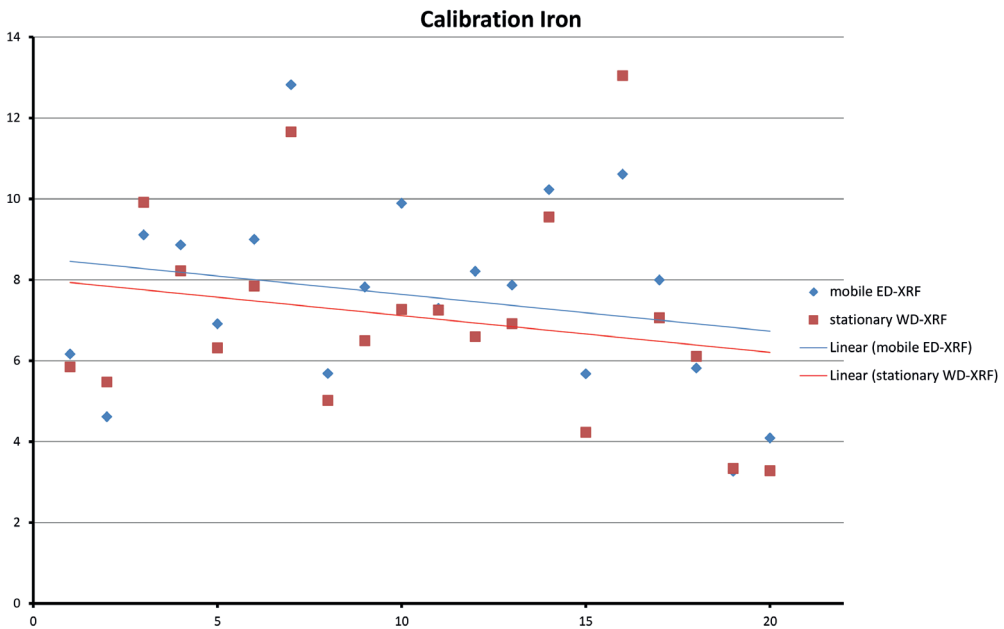
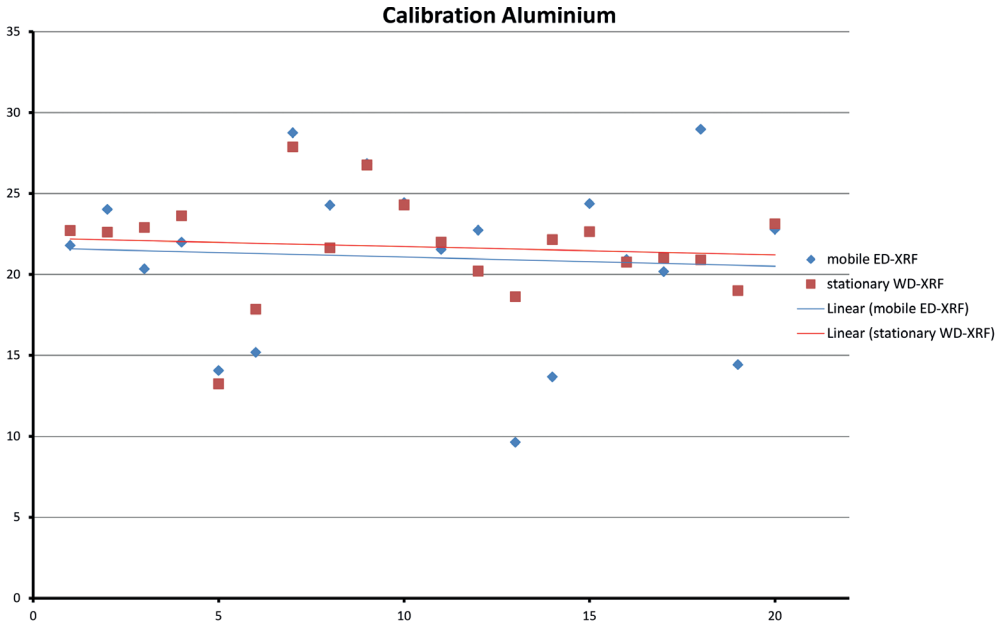
Annotation: The calibration curves show the comparison between the measurement values for each chemical element of the stationary WD-XRF (red square) and mobile ED-XRF (blue rhombus) for 20 samples as well as the corresponding best fit straight lines. The parallelism and distance between the two best fit straight lines is a measure for the quality of the calibration.

