STUDY OF THE ANCIENT IRON-SMELTING SITES AT PANTAKI, TSAUNI AND SAMARU-WEST, NORTHERN NIGERIA, USING NEUTRON ACTIVATION ANALYSIS

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Abstract

Neutron activation analysis was used to obtain concentrations of some elements in samples of potsherds and furnaces obtained from Pantaki, Tsauni and Samaru-west ancient iron-smelting sites in northern Nigeria. Using WARD method, cluster analysis of the elements was carried out to establish the relationship between the archaeological samples in term of similarity in elemental concentrations. It was found that there is some level of similarity among the potsherd samples from the three sites. Also, most of furnace samples display some similarity. It was established that there was cultural linkage between the iron-smelters at the different sites. The similarity between the samples lead to a conclusion that pot makers and furnace makers could have existed, who made and sold the products to users in different communities. The pot makers obtained their clays from common source. The furnace makers also obtained their clays from other common source, different from the source of clay used by pot makers.

Keywords: Iron-smelting, Pantaki, Tsauni, Samaru-West, Neutron activation analysis, WARD's method.

Introduction

Archaeologists have shown interest in the ancient iron-smelting sites at Pantaki, Samaru-west and Tsauni in Zaria, Northern Nigeria since around 1976 (Effah-Gyamfi, 1981). It was discovered that there was conspicuous evidence of large scale iron smelting activity at the sites. In attempt to determine age of the sites, Sutton (1976) submitted charcoal samples for radiocarbon dating and obtained some set of dates for iron-smelting at Samaru-west. It was intended to compare the age of the three sites. Unfortunately all excavations at Tsauni and Pantaki yielded no dateable charcoal. So, only Samaru-west was dated. As a follow up to this, Oladipo *et al.* (1992) analysed the elemental composition of potsherds from the three sites, using neutron activation analysis method. The result showed that samples from Samaru-west and Tsauni are not much similar, while those from Pantaki scatter among Tsauni and Samaru-west samples. The present study is a scientific follow up of those previous studies. Remnants of furnace samples that are still available at the sites are the main focus for analysis in this study.

Fig 1 shows the location of the three ancient iron-smelting sites. Pantaki (a) is the name of an area on the popular Kufena inselberg, at 11º06´N and 7º40´E. Two major sites are

recognized at this site. One is a low level, surrounded by a fence of rock. There was a layer of laterite on the rock, and some slag, which is an indication that iron smelting took place there in the past. The remaining part of this lower site is littered with potsherds. Some of them are thick and gritty while others are relatively thinner. On one part on the rock-top is an occupation site. This is covered with a layer of soil about 30cm thick, and is littered with potsherds. No furnace remnant was found from the site. At Samaru-west (b), located at 11º9½´N and 7º37´E, three different sites were found with slag heaps. Tsauni sites (c) are located along old Zaria-Brinigwari road at 11º06´N and 7º35´E. Two large iron-smelting sites were found there.

