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DEVELOPMENT OF TRANSLUCENT PORCELAIN FROM KANKARA, NAHUTA AND NSU CLAYS, USING LOCAL FLUXING CONTENT

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ABSTRACT

Translucent porcelain products are broadly based on K_2O, Al_2O_3, SiO_2 triaxial diagram, but precisely on the silica-leucite-mullite (SiO_2, $K_2O.Al_2O_3.4SiO_2$, $3Al_2O_3.2SiO_2$) tenary diagram. To achieve desirable translucency, primary white-firing kaolin, quartz, SiO_2 (glass) and fusible flux, must go into some combination. For this work three Nigerian white/near white-firing clays; Kankara (Katsina State), Nahuta (Plateau State) and Nsu (Imo State), were used to produce translucent porcelain with completely local flux, (Okene potasuim feldspar). After all preliminary beneficiations and characterizations, seventy-seven (77) compositions were made and fired at four different temperatures, 1200^o, 1220^o, 1240^0 and 1260^0C. Out of five best group compositions, five (5) best final compositions, coded; A, B, C, D and E were selected. While A, B and C were based on the original clays D and E were best from their combinations. All three clays were found good for different types of porcelains, sole Kankara, for soft porcelain of 1230^0-1250^0C, while sole Nahuta was good for any translucent/non-translucent hard porcelain fired at 1300^0-1450^0C. Sole Nsu would serve best as additive to the other two because it's Fe content would not yield good translucent porcelains. Percentage translucencies of groups were: 1.1%, 1.5%, 5.8% and 2.9% for B, C, D and E groups, respectively, with average porcelain samples thickness at about 1.997mm. A two-way ANOVA for comparing porcelain sample thickness (AT), incident light (IL), transmission co-efficient (t_c) and transmitted light percentage (TL%), was used on generated data. Cor-relation co-efficient calculations and scatter diagram plots were also generated from the data. There were no significant differences between the average thicknesses (AT) of the polished porcelain pieces at 5% ($P > 0.05$). But there were significant differences at 5% ($P < 0.05$), between the values of the incident light (IL), the transmission co-efficient (t_c) and the transmitted light percentage (TL%). Also the negative trend in the cor-relation between average sample thickness (AT) and transmission co-efficient (t_c) and between (AT) and transmitted light percentage (TL%), showed that as the (AT) increased, both the (t_c) and the (TL%) decreased.

Key words: Translucent porcelain, local clays, local fluxes, local processing and development.

INTRODUCTION

Translucent porcelain products like many of the whiteware group of products; tableswares, vitreous sanitarywares, vitreous tablewares, bone china wares and some stone-wares are based on the triaxial compositional triangle, but more precisely on the silica-leucite-mullite (SiO_2, $K_2O.Al_2O_3.4SiO_2$, $3Al_2O_3.2SiO_2$) triaxial diagram (kingery et al, 1976).

For translucent porcelain, a classical composition must comprise some primary kaolin, some white-firing plastic secondary clay, together with feldspar and flint (glass). The clay serves the dual purpose of providing the fine particulate and important plastic materials. The feldspar acts as a flux, forming a viscous liquid at the required firing temperature and aids adequate vitrification. Finally, the flint (quartz) provides capillaries for easy drying, serves as a filler material during firing and remains unreactive at very low temperatures, but at high temperatures, forms a highly viscous liquid. Great differences in products are encountered, based on the relative amounts and kinds of clays, feldspars and quartz used. Problems encountered with plasticity or implasticity during product forming or the product non-whiteness or non-translucency as an end-product, are taken care of through a balance struck with the proportioning of the above starting

materials. Firing temperatures lie between 1230^0 – 1450^0C, or a little lower.

