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DEVELOPMENT AND CHARACTERIZATION OF PLASTER OF PARIS (POP) FROM GYPSUM DEPOSITS OF FIKA (KWARRI) - YOBE STATE, NIGERIA BY CALCINATION PROCESS

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Abstract: *Gypsum is a common sulphate mineral of great commercial importance, it composes of hydrated calcium sulphate ($CaSO_4.2H_2O$). Calcination involves the practice of dehydrating gypsum into plaster through a batch or continuous process which includes heating the gypsum to evaporate the crystalline water. Gypsum is readily available at about 1.2 billion tonnes in our natural deposit sites in Nigeria with little or less exploration for industrial purposes especially in Plaster of Paris (POP) production. Overdependence on foreign/imported POP into the country is in the high rise, local economy is affected and local miners are not patronised. Thus, the study produced and characterized POP gypsum deposits in Fika Local Government Area of Yobe State- Nigeria (Kwarri deposit site). The Gypsum sample was collected at three layers of 0.5m each after excavating the top soil. The samples were beneficiated and calcined at the Civil Engineering Laboratory of Eighteenth Engineering Company at 170^0C. Porosity/absorbency, shrinkage, compressive strength and density tests were carried out on these samples. The gypsum collected were processed and characterized with the sample having porosity of $47.07g/cm^3$ on the average, 0% shrinkage, compressive strength of up to $23.03N/mm^2$ and $21.04N/mm^2$, density of $0.91g/cm^2$ and $1.01g/cm^2$. Water:POP ratio of 80:100 and 100:100 gave the best results. The properties obtained from the characterized POP revealed that Fika (Kwarri) sample when collected from the mining site and processed can replace the one obtainable in the market because of the excellent properties which surpassed the ones being used.*

Keywords: *Gypsum, Plaster of Paris, Calcination, Density, Porosity, Compressive Strength.*

1.0 INTRODUCTION

The common sulfate mineral known as gypsum has significant commercial value. It is mostly made up of hydrated calcium sulphate ($CaSO_4.2H_2O$). It is a white mineral of calcium sulphate in the earth crust (Mohammed, 2015). Gypsum is an essential material used for numerous construction purposes ranging from building and several others due to its aesthetics and insulative properties (Kerstin *et al.,* 2023). In nature, gypsum can be found as translucent, cleavable masses termed selenite and as flattened, frequently twinned crystals. It can also happen to be silky and fibrous, in which case it's referred to as Satin Spar. During precipitation, minerals like halite, anhydrite, calcite, and pyrite are included (Broadhurst, 2007; Sidney, 2008). Unwanted minerals inclusions are to be separated and removed during beneficiation stages from the valuables in accordance with the desired application requirements (Henkel and Gaynor 1996). According to the Federal Ministry of Science and Technology, (Raw Materials Research and Development Council (RMRDC), (2010), gypsum has been in existence in Nigeria since 1921 with grades from the various deposits in the country (at about one billion tonnes spread across the deposit sites) satisfying the specifications required by the cement industry: identified deposits in North Eastern Nigeria are found in Adamawa, Taraba, Yobe, Borno, Bauchi and Gombe States respectively. Locations like Yobe, Sokoto, Adamawa, Gombe, Benue state and others have gypsum deposits in substantial quantities (Madu *et al*., 2016). Plaster of Paris(POP) is a quick-setting gypsum plaster consisting of a fine white powder (calcium sulphate hemihydrate), which hardens when moistened and allowed to dry. It is known since ancient times as plaster of Paris, so called because of its preparation from the abundant gypsum found near Paris (Britannica, 2024). Calcination is the process of heating gypsum to evaporate the crystalline water in order to dehydrate it into plaster or stucco. This can be done in a batch or continuous manner. Free water is usually removed at 45°C steps in the initial process, while crystalline bound water is removed in 120–180°C steps in the second process. Many systems additionally include options for grinding capacity before, during and after calcination, depending on the product intended to be produced (Innogyps, 2021).

The present study will assess Fika (Kwarri) deposit site in Yobe State, Nigeria for the production of POP and compare their properties with POP currently available in the market(POPm). This study will also enhance and explore the use of gypsum from our readily available deposit sites, improve industrialization, create more job opportunities for local miners and boost the local economy through industrialization.

Madu *et al.,*(2016) assessed local production of POP from gypsum deposit in Nafada region by the use of electric burning kiln for calcination. In the study, water:plaster ratios were tested and it was discovered that the samples with water:plaster ratio of 90:100 and 100:100, gave excellent results of 14.47N/mm^{2}and 15.92N/mm^{2}of compressive strength, 1.08g/cm^{3}and 1.01g/cm^{3}of density, 42.9% and 54.9% of porosity respectively. On the basis of this investigation, the locally produced POP from Nafada gypsum deposit can adequately replace the foreign ones.

