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Experimental Evaluation of Local Agro Waste Using Modified Chapelle Test Method

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Abstract- This experimental study make use of the modified Chapelle test as a direct method for the determination of lime consumption in pozzolanic materials, in this case fonio husk ash is the pozzolanic material. The chemical test, performed during 16 hours at 90 °C, allows the quantification of Ca(OH)2 fixed by the fonio husk ash samples. The calcium hydroxide that was not consumed is quantified by hydrochloric acid titration, and the test result is expressed in mg of fixed calcium hydroxide by g of the fonio husk ash. According to the modified Chapelle test in respect to NFP 18 – 513 standard, for a material to be classified as highly pozzolanic, it must fix more than 700 mg of $Ca(OH)_2$ per gram of the material. The modified Chapelle pozzolanic activity of the seven fonio husk ash samples was experimentally evaluated between 720 and 1060 mg Ca(OH)2/g fonio husk ash. From the seven fonio husk ash samples produced between 650 ºC and 850 ºC, the FHA-850 sample has the highest modified Chapelle pozzolanic activity value of 1060 mg Ca(OH)2/g fonio husk ash, this is in agreement with some tested commercial metakaolin produced at industrial scale according to some established literatures. The fonio husk ash produced at 850 ºC resulting in a particle size reduction of ≈ 1.2% less and a consequent increase of 21 % in the pozzolanic reactivity. Therefore, Pozzolanic reactivity of the fonio husk ash produced at 650oC meet the standard minimum requirement to be used as a pozzolan.

Keywords: Fonio Husk, Fonio Husk Ash, Direct Method, Modified Chapelle test and Pozzolanic Reactivity.

I. INTRODUCTION

The pozzolanic activity of material is the ability to react with calcium hydroxide (He and Makovicky, 1995). According to Tironi, A., et al., (2012), the total quantity of calcium hydroxide with which pozzolanic material can combine depends on the nature and content of reactive phase in the pozzolan, SiO2 content of reactive phases, ratio of calcium hydroxide, the pozzolan and duration of curing. The rate of reaction of pozzolanic material with calcium hydroxide depends on the specific surface area of the pozzolan, water/solid ratio, and alkaline content in the Portland cement and temperature. The pozzolanic materials are generally high in SiO2 and Al2O3 and low in CaO, with or no reactivity when immersed in water. A wide range of test methods to assess the pozzolanic activity of materials have been developed. According to Vizcayno, C., et al. (2010), these methods can be categorized as either direct or indirect. The first evaluate the presence of Ca(OH)2, using analytical methods such as X-ray diffraction (XRD), thermogravimetric analysis (TGA) or classical chemical titration. The second ones measure a physico-mechanical property on a probe that indicates the extent of pozzolanic activity. This may involve measurement of properties such as mechanical strength, electrical conductivity or heat evolution by conduction calorimetry. Fratini (1950) pioneered in the development of a laboratorial methodology for the assessment of pozzolanic activity, based on the evaluation of the OH– and Ca2+ concentrations presented in a mixture of Portland cement and pozzolans.

The most common criteria to determine pozzolanic activity is its strength activity index according to the ASTM C311 Standard by applying the compressive strength test on a mix of cement-pozzolanic material. This test is indirect and does not give any information about the pozzolanic material itself, since the result depends on the chemical and physical characteristics of the cement. Since the C-S-H is the reaction product with lime, which improves the mechanical properties, a high content of silica is the most important parameter of the vitreous phase. Raask & Bhaskar proposed evaluating the fly ash quality by measuring the electrical conductivity of the dissolved silica content in hydrofluoric acid (0.1 M at 300°C, for 10 minutes) and the result inferred from a calibration curve plotted with silica fume. The authors admitted that in that concentration only the silica from the vitreous phase is solubilized. The most serious drawback to this method is that the authors did not prove that all silica present in the glass phase only was dissolved. Further, Majumdar & Larner also questioned the applicability of this method for fly ash Considering that lime consumption is an indicative of pozzolanic reactivity, Largent, R., (1978) proposed measuring the conductivity of a suspension of an opaline rock in saturated calcium hydroxide, stirring at 40°C. The conductivity decreased during the first 2 minutes then it stabilized. In the light of this result, this method was applied to a set of 100 natural materials, and a reactivity classification was proposed. The authors mentioned that during this period, the adsorption phenomenon is predominant, but did not seek to prove whether there was, in fact, early pozzolanic activity by chemical reaction. The method is therefore, questionable. However, Paya et al., (2001) applied this method with modifications, on different fly ashes and concluded that the results were affected by: the soluble salts, the fly ash: lime suspension ratio, and the method does not apply to high-calcium fly ash.

