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Elemental Comparison of Bones of Red Sokoto (*Capra Hiracus)* and West African Dwarf (*Capra Aeagurus)* Goats for Production of Glazes

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Abstract

Elemental characterization of glaze materials has not been of scholarly concern, especially in Nigeria where studies have also excluded animal by-products for glaze derivation and glaze modeling. This study therefore elementally examined ashes of bones of Capra hiracus (Red Sokoto Goats) and Capra aeagurus (West African Dwarf Goats) and produced glazes with them with a view to providing scientific modeling of the glazes. Raw bone samples of each animal were separately collected processed into ashes and analysed using particle induce X-ray emission (PIXE) and Rutherford Backscattering spectroscopic (RBS) techniques. Thirteen elements Magnesium, Aluminum, Silicon, Cholrine, Phosphorus, Potassium, Calcium, Titanium, Copper, Zinc, Lead and Iron (Mg, Al, Si, Cl, P, K, Ca, Ti, Cu, Zn, Pb and Fe) were determined and based on which the ashes of each sample with adobe clay, whiting, borax and soda ash were successfully modeled into five glaze batches, applied on bisque wares and fired up to 1150 ^{o}C in a down-draught kiln. Undiluted ash samples gave white and off-white colours. Their mixtures with other materials gave sienna and brown-ochre colours with opaque effect. In other words, the final glaze effects of the ashes were opaque. Visually, WAD combination was most successful, though with pocket of crazing, crawling and peeling off hosts. Conclusively, it is the hope of this article, that its finding will provoke further studies on comparative examination of animal by-products for glaze formulation, theorization and practical application.

Keywords: Glazing, Elemental characterization, Glaze formulation, Nigeria, Animal by-products

Introduction and Review of Scholarship

Glaze formulation and derivation have been of concern in ceramics research practice in Nigeria. This has continually opened new grounds in glazes and glazes derivation. Animal by-products, including Red Sokoto and West African dwarf goat bones, have been mainly utilized in Nigeria as supplement in poultry feeds. The use of animal by-products in glaze derivation and formulation is nascent. Attention to elemental composition and concentrations of the materials are also nascent in the study of ceramics and its practices in Nigeria. This has hampered a clear understanding of the elemental composition of glazes derived from such materials with the consequential negative impact on glaze modeling. Against the foregoing background, this study produced ash glazes largely from the bones of *Capra hiracus* and *Capra aeagurus* and comparatively examined the elemental concentrations of the ash glazes. The study was conducted with the aim of determining the elemental composition of the formulated glazes with a view to providing scientific modeling of the glazes.

Literature materials on ceramic glazes and glazing are rich. The literature materials on glazing are generally on derivation and application of glazes from horticulture materials or byproducts. Green (1963), examined glaze and its theorem especially ash glazes and identified woods and vegetables and their ash glazes derivable possibilities. He consequently observed that the elemental composition range of ashes is very wide and varied in tree from season to season. He also highlighted innovative possibilities in glazes
Singer and Singer (1963) examined industrial ceramic traditions that considered shaped products of clay, glass, enamel and cements, hardened by heat. They noted clay, flint and feldspar, often referred to as triaxial of clay-feldspar-flint as basic raw materials essential for glaze derivation. Nokes (1968) noted

innovations in scientific apparatus fabricated from glass as novel. His book paid general attention to equipment and operations as they concerned physical properties of glass, detection of strain, bloom and de-verification, annealing, storing and cleaning glass.

Cardew's (1970) book portrayed dexterity of Nigerian indigenous potters which had been developed centuries before the introduction of Western wheels and glazes. His practice and studies brought to light, Ladi Kwali, the most outstanding local potter of her time with prowess in both traditional and Western pottery skills. Also mentioned were other areas in pottery such as sources of raw material, chemical analysis of glaze type and glaze formation among others.