Soyinka (2015), in their research on the comparative analyses of Wurno, Dange and Weppa gypsums in Nigeria for plaster and ceramic production, it was deduced that the constant importation of plaster of Paris constitutes a drain on national economy and an added cost on ceramic mass production. Various tests were carried out; results showed that the ratio mix of 75-85 of the plaster to water ratio mix were suitable for ceramic slip casting process. 65 and 70 plaster:water ratio mix were better used for casting models and press moulding process. The gypsum obtained from the three locations can be used for ceramic application.

Similarly, Mohammed (2015) in suitability of Nafada Gypsum for the Production of Jute Fibre Reinforced Plasterboards pointed out that, high demand for Plaster of Paris (POP) in the Nigerian building industry, particularly within the north-east geopolitical zone has made local procurement of the raw gypsum inevitable. The result from the study showed that Nafada gypsum is suitable for the production of POP for use in plasterboards, with jute fibre as a reinforcing material.

Bukar *et al.,* (2011), in their research on, Geology and Geochemistry of Gypsum Deposits in Fika and Fune Local Government Area Of Yobe State, Nigeria: Implication to Industrial Applications, concluded that the gypsum of the study areas fall within the Cretaceous sequence and the gypsum occur as seams, vein let and lenses and based on these geochemical values the gypsum of the two areas is good quality for cement, plaster of Paris, solid wallboard, ceramic and ammonium sulphate fertilizers.

Also, Abidoye & Bello (2010), in restoration of compressive strength of recycled gypsum board powder, carried out a reversal of the state of hydration of waste gypsum powder via calcination to enable the reuse of the powder for gypsum board manufacturing. It was concluded in this study that, calcination of recovered waste POP has been found to be a satisfactory condition of treatment for the recovery of the compressive strength of the material. Calcination of the recovered POP returns the lost strength to the powder thereby enhancing its reusability. Of all the calcination temperatures investigated, 160^{0}C was optimal. Hence, the re-enhancement of waste plasterboard can be easily achieved with calcination of the waste at this temperature.

This study will explore the gypsum at three different layers, characterize the POP formed and compare the POP to see if properties of the POP will improve with depth of sample collection, thereby recommending the most suitable to use and at which layer of mining.

2.0 MATERIALS AND METHODS

2.1 Materials and Equipment

2.1.1 Materials Used

The materials used in this study include water and Gypsum rock from Fika (Kwarri) (Lat. 11.28336^0, Long. 11.315814^0) deposit mining site in Yobe Stater. Figure 1 shows Yobe State Map with all the Local Government Areas with the location of mining.

Figure 1: Map Showing Yobe State LGAs

2.1.2 Equipment Used

The equipment used for this study include; Electric blast drying oven (Type 10 -1A, Beijing Luda Waiye Technology) for calcination(at EEC Lab), digital weighing scale (model, Szegedi, OmhEng Type), electric hydraulic pressure testing machine (Huaxi, DYE-2000 type; EEC Lab) to determine compressive strength, shovel, pulley, torch light, handgloves, Vernier caliper, sieve (model, Bosch-112219), crucible for loading sample, stirrer for mixing gypsum and water.

2.2 Method

This work adopted the method of calcination used by Madu *et al.,*(2016). This included collection of gypsum samples, selection and cleaning, washing and drying, crushing, screening/sieving, calcination, storing, porosity, density and compressive strength was determined. The experimentation was done at the Eighteenth Engineering Company (EEC) Laboratory, Molai, Maiduguri, Borno State. Also, the chemical analysis was done at Spectral Laboratory Services, Tudun Wada, Kaduna, Kaduna State.

2.2.1 Preparation of the gypsum samples

The mineral was produced from underground mines which is below water level by excavation (digging). Three separate samples each at 0.5m, 1m and 1.5m respectively from each location after removal of the top soil were collected. The top soil was removed at 1.8m. It was then collected, stored in a bag and transported to the workshop where it was processed. Plate 1 shows the samples collected from the mining site.

Plate 1: Gypsum samples from deposit site

To produce good samples of gypsum that would be further processed, impurities were thoroughly cleared and cleaned from the samples. This was done by picking out and removing the larger clay and sand particles from the samples and washing it.

Gypsum was soaked for about 1hour to allow easy removal of the clay on the samples. It was then washed thoroughly to ensure it was free from impurities like sand and clay that got stuck to it. After proper washing, it was then allowed to dry under normal room temperature conditions.

