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Physicochemical Characterization of Cullet to Glaze for Environmental Sustainability and Entrepreneurial Development

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Abstract

Purpose: Inability of ceramists to formulate glazes locally and the cost of importing glazes had resulted to the closure of many cottage ceramic industries in Nigeria. The study, physicochemical characterization of cullet to glaze, was conducted with the aim of recycling cullet for glazing earthen wares and the intention was to conduct elemental analysis of waste glass for glaze recipes, determine its artistic usage, lessen cullet from environment and harness the chemical properties for industrial development and economic growth.

Methodology: Methodologies for the study involved laboratory analysis to determine oxides concentration in the cullet and studio artistic techniques to form glaze batches, batch-milling, glazes application on earthenware, and kiln firing. Four colours of windowpane cullet were used in order to determine their chromaticity effects as glaze. Cullet were pulverised with pulveriser machine (Rocklabs) and elemental analysis was carried out with tandem accelerator machine, using Particle Induce X-ray Emission to determine oxide compositions and concentrations.

Findings: Elemental analysis revealed Na₂O, MgO, K₂O, Al₂O₃, SiO₂, CaO, TiO₂, PbO and FeO. SO₃, P₃O₅, ZrO₂, Rb₂O, ZnO, MoO₃, Sb₂O₅, SrO and BaO in varying concentrations as the oxides in the samples. The glass ceramic glazes gave good matt, glossy, opalescent, translucent and opaque results at 950°C kiln temperature. The reactions of additive fluxes on each formulated glaze batch with windowpane cullet assist hydrokinetics nature of their crystallisation phases in the kiln.

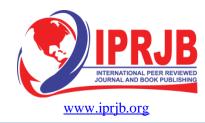
Unique Contributions to Theory, Practice and Policy: The study is hinged on the circular economy theory; an approach towards designing out of waste and regenerating natural system. It explicates artists' ideas in recycling cullet for glaze derivation and highlights recycling benefits of cullet for environmental sustainability and entrepreneurial development. The study enhances the understanding of ceramists on how cullet can be utilized for glaze production and contributes to the development of practical strategies and solutions for stakeholders in ceramic field. The adaptation will ameliorate the problem of unavailability and high cost of glazes; stimulate rapid development of the Nigerian ceramic industry and heighten economic growth of Nigeria.

Keywords: Glazes, Windowpane Glass, Entrepreneur, Environmental Sustainability, Elemental Analysis and Physical Characterization

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Vol.6, Issue 3, No.1. pp 1 - 12, 2023



INTRODUCTION

Environmental sustainability is one of the primal objectives of the Millennium Development Goals (MDGs). However, recycling complex products of cullet poses a challenge of right usage; a development that often causes environmental degradation and imminent hazard to its immediate community. Consequently, the inability of ceramists to formulate glazes locally and the cost of imported glazes had resulted in the closure of many cottage ceramic industries in Oyo State and Nigeria at large. Many of these outfits or centres were of the impression that compounding glazes locally is mystique. No wonder, Richard (2002) observed that glaze formulation maintains heightened value in the face of comprehensive scientific study and understanding. Nonetheless, readapting cullet properties to glaze recipes provide a focus for contemporary effort in addressing key issues in glazes derivation and to achieve Millennium Development Goals (MDGs) for environmental sustainability (Ajadi, 2019; Kalilu and Ajadi, 2021; Kalilu and Ajadi, 2023).

Waste glass is technically referred to as cullet. Cullet are fragments or worthless scraps of glass that melted down for reuse. According to Kalilu (2013) glazes are *sine qua non* as finishes in ceramic wares production. Glaze, a glass-like coat on ceramic products, provides hard wearing and non-porous cover against liquid or oily materials. Glaze makes ceramics wares matt, glossy, coloured, textured and easily cleanable. In the recycling process, cullet can be repeatedly used without any notable changes in their physical properties (Siikamaki, 2006; Kalilu and Ajadi, 2023) and could be considered as a substitute for glass former in recipes composition for ceramic glazes. Some glazes are formulated from frits as glaze slips and applied on the surface of clay body (Alavi, Mirhabibi, Van Landschoot, Van Landschoot, Kelder and Schoonman, 2009).