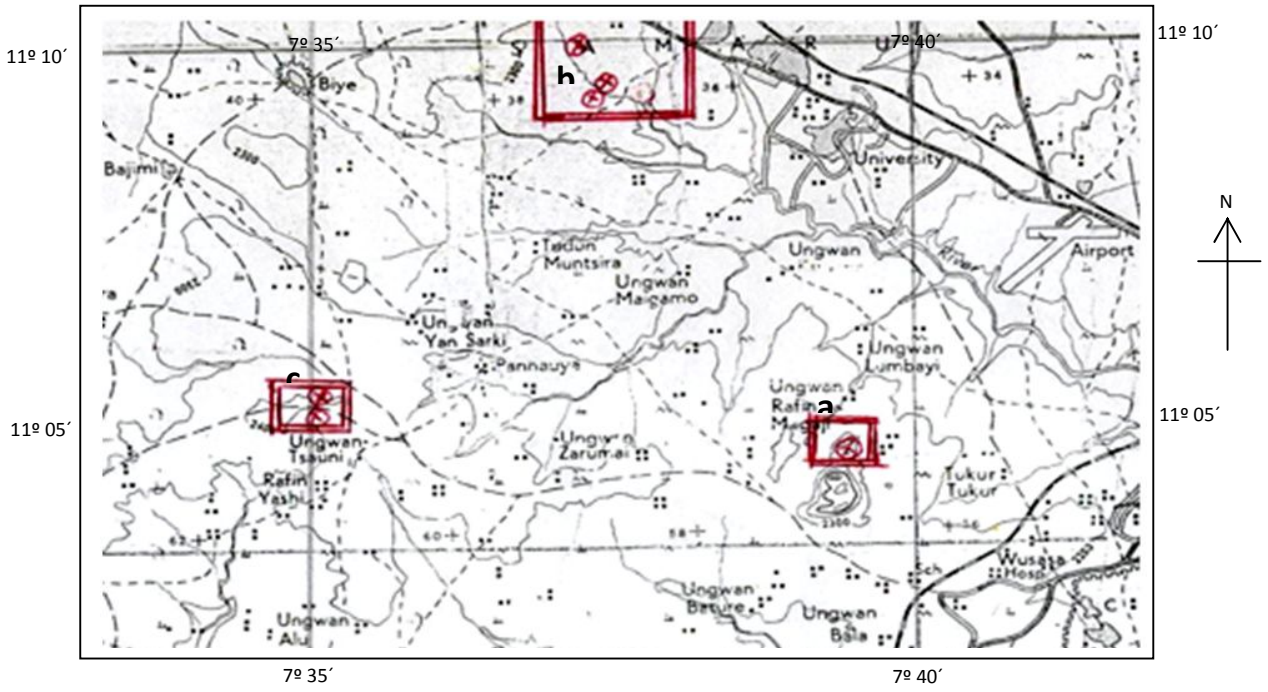

Fig. 1: Part of Zaria map showing (a) Pantaki, (b) Samaru-west and (c) Tsauni ancient iron smelting sites (Scale: 1:100,000)

Furnace samples were collected at Samaru-west and Tsauni site, while potsherd samples were collected at all of the three sites. Neutron activation analysis (NAA) using thermal neutron from Nigerian nuclear reactor (NNR-1), located at the Centre For Energy Research and Training, Ahmadu Bello University, Zaria, was employed to determine the concentration of some trace elements in the samples. The use of NAA to solving this problem is based on the premise that materials of which the ceramics are made will be finger-printed chemically thus reflecting their places of origin.

Experimental Procedure

The Samples

A number of representative samples were collected from each of the industrial sites. Table 1 is a presentation of samples and their description.

Table 1: Samples and Description

S/N	Sample	Description
1	SF 1	Samaru-west Furnace 1
2	SF 1CR	Samaru-west Furnace 1 (crumble-like)
3	SF 2	Samaru-west Furnace 2
4	SF 3	Samaru-west Furnace 3
5	SF 4	Samaru-west Furnace 4
6	SP E N	Samaru-west potsherd, excavated, thin sample.
7	SP E K	Samaru-west potsherd, excavated, thick sample.
8	SP S K	Samaru-west potsherd, surface found, thick sample.
9	TNF 1	Tsauni north furnace 1
10	TNF 2	Tsauni north furnace 2
11	TSOF	Tsauni South occupation site furnace
12	TSGF	Tsauni South gully side furnace
13	TSSF 1	Tsauni South slagheap top furnace 1
14	TSSF 2	Tsauni South slagheap top furnace 2
15	TSSF 3	Tsauni South slagheap top furnace 3
16	TSOP 1	Tsauni South occupation site potsherd 1
17	TSOP 2	Tsauni South occupation site potsherd 2
18	PUP TN	Pantaki upper-part thin potsherd
19	PUP TK	Pantaki upper-part thick potsherd
20	PLP TN	Pantaki lower-part thin potsherd
21	PLP TK	Pantaki lower-part thick potsherd

Samples were washed thoroughly with distilled water and then allowed to dry to a constant weight at temperature of 60°C in an oven. With aid of a diamond scraper, the outer layer of the samples was scraped to remove any trace of contamination. The dirt-free samples

were then powdered in an agate mortar. About 0.13 mg was carefully weighed and sealed as a capsule in a prewashed and dried polythene container. The sample was then placed in a plastic vial and packed with cotton wool before sealing with cellotape. Samples were prepared for both long-lived and short-lived irradiation. Three standards – soil-7, fly ash and IAEA-312 – samples were prepared and included in the vial for irradiation. These are for quality assurance test.