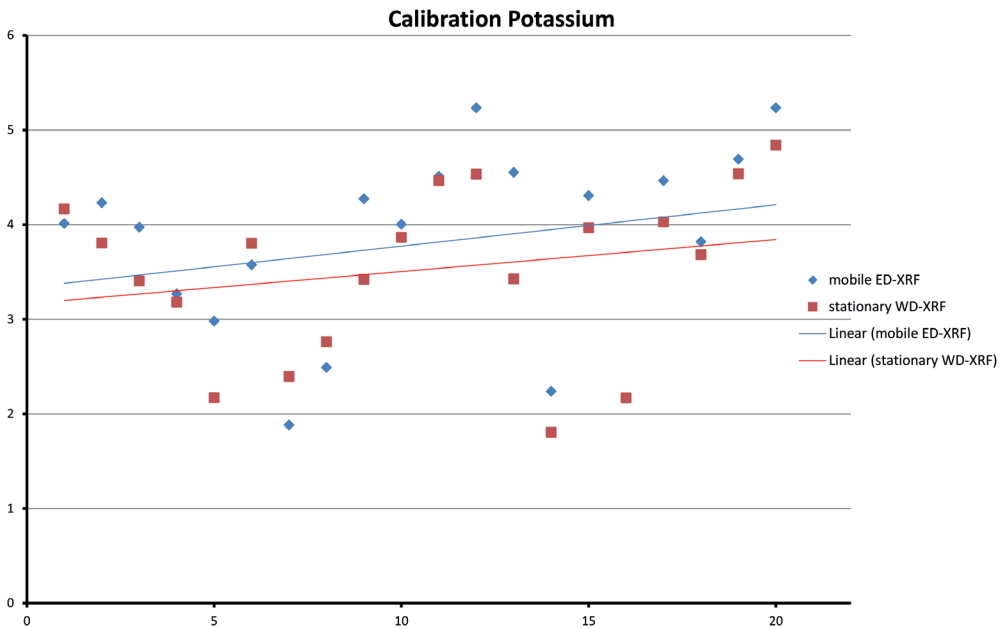
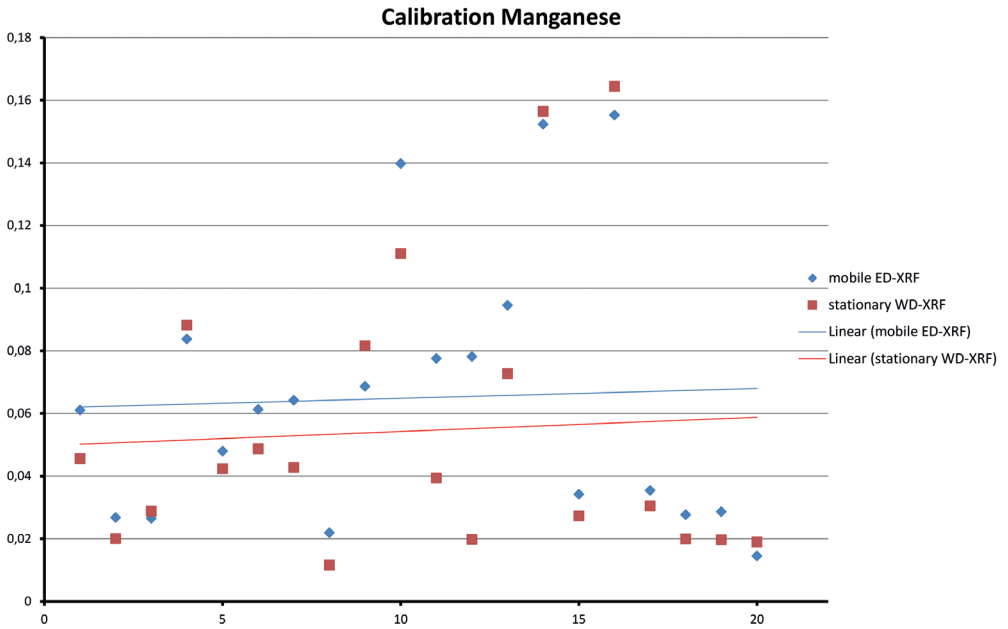
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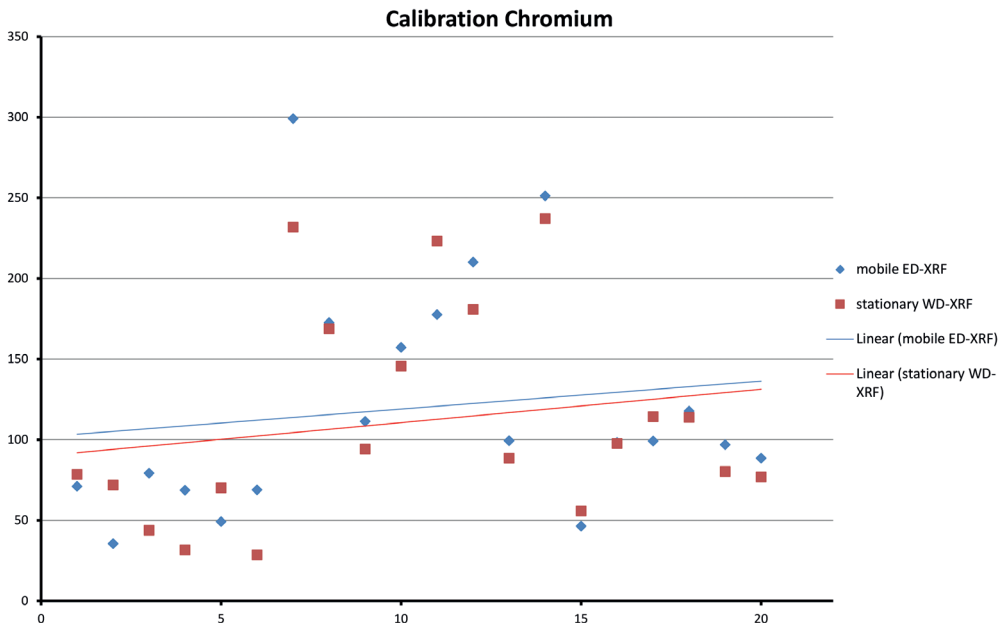
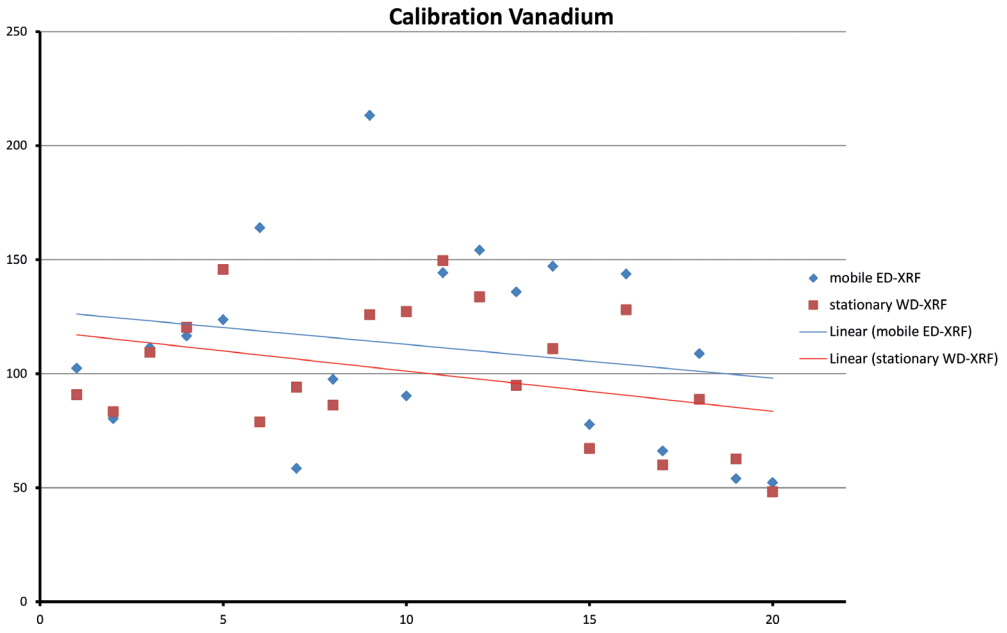


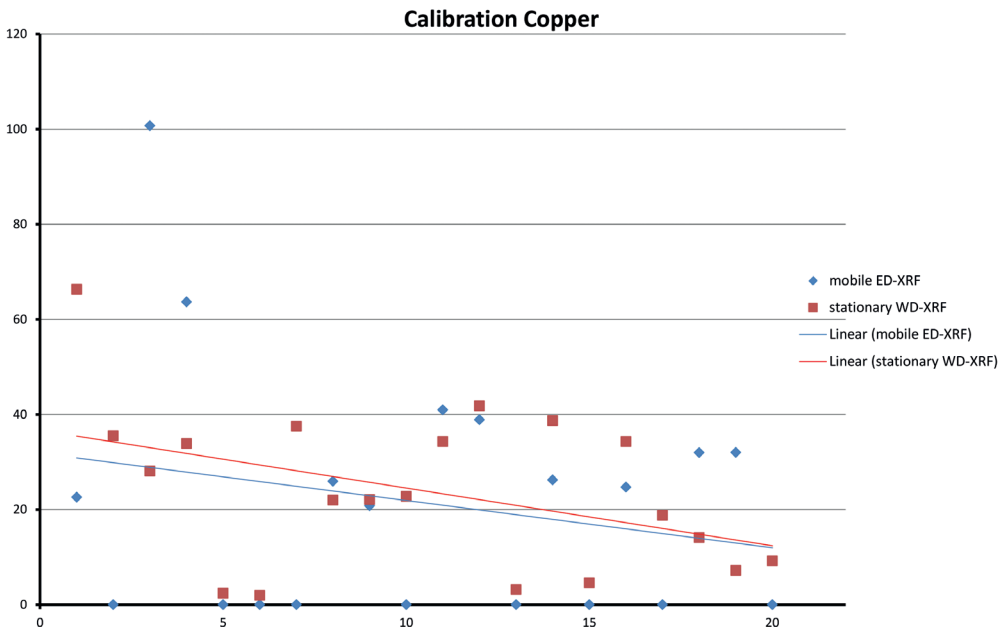
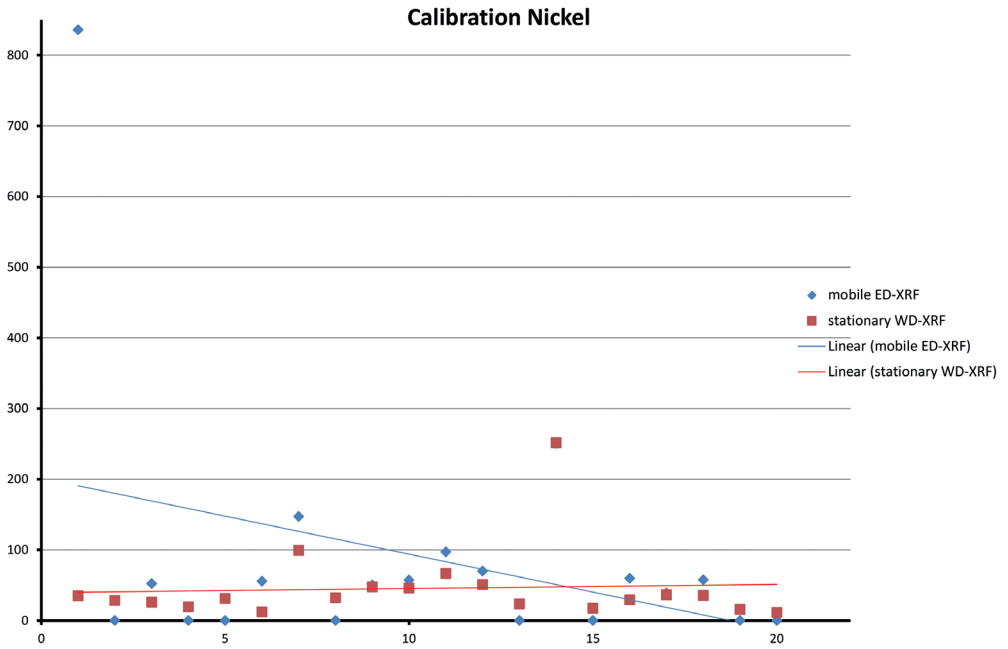
### Calibration Titanium