Glazes require a systematic composition, computation and recipe direction for proper chemical combination to prevent mistakes during formulation (Ajadi, 2021). Such directions will aid the attainment of requisite heat energy and positive behaviour of glaze on ceramic wares. However, glaze derivation has continued to pose serious challenges to intending formulators and users' alike; though, not for materials availability but for lack of requisite composition insight. A challenge that has led to the closure of many cottage ceramic industries in Oyo State and aside, the extant industries have limited their products to bisque ware despite the abundant ceramic raw materials in the state (Table 1). Also, some ceramic industries in the state are coating their ceramic products with gloss-paints in place of glaze recipes to render their wares impervious to liquids. This phenomenon came as the consequence of compositional problems usually encountered in glaze formulation. Meanwhile, no amount of gloss-paints can give sufficient scratchy resistance and provide surface characteristics such as brightness, colour and texture on wares as fired glazes.

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Ijebu Road, Ibadan

Ijebu Road, Ibadan

Ring Road, Ibadan

Monatan, Ibadan

New Adeoyo, Ibadan

Old Ife Road, Ibadan

Kosobo Area, Oyo

Fola Tyre, Oyo.

Orita Challenge, Ibadan

No 28, Lisabi Crescent,

University of Ibadan,

New Eruwa, Eruwa

Bolanta Area, Ogbomoso

Vol.6, Issue 3, No.1. pp 1 - 12, 2023

S/N	Cottage Ceramic Industries	Present Condition	Address
1	Akas Ceramic Industry	No longer in operation	Old Ife Road, Ibadan
2	Artias Ceramic Industry	No longer in operation	Old Ife Road, Ibadan
3	Asolom Ceramics	No longer in operation	Old Ife Road, Ibadan
4	Ediak Ceramics	No longer in operation	Asi-Basorun, Ibadan
5	De Craft Ceramics	No longer in operation	Asi-Basorun, Ibadan
6	Gbenga Ceramics	No longer in operation	Asi-Basorun, Ibadan
7	Sahara Ceramics	No longer in operation	Challenge Area, Ibadan
8	Red Clay Concepts	No longer in operation	Mabolaje, Oyo

No longer in operation

Extant (producing at minimal capacity)

Extant (producing at minimal capacity

Table 1: Condition of Small Scale Ceramic Industries in Oyo State

Source: Field Survey 2022

Boluwatife Pottery

Dapo Art Gallery

Sudit Ceramics

Sudef Ceramics

Ona Ara Ceramics

His Grace Ceramics

Alade Ceramic Industry

Fodacis Ceramic Entreprises

Saubana and Sons Ceramics

Musa Raymond Ventures

Women and Youth Art

Foundation (WY Art)

God's Grace Ceramic Industry

Aside from glaze derivation, the problem of inadequate and improper disposal of waste glass has become a major menace to environmental sustainability in Nigeria. Despite the fact that cullet composition is significant to glaze recipes in terms of chemical properties, non-utilization of these essential components for glaze derivation and industrial development in Nigeria is intriguing. Cullet are commonly disposed as landfills and are hazardous as a result of their non-biodegradable nature. Kalilu (2013) observed that such wastes could be harnessed for industrial development, as well as, to lessen environmental hazards. In recycling process, cullet can be repeatedly used without any notable changes in their physical properties (Siikamaki, 2006). Cullet could be considered as a substitute for glass former in recipes composed for ceramic glazes. In every practical sense, the pedagogical significance of converting cullet to suit glaze recipes for cottage ceramic industries will equally provide entrepreneurial development, crystallize the potential to boost the products of waste recycling industries and, consequently, lead to the nation's economic growth.

The study is the physical and chemical characterization of cullet to glaze for environmental sustainability and entrepreneurial development. It is aimed at recycling cullet for artistic usage, glaze production, environmental sustainability and entrepreneurial development. The study was conducted with two specific objectives. The first is to conduct physicochemical analysis of waste glass to formulate glass ceramic glaze recipes and determine its artistic usage. The second is to lessen cullet from environment and harness the elemental properties of this waste for industrial development and economic growth. For this study, soda ash lime glass was used and the glass is mostly made with composition of silicon dioxide, sodium oxide and lime. Glass Technology Services (2004) explains that a typical soda ash lime glass composition can be expressed in combination of silicon dioxide, sodium oxide, soda ash, calcium oxide and other oxide components with specific glass properties at low levels.

