DEVELOPING PYROMETRIC CONES FROM SILICATES OBTAINED IN NIGERIA

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Abstract
The bending of Pyrometric cone during firing indicates fusion temperature. Knowing this is useful in fuel and time management and, prevention of under or over firing. Efficient and accurate measurement of temperature within the kiln chamber is crucial to a successful round of firing. This study aimed at experimenting on the development of Pyrometric Cones, 7, 8, and 9 within the temperature range of 1230^0 C and 1280^0 C from silicate materials sourced around Nigeria. The standard Segar cone formula was adapted and the glaze formula recommended by Nosker was employed as a guide. The cone formulation was achieved using a constant proportion of feldspar and marble and a variation in the proportion of kaolin and quartz. Three rounds of firing were carried out to determine the fusibility of the formulated cones. The result of the test firing showed both the standard Segar cone 9 and the formulated test cone 9 bending almost at the same time. This provides convincing evidence that silicate materials from Bauchi, Kogi and Anambra states in Nigeria are viable for the development of Pyrometric cones
Keywords: Cones, Temperature, Feldspar, Blending

Introduction

Over the years, many heat measuring devices such as electric pyrometer with thermocouple, telescopic pyrometer, digital pyrometer, optical pyrometer, glass, liquid, and mercury and vapour thermocouples have evolved for use by both small and large industries for efficient heat measurement. Whilst pyrometer measures air temperature relationship within the kiln, the effect of heat also known as heat work on glaze materials can only be seen easily on Pyrometric cone. Pyrometric cones do not give the measurement of heat in the kiln but rather a measure of how much heat energy a ceramic material in the kiln has absorbed and the relationship between time and temperature (about.pottery.com, 2013).

Some of the earliest methods used for measuring heat work include the use of Bullers rings or Holcrofs (Daly, 1995).

The role played by Pyrometric cones in measuring the effect of the kiln atmosphere on a glazed being fired cannot be over-emphasized. Pyrometric cone is usually made up of refractory materials has a number inscribed on it; which corresponds to the bending temperature of that particular cone. These temperature ranges from 022 to 42 for Orton Cones (Sheffield Pottery, 2004) and 010 to 33 for Seger Cones (Segar Cones, 2000). Pyrometric cones provide assurances of control to the fireman, they are useful in monitoring soaking; a means of keeping the kiln at the same temperature for a

period of time. Soaking gives ceramic materials time to absorb more heat and the glaze to smooth out (Wallace, 2013).

Individual ceramicist, pottery workshops and even academic institutions in Nigeria have been depending on the use of test rings to determine the fusing temperature of glazes. This seems not to give an accurate measurement of the desired temperature; thus resulting in under or over firing of the kiln. Moreover, the slow progress from traditional low temperature to high temperature ceramics in Nigeria, in the authors view could also be attributed to the use of ineffective temperature measuring device.

The earliest method of measuring the maturity of glazed ware before the evolution of Pyrometric cones was the use of glaze draw rings; the old time potter learns to judge with surprising accuracy, using the colour of the pot as his indicator backed with test drawn from the kiln at intervals (Nelson, 1960, as Cited in Musoke, 1987). Temperature assessment has been determined either by using test ring which dealt with thermal change or the use of the Pyrometric cone which indicate temperature by bending and fusion

Laught and Voght were the first to attempt developing fusible Pyrometric cones, soon after a German named Hermann Segar of the Royal Porcelain factory Charlottenburg West of Berlin developed the first standard cone using the Segar cone system. Crammer and Hatch expanded the Segar formula to cover for lower temperatures and the developed the original cone temperature table in 1960 covering cones 01 to cone 022 (Abubakar, 2002), whereas Hofman in 1907 determined the fusion of cone 1 to cone 20 and extended it to cover cone 35 after two years and published temperature measurement of cone 2 to cone 17 and also studied cone 022 to cone 20. Moreover, Sosman pioneered the application of the laws of physical chemistry to the study of Pyrometric cones (Musoke, 1987). The manufacturing of cones in large scale came about after the development of a standard cone system by an American ceramic engineer (Musoke 1987)

Shipley formulated further experimental cone series in 1958, with an interval of 10^0 C + or - 20^0 C. which has the advantage of being introduced into hot furnace directly with a capability of uniform bending within a short period of time. Vickers and Theobald also manufactured Pyrometric cones by the addition of known ferruginous materials to their cone mixture, and observed that cones fired in air exhibited an increase squatting temperature with an increase in ferruginous material (Musoke, 1987).