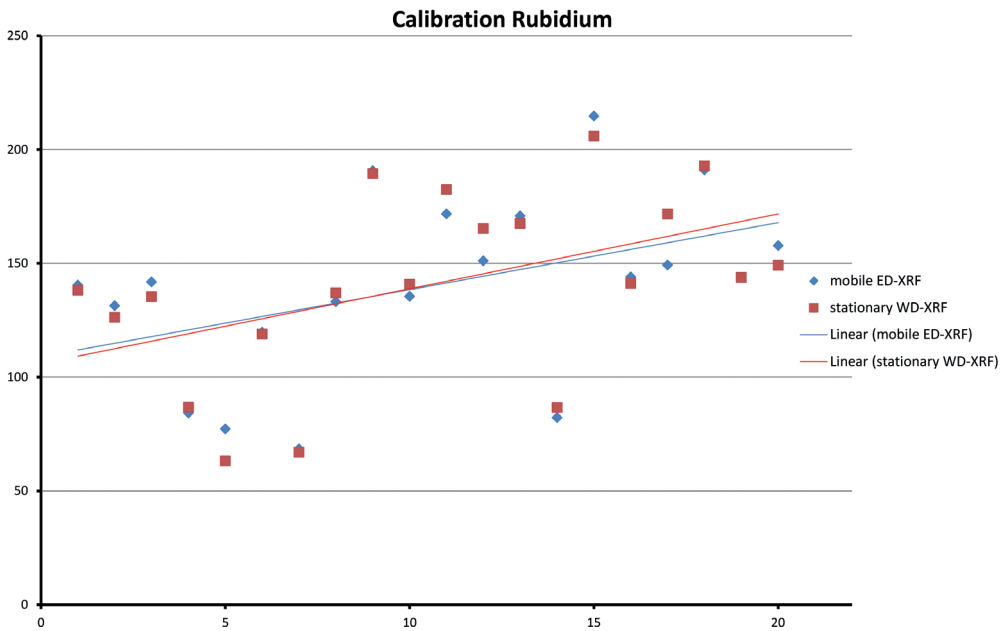
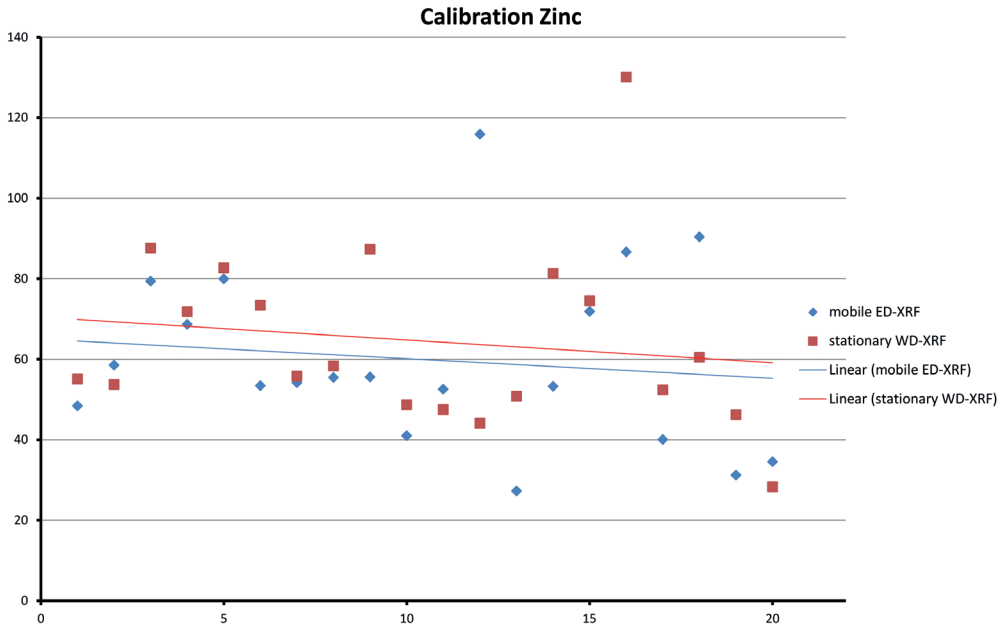




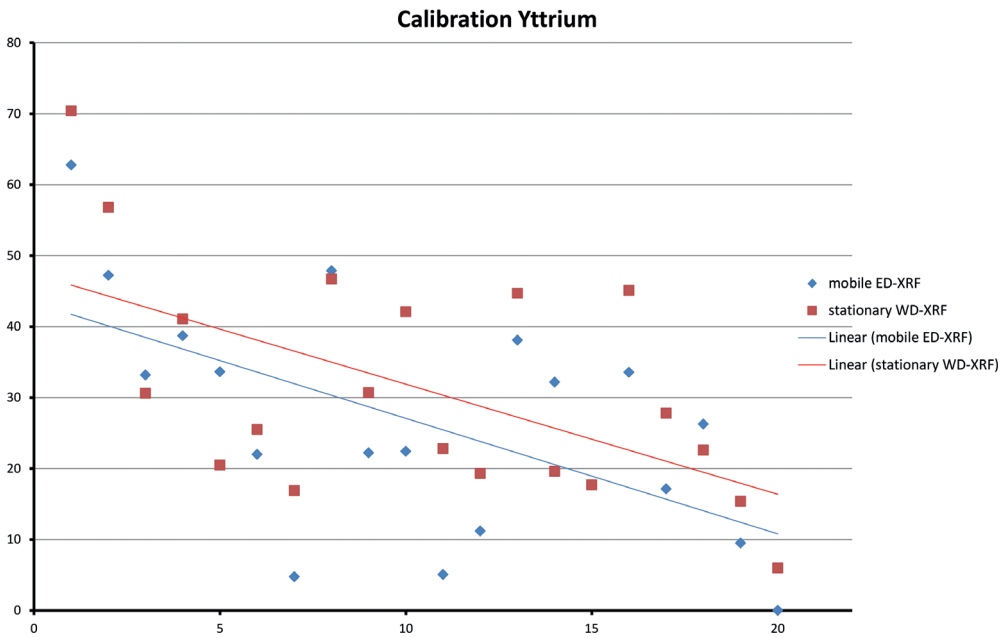
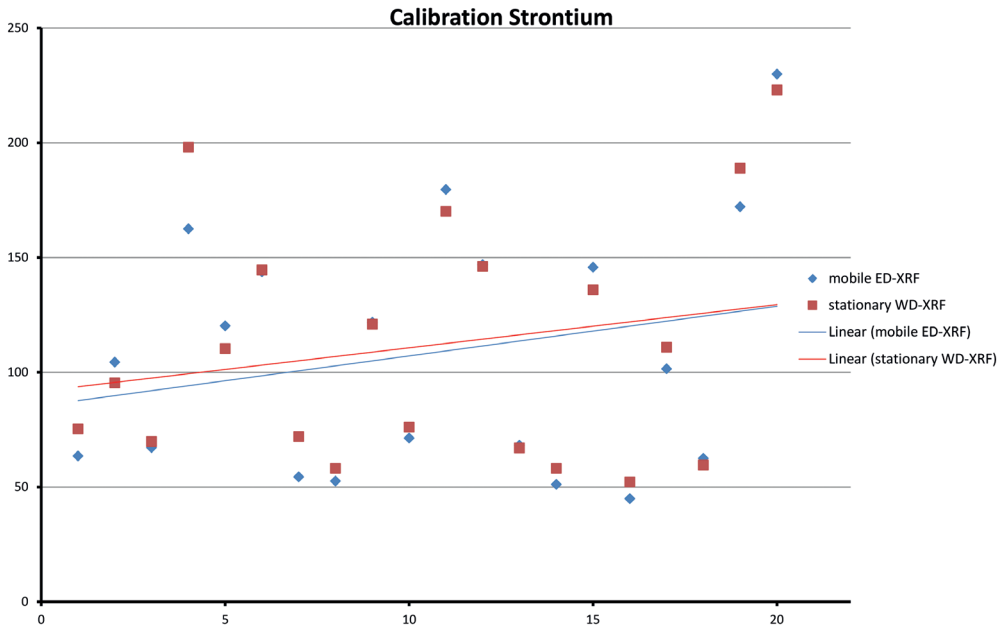