Vizcayno, C., et al., (2010), proposed to measure the resistivity of pastes of different materials with calcium hydroxide, steam cured at 70°C for 72 hours, determining the progress of the consumption of portlandite by XRD. No relationship was identified between the consumption of calcium hydroxide and the resistivity measurements.

The purpose of these methods was the speed which the pozzolanic activity could be determined, but in practice it did not happen. Surface phenomena interfere with these results, especially those relating to the high specific surface area of fly ash. In addition, the beginning of the specific reactivity for each pozzolan, it is highly important to determine the age at which the lime consumption is more significant, and the extension of the reaction contributing to the mechanical properties of cementitious materials.

The “Fratini test” is the basis of the actual EN 196-5 standard. In opposition to Fratini methodology, Chapelle (1958) developed a pseudo-dynamic test to evaluate the activity of a pozzolanic material, based on the consumption of Ca(OH)2 in a saturated water medium. Benoit (1967) improved the Chapelle methodology, particularly in terms of temperature (90 ºC) and duration (16 h) of the test, using 1 g of pozzolans, 1 g of calcium oxide and 100 ml of distilled water and gave the denomination “Chapelle test" to this methodology. Largent (1978) criticizing the “Fratini test”, because the system does not reach equilibrium and is performed in a static environment, introduced modifications to the Chapelle test, namely the continuous stirring during the duration of the test, and renamed the methodology to "modified Chapelle test". One of the most worldwide used pozzolanic material is the calcined kaolin (metakaolin), namely used as supplementary cementitious material. Tironi et al. (2012) and Fabbri et al. (2013) reported the influence of mineralogy, particle size distribution, moisture of the raw material, temperature and time of calcination, technique of calcination and the origin of the raw material (natural kaolinite clay or industrial kaolinite by-product) in order to obtain metakaolinized materials. This direct methodology (modified Chapelle test) performed in a aqueous lime-metakaolin system, during 16 hours at 90 °C with continuous stirring, allows the quantification of portlandite fixed by the metakaolin.

Literature proposes a few methods for fly ash, which provide an indirect determination of pozzolanic reactivity. However, to explain the hydrated cement composition and the microstructure resulting from the interaction with the calcium from the cement and the elements of the pozzolan vitreous phase, the best way is the direct determination of the interaction degree of pozzolan-calcium oxide or calcium hydroxide. In fact, the direct determination conceived by Chapelle is at present a Standard Method in France for metakaolin, and in Brazil for pozzolanic materials in general. In this work, direct method by modified Chapelle is adopted and the results of Chapelle's method applied to silica fume, fly ash, sugar cane bagasse and rice husk ashes, the effect were compared with the mass proportion lime: pozzolan of 2:1 as specified in Brazilian and French Standards.

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In France, the Chappelle's method was specified to select metakaolin for addition in concrete. The conformity criteria are: (a) the content of $SiO_2+Al_2O_3$ >90%, calcined between 600°C and 850°C; this result means that metakaolin has to be obtained from high quality kaolin; (b) the loss on ignition has to be <4.0%; compared to the theoretical ignition loss of pure kaolin, of 13.76%. Thus, we can conclude that the kaolin must have a transformation degree of at least 70%; (c) the specified calcium hydroxide consumption has to be 700 mg/g of metakaolin [700mg of $Ca(OH)_2$ is equal to 530 mg of CaO]. In this standard method, the reaction is one gram of metakaolin and two grams of lime. Chapelle's method was standardized too by ABNT - Associação Brasileira de Normas Técnicas (NBR 15895:2010), being applicable to natural and artificial pozzolans - calcined clay minerals, fly ash, silica fume and metakaolin but not to siderurgical slags. In fact, it is also not applicable to metallurgical slags, as granulated blast furnace slag.