For a quality translucent porcelain, the fraction of the incident light which emerges as a diffuse transmission and the quality of diffuse reflection produced by internal scattering by the porcelain are important factors that enhance or assure its translucency quality. (Kingery et al, 1976). Inherent pore sizes and concentrations are also vital; they must be small in size and of very low percentage. This is so vital that a porosity as low as 3% reduces light transmission to as low as 0.01%. Porosities of about 0.3% have been found to enable samples yield only about 10% of the transmissions given by a completely dense (non-porous) sample (Bowen et al, 1976).

To increase translucency therefore, it is necessary to increase the glass content of the finished product, thereby decreasing the amount of mullite present in the fired product. An increase in the ratio of feldspar to clay, ensures a better translucency. This increases the amount of glass in the fired product, at the expense of mullite formed although it lowers the strength of the porcelain produced.

Historically it had been discovered in the early 18th century by the Europeans that the Chinese and the Far East people had an early knowledge advantage in making translucent porcelain. This was later seen to be because of the nature of their white-burning iron-free clay deposits. Thereafter, European nations began the research into the transluceny factor, especially since the industrial revolution. Taking Chinese better improved furnace technology and advanced works with white-burning kaolins back to about 600 AD, that date clearly far predates Europe's first porcelain manufacture by about 1000 years. This was so because during Chinese Han Dynasty of about 200BC, porcelain production had started in China and the best treasured porcelains belonged to the Sung Dynasty of 960-1259 BC.

The travels of Marco Polo and Vasco Da Gama's discoveries spurred the European research into porcelain composition far into early 1700^s AD. After several false attempts in France, Italy, Germany and Britain, the search ended in 1708/1709 with the German, Ehrenfried Walter Von Tschirnhaus and Johann Friedrich, Bottger's discovery, which Bottger formally reported to their financier, Augustus by March 1709, after the death of his co-researcher Tschirnhaus by October 1708 (Harry, 1992).

Thereafter William Cook-worth had a break-through in Britain, following his discovery of the still existing Cornwall China clay at Plymonth, in 1768 (Burton, 1906). All the above history notwithstanding, today the translucency factor is still a problem to many, even in advanced nations like U.S, U.K; with people still asking what "raw material to use" to achieve translucency and this is evident in today's internet. Most people keep asking what clay proportion to incorporate and what proportions of fluxing material? There are so many already confirmed clay sources and other materials in different parts of the world, as is the case here in Nigeria. What is needed in our own case is to start characterizing our clays and other raw materials, fluxes and silica deposits. However, researches into this area of the ceramic sector is yet to begin in earnest. This is because we do not as of now have ceramic departments in any of our universities or many trained ceramic engineers to go into this research area. Worse still, there are no government awareness, financial and physical provisions for such studies to take off. It is then at a time like this and in the above situation that this research effort was born.

MATERIALS AND METHODS:-

The materials for this research work were all obtained locally. The clays were, Kankara clay from Kastina Sate, Nahuta clay from Plateau State and Nsu clay from Imo Sate. Other materials include quartz (silica) and feldspar (flux), from Kogi State, including magnesium carbonate from Tagasinta in the North East of Nigeria. Yet other materials like sodium silicate (Na_2SiO_3), Plaster of Paris (P.O.P) and other laboratory equipment were located at the local markets and organizational laboratories.

Raw materials chemical analyses for this work were done by Plateau Minerals Development Company and Projects Development Institute (PRODA), Enugu. The initial raw

material firings were done at Science Equipment Development Institute (SEDI), Enugu, while the later firings were done with a G.K.4, 1300^0C kiln in Owerri, Imo State.

Primary and secondary comminutions of raw material, clay, quartz and feldspar were done in PRODA workshop, Enugu while the dry sieve analysis was done in the Nigerian Standard Organization, Engineering Lab. (SON); at Emene, Enugu. Finally all test samples at different stages of project designs were prepared with the standard specifications for preparing ceramic test piece for different parameter determinations.