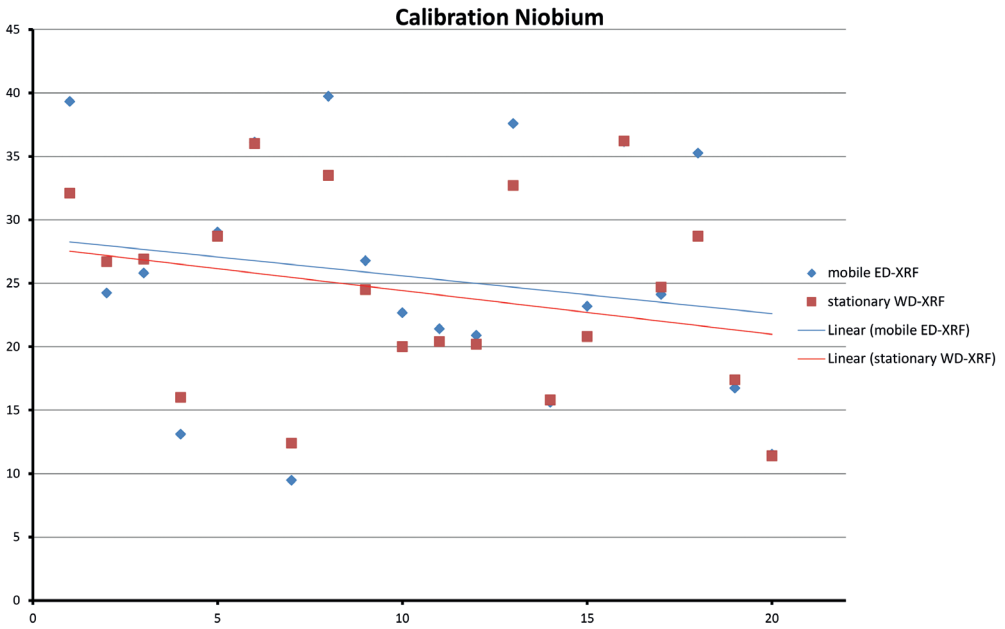
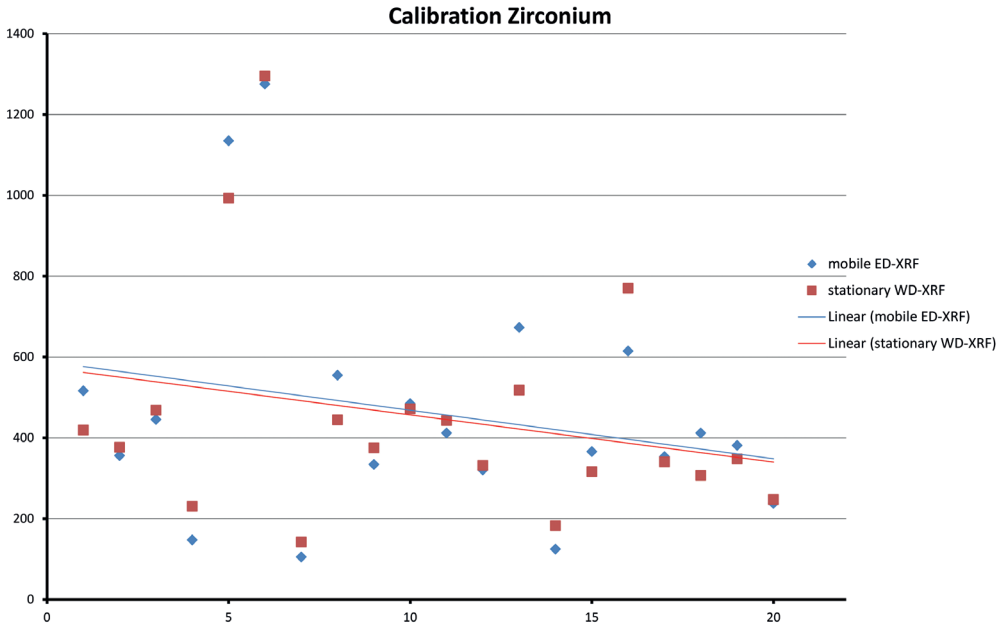


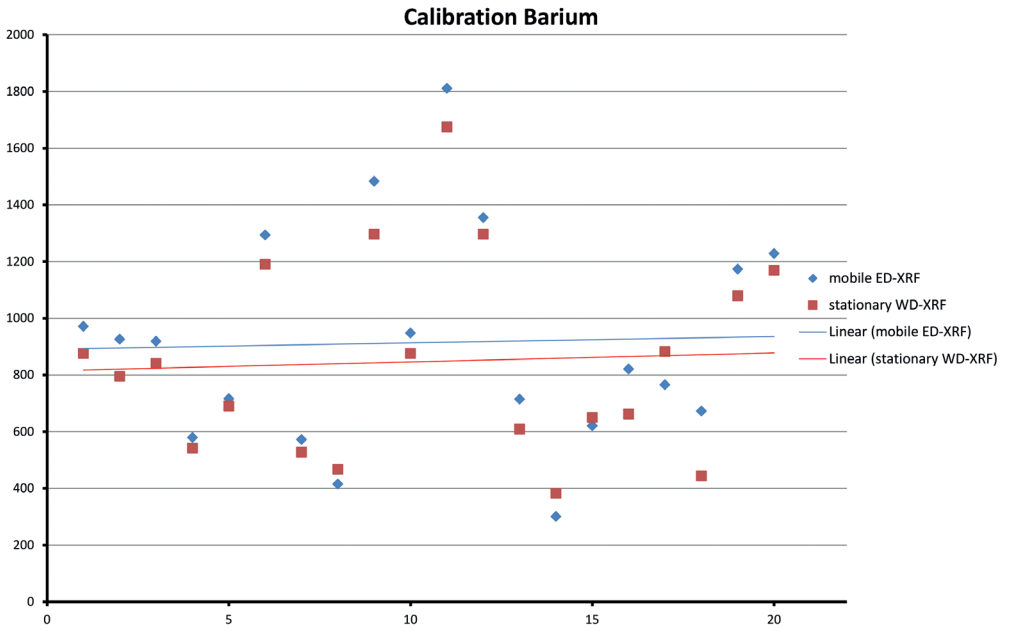












## **Appendix 2**

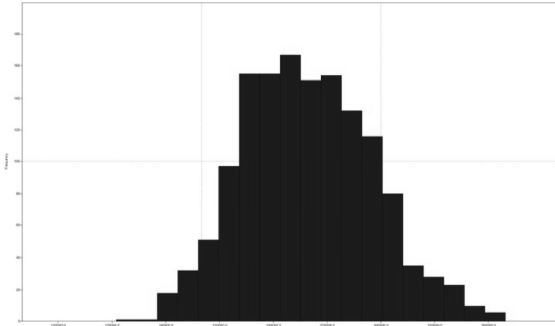
**Statistical key figures of the raw data of all chemical elements**