Peterson (1992 and 1998) investigated embellishment or clay works with indentations and sometimes brush strokes of different coloured clays, known today as glazes, which he observed started long in human history. The studies further, observed that the development of porcelain clay body and glazes, pioneered by the Chinese, triggered further experiments in Europe, springing up outstanding glazes such as Delft, Luster and Majolica. On glaze improvisation, Peterson argued that anything that can melt at heat temperature can glaze.

Ogunsina (1997), in another effort examined maize sheaths as an alternative glaze oxide. The study observed that glaze materials are numerous. He presented chemical analysis of the ash oxide derived from maize sheaths, noting it as being dominated by silica, quartz and what he termed sheathpar derivable from soft calcium arguably synonymous to feldspar (soft rock) mined from the field.

Rhodes (1998) argued that advancement in man's standard of living may not have commenced without the knowledge in clay and firing. He noted bricks, tiles, drains and water pipes, dishes, bowls, cooking pots and sarcophagi as some of the contributions derived from clay. The book provides information on fundamental issues of origin of ceramics, its chemical and physical properties and preparation of glazes.

In his own case, Ogunsina (1992) conducted a general survey of craft, science, technology and craft of ceramics in Nigeria. His book provides information on some of the primary and secondary clay deposits in Nigeria and the prospects of making ceramic wares from them. It further provided information about formulation of glaze, the poisonous nature of lead glazes and suggestion on the use of grass ash and bone ash for glazes.

Fournier (2000) worked on virtually anything ceramics with instances in ash glaze, ash preparation, glass, glass former, glaze source and glaze mixing, among others. He further emphasized on the difference between window glass, glassware and ash. It consequently, observed that natural ash compounds can give unique results when applied as glaze and are better exploited in reduction rather than in oxidation firing. Akinde (2009) studied ash glazes derivable from fruit peelings sourced locally in the southwestern part of Nigeria. This was a culmination of scientific and artistic enquiries on ashes of plantain, pineapple, orange and sugarcane peelings chemically analyzed and applied as glazes.

Ologunwa, Akinbogun and Kashim (2013) stressed the need to salvage Nigeria's small scale ceramic industry from her perpetual dependence on imported glaze; particularly as it has to do with replacing opaque glaze derivable from tin and zirconium oxides with calcium, phosphorus and fluorine inherent in natural bone ash, which they argued abound in Nigeria and are accessible at little cost.
Willard (2015) examined that creative idea behind decorative techniques was primarily manipulation of real or illusory depth through juxtaposition of different textures, patterns, colours and proportions. He noted in his glaze layered decoration that, using different techniques and tools on the same glazed piece adds to its contrast; drawing inspiration from quilts. Willard further observed that, separate patches create

repeat patterns that ultimately become a complete and unified form. Its result when translated to clay as glazes, gives one the immediacy of working with colour in painterly ways.

Almamari (2016) examined ceramic textured matte glazes using Omani plant ash. He argued that plant ash is very useful for making unique glazed surfaces on pottery. He further noted that the organic elements found in plant ash supply potters with calcium, potassium, magnesium and sodium, which are essential for making unique glazes. In fact, these elements supply potters with the proportion of fluxes and hardeners and these materials are necessary for making glaze recipes.

Akinde's (2016) thesis was a comparative examination of strengths of glazes derived from horticulture and animal by-products comprising bran of cereals, pod of legumes, stone of tree fruits and bone of ruminants. These samples were collected, prepared into ashes and characterized for elemental contents. They are consequently batched and applied on wares as glazes firing to 1200 ^{o}C into glazes and their strength capacities were determined using universal testing machine (UTM). Comparatively, bones of ruminant stand out at maximum compressive stress of N25082.17.

Kalilu and Ajadi (2021) investigated derivation of glazes from cullet; a non-biodegradable waste that is hazardous, indiscriminately discarded and unutilized. Its solid waste challenge, poly-chemical oxides and low firing potentials were analyzed elementally and batched into glazes firing to maturity at 1100 ^{o}C. The study, apart from contributing to waste management, economic and industrial advancement, has a far-reaching effect on cleaner earth.

