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Physicochemical Analyses of Feldspar Ceramic Glazes Produced with Borax and Potash

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ABSTRACT
The significance of glaze in ceramics is beyond vitreous brilliant radiant beauty, it is a prerequisite to all finished ceramic wares. Feldspar glazes however, appears to be one of the most explored glazesparticularly in nations like Nigeria, perhaps abundance of these transparent gems.Feldspathic materials are under-utilized in Nigerian ceramic industries peradventure temperatures at which they arematured for glossy effects. And as such, study on fluxes capable of melting reduction is imperative. The study experiment on the derivation of ceramic glazes from feldspathic material; give a scientifically clear and concise description of the physical and elemental properties of the substances; and determine melting capacity after combined with flux samples. Field investigation, laboratory experimentation and studio practice were combined. Sample was collected from Ila-Orangun, Osun State, Nigeria; pulverized and sieved; subjected to Particle Induce X-ray Emission Technique for oxide capacities; and fired at 1100^{o}C and 1250^{o}C. The findings revealed the presence of silicon, aluminum, calcium, potassium, magnesium, iron, sodium, borate and phosphorous both in feldspar and flux samples of borax and potash though, at varying percentages. Their results were matt, melted and glossy after fired. Protagonism and restoration of the result will help in repositioning ceramic industries in terms of glaze formulation and production.

Keywords: Ceramic**,** Feldspar,Glaze,Particle Induce X-ray Emission, fluxes,Oxide capacities.

I. BACKGROUND TO THE STUDY

The resoluteness of ceramists cum chemists in material explorationshas proffered numerous solutions to universal challenges in arts, sciences and engineering. These exhaustive studies and explorations are obvious in ceramic glaze productions.Glazesresplendency has proven their requisite to finished ceramic wares. Glaze is a glass-like coat capable of viscosity and non-porous cover against liquid or oily materials on ceramic products. It provides hard wearing coating for ceramic wares after being melted in the kiln. Glaze covers ceramic wares with matte, glossy, coloured and textured effects to ensure easy cleaning. Ceramic processes however, showed nature and environment a product of explorations and experimentations. Dexterous of ceramists explicate proficiency of these universal materials. Although the quality of these materials having the right properties between locations cannot be fictitious, divergence has to be demonstrated to ascertain appropriateness for the production [1].Among the most copious group of minerals found on the earth's crust are feldspars, formed as a result of magna crystallize veins in igneous and metamorphic rock.

Feldsparsare group of hard crystalline minerals that consist of aluminum silicate of potassium, sodium, calcium or barium having the requisite qualities for glaze composition. Mostly, feldspars progressively contained higher degree of silica undersaturation [2]. Feldspars show broad range of compositional and structural characteristics that present rich information of their origin [3]. Feldspathic materials are transparent gems of different properties which are typically classified into potassium feldspars (sanidine, orthoclase, and microcline) and plagioclase feldspars (albite,bytownite, moonstone, oligoclase, andesine, labradorite and anorthite) [4].Pliability tendency of some of thesefeldspathic materials revealed array of new innovations in ceramic products.

Ceramics have moved beyond traditional clay materials to high substances that transmuteto objects of permanent, utility and beauty after vitrification which is the way to stabilizing inorganic raw materials. The process of becoming vitreous manifested in highly technology materials of refractory, glass and glaze [5].Glaze formulations at low melting temperatures are of concern in Nigerian ceramic industries [6] and this challenge is obvious in materials processing to the requisite metamorphose. No wonder glaze formulation maintained its artistic mystique even in the face of comprehensive scientific study and understanding [7].

Several scholars have carried out experiments on different materials for glaze and glaze formulation using substances of various recipes. [8][9][10] and[11] have documented the results of ash glazes derived from vegetables, fruit peelings and animal by-products. [12] [13] [14] [15]also analyzes fly ash of electric power generating station and blast furnace slag of granite rock as additive for glaze compositions. [16][17][18] [19][20][21] [22] and [23] studies physical and chemical properties of different glass types as possible materials for glaze derivations. It is apparent in their results that most of these materials possess qualities of glaze after transubstantiation.

II. STATEMENT OF PROBLEM

Glaze is not industrially produced in Nigeria and this is the background and inspiration for this study. Bulks of glazes using in Nigeria are imported and invariably firing at high temperatures. Often, some of these glazes threaten sustainable development as a result of carbon emission and energy consumption. Besides, many of low imported glazes could not provide better surface resistance against scratches and stress.Feldspars seems to be one of the most explored material for glazes apart from ashes, particularly in nations like Nigeria, perhaps it is due its abundance and accessibility. Under-utilization of these materials as glaze substances for Nigerian ceramic industry is still evident, even now. It is as a result of the temperatures at which they are melted. Hence, study on fluxes capable of melting reduction is imperative.