**Terracotta**

**Pottery**

**Clay samples**

Silicon



Standard deviation (ppm): 36192.03

Mean (ppm): 259390.97

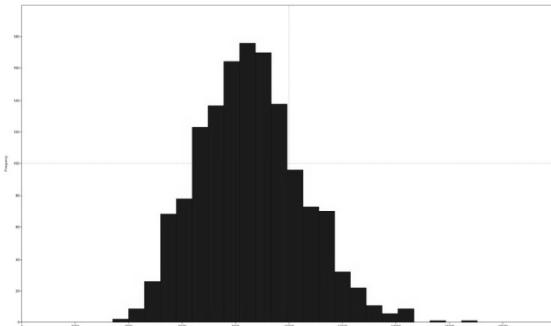
Median (ppm): 257357.87

Skewness: 0.24

Kurtosis: -0.25

**Terracotta**

Titan



Standard deviation (ppm): 1953.11

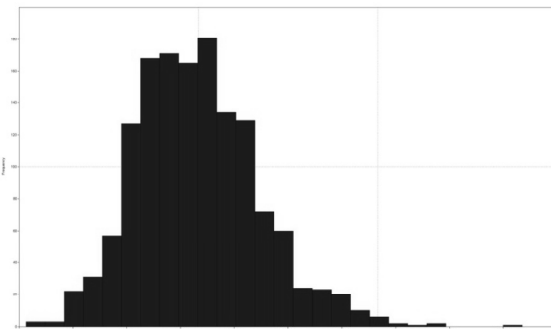
Mean (ppm): 8567.88

Median (ppm): 8465.62

Skewness: 0.37

Kurtosis: 0.15

Aluminium



Standard deviation (ppm): 34335.82

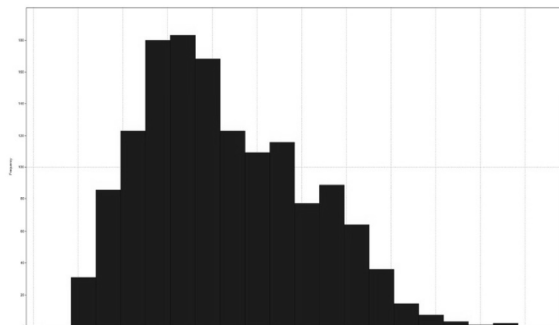
Mean (ppm): 99418.43

Median (ppm): 97203.33

Skewness: 0.58

Kurtosis: 0.91

Iron



Standard deviation (ppm): 18040.15

Mean (ppm): 52512.54

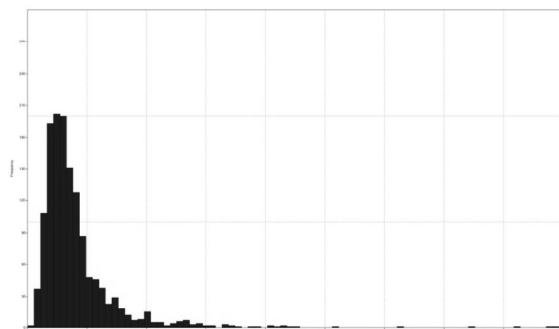
Median (ppm): 49564.89

Skewness: 0.49

Kurtosis: -0.39

**Terracotta**

Manganese



Standard deviation (ppm): 730.20

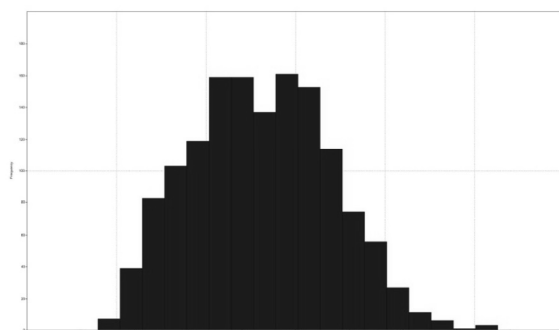
Mean (ppm): 817.30

Median (ppm): 636.41

Skewness: 4.89

Kurtosis: 38.40

Potassium



Standard deviation (ppm): 7816.77

Mean (ppm): 26145.18

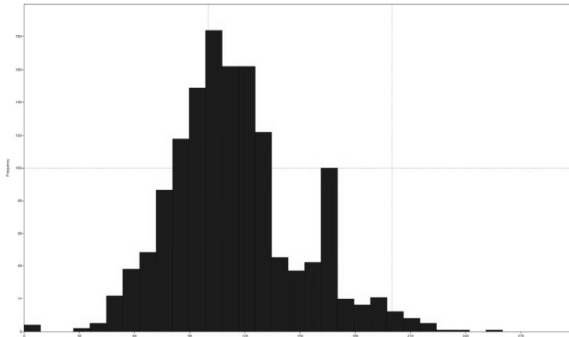
Median (ppm): 25858.35

Skewness: 0.17

Kurtosis: -0.47

**Terracotta**

Vanadium



Standard deviation (ppm): 34.88

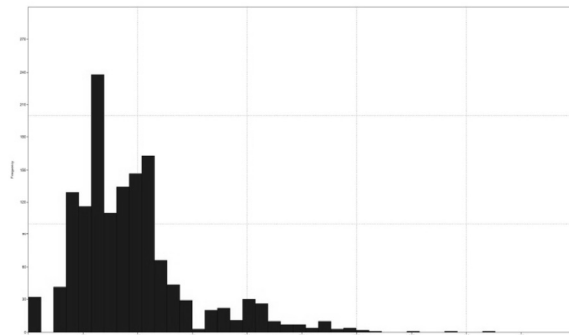
Mean (ppm): 114.29

Median (ppm): 110.24

Skewness: 0.53

Kurtosis: 0.54

Chromium



Standard deviation (ppm): 54.36

Mean (ppm): 93.84

Median (ppm): 83.29

Skewness: 1.55

Kurtosis: 3.53

Zinc



Standard deviation (ppm): 114.38

Mean (ppm): 80.54

Median (ppm): 62.34

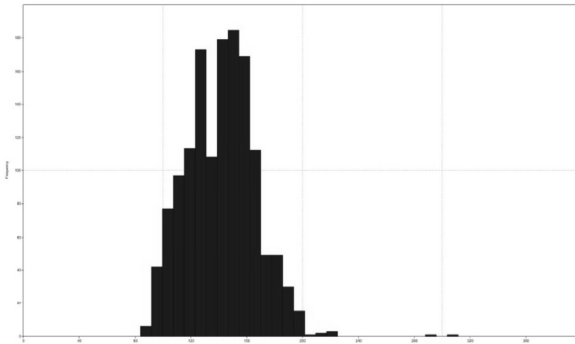
Skewness: 10.94

Kurtosis: 152.30



**Terracotta**

Rubidium



Standard deviation (ppm): 24.90

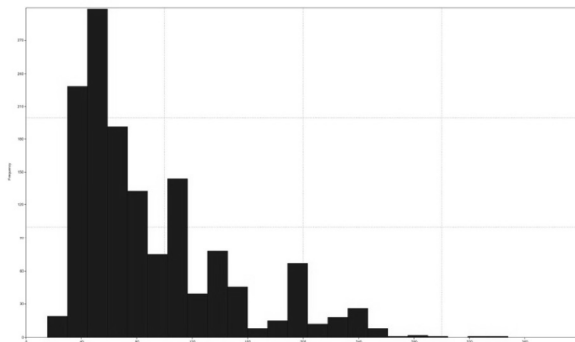
Mean (ppm): 141.73

Median (ppm): 143.25

Skewness: 0.48

Kurtosis: 1.91

Strontium



Standard deviation (ppm): 54.56

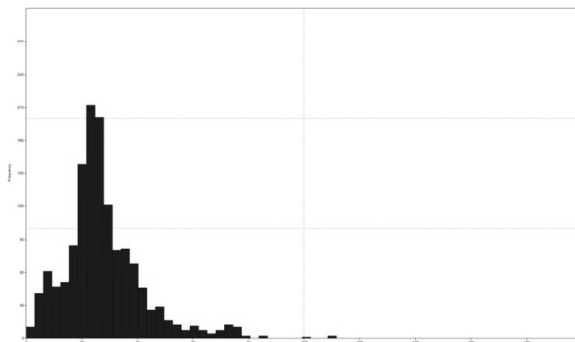
Mean (ppm): 89.75

Median (ppm): 71.13

Skewness: 1.35

Kurtosis: 1.34

Yttrium