Benkacem *et.al* (2021) focused on composition of glazes from a local kaolin deposit of Djebel Debbagh, code-named DDI as a result of its natural abundance, low price and good chemical content which stood at 38.49 wt% for Alumina and 44.8 wt % for Silica. Two batches were formulated from DDI, applied and fired to 1250 ^{o}C. Their findings revealed the presence of Zircon and Quartz as the dominant crystalline components of the glazes. The degrees of whiteness were around 93.30 %, while water absorption coefficients were low. They therefore, advocated the adoption of DDI glazes in commercial sanitary wares application locally and possibly globally.

The foregoing indicates that scholarship had concentrated on derivation and application of glazes. Elemental characterizations of the materials explored for the production of glazes have however not been looked into. This indicates a significant gap in the body of knowledge concerning ash glazes in Nigeria. It also indicates that the scholarly and artistic efforts have largely excluded studies on animal by products for glaze derivation, especially in Nigeria

Materials and Methods

The main materials used are Red Sokoto goat and West African dwarf goat bones sourced from Ogbomoso and Oyo in Nigeria. Ogbomoso and Oyo townships are located in the vegetation transitional belt between the forest and the savannah areas of Nigeria. Both towns are important market hubs for agricultural produce. Raw bones (8.15kg) of West African Dwarf goat (*Capra aeagurus)* were collected at a cafeteria at Adenike area in Ogbomoso while 7.57 kg raw bones of Red Sokoto goat (*Capra hiracus)* were collected from another cafeteria at Sabo area of Oyo town (plates 1-2).

Plate 1: Red Sokoto Goat Bones
Mama T cafeteria, Adenike Area, Ogbomoso.
Photograph generated from the field research.

Plate 2: West African Dwarf Goat Bones
Olaiya Cafeteria, Sabo Area, Oyo.
Photograph generated from the field research.

The bones were sun dried and subsequently dehydrated. They were consequently, calcined at 350 ^{o}C in a kiln and the residue ground into powdery state. The ashes produced were washed by soaking separately in 15 litres transparent buckets filled with clean water. On settling down, their supernatant liquid water was decanted. After each decantation of the surface water, the containers were refilled with clean water, stirred rigorously and allowed to settle for several hours, (at least an average of five (5) hours) before decanting again. This operation was repeated as many times as possible until the soluble flux of the ash was relatively removed. The bone ash slurries (Plate 3) were thereafter sun dried. Details of the processes and weight changes of the collected animal bone samples are shown in Table 1.

Plate 3: Sieving with 160mm mesh
Photograph generated from the field research.

Table 1: Weight changes of collected sample in kilogram (kg)

Horticulture and animal by-products	Actual weight (kg)	Dry weight (kg)	Calcined weight (kg)	Weight after decanting (kg)
WAD goat bones	8.15	7.45	5.68	2.86
RS goat bones	7.57	6.91	5.10	2.64

The ashes were qualitatively and quantitatively analyzed for their elemental composition at the Centre for Energy Research and Development (CERD), Obafemi Awolowo University, Ile-Ife, Nigeria using particle induced X-ray emission (PIXE) and Rutherford Backscattering spectroscopic (RBS) techniques. Tandem accelerator machine was used for the particle induced X-ray emission (PIXE) and Rutherford Backscattering spectroscopic (RBS) techniques. The first phase of this exercise was preparation. It involved grinding of the samples in an agate mortar and thereafter pelletizing them with a mechanically powered compressing machine with dices to form thick tablet of 13 mm diameter without a binder. This is followed by the ionization and acceleration of the pelletized samples using PIXE technique for the West African Dwarf goat bone ash and the Red Sokoto goat bone ash and Adobe, while RBS technique was adopted for Borax, whiting and Soda ash.