In Nigeria, Musoke a Ugandan attempted to develope a fusible Pyrometric cones during his post graduate research work at Ahmadu Bello University Zaria in 1987, using local materials such as Kankara kaolin from Kaduna state, Jakura Marble from Lokoja in Kogi state, feldspar and quartz from Oguda an area along Auchi-Okene road in Kogi state (Abubakar, 2002).

Cones are very useful in determining the fusion temperature of either low temperature ceramics for decoration or high temperature ceramics for utilitarian use**.** Most firings done by either individual ceramicist or higher institutions of learning in Nigeria depend largely on the use of test rings and trial firing for assessment of temperature especially when they run short of temperature measuring device; this is a major problem for the advancement of ceramics in Nigeria. Other problems this study reflects include lack of fuel management, inefficient control and inaccurate assessment of the kiln temperature and over dependence on temperature measuring device.

In view of the importance of Pyrometric cones in firing, this study therefore focused on developing Pyrometric cones (7, 8 and 9) from silicate materials sourced from Bauchi, Kogi and Anambra, states to replace the use of test rings for temperature measurement and the over dependence on imported cones

The scope and objectives of this study include identification of suitable materials for the development Pyrometric cones, formulation of materials for Pyrometric test cones and determination of the fusion point of the Pyrometric cones. Moreover, this research focused on producing three Pyrometric cones 7, 8 and 9 with a bending temperature of 1230^0C, 1250^0C and 1280^0C respectively and the silicate materials sourced from the aforementioned geographical areas.

Materials and Methods

The constituent raw materials used in this study were collected from Bauchi, Kogi, and Anambra State respectively. Kaolin and quartz were collected from Gwaram villege in Alkaleri Local government area and Maryam ward in Tafawa Balewa Local Government Area all in Bauchi state. Marble was collected from Citizens Chemical Company Nigeria limited, Kilometer 2 Nkpobo-Obasi road Onitsha, Anambra State, while feldspar was collected from Okene in Kogi State. Okene feldspar and Maryam quartz were all named after the area they were collected from. Marble was procured and processed form from Onitsha and it is also called Onitsha marble. Feldspar, Kaolin, Quartz and Mable were the constituent raw materials used for the development of the pyrometric cones

Empirical method was adapted for the research beginning with the identification, sourcing/ procurement, beneficiation or treatment, compounding of materials and test firing. The constituent raw materials used in this research for the formulation of the Pyrometric cones passed through a systematic processing procedure using studio available tools and equipment such as lead cell digital scale for weighting, hammer and jaw crusher for reducing particle size, 121 micrometer mesh for sieving and bisque fired pot for drying. The processing of materials was carried to maintain, purity, particle size, and plasticity (Kingery, 1960, as Cited in Musoke, 1987). Feldspar was calcined for one hour; this is within the temperature range of 950^0 C to 1000^0 C and 925^0 C to 980^0 C, recommended by Petrie and Segar (1940) and the American Society for Testing Materials respectively. The powder obtained from the calcined feldspar was then milled for eighteen hours.

Marble was obtained in purified powdered form, whereas Quartz was calcined to a temperature of 600^0 C within 2hrs and reduced to smaller grain particles size. The reduced particles were jaw-crushed, sieved and soaked in water for 24hrs, after which the mixture was stirred and sieved again.

Materials were separately measured using a digital weighing machine “load cell” kitchen model 323 with a capacity of 5000g and graduation of 1g was used and mixed together and converted into cone batches. Each cone batch was soaked in water for twenty-four hours (24hrs) to enhance the intimate blending of the components, the mixture was stirred and sieved to substitutes for ball milling as recommended by Gukas in Musoke(1987). Standard Seger cone formula and Norsker glaze formulation were used as a guide and the cone batches were developed by increasing the refractoriness of the standard formulation; Swan glue was used as a binder to induce plasticity and leather hard strength

Excess water was decanted to enhance a slow and even drying process and to prevent warping and cracking, the formulations were placed on a bisque fired pot and later on a wooden board and dried

at room temperature ensuring even moisture content as recommended by Keey, (1972), as Cited in Musoke, (1987). The powder form was admixed with water and a binder (Swan Glue) was added to the paste to workable state. The paste was pressed into a wooden mould with the same dimension as described by the American Standard for Testing Materials (A.S.T.M. 484) using plastic spoon and polyethylene, placed in between the grove of the mould to prevent the cone from sticking to the walls of the mould as shown on plate 1 and 2. The formed cones were allowed to harden and removed from the mould together with the polyethyelene which served as a separator and dried at room temperature, while marble being obtained in powder beneficiated form was used without further treatment due to its high level of purity.