Standard deviation (ppm): 14.21

Mean (ppm): 27.65

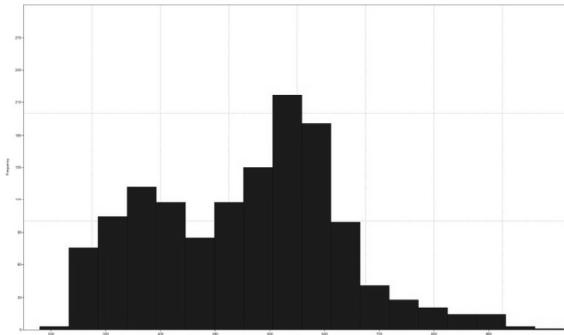
Median (ppm): 25.43

Skewness: 1.35

Kurtosis: 3.62

**Terracotta**

Zirconium



Standard deviation (ppm): 136.93

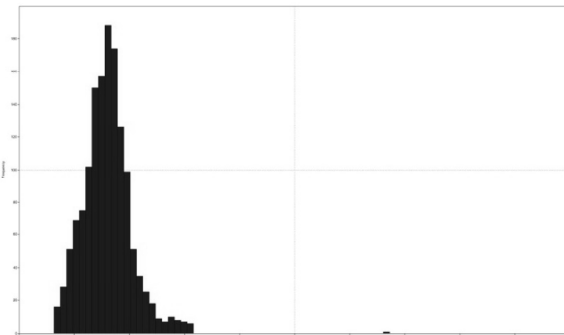
Mean (ppm): 525.30

Median (ppm): 545.25

Skewness: 0.14

Kurtosis: -0.43

Niobium



Standard deviation (ppm): 8.85

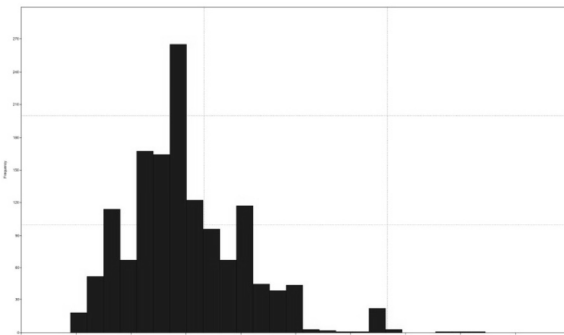
Mean (ppm): 31.93

Median (ppm): 31.68

Skewness: 1.57

Kurtosis: 13.13

Barium



Standard deviation (ppm): 319.54

Mean (ppm): 899.25

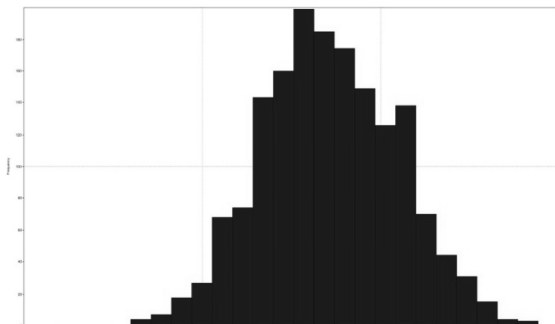
Median (ppm): 853.95

Skewness: 0.94

Kurtosis: 1.69

Silicon

**Pottery**



Standard deviation (ppm): 37980.50

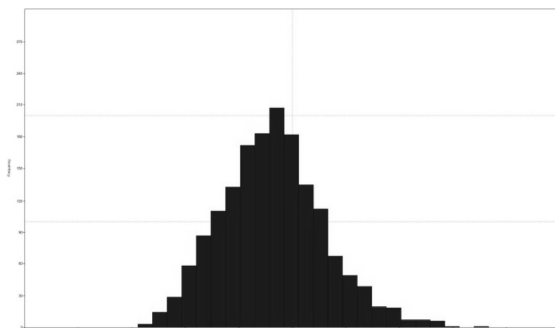
Mean (ppm): 270938.90

Median (ppm): 270162.04

Skewness: 0.09

Kurtosis: -0.31

Titan



Standard deviation (ppm): 1978.02

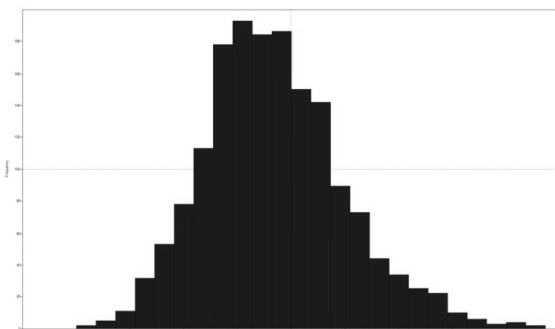
Mean (ppm): 9284.33

Median (ppm): 9217.56

Skewness: 0.42

Kurtosis: 0.26

Aluminium



Standard deviation (ppm): 26147.72

Mean (ppm): 94112.81

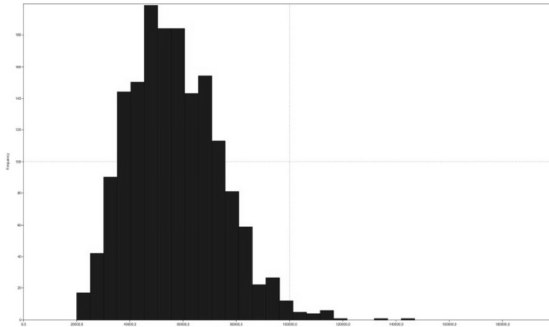
Median (ppm): 92226.10

Skewness: 0.48

Kurtosis: 0.41

**Pottery**

**Iron**



Standard deviation (ppm): 17199.33

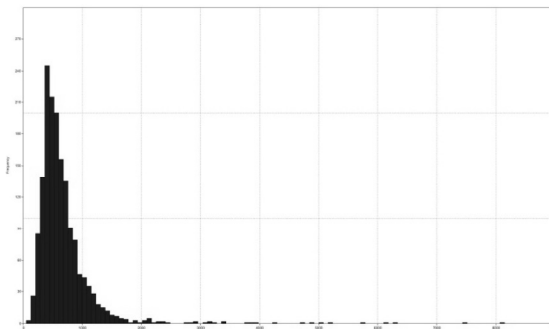
Mean (ppm): 56864.31

Median (ppm): 55362.61

Skewness: 0.57

Kurtosis: 0.56

**Manganese**



Standard deviation (ppm): 570.72

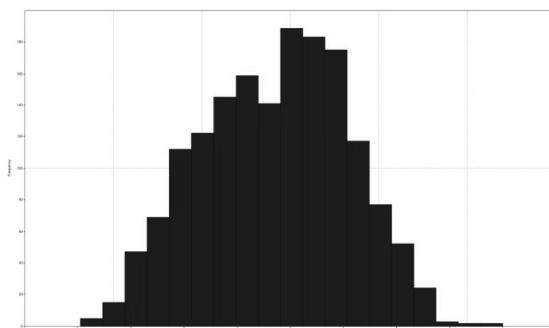
Mean (ppm): 687.27

Median (ppm): 565.59

Skewness: 6.02

Kurtosis: 53.72

**Potassium**



Standard deviation (ppm): 8625.47

Mean (ppm): 28458.14

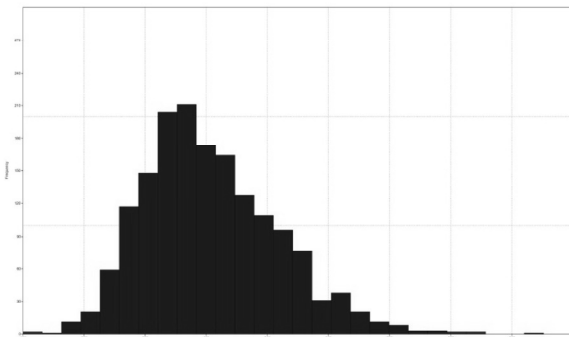
Median (ppm): 29046.17

Skewness: 0.64

Kurtosis: 5.65

Vanadium

**Pottery**



Standard deviation (ppm): 111.74

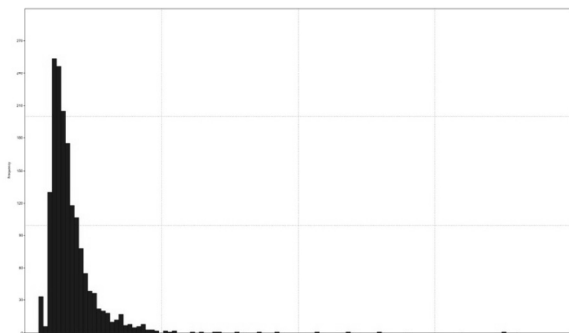
Mean (ppm): 404.73

Median (ppm): 390.35

Skewness: 0.55

Kurtosis: 0.62

Chromium