The PIXE experiments were performed using a 2.5 mega electron volt (MeV) proton beam. The measurements were carried out with a beam spot of 4 mm in diameter and a low beam current of 3-6 nA. The irradiation was for about 10-20 minutes. A Canberra Si (Li) detector Model ESLX 30-150, beryllium thickness of 25µm, with full width half maximum (FWHM) of 150 eV at 5.9 keV, with the associated pulse processing electronics, and a Canberra Genie 2000 (3.1) MCA card interfaced to a PC were used for the X-rays data acquisition. With respect to the beam director, the sample's normal was located at 0^o and the Si (Li) detector at 45^o. The PIXE set-up was calibrated using some pure element standards and NIST geological standard, NBS278. This process is often halted by the absence of liquid nitrogen, a liquefied nitrogen gas housed in an aluminium chamber, primarily functioning as regulator and coolant to the accelerator during experimental extermination (Obiajunwa, 2015).

Laboratory experimental and studio application approaches were the methodologies used for this study. These processes are reflected in the flowchart in Figure 1:

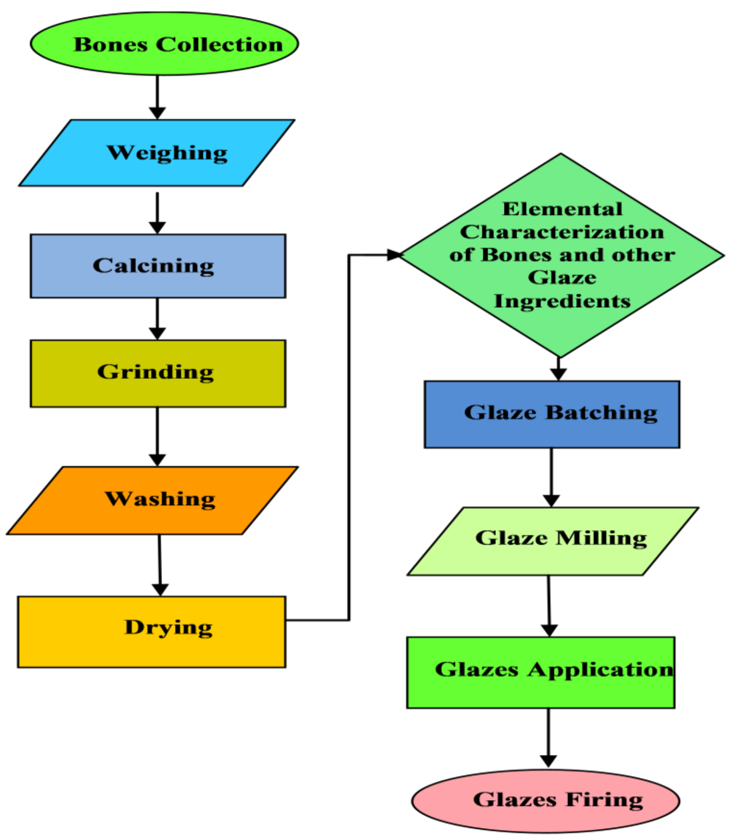

Figure 1: Laboratory Experimental and Studio Application Flowchart

Results and Discussion

Laboratory examination: The results of the laboratory examination of the animal bone ash and other samples are presented in Tables 2 to 7:

Table 2: Elemental concentration of West African Dwarf (WAD) goat bone ash

Element	Symbol	Concentration (ppm)	Oxides	Oxide Concentration (%)
Magnesium	Mg	8133.0 ±505.87	MgO	1.346
Silicon	Si	15617.6 ±432.61	SiO_2	3.341
Phosphorus	P	223068.3 ±624.59	P_2O_5	51.12
Calcium	Ca	348858.4 ±279.09	CaO	44.11
Iron	Fe	389.8 ±12.55	Fe_2O_3	0.056
Zirconium	Zr	132.6 ±11.50	ZrO_2	-
Strontium	Sr	330.2 ±35.60	SrO	-