Other additional equipment for this project were a table-top screw press for pressing the experimental porcelain thin test pieces for characterization and a wide measuring angle, high resolution Chinese digital lux (light) meter with auto-zero adjustment, (MODEL LX 1010B), and its separate powerfully configured light sensor; both purchased as a working unit.

RAW MATERIAL PREPARATION:-

The requisite raw materials had to be beneficiated and technically synthesized in order to upgrade them for incorporation into successful porcelain bodies. These steps included: primary and secondary crushing of the clays and other harder/additive raw materials, wet and dry sieve analysis of the clay and other materials, determination of the casting properties of the project clays, preliminary tests for colours, plasticities, fusibilities and dimensional stabilities at temperature increases, preliminary selective body composition for this work and even porcelain glaze formulation with these local raw materials.

Wet sieve analysis for the clays showed that only 44.38% and 55% pure clay material were recoverable from Kankara and Nahuta clays respectively, all others being composed of quartz and feldspar inclusion, organic matter and process losses. For Nsu clay the recoverable clay material was quite high, 97%, while only 3% went to process losses.

Dry sieve-shaker machine analysis for crushed and 12hours–milled quartz and feldspar with B.S.S requirements, showed that a large part of the particles, 10 to 80% did fall within the range of 90µm to 350µm for the quartz additive. For the milled feldspar 20 to 80% fell between 75µm to 300µm, so that the greater percentage of the particles used passed the 150µm, but remained above the 75µm sieve.

Of the three clays, Kankara came out most coarse, followed by Nahuta and fine-grained Nsu, hence Kankara provided the fastest but coarse/porous cast, followed again by Nahuta and slow casting Nsu clay. While the first two needed to be closed up for better cast product, Nsu needed to be opened up instead for the same purpose.

White–firing clays being strong pre-requisites for translucent porcelain bodies, investigation showed Kankara whitest among the three, followed by Nahuta, while Nsu was rather purplish white on firing and showed the highest fusion and shrinkage of 6.74% at just 1100^oC. Kankara clay mixed with Nsu clay was seen to fuse better than Nahuta mixed with Nsu.

PROJECT'S BODY COMPOSITIONS:-

In the technical upgrading stage of the raw materials, 28 compositions were made to cover purely starting clays and their several combinations, with choice emphasis based on these parameters:- whiteness, fusibility, shrinkages, densities, porosities, water absorption, physical ring on impact and other visual appearances.

For the second compositional stage, eleven out of the 28 compositions of the first stage, were previewed before seven (7) were finally chosen for actual classical porcelain compositions, which now involved the incorporation of quartz and feldspar.

Out of these incorporations, seventy-seven (77) compositions were made into three hundred and eighty-five

(385) test pieces, fired differently at 1200°C, 1220°C, 1240°C and 1260°C. Since random appearance of every composition in the four (4) firing temperatures were provided, a large number of good results for various porcelain types emerged in just five (5) firings.

Finally with a review of the characterized results of the seventy-seven (77) compositions, based this time purely on the porcelain translucency–sensitive parameters of; densities, porosities and water absorptions, five (5) final choice was made for the third and last stages compositions. The final compositions comprised, three best compositions made purely from the three (3) starting clays; Kankara, Nahuta and Nsu respectively and two (2) best compositions from their combinations. These were coded A, B, C, D and E. They were recomposed and fired at 1255°C to avoid bloating and overfiring noticed at the 1260°C, where sample A got overfired.

Table 1: Third stage compositions' fired results at 1255°C

Composition Codes	(%) fired shr	D_{as} (g.ml)	D_b (gr/ml)	P_{ap} (%)	W_{abs} (%)	At 1255°C
(1a 1.1) A	15.26	1.9	1.89	0.14	0.05	Over-fused/distorted
(2a 6.1) B	7.48	2.26	1.67	26.9	16.07	Still too porous/unacceptable
(3a 3.1) C	9.5	2.06	2.04	1.49	0.74	Acceptable range
(4b 3.1) D	9.99	1.92	1.89	1.6	0.78	Acceptable range
(10a 2.1) E	11.82	1.93	1.92	0.66	0.3	Acceptable range