A grinder was used to first reduce the size of the dried gypsum rocks to smaller pellets, then it was taken to another grinder which crushed the samples to powdered form.

A manual sieve of 300mm was used for sieving the crushed powder in order to remove the large grains which were not properly crushed so as to obtain a fine powder.

2.2.2 The Calcination Process

The process of calcination to form plaster of Paris was one involving heating of the cleaned, dried, and crushed gypsum to a certain temperature ranging from 120°C to 180°C to drive off a portion of the water of crystallization (Tesfaye, 2015). The gypsum powder after preparation, waspoured into a crucible and loaded into the electric blast drying oven for calcinations (Plate 2 shows the electric blast drying oven). Crucible was used in the kiln for calcinations of gypsum because POP produced from a crucible is denser and stronger. Sample was heated to a temperature of 170^0C, to ensure that the combined water was released from the samples. After heating it to the required temperature, the oven was turned off and the powder allowed to cool down inside the oven for a period of 18 hours before it was opened.

Plate 2: Electric Blast Drying Oven

After calcination, the powder was kept dry and enclosed in a polythene bag to stop moisture intrusion/infiltration.

2.3 Characterization of the POP

The samples were subjected to various tests (characterization) to determine the porosity/absorbency, density, shrinkage and compressive strength.

2.3.1 Porosity & Absorbency

Porosity/Absorbency is a measure of the void spaces in a material. High porosities results in micro-cracks forming in regions where stress concentration is high thereby affecting the strength of the material (POP) (Mahendra, 2018). The porosity/absorbency test was carried out at the Eighteenth Engineering Company (EEC) civil engineering Laboratory at Molai, Maiduguri.

The samples were dried in the oven for 24 hours at a temperature of 45^0C and cooled before the porosity/water absorption test was performed. The weight of the dried samples were measured. The samples were then soaked in water for a period of two hours to ensure optimum saturation (Madu *et al,* 2016). The POP was further subjected to porosity test with a ratio mix of POP/water at about 80ml to 150ml (Madu *et al,* 2016). Plate 3 and 4 shows how the porosity test was carried out. Samples were moulded (Plate 4) according to the mix ratio of water:POP.

The porosity was determined using Equation 1;

$$\text{Percentage of Water Absorption} = \frac{\text{Ws} - \text{Wd}}{\text{Wd}} \times 100 \qquad 1$$

where;

Ws = Saturated Weight and Wd = Dry Weight

Plate 3: Samples being weighed to determine porosity

Plate 4: Samples being soaked

2.3.2 Density

The mass of the samples were measured using the electric weighing scale and the volume was calculated using equations 1 and 2 respectively;

$$\text{Volume of Cylinder} = \pi r^2 h \ (\text{cm}^3) \qquad 1$$

$$\text{Dry Density} = \frac{\text{Mass}}{\text{Volume}} (g/\text{cm}^3) \qquad 2$$

2.3.3 Shrinkage

Shrinkage means a decrease in single dimension of a sample when moisture is reduced (Madu *et al,* 2016). The samples were placed in a mould and allowed to solidify. After it was dried completely, it was observed that there was zero reduction in their sizes.

2.3.4 Compressive Strength

Compressive strength of a material is by definition that value of uniaxial compressive stress reached when the material fails completely. The compressive strength obtained usually by experimental means using a compressive strength test Plate 5 shows the compressive Test Procedure using the Electro-hydraulic pressure testing machine. The apparatus used for this experiment is the same as that used in a tensile test. However, rather than applying a uniaxial tensile load, a uniaxial compressive load is applied as shown in Plate 6. The specimen (usually cylindrical) is shortened as well as spread laterally (Tesfaye 2015). The compressive strength of the POP was be determined using the ratio mix of 80ml to 130ml to 100g of water:pop. To determine the compressive strength Equation 3 was used and Equation 4 to calculate the cross-sectional area;

$$\text{Compressive Strength} = \frac{\text{Applied Force}}{\text{Cross} - \text{Sectional Area}} (\text{N/mm}^2) \qquad 3$$

Where;

$$\text{Cross} - \text{Sectional Area of Cylinder} = 2\pi rh + 2\pi r^2 h \ (\text{mm}^2) \qquad 4$$

Plate 5: Compressive Strength Test (EEC Lab) **Plate 6: Sample loaded to the press jaws**

3.0 RESULTS AND DISCUSSION

3.1 Porosity/Absorbency Test Observation

The measure of the void spaces in the POP was determined and presented in Figures 1, 2 and 3.

3.1.1 Porosity/ Absorbency Test result for Fika (Kwarri) sample

Comparison between porosities of the POP obtained in the market (POPm) and that of the samples obtained from Fika (Kwarri) deposit site at 0.5m, 1.0m and 1.5m depth is presented in Figure 1.