Table 3: Elemental concentration of Red Sokoto (RS) goat bone

Element	Symbol	Concentration (ppm)	Oxides	Oxide Concentration (%)
Magnesium	Mg	5757.4 ±473	MgO	0.955
Silicon	Si	37507.6 ±439	SiO_2	8.025
Phosphorus	P	208419.4 ±625	PO_3	45.759
Chlorine	Cl	259.1 ±28	Cl_2O	-
Calcium	Ca	325323.6 ±260	CaO	44.519
Iron	Fe	312.5 ±11	Fe_2O_3	0.048
Zirconium	Zr	166.9 ±13	ZrO_2	-
Strontium	Sr	321.8 ±33	SrO	-

Table 4: Elemental concentration of Adobe clay

Element	Symbol	Concentration (ppm)	Oxides	Oxide Concentration (%)
Magnesium	Mg	2564.5 ±205	MgO	0.425
Aluminum	Al	129123.0 ±439	Al_2O_3	24.395
Silicon	Si	191125.9 ±363	Si_2O_3	40.891
Chlorine	Cl	315.7 ±55	Cl_2O	-
Potassium	K	4823.6 ±47	K_2O	0.581
Calcium	Ca	4106.6 ±38	CaO	1.221
Titanium	Ti	7317.8 ±46	Ti_2O	-
Vanadium	V	325.8 ±35	V_2O_3	-
Iron	Fe	105081.7 ±168	Fe_2O_3	15.025
Copper	Cu	583.3 ±115	Cu_2O_3	-
Zinc	Zn	1172.2 ±167	ZnO	-
Zirconium	Zr	287.0 ±57	Zr_2O	-
Lead	Pb	138.6 ±95	PbO	-

Table 5: Elemental concentration of Whiting

Element	Symbol	Concentration (%)	Oxides	Oxide Concentration (%)
Calcium	Ca	17.73	CaO_2	24.808
Carbon	C	12.62	CO_2	-
Oxygen	O	69.66	O_2	-

Table 6: Elemental concentration of Borax

Element	Symbol	Concentration (%)	Oxides	Oxide Concentration (%)
Sodium	Na	11.39 %	Na_2O	15.352
Boron	B	16.97 %	B_2O_3	-
Oxygen	O	43.77 %	O_2	-
Hydrogen	H	27.88 %	HO_2	-

Table 7: Elemental concentration of Soda ash

Element	Symbol	Concentration (%)	Oxides	Oxide Concentration (%)
Sodium	Na	33.54 %	Na_2O	45.208
Carbon	C	14.09 %	CO_2	-
Oxygen	O	52.37%	O_2	-

From the above analyzed West African Dwarf goat bone ash, Red Sokoto goat bone ash and adobe, a range of 7-13 elements were qualitatively detected by PIXE while 3-4 elements were detected by RBS from whiting, borax and soda ash. On the average, ten (10) major elements (Na, Mg, Al, Si, P, K, Ca, Ti, Mn and Fe) and seven (7) trace elements (S, Cl, Cr, Zn, Rb, Sr and Zr) were detected and quantified.

Glaze Batching and Milling: Batch is synonymous to recipe. It is an aggregate quantity of a group or collection of samples produced at one operation, forming a union or mixture of homogeneous particles which is synonymous to a solution (Hamer, 1975: 22). Technically, batch or batching in ceramics is the blending and grinding of glaze recipe through hand milling and ball milling or jar milling (Fournier 2000: 16 and 168).

In the ash batching exercise, the jar mill employed was electrically powered with maximum water capacity of 4 litres, programmed to run for an average of six (6) hours per batch. Red Sokoto goat bone ash, West African Dwarf goat bone ash, borax, soda ash and whiting were the samples for milling. All the samples were in dried states and in milling them, each batch sample was measured and weighed in part(s). This procedure was done one at a time. To start, the jar was disengaged from its horizontal base propeller, and its cover bolts and nuts were unscrewed. The jar was then filled simultaneously with whitish flint pebbles numbering precisely ninety-seven (97) pieces of the ash sample and one and half litres (1.5ltrs) of clean portable water. Thereafter, the jar was covered and screwed to be water tight, so as avoid spillage and then placed back to its motor and switched on for milling of the ash batch. The batches and their varying alterations are as shown in Table 8.