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Vol.6, Issue 3, No.1. pp 1 - 12, 2023



This study is hinged on the circular economy theory; an approach towards transformation or redesigning out of waste, ensuring products and material in use, and regenerating natural system. This theory account for production and consumption, which involves sharing, leasing, reusing, repairing, refurbishing and recycling existing materials and products as long as possible (Lacovidou, Hahladakis and Purnell, 2021). Circular economy became a symbol of progressive exploration and innovation to entwine new usages in complementary relationships with wastes and products. However, the study demonstrate the innovation in producing products from waste, explicate artists' ideas in recycling waste glass for glaze derivation and highlight recycling benefits of cullet for environmental sustainability and entrepreneurial development. The result gave reduction to the resources used; waste and leakage created, helps to reduce environmental pollution and connotes the implication of challenging artistic status quo in intellectual and methods of material derivation and ceramic production. Conceptually, the study serves as a revolution for ceramic industries with exploration of cullet as glaze material and enhances the understanding of ceramists on how cullet can be effectively utilized for glaze production. It will also contribute to the development of practical strategies and solutions for stakeholders in ceramic industry.

MATERIALS AND METHODS

Windowpane glass type is the major composite for the experiment. Four different colours: green, blue, brown and transparent white were selected for colouration. The rationale for the colour selection was based on comparative analysis of physical characterization, influence of glasses' colour on glaze production and thermal behaviour after kiln trial temperatures. Methodologies for the study involved laboratory analysis and studio artistic practice. Laboratory analysis was conducted to determine the oxides in the samples. Studio application was done to formulate, apply and fire the glazes

Cullet Collection and Processing

Samples were sourced from building construction sites and waste dumps of glass seller. Collected samples were drenched and washed repeatedly with clean water and cleansing agent (detergent) that emulsified and extinguished unwanted materials. Contaminants and impurities in cullet have great influence on glass properties and some of these effects, according to (Reynold, 2002; Dhir, & Dyer, 2004; Siikamaki, 2006) are linear discolouration, optical failure, achromaticity, disgorge, oxide reduction issues and vesicates. Thereafter, the samples were hammer-milled in separate colour before sieved 150nm mesh to further remove solid impurities that might have mixed with the cullets during the hammer-milling process.

To remove the remaining impurities, leftover hammer-milled samples in the mesh were soaked in water, washed and decanted before dried up under solar. After thorough drying, the samples were pulverised with pulveriser machine (Rocklabs) to obtain lowest particle size in a small mesh 350nm. To ensure purity of the powder, each pulverized sample was drenched again in five litres transparent buckets almost filled to the brim with clean water and stirred for minutes to ensure separation. The water drenched treatment caused matter separation energy, slight water foamy bubble and contaminants suspension.

Meanwhile, the pulverised samples fall slowly to stable position and settled at the bottom of the buckets. In the process, the water was a little impacted with the colour of the cullet samples as a result of residues interaction with water. The excess water was decanted after satisfactory washing, leaving the pulverised settled recipes in the containers. The washed pulverised samples were then poured and entangled in 500mn mesh, hanged free with rope for about five

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Vol.6, Issue 3, No.1. pp 1 - 12, 2023



hours to drain extra water before being spread in a clean plastic tray covered with thermoplastic for drying under solar. The statistical weight changes of collected sample in kilogram (kg) were taken before and after hammer-milling and after pulverisation and washing (Table 2).

S/N	Structural Typology	Colour Typology	Actual Weight (kg)	Weight After Hammer	Weight After Grinding	Weight After Washing	Weight after Measuring
1	Windowpanes		× 8/	Mill (kg)	(kg)	(kg)	Sieve
	glass (WPG)	Green	5.98	5.77	5.03	4.79	(kg)
		Clear white	8.25	8.07	6.77	6.43	4.78
		Brown	3.70	3.49	2.57	2.33	6.43
		Blue	5.00	4.86	3.99	3.76	2.33
							3.75

Table 2: Statistical Weight Changes of Collected Sample in Kilogram (kg)

Chemical and Elemental Analyses

The desiccated pulverised residues were packed in transparent plastic cans and stored in a cool place for oxides analysis. Oxides analysis of the selected samples were examined in tandem accelerator machine, using Particle Induce X-ray Emission to determine oxide compositions and concentrations. However, each sample was trial milled into eight (8) compositions with varying quantity of additive materials. Cullet with additive materials in compositions was thirty-two (32). The batch trials milled of a cullet sample were highlighted in simple addition ratio of ten (10) in (Table 3) below.