Standard deviation (ppm): 202.54

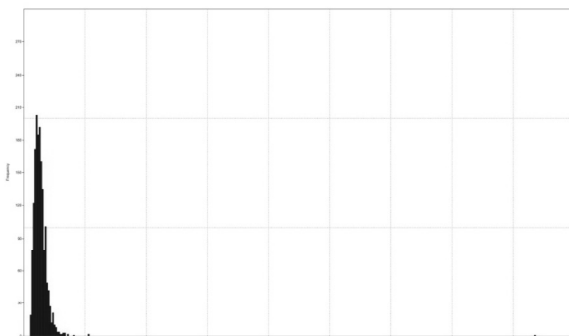
Mean (ppm): 332.87

Median (ppm): 287.42

Skewness: 5.66

Kurtosis: 60.49

Zinc



Standard deviation (ppm): 222.94

Mean (ppm): 271.73

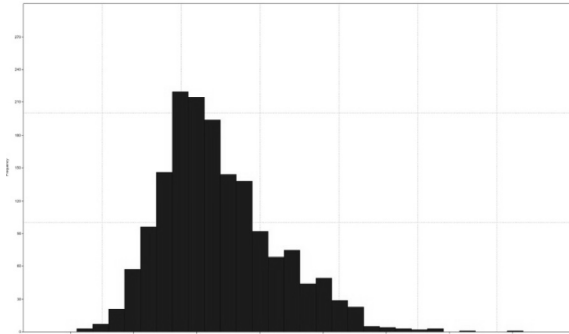
Median (ppm): 251.67

Skewness: 29.10

Kurtosis: 1049.58

## Pottery

Rubidium



Standard deviation (ppm): 75.67

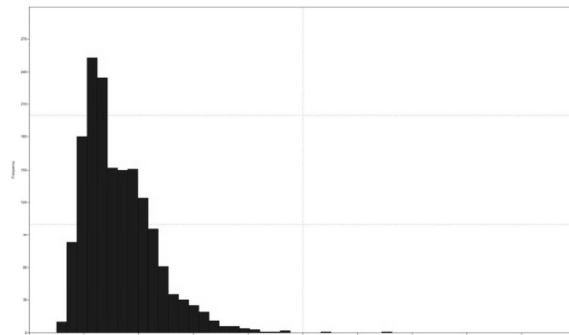
Mean (ppm): 345.06

Median (ppm): 332.70

Skewness: 0.60

Kurtosis: 0.94

Strontium



Standard deviation (ppm): 133.08

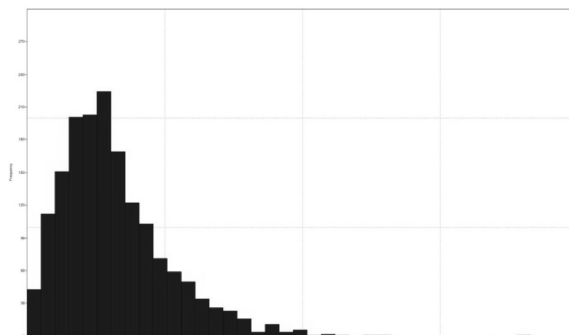
Mean (ppm): 327.01

Median (ppm): 298.19

Skewness: 1.31

Kurtosis: 3.58

Yttrium



Standard deviation (ppm): 40.83

Mean (ppm): 161.55

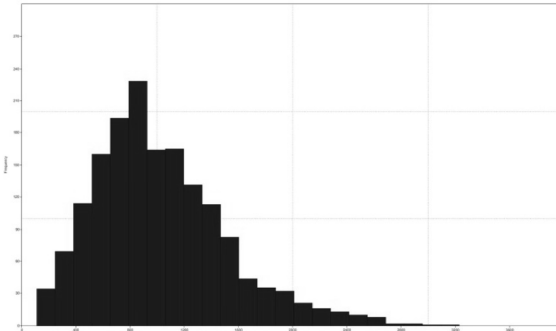
Median (ppm): 155.57

Skewness: 0.95

Kurtosis: 3.60

**Pottery**

Zirconium



Standard deviation (ppm): 491.90

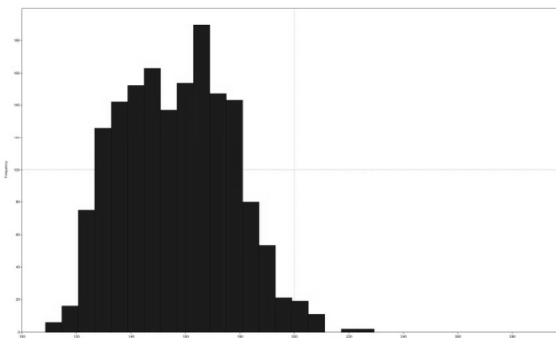
Mean (ppm): 1024.97

Median (ppm): 941.67

Skewness: 0.85

Kurtosis: 0.89

Niobium



Standard deviation (ppm): 24.56

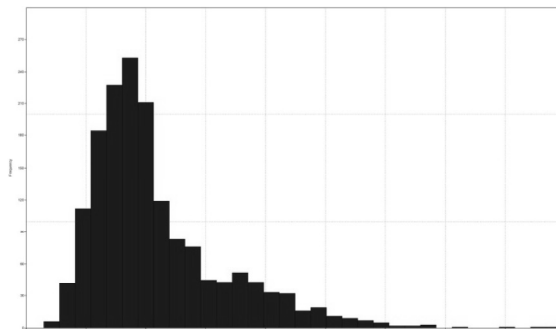
Mean (ppm): 155.38

Median (ppm): 156.88

Skewness: -1.51

Kurtosis: 7.37

Barium



Standard deviation (ppm): 1136,79

Mean (ppm): 2172.40

Median (ppm): 1857.46

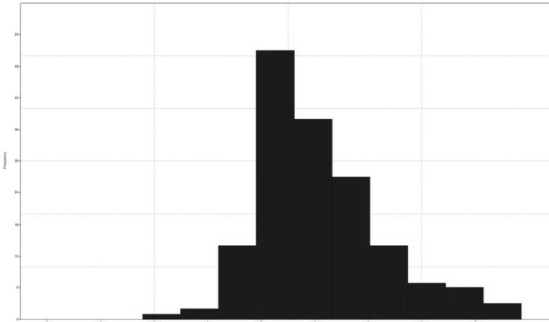
Skewness: 1.49

Kurtosis: 2.76



**Clay samples**

Silicon



Standard deviation (ppm): 47084.14

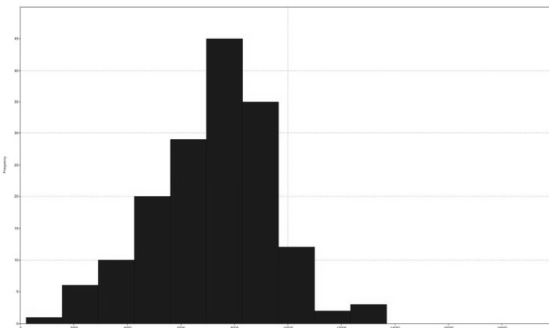
Mean (ppm): 323464.48

Median (ppm): 312068.41

Skewness: 0.76

Kurtosis: 0.78

Titan



Standard deviation (ppm): 2198.08

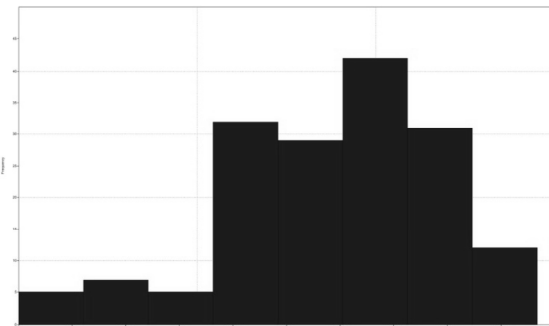
Mean (ppm): 7205.39

Median (ppm): 7404.12

Skewness: -0.12

Kurtosis: 0.54

Aluminium



Standard deviation (ppm): 59937.15

Mean (ppm): 175385.84

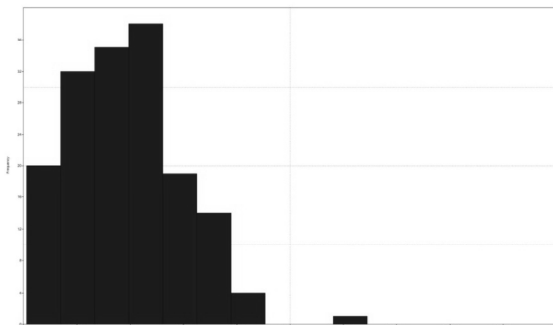
Median (ppm): 183120.72

Skewness: -0.67

Kurtosis: 0.42

Iron

**Clay samples**



Standard deviation (ppm): 20208.33