The glaze formula of 4 feldspar, 3 quartz, 2 whiting and 1 Kaolin as recommended by Nosker, (1987) was employed as a guide alongside the constant variation of kaolin and quartz for all the cone mixtures of 7, 8 and 9 as thus;

Cone 7 $0.3k_20$, $0.7Ca0$, $0.7Al_20_3$, $0.7SiO_2$
Cone 8 $0.3k_20$, $0.7Ca0$, $0.8Al_20_3$, $0.8SiO_2$
Cone 9 $0.3k_20$, $0.7Ca0$, $0.9Al_20_3$, $0.9SiO_2$

The Test for the efficiency of binding agent was done by mixing Evostick and swan glue and was tested for their binding efficiency. Kerosene fired kiln measuring 100 cubic meters with a capacity of four to six shelves was used for the firing. Three different sets of firings and arrangements with durations of three to six hours in a reducing kiln atmosphere were employed. The first round of firing lasted 4hrs, with Orton cone and the formulated cone placed at an angle of 82^0 to the horizontal on the same cone plaque made purely from kaolin (Fiq.1). The second round of firing lasted 5hrswith Orton cone placed close to the spy hole and the formulated cone placed towards the bag wall (fig.2.). The third round of firing lasted 6 hrs with a repeated arrangement of fig 1.

Discussion

The first round of test firing lasted four hours; by the third hour of firing, Orton cone had started bending while the formulated cones (7a, 7b, 7c) were still standing. Three hours twenty minutes (3hrs, 20min.) into the firing, Orton cone was down whilst the formulated test cones (7a, 7b, 7c) had started bending

By the fourth hour of firing, both Orton cone and the formulated cone (7a, 7b, and 7c) were down flat. Orton cone was down thirty-five minutes (35 min.) earlier than the formulated test cone and a close look at the formulated test cone shows a rough texture on its surface and this was attributed to poor sieving as shown on plate 3

The second test firing lasted five hours (5hrs); after three hours (3hrs) of continuous firing, both the standard Segar cone 9 and the formulated test cones were still standing. Three hours forty minutes (3hrs, 45min.) into the firing, formulated test cone 7 started bending at which time cone 8 and cone 9 and the standard Segar cone 9 were still standing; When the firing was four hours (4 hrs), formulated cone 7 was down, cone 8 had started bending while cone 9 and the standard Segar cone 9 were still standing. By the fifth hour of firing formulated test cone 8 and 9 and the standard Segar cone 9 were all down.

The third test firing lasted six hours (6hrs); halfway into the firing (3hrs), the three formulated test cones (cone 7, 8 and 9) and the standard Segar cone were all standing. When the firing was three hours and forty-five minutes (3hrs, 45min.), the formulated test cone 7 had started bending while the formulated test cone 8, 9 and the standard Segar cone 9 were still standing. Formulated cone 7 was down when the firing was four hours (4 hrs) at which time cone 8 had started bending while cone 9 and the standard Segar cone

9 were still standing. By the fifth hour of firing, cone 8 was down and both cone 9 and the standard Segar cone 9 had started bending

Five hours thirty minutes into the firing; the formulated test cone 9 was down together with the standard Seger cone 9.When the firing was five hours thirty minutes (5hrs, 35min.) formulated test cone 9 was down. The standard Seger cone 9 and the formulated test cone 7, 8, 9 had started fusing at the 6th hour of firing. The firing was then stopped.

The level (degree) of fusion of the Pyrometric cones was determined by the lustrous appearance of the cones. The more fusible the cone was, the glassy the appearance. Heat distribution within the kiln chamber greatly affects the bending (deformation) of the Pyrometric cones. Uneven circulation and distribution of heat within the kiln chamber delayed the bending of the cones

It was difficult to keep the kerosene burner at a constant pressure; as the pressure on the kerosene tank reduces, the kerosene-air flow increase, this had impacted on the heat intensity generated by the burner. To keep the pressure constant, therefore, air has to be pumped into the kerosene tank at an interval of ten minutes.