III. AIM AND OBJECTIVES

The aim of this paper is to derive low temperature glazes from feldspar and addition offlux recipes. The objectives are to give analyses on physical and chemical properties of studied feldspar as a possible material for glaze derivation; to ascertain suitable fluxes capable of feldspar melting reduction; and to determine melting capacity after addition of flux materials.

IV. MATERIALS AND METHODS

Methodology for this research combined three research components: field research, laboratory analysis, and studio artistic practice. Sample was collected fromfeldspathic rocks inIla-Oragun, Osun state, Nigeria. The collected samples of feldspar, borax and potash were spread in clean plastic trays to dry under sunlight in order to remove shards, stones and unwanted materials. After thorough desiccated of thesamples, they were sieved before pulverized with pulverizer machine (Rocklabs: model CRC3E) to obtain lowest particle size. Five grams (5g) of each pulverised samples were packaged in Petri dishes for the oxides analysis. Particle Induce X-ray Emission (PIXE) technique of 1.7 MV Tandem Pelletron (model: 5SDH) was used to determine sample oxides concentration.Thereafter, samples were subjected to studio artistic practice of formulation of glaze samples, batch-milling, clay collection and preparation for tiles production, application of glazes and glaze firing.

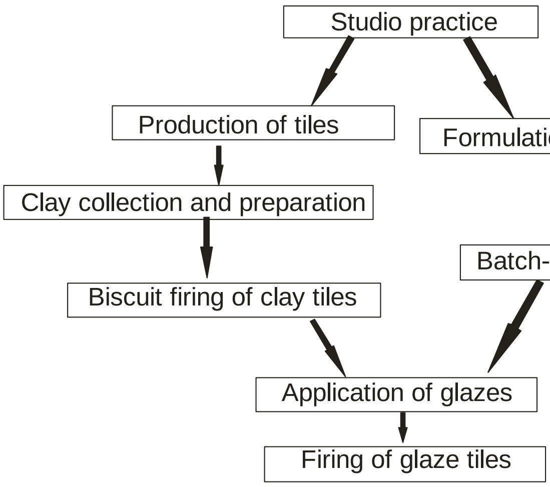

Figure 1

Flow chart of studio artistic practice
Source: Fieldwork: 2019

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V. DATA PRESENTATION

The feldspar sample was experimented with borax and potash in twenty-four (24) feldspar-ceramic-glaze trial phases with varying degree of these flux materials. The samples were carefully measured on electronic poyear digital scale to take into account the kilogramme (kg). Each experimented composition was batch-milled in Jar mill for average of two and half hours to get rid of immiscible droplets and bubbles. The millings were done in eight (8) phases of pulverized feldspar to each of the flux and in combination of the two fluxes. The batch-milled compositions were encoded with their samples combination. Initial alphabets of feldspar and flux materials (borax and potash) were used for batch sample labeling and easy identification: feldspar + borax ($FB_{1 \text{ to } 8}$) feldspar + $potash_{11}$($FP_{9 \text{ to } 18}$) and feldspar + potash + $borax_{22}$($FPB_{19 \text{ to } 24}$).The batch-milled composition samples are highlighted in simple addition ratio of percentage (100%) in Table 1.

Table 1: Batch compositions and Percentage ratios of feldspar to additive flux materials

Sample Batch compositions	Batch FB_1	Batch FB_2	Batch FB_3	Batch FB_4	Batch FB_5	Batch FB_6	Batch FB_7	Batch FB_8
Feldspar/Borax	60/40	65/35	70/30	75/25	80/20	85/15	90/10	95/05
Feldspar/ Potash	**Batch FP_9**	**Batch FP_{10}**	**Batch FP_{11}**	**Batch FP_{12}**	**Batch FP_{13}**	**Batch FP_{14}**	**Batch FP_{15}**	**Batch FP_{16}**
	60/40	65/35	70/30	75/25	80/20	85/15	90/10	95/05
Feldspar/Potash/Borax	**Batch FPB_{17}**	**Batch FPB_{18}**	**Batch FBP_{19}**	**Batch FBP_{20}**	**Batch FBP_{21}**	**Batch FBP_{22}**	**Batch FBP_{23}**	**Batch FBP_{24}**
	60/20/20	65/17.5/17.5	70/15/15	75/12.5/12.5	80/10/10	85/7.5/7.5	90/5/5	95/2.5/2.5