Table 8 shows batches weight, volume, percentage and sample combination in phases. For instance, the first phase was identified with its actual name; the second phase was tagged mixed, while the third and the fourth phases were acronym as in RSWAD ash; their percentages are as presented in Table 9.

Table 8: Composition of sole and combine samples batches 1-5

Batch 1 (**WAD ash**)				
	0.45 kg	13 parts	100 %	WAD goat bone ash
	0.45 kg	**13 parts**	**100** %	**WAD ash**
Batch 2 (**WAD ash mixed**)				
	0.46 kg	13 parts	50 %	WAD goat bone ash
	0.41 kg	9 parts	35 %	Borax
	0.14 kg	3 parts	12 %	Adobe
	0.07 kg	1 part	3 %	Soda ash
	1.08 kg	**26 parts**	**100 %**	**WAD ash mixed**

Batch 3 (**RS ash**)				
	0.09 kg	3 parts	100 %	RS goat bone ash
	0.09 kg	**3 parts**	**100 %**	**RS ash**
Batch 4 (**RS ash mixed**)				
	0.09 kg	3 parts	30 %	RS goat bone ash
	0.14 kg	3 parts	30 %	Borax
	0.07 kg	1 part	10 %	Adobe
	0.28 kg	3 parts	30 %	Whiting
	0.58 kg	**10 parts**	**100 %**	**RS ash mixed**
Batch 5 (**RSWAD ash**)				
	0.16 kg	4 parts	23.5 %	RS goat bone ash
	0.18 kg	4 parts	23.5 %	WADG bone ash
	0.28 kg	5 parts	29.4 %	Borax
	0.07 kg	1 part	6 %	Adobe
	0.23 kg	3 parts	17.6 %	Whiting
	0.92 kg	**17 parts**	**100 %**	**RSWAD ash**

Table 9: Sole and combine samples batches in percentage compositions 1-5

Batches no	**1**	**2**	**3**	**4**	**5**
WAD goat bone ash	100	50			23.5
RS goat bone ash			100	30	23.5
Borax		35		30	29.4
Soda ash					
Whiting		3		30	17.6
Adobe		12		10	6
Total	**100**	**100**	**100**	**100**	**100**

Kiln Firing and Visualization: The milled ash batches were applied on bisque ceramic ware by dipping. As the wares are dipped (plate 4) in the glaze with care, they were immediately exposed to dry in the open air. All the wares were allowed to dry before excess glazes were cleaned off their bases. For proper identification of the batches, each bisque ware was labelled, stalked and fired (plates 5-6).

Plate 4
Dipped bisque tile
Ceramics Studio, The Polytechnic Ibadan
Photograph generated from the field research.

Plate 5
Placing pyrometric cone in the kiln
Ceramics Studio, The Polytechnic Ibadan
Photograph generated from the field research.

Plate 6
Concurrent firing of the kiln
Ceramic studio, The Polytechnic Ibadan
Photograph generated from the field research.

The kiln was consequently, sealed up with bricks leaving two spy holes as windows into the kiln. This firing commenced at exactly ten twenty-four hours (10: 24) and halted at precisely eighteen fifty-five hours (18:55). Sintering is attained at 600 ^{o}C. This phase was subsequently followed by series of other melting processes until eutectic was reached, a thermodynamic equilibrium condition where a liquid coexists with two solid phases. Details of the various visual effects of firing on wares as observed are presented in Tables 10-12 and plates 7-11.