Glaze firing in the kerosene fired kiln lasted between five to six hours. The bending of the standard Segar cone 9 and the formulated test cone 9 at almost the same time provided convincing cone behaviours comparable with the standard cone batches. Although it was expected that the time difference between the falling of cone 7 and cone 9 is enough for cone 7 to melt, it was however not so and this was attributed to the quality (purity) of the constituent raw materials.

In the three sets of different firing arrangement, the standard cone was more lustrous in appearance than the formulated test cones; this was also attributed to the quality of the constituent raw materials as shown on plate 4

Findings

It was observed that cone batches mixed with Evostick, shows a lesser binding ability, the grain particles of the mixture were not held very strongly and when leather hard it crumbles easily; while cone batches mixed with swan glue shows a stronger binding ability; the grain particles of the mixture were held very strongly (cone less fragile). It was also observed that cone batches admixed with water only was very fragile and crumbled to powder, indicating a very poor handling property.

Cone batches admixed with a mixture of Swan glue and Evostick® exhibited almost the same characteristic as that admixed with swan glue. When the cones admixed with binders were finally test fired in a reducing kiln atmosphere, the cone admixed with swan glue did not bloat, burst, disintegrate or brake. The cones deformed at the temperature range of 1230^0 C to 1280^0 C after four hours of continuous firing. The result of the test informed the choice of Swan glue as the binding agent for the research

Conclusion

The standard batch formula of cones 7, 8 and 9 can be used successfully in the manufacture of cones 7, 8 and 9 using raw materials locally sourced. These raw materials kaolin, quartz, marble are available in Nigeria. A wooden mould can be successfully employed for moulding of Pyrometric cones. Intimate binding of the raw materials can be achieved in three hours. Jaw crushing using an iron based jaw crusher introduces iron components into the powder



Plate 1: Cone Formulation



Plate 2: Cone Mould



Plate 3: Rough Textured Cone



Plate 4: Segar Cone and Developed Cone



Plate 5: Feldspar and Marble



Plate 6: Quartz



Plate7: Formulated Cone 7



Plate 8: Formulated Cone 7 and 9

Table 1: Cones 7,8,9 Compositions

Cone 7 (g)	Cone 8 (g)	Cone 9 (g)
Feldspar 39	Feldspar 39	Feldspar 39
Marble 13	Marble 13	Marble 13
Quartz 52	Quartz 62	Quartz 72
Kaolin 26	Kaolin 31	Kaolin 36

Table 2: Result of first round of firing

Time(hrs)	Standard Cone	Result	Formulated Cone	Result
2 hrs	Orton cone 9	Standing	7a, 7b, 7c	Standing
3 hrs	Orton cone 9	Started bending	7a, 7b, 7c	Still standing
3hrs, 25min	Orton cone 9	Down	7a, 7b, 7c	Started bending
4 hours	Orton cone 9	Down flat	7a, 7b, 7c	Down

Table 3: Result of second round of reduction firing test

Time (hrs)	Standard cone	Result	Formulated cone	Result
2	Segar cone 9	Standing	Cone 7, 8 and 9	Standing
3	Segar cone 9	Still standing	Cone 7, 8, and 9	Still standing
3.45	Segar cone 9	Still standing	Cone 7	Started bending
	Segar cone 9	Still Standing	Cone 8 and 9	Still standing
4	Segar cone 9	Still standing	Cone 7	Down
			Cone 8	Started bending
			Cone 9	Still standing
5	Segar cone 9	Down	Cone 8 and 9	Down

Table 4: Result of third round of firing test

Time (hrs)	Standard cone	Result	Formulated cone	Result
2	Segar cone 9	Standing	Cone 7, 8, 9	Standing
3	Segar cone 9	Still standing	Cone 7, 8,9	Still standing
3.30	Segar cone 9	Still standing	Cone 7	Started bending
			Cone 8 , 9	Still standing
4	Segar cone 9	Still standing	Cone 7	Down
			Cone 8	Started bending
			Cone 9	Still standing
5	Segar cone 9	Started bending	Cone 8	Down
		Bending	Cone 9	Started bending
5.30	Segar cone 9	Down	Cone 9	Down
6	Segar cone 9	Started melting	Cone 7, 8 and 9	Started melting

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