Source: Fieldwork: 2019

The selected temperatures for the glaze firing were 1100°C and conventional stoneware 1250°C. Three centimeters by five centimeters (3cm x 5cm) rectangular tiles with one to twenty-four (1-24) inscriptions at the back were designed for identification. The inscriptions represented each sample of the batch-milled compositions. Forty-eight (48) ceramic tiles were produced for feldspar ceramic-glazes application. These numbers of tiles were made for the twenty-four (24) batch-milled feldspar ceramic-glazes for two temperature trials of 1100°C and 1250°C. The trial temperatures were to determine suitable melting temperature for the batches. Surface textures for the two trial temperatures tiles were differentiated with smooth and rough. The first glaze coated tiles for the 1100°C was fired for twelve hours (12hrs); commencing at 8:32pm and stopped at 8:22am. The second firing of 1250°C commenced at exactly 12:00am and was halted at 3:15pm. The stoppage of each firing was based on the observed bent of the pyrometric cone for temperature accuracy. However, the differences in firing time in a simple sense justified the firing temperature.

VI. DISCUSSION OF FINDINGS

The Particle Induce X-ray Emission results of the feldspar, borax and potash used in this study expressed in oxide form is included in Table 2. The feldspar sample is rich in silica, aluminum, iron, calcium, sodium and potassium. Hence, devitrification of phases for this feldspar sample belonging to the Si-Al-Ca-Fe-Na-K system. Consequently, both borax and potash fluxes exhibit different chemical compositions, revealing that a melting differences are likely to be achieved. The chemical analysis of the feldspar is shown that the sample is a silica-based composition with aluminum and calcium. The concentration of the oxide components were analysed in the sum to 100%.

Table 2: Chemical analysis determined by Particle Induce X-ray Emission of Feldspar, Borax and Potash

Feldspar		**Borax**		**Potash**	
Oxides	Oxides Concentration(%)	**Oxides**	**Oxides Concentration(%)**	**Oxides**	**Oxides Concentration(%)**
Na_2O	1.96	Na_2O	32.33	Na_2O	29.0
MgO	0.24	MgO	0.22	MgO	0.20
Al_2O_3	16.24	Al_2O_3	0.14	Al_2O_3	0.16
SiO_2	62.72	SiO_2	1.82	SiO_2	6.61

P_2O_5	0.08	P_2O_5	0.02	P_2O_5	0.33
SO_3		SO_3	0.24	Cl	3.65
K_2O	18.37	B_2O_3	64.70	K_2O	58.20
CaO	0.27	CaO	0.47	CaO	0.35
FeO	0.12	Fe2O3	0.04	Fe2O3	0.12
PbO		PbO	0.03	PbO	1.31
Total	**100**	**Total**	**100**	**Total**	**100**

Source: Fieldwork: 2019

Four (4) parametric quantities were used for the visual analysis. The parameters were used to comparatively determine the effects of fluxes percentages and tri-trial kiln temperatures on the feldspar ceramic-glaze recipes. The factors that were used to define the visual qualities of thesefeldspar-ceramic-glazes behaviour in batches are the firing output qualities and physical characteristics of the glazes. The physical qualities of the glazes ranged from matt-bubbled, melted and glossy, melted and bubbled and bloated. These visual qualities are detailed in Tables 3 and 4. The tables revealed the kiln trial temperature results and surface quality of the glazes.

Table 3: Visual results of batch-milled compositions after fired at 1100^{o}C

Material compositions	Batch-milled samples	Matt-bubbled	Melt-glossy	Melt-bubbled	Bloated
Feldspar/Borax	FB_1 FB_2 FB_3 FB_4 FB_5 FB_6 FB_7 FB_8	FB_8	FB_6 FB_7	FB_2FB_3 FB_4	FB_1
Feldspar/Potash	FP_9 FP_{10} FP_{11} FP_{12} FP_{13} FP_{14} FP_{15} FB_{16}	FP_{16}	FP_{13} FP_{14}	FB_{11} FB_{12}	FP_9 FB_{10}
Feldspar/Potash/Borax	FPB_{17} FPB_{18} FPB_{19} FPB_{20} FPB_{21} FPB_{22} FPB_{23} FPB_{24}	FPB24	FPB_{21} $FPB_{22}FPB_{23}$	FPB_{19}	FPB_{17}