Translucency parameter determination:

The transmission of light through translucent porcelain can be approximated by the following equation:

$I/I_o = t_c^x$ - - - - - - - - - - - - - - - (1)

So that the amount of light actually passing through that porcelain material can be approximated to:

$I = I_o\, t_c^x$ - - - - - - - - - - - - - - - (2)

This works out for a small range of thickness of porcelain where:

I_o stands for the intensity of the incident beam,

I stands for the fractional amount of incident radiation passing through the porcelain sample.

X stands for the thickness of the porcelain sample.

t_c stands for a constant of the material (sample), defined as the transmission co-efficient, being the ratio of the intensity of the incident bean and that fraction of the beam passing through the porcelain sample of unit thickness.

There are three methods of studying the translucency of real porcelain or human dental piece (Brodbelt et al, 1980; Johnson et al, 1996; Nicoleta Lile et Reinhard Hickel, July, 2008)

One method is the direct transmission method, the other is the total transmission method, involving an integrated scattering sphere and the third is using the spectral reflectance method. This work used the direct transmission method involving passing a light beam across a thin piece of translucent material and recording the amount of light passing through the sample to the other side of the material.

To do this study, thirty–five (35) thin porcelain pieces approximating to 3mm thickness by 30mm x 30 mm squares were pressed from a screw press. The total number of thin–sectioned squares pressed from the five (5) chosen porcelain compositions was one hundred and seventy – five (175) pieces. They were duely dried and fired to 1255°C, with 15 minutes soaking at the end. Next the thin porcelain squares

were vigorously polished, using rough to fine–finishing 600 grit silicon nitride papers. Best twenty–five (25) out of the thirty–five (35) pressed pieces from only four (4), out of the five A, B, C, D and E groups, were used for translucency determinations; unfortunately group A's thin porcelain samples again got overfired and distorted at the 1255°C final firing. While these thin pieces were used for translucency determination the other parameters in table 1 were determined with rectangular test pieces of 6cm x 1.5cm x 1.5cm dimensions.

The average thickness of the thin porcelain samples were obtained from three measurements along the 30mm side, using a caliper in close contact with the sample sides, after samples polishing. For the direct transmission measurements each polished sample was glued to the tip of 2cm wide by 8cm long cardboard paper sample–holder, with "top–bond" general purpose white glue. The hundred (100) fine–polished samples, twenty–five (25) each from sample codes: B, C, D and E used for translucency characterization are shown in table 2.

Table 2: Polished porcelain samples of codes B, C, D and E

S/N	Sample code	Average thickness (mm)	Sample code	Average thickness (mm)	Sample code	Average thickness (mm)	Sample code	Average thickness (mm)
1.	B4	2.18	C22	2.0	D22	2.13	E15	1.88
2.	B26	2.12	C2	2.5	D16	1.92	E26	1.94
3.	B22	1.91	C18	2.15	D24	1.92	E4	2.02
4.	B6	1.13	C21	2.12	D14	2.15	E21	2.02
5.	B14	1.8	C24	1.94	D4	2.0	E13	1.97
6.	B15	1.9	C8	1.9	D19	2.16	E2	2.04
7.	B8	2.11	C26	1.94	D6	1.91	E20	1.84
8.	B21	1.87	C15	1.93	D23	1.83	E25	1.92
9.	B17	1.87	C1	1.95	D3	1.82	E19	2.12
10.	B2	1.97	C3	2.03	D21	1.96	E16	1.94
11.	B24	1.83	C28	2.14	D9	1.95	E23	2.02
12.	B16	1.83	C10	2.15	D5	1.98	E24	2.15
13.	B5	1.97	C30	1.88	D1	2.13	E7	2.14
14.	B28	1.76	C6	2.30	D7	1.91	E8	2.04
15.	B20	1.93	C23	2.12	D15	1.92	E18	2.04
16.	B19	2.1	C12	2.00	D20	2.14	E22	2.19
17.	B18	2.02	C17	1.59	D26	2.0	E14	2.14
18.	B23	2.11	C9	1.74	D25	2.17	E10	2.08