Fonio husk ash as a Pozzolana

The XRF investigation conducted showed that the Plateau State Fonio husk ash is a pozzolana because of its significant quantity in chemical oxide composition which can be classified as natural pozzolana due to the fact that it is a calcination by-product of an agricultural waste, with the summation of SiO_2 + Al_3O_2 + Fe_3O_2 be >70%) according to BS 1881: Part 103 and ASTM C618-17, 2017. Like all pozzolans, fonio husk ash does not possess any cementing properties of its own, but it contains silica and alumina in reactive form. Ancient Romans produced exceptional cement by mixing pozzolanic materials with lime to build structures some of which are standing today (Antoni, M., et al., 2012).

By investigating the reactivity performance of fonio husk ash by means of modified Chapelle test, this study aims to contribute to the development of a sustainable environment by reducing the environmental impact of the construction industry through reducing waste material and decreasing demand for cement, as well a significantly reduces green gas emissions. This study could addresses a significant knowledge gap in the understanding fonio husk ash effects on concrete properties

II. MATERIALS AND METHODOLOGY

Materials

The materials used for this study include:

Fonio Husk ash

The Fonio husk from which fonio husk ash was obtained was locally sourced from mills within Barikin Ladi town in Plateau State of Nigeria. The fonio husk was calcined at ATBU industrial design workshop.

Lime (CaO)

The lime powder used in this study was purchased from Bauchi chemicals and Equipment store in Bauchi city, the Bauchi State Capital, Nigeria.

Water

Distilled water used in this study was obtained from department of Industrial Chemistry laboratory ATBU, Bauchi.

Table1: Fonio Husk Ash produced and used for this study.

Sample	Description
Fonio Husk Ash produced at 650 ºC	FHA-650
Fonio Husk Ash produced at 700 ºC	FHA-700
Fonio Husk Ash produced at 720 ºC	FHA-720
Fonio Husk Ash produced at 740 ºC	FHA-740
Fonio Husk Ash produced at 750 ºC	FHA-750
Fonio Husk Ash produced at 800 ºC	FHA-800
Fonio Husk Ash produced at 850 ºC	FHA-850

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The raw material (referred to as "Fonio husk") used in the production of the fonio husk ash was obtained from a small scale industry plant in Barikin Ladi town, the headquarters of Barikin Ladi Local Government Council in Plateau State, Nigeria. The fonio husk ash samples used in this study are presented in Table 1.

Seven fonio husk samples (referred to as "FH") were calcined from 650 ºC to 850 ºC to produced fonio husk ash (referred to as "FHA-650" to "FHA-850"). Overall, seven fonio husk ash samples were considered for analyses. The fonio husk was subjected to chemical and particle size determinations. The calcination of the fonio husk was performed at the Department of Industrial Design, Abubakar Tafawa Balewa University, Bauchi in a locally designed kiln, at a temperature of 800°C for 3hours to form fonio husk ash. The burning temperature was carefully monitored by using thermocouple. The chemical analysis of the major elements was performed by X-ray fluorescence (XRF) spectrometry using a PanAnalytical equipment in the Nigerian Geological Survey Agency Kaduna. The characterization of the obtained fonio husk ash samples (FHA-650 to FHA- 850) includes chemical and particle size analyses in accordance with the procedures spelled out in relevant standards described for fonio husk raw material. For the sake of statistical evaluation of the fonio husk ash pozzolanic reactivity. Response surface methodology technique was used to statistically estimate the effects of temperature, particles size and specific surface area on the pozzolanic reactivity of the fonio husk ash.