Source: Fieldwork: 2019

The surface morphology of feldspar ceramic-glazes formulated with larger percentage of borax was characterized by bubbles and bloats. These features are common to Batches FB_1, FB_2, FB_3 and FB_4 with hierarchical sizes of pores and cracks. Although cracks were more conspicuous in glazes production when host bodies did not agree with glaze composition, pore formation occurred less; crack formation may also be attributed to thermal stresses generated during high heat in the chamber. The result shows that, glazes formulated with high percentage of additive flux may demand low thermal conductivity in terms of heat expansion. However, if higher heat occurs during the melting-point in the kiln chamber, this will generate thermal stresses on the glaze and caused defects like bubbling and bloating. The result of batches FB_6 and FB_7 gave good glossy output and fitted well on the host bodies. Batches FP_{13} and FP_{14} also gave desirable result with smooth and glossy surface while FB_{16} and FB_{10} are characterized with bubbles and bloats. Intermixture of borax and potash appeared more effective on batches $FPB_{21,}$ FBP_{22} and FPB_{23} in the above glaze batches result, though batches FPB_{24}, FPB_{19} and FPB_{17}still exhibited unsought bubbles and bloats. Evidently, the results of batches mixed with 10 to 20 percents of flux gave good result at 1100^{o}C and compositions below or above these percentages exhibited defects. All the batches resistance to flow between solid and liquid after fired at 1100^{o}C appeared thick and fitted well on host bodies.The surface morphology of the devitrified $FPB_{21,}$ FBP_{22} and FPB_{23} of potash and borax intermixture samples shows uniform in crystallization with fine surface microstructure at 1100^{o}C. A considerable glossy effect was equally obtained in FPB_{19} except, bubble defects exhibited on its surface. Intermixed of the two additive fluxes in the glaze compositions improves both their thermal expansion and solidification at maturation phase.

Table 4: Visual results of batch-milled compositions after fired at 1250^{o}C

Material compositions	Batch-milled samples	Matt-bubbles	Melt-glossy	Melt-bubbles	Bloated
Feldspar/Borax	FB_1 FB_2 FB_3 FB_4 FB_5 FB_6 FB_7 FB_8	FB_6	FB_7 FB_8	FB_1	FB_3, FB_4, FB_5
Feldspar/Potash	FP_9 FP_{10} FP_{11} FP_{12} FP_{13} FP_{14} FP_{15} FB_{16}	FP_{12}, FP_{13}	FP_{16}, FP_{15}	FP_{14}	FP_9, FP_{10}, FP_{11}
Feldspar/Potash/Borax	FPB_{17} FPB_{18} FPB_{19} FPB_{20} FPB_{21} FPB_{22} FPB_{23} FPB_{24}	FPB_{21}, FPB_{22}	FPB_{23}, FPB_{24}	FB_{16}, FB_{17}	FB_{18}, FB_{19}

Source: Fieldwork: 2019

Morphology characteristics of all the glaze compositions prepared with ratio of 90:10 and 95:05 were somewhat similar in appearance at 1250^oC, although the micro-structural features were quite different in nature. Results of FB_7, FB_8, FP_{15}, FP_{16} and FPB_{24}were more successful in terms of viscosity and fusion. Bloats and bubbles appeared onFB_3, FB_4 FB_5 and FB_6 are symptoms of thermal stress in the kiln, causing compositions pulling tension after exceeding requisite temperature. However, the reaction of these glaze batches to the two selected kiln trial temperatures assist hydrokinetics nature of their crystallization phases in the kiln. By temperature comparison, thermal compatibility between glaze and requisite temperature is of great importance. Incompatibility of the heat with glaze recipe temperature cause unsought defects. Decrease in glossiness and viscousness occurred in batches FB_{12}, FB_{13}, FPB_{22} and FPB_{23} due to excessive and undesirable heat persistence on the compositions. The crack defects in the batches FB_{12}, FB_{13}, FPB_{22} and FPB_{23}can equally be colligated to higher percentage of chemical reaction of fluxes to the feldspar sample.Although their viscosities were good the resultant micro-cracks and pores may be improved upon by reducing flux quantity for easy flow during crystallization phase. This will increase melting flow of cracks at vitrification stage for opacity.

VII. CONCLUDING REMARKS

In conclusion, ceramic glazes on ceramic tiles have been formulated from feldspar and two additive flux materials of borax and potash using two different temperatures of 1100^oC and conventional stoneware 1250^oC. In the process, the percentage ratios of additive flux materials to the principal material (feldspar) were varied to ascertain suitable degree capable of low melting temperature, good crystallization and micro-structural effects. The compositions exhibit high glaze quality at ratios 85:15 and 90:10. The two additive fluxes gave good results at 1100^oC with sole mixture and intermixture of the two materials. Evidently, the practicalities and economic benefits of these two flux sample in the batches prove their suitability for industrial usage in the production of feldspar ceramic-glazes.

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