19.	B25	2.11	C5	2.14	D30	1.92	E12	2.11
20.	B27	1.9	C11	1.9	D18	1.82	E1	1.93
21.	B7	2.11	C7	2.4	D17	1.83	E11	2.01
22.	B9	1.77	C25	1.93	D27	1.95	E17	1.94
23.	B13	2.0	C14	1.92	D28	1.96	E27	2.12
24.	B12	1.9	C13	2.01	D26	1.97	E30	2.03
25.	B30	1.87	C4	1.84	D29	1.98	E28	2.02

Porcelain sample characterization:

On switching the instrument's LED flashlights directly into the light sensor compartment, a quick taking of the digital wavelength (nm) reading was done. Then quickly slotting the porcelain thin sample into the instrument's sample slot, before the sensor compartment, a second digital reading (nm) was quickly taken too. Both readings were done so fast as to beat the digital light meter's prescribed <4 seconds sample–taking (reading) time.

By passing all the polished samples of the five groups through the configured digital light meter, all the data for calculating translucency–related properties were generated. These data comprise: incident light (Io) nm, transmitted light I (nm), transmission co-efficient (t_c) or (I/I_o) mm^{-1}, transmitted light % (TL%) and Naperian log of transmission co-efficient ($\ell n\ I/I_o$)

Table 3: Table of raw data for calculating translucency-related properties for sample D only.

Sample code	Average thickness (mm)	Incident light I_o (nm)	Transmitted light I (nm)	Transmission coefficient t_c $(I/I_o)_{mm}^{-1}$	Transmitted Light % t_c x 100	ln I/I_o
D22	2.13	354	17	0.05	5.0	-2.99
D16	1.92	340	24	0.07	7.0	-2.66
D24	1.92	359	20	0.06	6.0	-2.8
D14	2.15	363	16	0.044	4.4	-3.0
D4	2.0	368	22	0.06	6.0	-2.8
D19	2.16	342	18	0.053	5.3	-2.7
D6	1.91	342	22	0.064	6.4	-2.7
D23	1.83	357	22	0.06	6.0	-2.8
D3	1.82	251	21	0.084	8.4	-2.48
D21	1.96	335	19	0.60	6.0	-2.8
D9	1.95	355	22	0.062	6.2	-2.8
D5	1.98	328	18	0.055	5.5	-2.9

D1	2.13	352	17	0.05	5.0	-2.99
D7	1.91	340	22	0.065	6.5	-2.73
D15	1.92	357	20	0.056	5.6	-2.88
D20	2.14	353	17	0.048	4.8	-3.01
D26	2.0	364	20	0.055	5.5	-2.90
D25	2.17	339	17	0.05	5.0	-2.80
D30	1.92	356	20	0.06	6.0	-2.79
D18	1.82	346	21	0.061	6.1	-2.79
D17	1.83	354	22	0.062	6.2	-2.78
D27	1.95	351	21	0.060	6.0	-2.8
D28	1.96	353	22	0.062	6.2	-2.77
D26	1.97	324	19	0.058	5.8	-2.85
D29	1.98	355	20	0.056	5.6	-2.88

ANOVA and correlation co-efficient calculations:

A two way ANOVA at 5% and correlation co-efficient calculations, including correlation scatter diagrams, were done on the translucency data generated from the sensored digital light meter, for all groups: B, C, D and E, as showcased with only group E in table 4 and figs 1, 2 and 3.

Table 4: Correlation table for average thickness (AT) Vs transmission co-efficient (t_c), transmitted light percentage (TL%) and $\ell nI/I_o$ of sample E.