Measurement of Calcium Hydroxide Fixed Quantity by Modified Chapelle Test

The pozzolanic activity of the fonio husk ash was assessed with the modified Chapelle test according to NF P18-513 standard (annex A). Procedures spelled out in French Norm NF 18-513 was adopted to measure the fixed quantity of calcium hydroxide on the fonio husk ash which helps to establish the pozzolanic activity of the ash. Typically, the modified Chapelle test is standardized and performed in a lime–pozzolan aqueous system for 16 h at 90 ◦C with continuous stirring. In this process, 2 g of freshly calcined pure virgin lime (CaO) is used in a mixture with 1 g of the pozzolanic material in 250 mL of deionized water at 90 ◦C for 16 h on average. Concomitantly, a blank test is performed to compare the lime setting index using an equal container with only calcined lime and deionized water. After the thermal treatment, the mixtures should be cooled to room temperature and stirred in circular movements. The unreacted lime is then extracted by sucrose solution and titrate with a burette using the hydrochloric acid (HCl 0,1N) solution using the phenolphthalein as indicator. The titration reaction is

CaO + 2HCL ----------- CaCL2 + H2O

Ca (OH)2 + 2HCL --------- CaCL2 + H2O

Check of the blank reaction

56/2 x V1 must be under 1000

The Ca (OH)2 consumed was estimated by the below expression:

Mg of Ca (OH)2 fixed = 2 x (V1-V2)/V1 X 74/56 X 1000 ----------------- (1)

Where:

- V1 (ml) is the volume of 0,1N HCl necessary for the 25 ml of solution obtained by the blank sample
- V2 (ml) is the volume of 0,1N HCl necessary for the 25 ml of solution obtained by the reaction of fonio husk ash.

The result is expressed in mg Ca (OH)2 fixed per gram of the pozzolanic materials (fonio husk ash).

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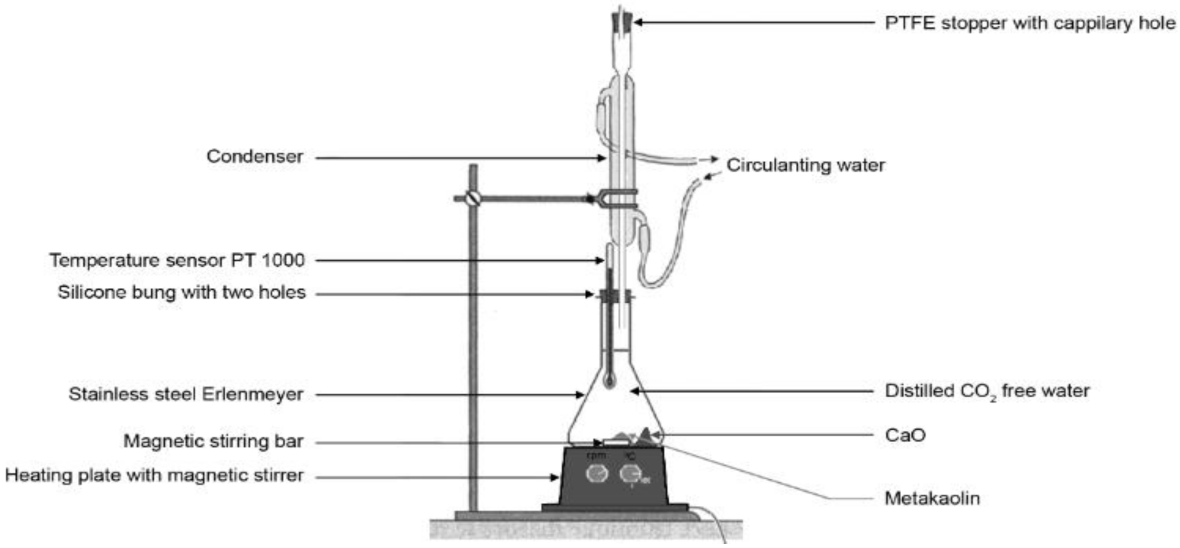

Fig. 1 Assembly of the modified Chappelle test equipment (adapted from NF P18-513, 2010).