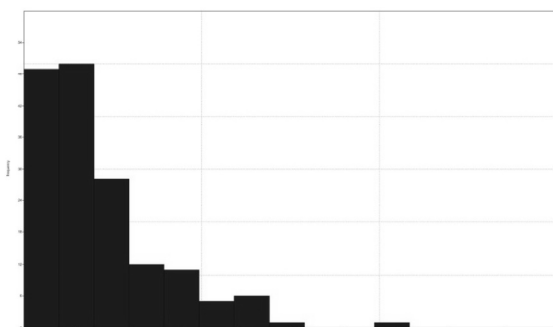
Mean (ppm): 38602.00

Median (ppm): 38698.64

Skewness: 0.73

Kurtosis: 1.44

Manganese



Standard deviation (ppm): 365.94

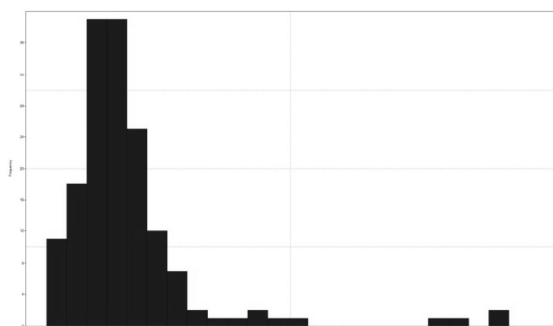
Mean (ppm): 421.93

Median (ppm): 318.39

Skewness: 1.58

Kurtosis: 3.24

Potassium



Standard deviation (ppm): 26075.39

Mean (ppm): 38366.99

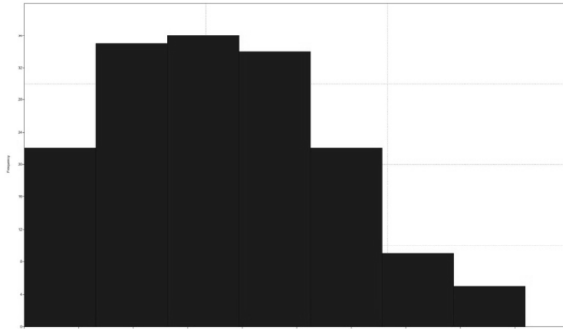
Median (ppm): 33029.77

Skewness: 3.38

Kurtosis: 14.40

**Clay samples**

Vanadium



Standard deviation (ppm): 64.55

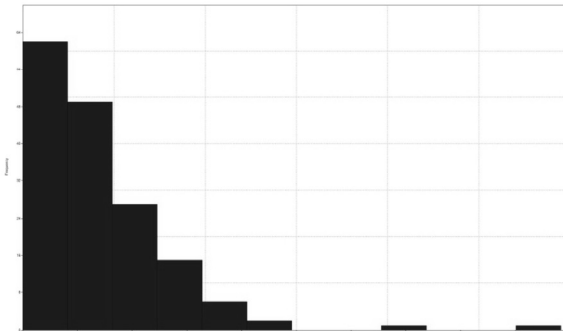
Mean (ppm): 108.94

Median (ppm): 106.57

Skewness: 0.21

Kurtosis: -0.34

Chromium



Standard deviation (ppm): 79.60

Mean (ppm): 79.44

Median (ppm): 66.41

Skewness: 2.32

Kurtosis: 10.59

Zinc



Standard deviation (ppm): 123.89

Mean (ppm): 115.96

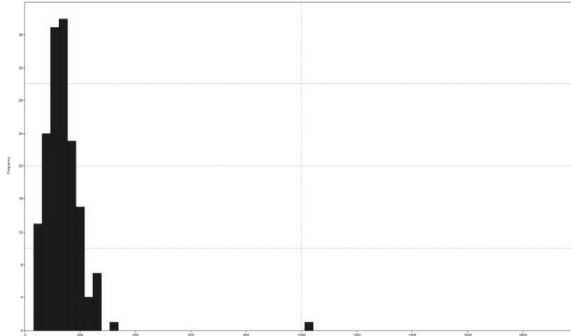
Median (ppm): 95.69

Skewness: 5.15

Kurtosis: 41.93

Rubidium

**Clay samples**



Standard deviation (ppm): 89.20

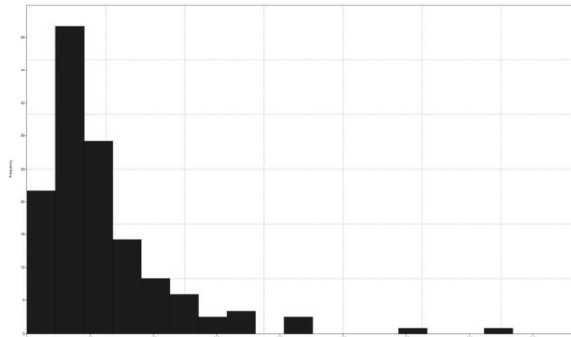
Mean (ppm): 137.79

Median (ppm): 128.20

Skewness: 6.54

Kurtosis: 64.33

Strontium



Standard deviation (ppm): 84.72

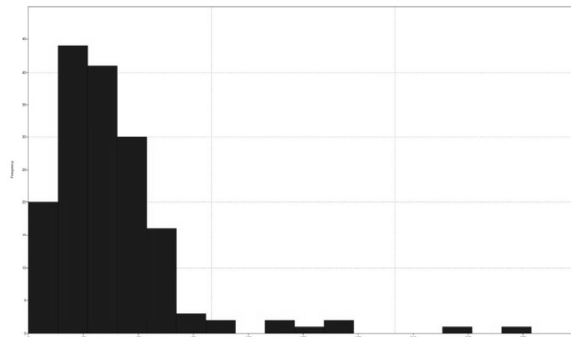
Mean (ppm): 95.81

Median (ppm): 71.01

Skewness: 2.75

Kurtosis: 11.22

Yttrium



Standard deviation (ppm): 36.95

Mean (ppm): 45.42

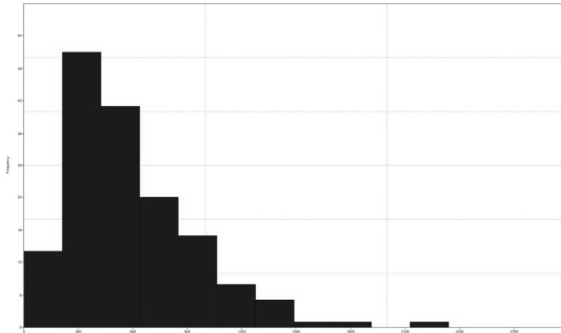
Median (ppm): 38.18

Skewness: 3.02

Kurtosis: 13.60

**Clay samples**

Zirconium



Standard deviation (ppm): 351.30

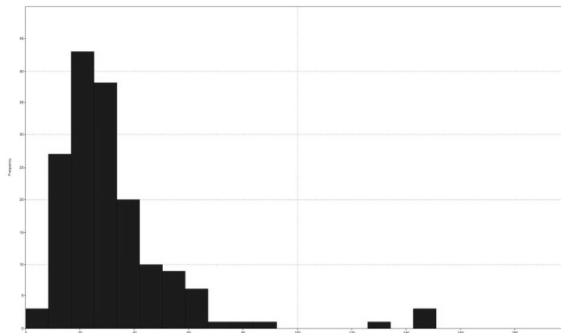
Mean (ppm): 584.59

Median (ppm): 501.54

Skewness: 1.40

Kurtosis: 3.46

Niobium



Standard deviation (ppm): 23.09

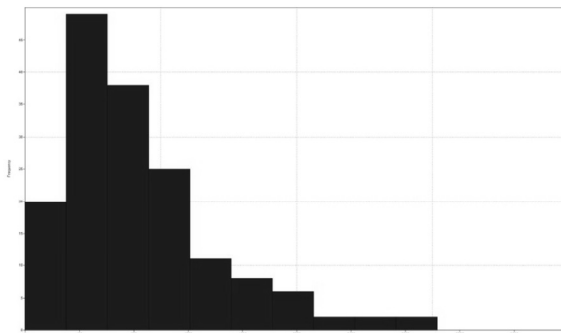
Mean (ppm): 31.96

Median (ppm): 27.06

Skewness: 2.92

Kurtosis: 11.43

Barium



Standard deviation (ppm): 566.46

Mean (ppm): 828.88

Median (ppm): 715.25

Skewness: 1.35

Kurtosis: 2.00