	Average thickness (mm)	Transmission Coefficient t_c (I/Io)	Average thickness (mm)	Transmitted Light % (t_c x100)	Average thickness (mm)	*L*n I/Io
Sample codes	X	Y	X	Y	X	Y
E15	1.88	0.033	1.88	3.3	1.88	-3.41
E26	1.94	0.028	1.94	2.8	1.94	-3.57
E4	2.02	0.030	2.02	3.0	2.02	-3.51
E21	2.02	0.021	2.02	2.1	2.02	-3.9
E13	1.97	0.027	1.97	2.7	1.97	-3.61
E2	2.04	0.026	2.04	2.6	2.04	-3.65
E20	1.84	0.032	1.84	3.2	1.84	-3.44

E25	1.92	0.038	1.92	3.8	1.92	-3.27
E19	2.12	0.025	2.12	2.5	2.12	-3.69
E16	1.94	0.028	1.94	2.8	1.94	-3.57
E23	2.02	0.029	2.02	2.9	2.02	-3.54
E24	2.15	0.031	2.15	3.1	2.15	-3.47
E7	2.14	0.022	2.14	2.2	2.14	-3.82
E8	2.04	0.030	2.04	3.0	2.04	-3.51
E18	2.04	0.025	2.04	2.5	2.04	-3.69
E22	2.19	0.027	2.19	2.7	2.19	-3.61
E14	2.14	0.031	2.14	3.1	2.14	-3.47
E10	2.08	0.028	2.08	2.8	2.08	-3.57
E12	2.11	0.025	2.11	2.5	2.11	-3.69
E1	1.93	0.029	1.93	2.9	1.93	-3.54
E11	2.01	0.028	2.01	2.8	2.01	-3.57
E17	1.94	0.038	1.94	3.8	1.94	-3.27
E27	2.12	0.028	2.12	2.8	2.12	-3.57
E30	2.03	0.031	2.03	3.1	2.03	-3.47
E28	2.02	0.03	2.02	3.0	2.02	-3.51

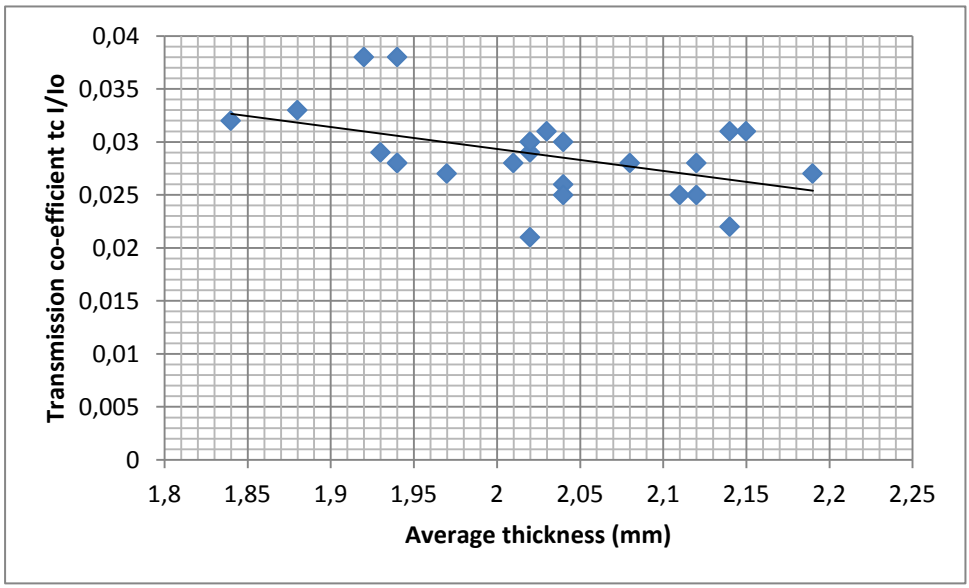

Fig. 1: Correlation co-efficient plot for average thickness (AT) Vs transmission coefficient (t_c) for sample E.