III. RESULTS AND DISCUSSION

The chemical oxides of the fonio husk ash determined by XRF technique is presented in Table 2. The major oxides present in the Ash are Al2O3, 33.18% and SiO2, 59.05% in the ratio of 1.78, which are mainly in similarity with quartz and kaolinite minerals. The trace amount of other chemical oxides such as Fe_2O_3, 2.92%, K_2O, 0.3%, Na_2O, 0.4%, CaO, 1.28%, TiO_2, 0.3% and MgO, 1.23% are connected with the exchanged cations presented in fonio husk ash crystallochemical structure, as well as to the presence of muscovite (iron and magnesium) as a penalizing mineral. The loss on ignition of 1.09%, means the ash is well calcined with little mass is lost. This result has met the chemical oxides requirement defined by ASTM C618-17, 2017, and classified the Ash as class N pozzolan. Having Si/Al ratio of 1.78, there is likelihood that the paste to be prepared with this Ash could experience slow dissolution, stiff paste and low workability as observed by Usman N.D., et al., (2020), that a trial mix with Si/Al ratio >1.6 <2.0, the dissolution of the Ash was very slow and the resulting paste was stiff which will gives low workability.

Table 2: Chemical Oxides of the Fonio Husk Ash (FHA)

Properties	(Weight %)
MgO	1.23
SiO_2	59.05
Fe_2O_3	2.92
MnO_2	0.25
Al_2O_3	33.18
CaO	1.28
K_2O	0.30
Na_2O	0.40
TiO_2	0.30
LOI	1.09

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According to the modified Chapelle test result (Pozzolanic activity) shown in table 3, the FHA-850 and FHA-800 exhibit higher pozzolanic activity values of 1060 and 880 mg Ca(OH)2/g Fonio Husk Ash, respectively. The FHA-650 showed the lowest value of 720 mg Ca(OH)2/g Fonio Husk Ash). FHA-850 Fonio Husk Ash present the higher value of modified Chapelle pozzolanic activity. This material is classified as "high reactive fonio husk ash" in cementitious systems (Antoni et al., 2012). The NF P 18-513 standard defines that the pozzolanic activity of a pozzolanic material should not be less than 700 mg Ca(OH)2/g pozzolanic ash. It can be observed from the table 3 that the higher the Specific Surface Area is, the lower the D50 and higher reactive the pozzolanic ash is. The effect of particle size reduction in the calcined product is a key factor for the increase of the modified Chapelle pozzolanic activity, as can be observed from FHA-800 to FHA-850 samples. The FHA-800 sample was submitted to further calcination process at 850 ºC, which has resulted in a consequent reduction of about 1.2% less of the particle size from 36 µm to 30 µm (Table 3) and a consequent increase in the specific surface. With this additional process a relative gain of 21 % in the modified Chapelle pozzolanic activity was recorded.

Table 3: Physical characteristics of the Fonio Husk Ash

Sample	D50 (µm)	Specific Surface Area (m^2/Kg)	Pozzolanic Activity (mg $Ca(OH)_2$)/g FHA
FHA-650	51	47	720
FHA-700	50	48	730
FHA-720	48	50	760
FHA-740	45	53	790
FHA-750	41	59	800
FHA-800	36	67	880
FHA-850	30	80	1060

Figure.2 displays the result of the particle size distribution analysis for the fonio husk ash powder. It can be observed from the figure, that the particle size of the ash is distributed across the range (2.25µm---600µm) with a D50 of 30µm. The width of the distribution is 1.5 indicating how spread out the particle sizes are. The particle size distribution for the fonio husk ash present fine particle size (d50 =30 µm) which results from the origin of the material (industrial sand washing) and BET specific surface area of 72 m2/Kg. Most of the ash particles are close to the same size, when this ash is to replace cement in concrete mix, it will be good for flow, packing, consistent reactivity and less bleeding.