Sample Irradiation

For short-lived irradiation, samples were subjected to two minutes irradiation in a neutron flux of 1×10^{11} n/cm^{2}sec. After removal from the reactor, the samples were allowed to cool till radiation dose fall to a level save for handling (ca 30μSv). For long-lived irradiation, samples were irradiated for six hours in neutron flux of 5×10^{11} n/cm^{2}sec. The samples were allowed to cool for four days to allow short-lived species to decay away before counting, for the first set of long-lived radionuclides. The counting was repeated after nine days in order to take measurement for the next set of radionuclides.

Counting of Neutron Activated Samples

Counting was done with high purity Germanium detector with resolution of 2 keV per channel. The counting of samples was usually preceded by system calibration with a standard Cs-137 (662 keV) and Co-60 (1173- and 1332.5 keV) source. The spectra acquired from the counting were processed using a programme called “WISPAN”. This programme calculates background by integrating areas indicated by the analyst on each side of the desired peak. This is then subtracted from the total peak area. The results were corrected for dead time, sample weight and half-lives. The programme has a built-in library of radionuclides. The analyst has to indicate the elements of interest to his investigation before the names and concentration of such elements are displayed if they are present in the sample whose energy spectrum is being analysed. Table 2 shows the elements of interest in this investigation with their nuclear characteristics.

Table 2: Elements of interest and their nuclear characteristics

Target isotope	Product Isotope by(n,γ) reaction	Half life ($t_{½}$)	Gamma (γ) ray Energy (keV)
^{23}Na	^{24}Na	14.96 h	1368, 2754
^{41}K	^{42}K	12.36 h	1524
^{55}Mn	^{56}Mn	2.58 h	846, 1810
^{164}Dy	^{165}Dy	2.33 h	95
^{26}Mg	^{27}Mg	9.46 min	1014
^{27}Al	^{28}Al	2.24 min	1778
^{45}Sc	^{46}Sc	83.81 d	889
^{48}Ca	^{49}Ca	8.72 min	3084
^{50}Ti	$_{51}Ti$	5.76 min	320
^{50}Cr	^{51}Cr	27.7 d	320
^{51}V	^{52}V	3.75 min	1434
^{58}Fe	^{59}Fe	44.5 d	1099
^{59}Co	^{60}Co	5.27 y	1173, 1332
^{64}Zn	^{65}Zn	243.9 d	1115
^{75}As	^{76}As	26.3 h	559
^{81}Br	^{82}Br	35.3 h	776
^{85}Rb	^{86}Rb	18.8 d	1076
^{121}Sb	^{122}Sb	64.8 h	564
^{139}La	^{140}La	40.3 h	1596
^{151}Eu	^{152}Eu	13.3 y	1408
^{152}Sm	^{153}Sm	46.3 h	103
^{159}Tb	^{160}Tb	72 d	879
^{174}Yb	^{175}Yb	4.19 d	396
^{176}Lu	^{177}Lu	6.7 d	208
^{180}Hf	^{181}Hf	42.4 d	5482
^{181}Ta	^{182}Ta	115 d	1221
^{232}Th	^{233}Pa	27 d	312
^{238}U	^{239}Np	2.36 d	277

Results and Discussion

Elemental concentrations for fourteen elements in twenty-one ceramic samples have been obtained. Qualitative information were established for Na, K, Mn, and Dy, through short-lived irradiation, and for Sc, Cr, Cs, Fe, Co, Rb, Eu, Th, U and Hf through long-lived irradiation. To find the similarity between the samples, a multivariate statistical package, MINITAB on Windows version 10, containing a body of techniques called "cluster analysis" was employed. Clustering of the samples was performed using WARD methods, as shown in figure 2. This method commences with the number of cluster equal to the number of samples and links these together until only one cluster remains. The criteria for linking clusters is minimization of the error sum of squares given by

$$SS = \sum_{\text{cluster (c)}} \sum_{\text{samples (i)}} \sum_{\text{elements (j)}} \left(L_{ij} - L_{ij(c)}\right)^2$$

Where , $L_{ij(c)}$ = mean value of j for (c) to which sample i is assigned. WARD's method calculates the squared distance from each point in a cluster to the centre and sums these to produce an "error sum of squares". The smaller and more tightly packed the cluster, the smaller the error sum of squares. Merging two clusters increases "SS" value, but the steps involving the smallest increases in SS are the ones considered by WARD's method (Oladipo, 1992)