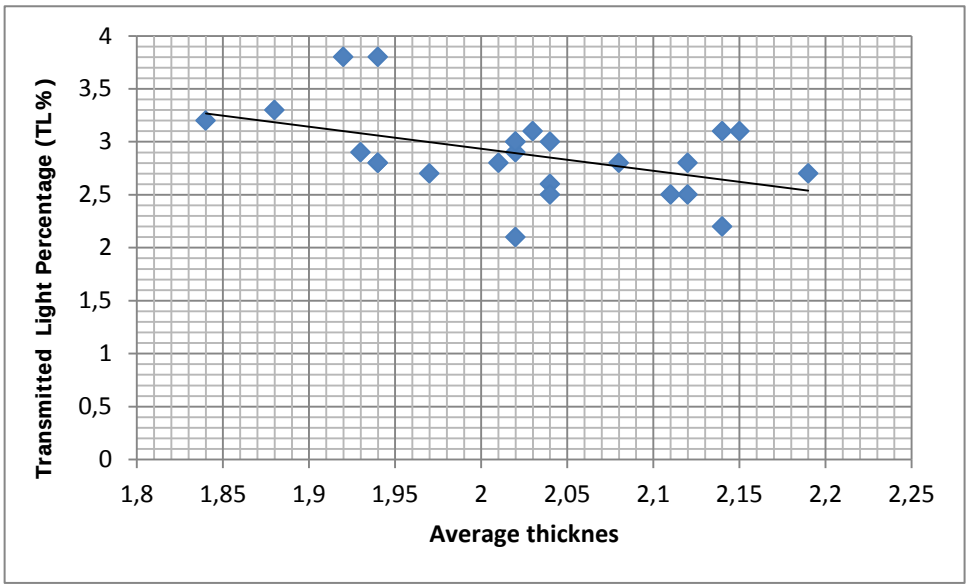

Fig. 2: Correlation co-efficient plot for average thickness (AT) Vs transmitted light percentage (TL%) for sample E.

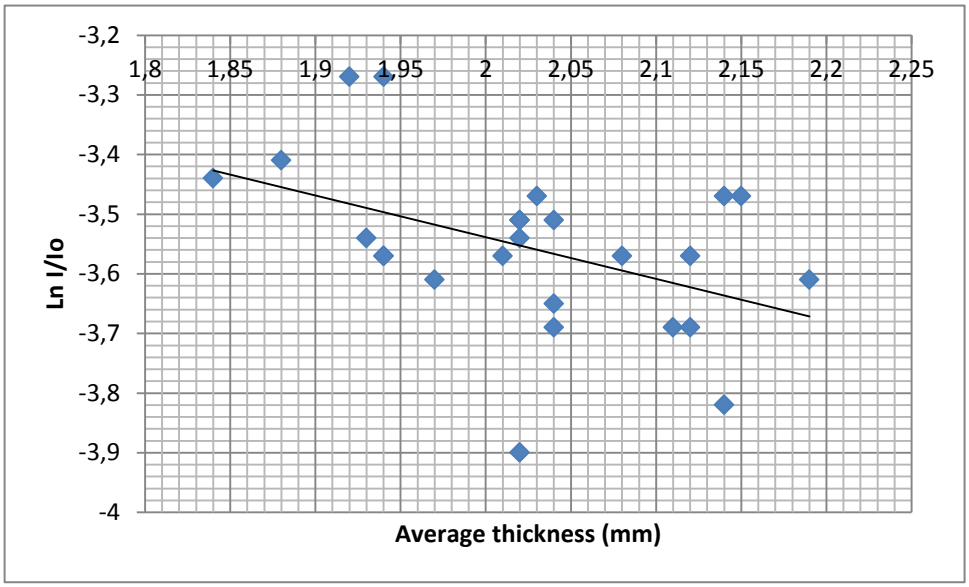

Fig. 3: Correlation co-efficient plot for average thickness (AT) Vs ($\ell nI/I_o$) for sample E.