Table 10: Glaze firing trials 1 and 2 for RS Goat Bone Ash sole and combine mixtures

Glaze trial log for WAD Goat Bone Ash				
Test no	*1*		*2*	
Recipe	WAD goat bone ash	100	WAD goat bone ash	50
			Borax	35
			Adobe	12
			Soda ash	3
Test Piece	Small vase and two tiles		Small vase and two tiles	
Test Surface	Plain and impressed		Plain and impressed	
Glaze Thickness	Thick		Thick	
Kiln and Firing	Down draught kiln, 991^{o}C-1120^{o}C		Down draught kiln, 991^{o}C-1120^{o}C	
Type	Opaque		Opaque	
Colour	Off white		Umber	
Surface	Non-shiny		Non-shiny	
Notes	Glaze fit though peels		Glaze runs with pocket of cracks	
Assessment	Good		Good	
Alterations	Needed		Needed	
Name	WAD bone ash glaze		WAD bone ash glaze mixed	

Plate 7
WAD goat bone ash
Photograph generated from the field research

Plate 8
WAD goat bone ash mixed
Photograph generated from the field research

It was observed that both samples fitted on the wares with opaque results. The 100% WAD gave off white colour while mixed WAD yielded umber colour. They both had opaque effects but cracked. This result may be improved by adding more flux and fire at a stone ware temperature of 1200 ^{o}C.

Table 11: Glaze firing trials 3 and 4 for RS Goat Bone Ash sole and combine mixtures

Glaze firing trial log for RS Goat Bone Ash				
Test no	*3*		*4*	
Recipe	RS goat bone ash	100	RS goat bone ash	30
			Borax	30
			Adobe	10
			Soda	30
Test Piece	Small vase and two tiles		Small vase and two tiles	
Test Surface	Plain and impressed		Plain and impressed	
Glaze Thickness	Thick		Thick	
Kiln and Firing	Down draught kiln, 991^{o}C-1120^{o}C		Down draught kiln, 991^{o}C-1120^{o}C	
Type	Opaque		Opaque	
Colour	Off white		Raw sienna tint	
Surface	Non-shiny		Non-shiny	
Notes	Glaze fit and run well		Glaze fit and run well	
Assessment	Good		Good	
Alterations	Needed		Needed	
Name	RS bone ash glaze		RS bone ash glaze mixed	

The samples below 100% and mixed Red Sokoto glazes run and fit on their hosts. They however, gave opaque effect and their colorations were off white and raw sienna respectively.

Plate 9
RS goat bone ash
Photograph generated from the field research

Plate 10
RS goat bone ash mixed
Photograph generated from the field research

Table 12: Glaze firing trial 5 for RS and WAD Goat Bones Ashes combine mixture
Table 13: Trials 5

Glaze firing trial log for RS and WAD Goat Bones Ash		
Test no	*5*	
Recipe	RS goat	23.5
	WAD goat	23.5
	Borax	29.4
	Adobe	6
	Whiting	17.6
Test Piece	Small vase and two tiles	
Test Surface	Plain and impressed	
Glaze Thickness	Thick	
Kiln and Firing	Down draught kiln, 1120^{o}C	
Type	Matt	
Colour	Cream/Brown tint	
Surface	Non-shiny	
Notes	Glaze runs with pocket of cracks	
Assessment	Good	
Alterations	Needed	
Name	RSWAD ash glaze	

Plate 11
RSWAD bone ash glaze
Photograph generated from the field research

The above glaze effect was opaque though it appeared matt. RSWAD coating results crazed and peeled off its bisque body. The latter may be improved by adding more binders to the batches and fire at below 900 ^{o}C.

Conclusion

Glaze functions are basically to add colour, shine, texture and strength values on earthenware. Compounding and formulating glazes were age long tradition, explored up till this present time with more innovating conversions from plant leaves, fruit peeling, animal shells, and bones. In this study, the elemental comparison of bones from Red Sokoto and West African Dwarf goats was made for possible glaze production. WAD has five (Mg, Si, P, Ca and Fe) and Red Sokoto has five (Mg, Si, P, Ca and Fe), respectively. The final glaze effects of the ashes were opaque. Evidently, WAD combination appeared the most successful, though they crazed, crawled and peeled off their hosts. Consequently, it is hereby recommended that further studies on comparative examination of animal by-products for glaze formulation, theory and practical application be carried out.

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