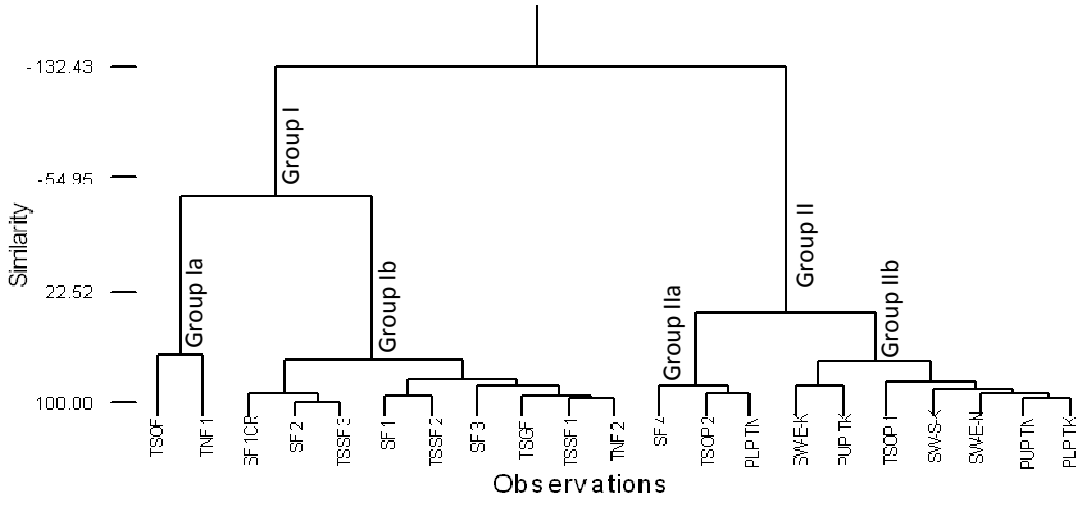

Figure 2: Dendrogram of furnace and potsherds

As can be seen in figure 2, the samples break into two major groups: I and II. Group I consists of only furnace samples, while group II consists of all potsherds with a furnace sample,

SF4. Group I can be sub-divided into groups Ia and Ib. Group Ia consists of only two of Tsauni samples (TSOF and TNF1). Either this group is an outlier or the two samples were made with clay of different origin. Group Ib is sub-divided further into two groups. Each of these is heterogeneous group consisting mixture of furnaces from Samaru-west and Tsauni (SF1 CR, SF2, TSSF3, SF1, TSSF2, SF3, TSGF, TSSF1, and TNF2). The group II that consist mainly the potsherds can also be subdivided into groups IIa and IIb. The similarity between these groups is however higher, compared to groups Ia and Ib. Group IIa contains, in addition to two potsherds, a furnace sample (SF4), which is an outlier. The remaining two potsherds are a mixture of two sites (TSOP2 and PLPTN). Group IIb is heterogeneous in composition as it contains potsherd samples from all the three sites. (SWEK, PLPTK, TSOP1, SWSK, SWEN,PLPTN and PLPTK). It can be deduced from figure 2 that there is no distinct differences between the potsherds used in each of the sites. This agree to some extent with Oladipo *et al* (1992) who showed that Pantaki samples cluster with both Samaru-west and Tsauni sample. Two of Tsauni furnaces show higher dissimilarity with the rest, while the remaining five show great similarity with each other and with Samaru-west furnaces. The implication of this result is that there was strong interaction between iron-smelters at these sites, as Effah-Gyamfi (1981) suggested. It is possible that some members of one of the site specialised in art of pot making and/or furnace making, and sell these to other communities that need them. This can explain why there could be great similarity between samples in different sites that are a few kilometres apart. It is however clear that the group of people making pot might be different to those moulding furnace, and use clay from different origin to that used for making the furnaces.

Conclusion

Using WARD procedures, Cluster analysis was conducted on samples of potsherds and furnace obtained from Samaru-west, Tsauni and Pantaki. The elemental concentrations in the samples were obtained by method of neutron activation analysis, using neutrons from a nuclear reactor. It was found that samples from the three sites are similar. There was dissimilarity however between potsherd and furnace samples. It was concluded that pot makers or furnace makers existed probably in one of the communities, whose primary occupation was to make and sell the product to others within and outside their communities. The pot makers are different from furnace makers. The pot makers obtained clay from common source. Similarly,

the furnace makers obtained clay from a common source, which however, is different from that of pot makers.

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