Results and discussions:

Kankara and Nahuta clays were found to be requisite white–firing kaolinite clays, well suited for the production of translucent porcelains. While sole Kankara clay could be good for classes of soft porcelain, 1230° to 1250°C, due to its lower fusion temperature, Nahuta was found suitable for hard porcelains 1300° - 1450°C. Nsu clay on the other hand was of pinkish white colour and could only serve as plasticizing additive to porcelain compositions. Kankara clay was found to be probably the whitest presently known Nigerian kaolin.

For this reason it could find useful applications in other industrial uses for high quality paper-making, pharmaceuticals, bioceramics , ceramics, paints, plastics and rubber products.

Both Kankara and Nahuta clays were also good for any kind of whitewares, sanitary wares, stone wares, building material units etc.

The project's average transmitted light percentage (TL%) (translucencies) were as follows:

Sample A – test pieces overfired/distorted at 1255°C, sole Kankara clay.

Sample B – 1.1% (AT, 1.96mm), sole Nahuta clay, unmatured even at 1255^{o}C.

Sample C – 1.5% (AT, 2.02mm), sole Nsu clay, slightly overfired/ bloated at 1255^{o}C .

Sample D - 5.8% (AT, 1.98mm), kankara/Nahuta combination, good at 1255^{o}C.

Sample E – 2.9% (AT, 2.03mm), Kankara/Nahuta/Nsu combination, fair at 1255^{o}C.

Where AT stands for average porcelain sample thickness.

A two–way ANOVA was used to compare differences between average thickness (AT) of projects polished porcelain samples, the incidents light passed through them (IL), the transmission co-efficient (t_c) recorded from them and the transmitted light percentages (TL%) obtained through them.

Average thickness (AT) of porcelain samples were found not significantly different at 5% ($p>0.05$). However the incident light (IL), the transmission co-efficient (t_c) and the transmitted light percentage (TL%) averages were found significantly different at 5% ($p<0.05$).

The negative trends of the calculated correlation co-efficient between samples average thickness (AT) and transmission co-efficient (t_c) and between sample average thickness (AT) and transmitted light percentage (TL%) and even between (AT) and ($\ell nI/I_o$), indicated that as the average sample thickness increased, the (t_c), (TL%) and the ($\ell nI/I_o$) decreased as was evident in figs. 1, 2 and 3. This was quite in agreement with previous researchers results.(Brodbelt et al, 1980; Lee, 2006)

Conclusion:

Sole Kankara clay (A) porcelain composition could make most whitish translucent "soft" porcelain at a slightly lower firing temperature, or with some upward adjustment of its kaolin or slight reduction of either the feldspar content or the firing temperature. This was because of the clay's lower fusibility, due to its natural feldspatic (especially K_2O) gangue content.

Sole Nahuta clay (B) had the refractory potentials which made it less fusible and so could be adjusted for any type of porcelain, "soft" or hard even up to 1450°C. The composition of B sample was yet unmatured (unglassified) at the test temperature of 1255°C, hence its low translucency of 1.1%.

Sole Nsu clay (C) was so fusible for that porcelain test temperature due to its secondary clay nature, with highest Fe content, which shaded its ability to show whitish, and also caused bloating/porosity which negates translucency in porcelains by light scattering effects. Its translucency of 1.5% was just modestly hopeful.

But the combination of Kankara and Nahuta clays in sample (D), yield an encourageable result which in colour and other parameters could still be improved. Its 5.8% translucency was a good result.

Finally, the group (E), a composition with the three clays was equally fair, though not as good as sample D, even with its translucency approximating to 3.0%. It provided wide room for adjustments, because it suffered a little from slight overfluxing or overfiring, which encouraged open porosities with light scattering, impairing translucency. On the whole the three clays had useful contributions that work for successful translucent porcelain production.

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