Table 3: Ball Mill Composition Analysis in Batch Samples Trial of Ratio Ten (10)

S/N	Batch Samples	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Trial 6	Trial 7	Trial 8
1	Cullet sample	9	7	7	7	7	7	7	7
2	Ball clay	1	1	1	1	1	1	1	1
3	Potash	-	2	-	-	1	1	-	0.67
4	Soda ash	-	-	2	-	1	-	1	0.67
5	Borax	-	-	-	2	-	1	1	0.66
	Grand total	10	10	10	10	10	10	10	10

Tiles with low numeric inscriptions of one to thirty-two (1-32) at the back were mould for the experiment. The inscriptions represented each sample batch mill compositions. These tiles were made for the thirty two (32) batch milled cullet glazes. The coated tiles with cullet glazes were subjected to kiln firing temperature of 950°C. A gas kiln powered by three burners was used for firing. The stoppage time was based on the result of the pyrometric cone for temperature accuracy.

RESULTS AND DISCUSSION

The oxide compositions from Particle Induce X-ray Emission results of each sample are highlighted in Table 4.



Vol.6, Issue 3, No.1. pp 1 - 12, 2023

Colour	Blue	Transparent white	Green	Brown	Bal	l clay	Bo	rax	Po	tash	Soda	ash
Oxides	Oxides Concentration (%)	Oxides Concentration (%)	Oxides Concentration (%)	Oxides Concentration (%)	Oxides	Oxides Concentration (%)	Oxides	Oxides Concentration (%)	Oxides	Oxides Concentration (%)	Oxides	Oxides Concentration (%)
Na_2O	13.70	13.85	13.18	12.79	Na ₂ O	1.06	Na ₂ O	32.33	Na ₂ O	29.08	Na ₂ O	98.70
MgO	3.17	3.24	3.37	3.10	MgO	1.48	MgO	0.22	MgO	0.20	MgO	-
$A1_2O_3$	0.26	0.18	0.25	0.57	A1,01	6.04	A1203	0.14	A1203	0.16	A1203	0.04
SiO_2	76.26	76.51	63.73	70.14	SiO ₂	81.46	SiO ₂	1.82	SiO ₂	6.61	SiO,	0.77
P_2O_5	0.09	0.06	0.07	0.22	P205	0.18	P205	0.02	P205	0.33	P205	0.00
SO3	0.32	0.34	-	0.39	SO3	-	SO3	0.24	SO ₃	-	SO ₃	0.13
K_2O	0.33	-	-	-	K ₂ O	1.74	B-03	64.65	K ₂ O	58.16	K ₂ O	-
CaO	5.56	5.55	4.94	11.72	CaO	0.49	CaO	0.43	CaO	0.33	CaO	0.07
TiO ₂	0.31	0.02	0.03	0.41	TiO_2	0.53	TiO_2	-	TiO_2	0.02	TiO ₂	-
FeO	0.26	0.09	036	0.30	FeO	6.45	FeO	0.04	FeO	0.12	FeO	0.02
ZrO_2	-	0.01	0.00	0.02	ZnO	0.01	ZnO	-	ZnO	-	ZnO	-
ZnO	-	-	-	0.09	ZrO ₂	0.09	ZrO ₂	0.04	ZrO ₂	-	ZrO ₂	-
Sb_2O_5	-	0.12	-	-	Sb ₂ O ₃	0.42	Sb ₂ O ₅	-	Sb ₂ O ₅	0.04	Sb ₂ O ₅	-
MoO_3	-	-	0.01	-	Rb ₂ O	0.03	Rb ₂ O	-	Rb ₂ O	-	Rb ₂ O	-
SrO	0.01	-	0.01	-	SrO	0.01	SrO	0.03	CI	3.65	CI	0.18
PbO	-	-	-	0.24	BaO	-	BaO	-	BaO	-	BaO	-
BaO	-	-	-	0.01	PbO	-	PbO	0.05	PbO	1.31	PbO	0.09
Total	100.00	100.00	100.00	100.00	Total	100.00	Total	100.00	Total	100.00	Total	100.00

Table 4: Oxide Composition of Pulverised Glasses and Additive Materials

Source: Fieldwork 2023

The concentration of the oxides components were analysed in percentage (100%). The principles of differences in oxides that exist in each cullet were used to determine chromaticity in oxides. Percentage oxides concentration that determined dominant chromaticity capacity in each glass sample was traced and quantitatively analyzed. The visual results of the cullet ceramic glazes after the 950°C kiln temperature are presented by the tables below. The discussion is based on the output results of the glaze compositions on the surface of biscuit tiles. The parametric qualities for the analysis are output colour, matured surface quality, composition evaluation and resistance to flow.