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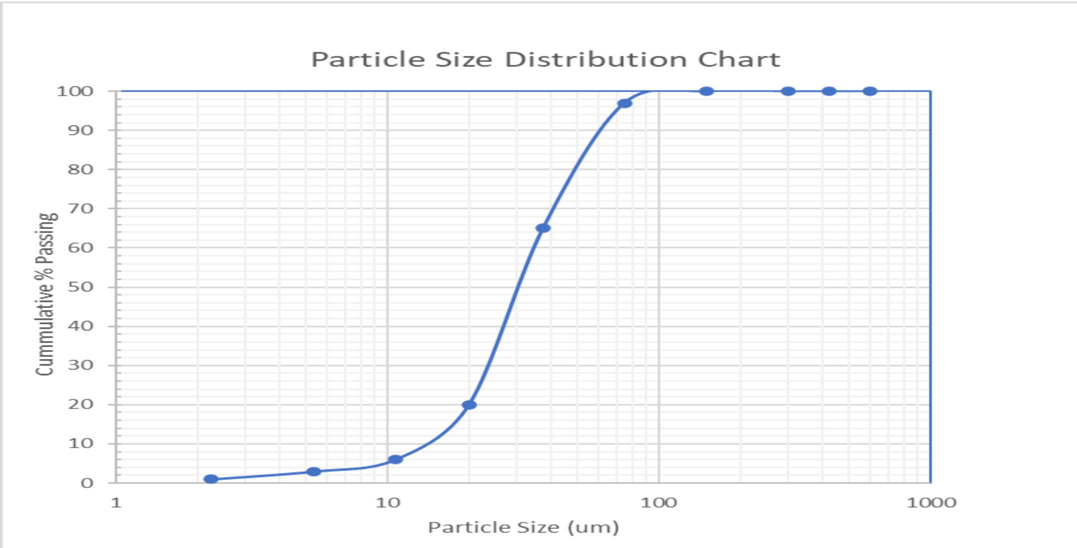

Figure 2: Particle Size Distribution Analysis for the Fonio Husk Ash
D10 = 15 D50 = 30 D90 = 60 Distribution Width = 1.5

IV. CONCLUSION

Based on the goal of this research and the obtained results, discussion and interpretation of the data, the following remarks can be stated about the direct methodology that allows the evaluation of the pozzolanic activity of fonio husk ash by the French standard of the modified Chapelle test.

1. The direct methodology is exceptionally fast, with ease and quick execution, allowing for the direct experimental evaluation (non- strength tests) of the pozzolanic activity of fonio husk ash.

2. The values obtained from the modified Chapelle pozzolanic activity test conducted on the seven fonio husk ash samples are above the standard limit (700 mg Ca(OH)2/g pozzolanic material).

3. The modified Chapelle pozzolanic activity of the fonio husk ash presented values between 720 and 1060 mg Ca(OH)2/g fonio husk ash, indicating that the seven fonio husk ash produced at different calcination temperatures are not of same quality.

4 The FHA-850 sample has the highest modified Chapelle pozzolanic activity value of 1060 mg Ca(OH)2/g fonio husk ash. This is in comparison with the estimated metakaolin minimum value of 1000 mg Ca(OH)2/g metakaolin powder according to many literatures.

5. In the experimental evaluation of the fonio husk ash, the modified Chapelle pozzolanic activity values increase with decrease in particle size values (d50). The calcination levels that produced the study materials (FHA-650 to FHA-850) significantly improves the modified Chapelle pozzolanic activity values by about 32%

6. It was observed that significant and interesting relationship exist among the physical parameters (D50, Specific Surface Area and Pozzolanic Activity (mg Ca(OH)2/g FHA) of the fonio husk ash. The pozzolanic activity of the ash is directly proportional to the specific surface area and inversely proportional to the particle size.

Authors Contributions Statement
UAA conceived this experimental study and designed the experiments. UAA and ASG participated in the design and interpretation of the data. UAA performed experiments and analysis. UAA wrote the manuscript. UAA and ASG participated in the revisions of the manuscript. All authors read and approved the final manuscript.

Conflict of Interests
The authors of this paper declared that no conflict of interests regarding its publication.

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