Table 5: Physica	l Analysis of	Trial Batches 1	to 4 after Kiln	and Firing
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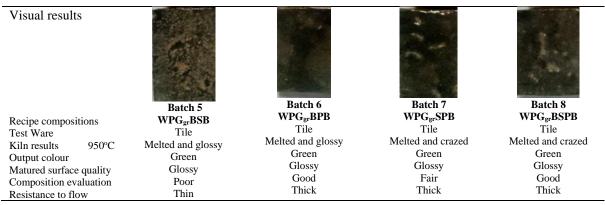
Visual results					
		Batch 1	Batch 2	Batch 3	Batch 4
Recipe compositi	ions	WPG _{gr} B	WPG gr BB	WPGgrBS	WPGgrBP
Test Ware		Tile	Tile	Tile	Tile
Kiln results	950°C	Matt	Melted and glossy	Melted and glossy	Melted and glossy
Output colour		Green	Green	Green	Green
Matured surface	quality	Glossy	Glossy	Glossy	Glossy
Composition eval	luation	Fair	Good	Good	Poor
Resistance to flow	w	Thick	Thick	Thick	Thick

WPG_{gr}B, the name given to Batch 1, is the acronym for composition of green windowpanes glass and ball clay. The composition resulted into matt without additive chemical flux and gave a transparent green colour effect. The glass glaze mixture requires fluxes for economic benefit in terms of melting capacity and glossiness. The result from the addition of borax (Batch 2) and potash (Batch 3) gave quick melting temperature at 950°C and good glossy surface output. Likewise, addition of soda ash flux (Batch 4) lowered in melting temperature but cracked and crazed the composition at both application and matured fired phase.



Vol.6, Issue 3, No.1. pp 1 - 12, 2023

Table 6: Physical Analysis of Trial Batches 5 to 8 after Kiln and Firing



Combination of borax with soda ash in Batch 5 improved soda ash reactions both in application and fired stage. The results of batches 7 and 8 had certain outward effects of crazing but melted, glossed and fitted well on their biscuit host. Some of these glaze batch compositions are glossy on their host bodies. Therefore, the conforming preciseness temperature for these compositions is 950°C. Addition of the three chemical fluxes in Batch 8 also gave positive effects on glaze residue and biscuit host after firing. Nonetheless, soda ash combination with other fluxes yielded a better result.

Table 7: Physical Analysis of Trial Batches 9	9 to 12 after Kiln and Firing
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The glazes from Batches 9 and 12 successfully run at selected temperature degree. Batch 9, (WPG_{bl}B), resulted in matt and gave opalescent blue on its biscuit ware at 950°C temperature, perhaps, due to the absence of additive chemical materials. Batches 10, 11 and 12 are compositions of cullet with additive chemical fluxes of borax, soda ash and potash. The batches fitted and ran well on their host bodies. Their quick melting capacities were based on additive fluxes in the compositions. However, the result of Batch 11 with soda ash mixed composition appeared with undesired micro cracks and bubbles on its host body as a result of hygroscopic and moisture absorbing propensity.



Vol.6, Issue 3, No.1. pp 1 - 12, 2023

Table 8: Physical Analysis of Trial Batches 13 to 16 after Kiln and Firing

Visual results				
	Batch 13	Batch 14	Batch 15	Batch 16:
Recipe compositions	WPG _{bl} BSB	WPG _{bl} BPB	WPG _{bl} SPB	WPG _{bl} BSPB
Test Ware	Tile	Tile	Tile	Tile
Kiln result s	Melted and glossy	Melted and glossy	Melted and bubbled	Melted and glossy
950°C	Blue	Blue	Blue	Blue
Output colour	Glossy	Glossy	Glossy	Glossy
Matured surface quality	Good	Good	Poor	Good
Composition evaluation Resistance to flow	Thick	Thick	Thick	Thick

Intermixture of two additive chemical fluxes appeared more effective as shown in the result of table 8 with batches 13-16. Though, the batches with soda ash still exhibited unsought cracks. The results of other fluxes were outstanding both in melting temperature and output effects. The combination of potash and borax aids quick response of their composition on the host body and matured at 950°C. All the batches were resistance to flow in their transformation of liquid to solid after firing. They appeared thick and their glaze effects were glossy. Pore formations on soda ash and potash mixed can be minimized through the increase in percentage of compoundable flux; perhaps in ratio 30:70 respectively in order to lower the elements that caused defects.





WPG_{br}B, WPG_{br}BB, WPG_{br}SB and WPG_{br}PB are acronyms from the composition of brown windowpanes glass with ball clay, borax and ball clay, soda ash and ball clay and potash and ball clay respectively. Their results gave opalescent brown effects. The WPG_{br}B resulted in matt after the kiln temperature. The other three batches gave quick response to melting temperature at 950°C and good flow resistance in terms of thickness. The cracks and pore formations that appeared on their surfaces may be attributed to the stress generated during thermal expansion and thermal compatibility with the host bodies.



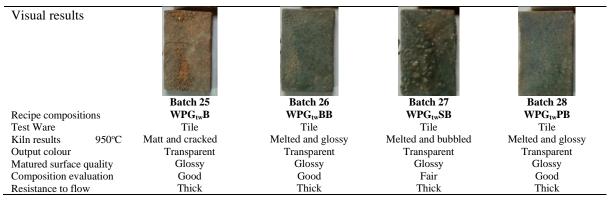
Vol.6, Issue 3, No.1. pp 1 - 12, 2023

Table 10: Physical Analysis of Trial Batches 21 to 24 after Kiln and Firing

Visual results				
	Batch 21	Batch 22	Batch 23	Batch 24
Recipe compositions	WPG _{br} BSB	WPG _{br} BPB	WPG _{br} SPB	WPG _{br} BSPB
Test Ware	Tile	Tile	Tile	Tile
Kiln results 950°C	Melted and crazed	Melted and glossy	Melted and bubbled	Melted and bubbled
Output colour	Brown	Brownish-green	Brownish-green	Brownish-green
Matured surface quality	Glossy	Glossy	Glossy	Glossy
Composition evaluation	Fair	Good	Fair	Good
Resistance to flow	Thick	Thick	Thick	Thin

The surface morphology of batches formulated with soda ash in table 10 was characterized by cracks. It is noteworthy however, that generally in glazing, cracks do normally occur when host body did not agree with glaze compositions. In the same vein, crack formation may also be attributed to thermal stresses generated during high heat in the chamber. Also, glazes formulated with low flux may demand low thermal conductivity in terms of expansion. If higher heat occurs during the melting period, this will generate thermal stresses on the glazes and caused defects like crazing and cracking.

Table 11: Physical Analysis of Trial Batches 25 to 28 after Kiln and Firing



The transparent windowpanes glass ceramic glazes appeared successful. Batch 25 was a composition from transparent white windowpanes glass at ratio 90% glass and 10% ball clay without additive flux. This composition matt-peeled and gave opalescent white colour effect. The surface result had underneath colour, perhaps the effects of the addition of geological material. The compositions of batches 26, 27 and 28 ran effectively on their host bodies at the selected temperature. The additive flux in each composition gave glossy effects.



Vol.6, Issue 3, No.1. pp 1 - 12, 2023

Table 12: Physical Analysis of Trial Batches 29 to 32 after Kiln and Firing



Mixtures of two chemical fluxes with pulverised transparent windowpanes glass as indicated in Table 12 were more successful in terms of viscosity and fusion. The mixture gave good result at 950°C. The micro cracks and crazes on Batch 31 are symptoms of hygroscopic reaction of soda ash causing composition pulling tension before and during firing. However, the reactions of additive fluxes on each formulated glaze batch with windowpanes glasses assist hydrokinetics nature of their crystallisation phases in the kiln.

Conclusion

This research has proven that low temperature glazes can be formulated from cullets with addition of borax, soda ash, potash and ball clay. The three additive flux materials in sole mixture of glaze composition and in combination gave good surface character and glossy quality at the 950°C. The paper has also successfully demonstrated the recycling possibilities of cullet for glaze productions. The usage of cullet for local production of glazes is recommended. Further research on more glass types is required to enlarge the scope of glass to glaze productions. The adaptation of the findings of this research in studio ceramic art practices will ameliorate the problem of unavailability and high cost of glazes, stimulate rapid development of the ceramic industry and heighten economic growth of Nigeria.

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Vol.6, Issue 3, No.1. pp 1 - 12, 2023

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