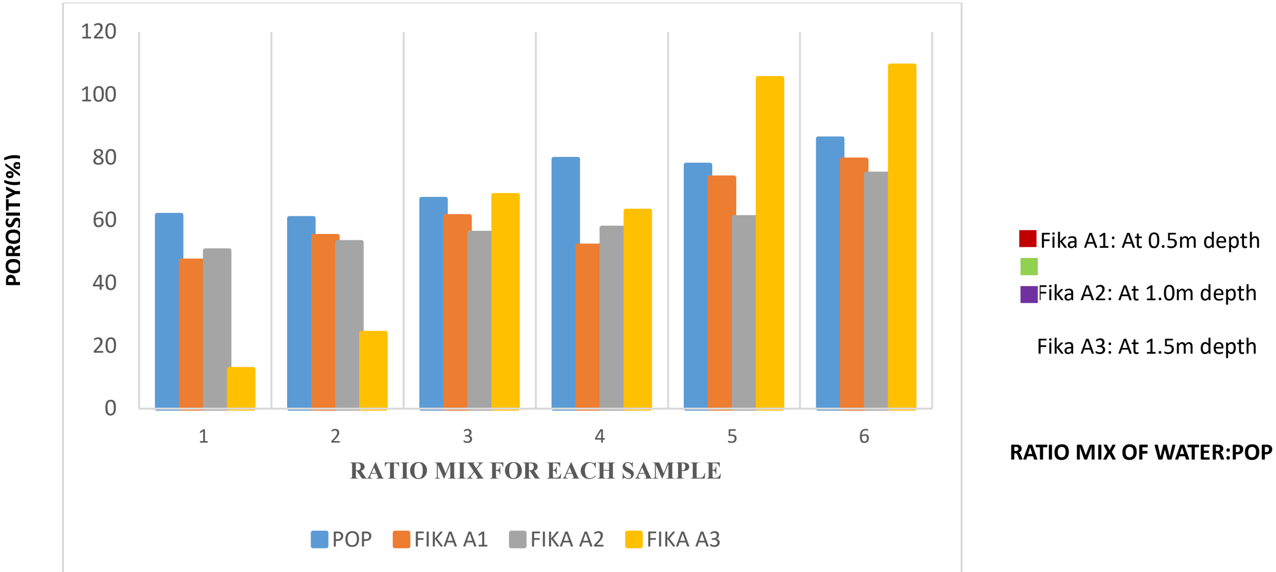

Figure 2: Comparison between porosities of POPm and Fika (Kwarri) POP

From Figure 2, percentage in porosity increases as the volume of water increases. The POPm had its highest porosity of 85.92% at 100:130 ratio mix of POP to water and the lowest percentage porosity at 61.65% at 100:80. At 0.5m, the highest percentage porosity was 79.25% of POP to water mix ratio at 100:130 and the lowest to be 47.07% at 100:80. For Fika (Kwarri) at 1.0m, it also had a similar steady increase in porosity as the volume of water increased. At 100:80 ratio mix of POP to water, the porosity was 50.37% and the highest was at 100:130 which was 74.81%. At 1.5m, the least porosity which was 12.58% at 100:80 POP to water ratio mix and the highest was 109.19%.The increase in porosity was as a result of the increase in the volume of water added to the POP. As stated by Mahendra *et al*., (2018), micro-cracking of the POP is increases as the percentage of porosity increases which lowers the strength of the POP. The porosity at 1.5m at 100:100 ratio mix of POP to water which was 55.94% was also similar to that of Soyinka, (2015) for Dange sample which was 56.45%.

3.2 Shrinkage Test Result and Observation

Shrinkage Test was carried out to determine the shrinkage (reduction in the cross-sectional area and volume). The shrinkage percentage of POPm compared to Fika(Kwarri) samples at 0.5m, 1.0m and 1.5m is presented in Table 1.

Table 1: Shrinkage Test Result for POPm with Fika (Kwarri) sample at 0.5m, 1.0m and 1.5m depths

Parameters	Samples					
	1	2	3	4	5	6
Plaster(g)	100	100	100	100	100	100
Water	80	90	100	110	120	130
Shrinkage(%) (POPm)	0	0	0	0	0	0
Shrinkage(%) (Fika, Kwarri)	0	0	0	0	0	0

Table 1 shows the shrinkage percentage for Fika (Kwarri) sample at 0.5m, 1.0m and 1.5m depth. It can be noticed that the percentage in shrinkage is at 0% for all the water:POP mix ratio.

3.3 Compressive Strength Test

Compressive strength test is carried out on POP because it falls into the category of earthen materials which lacks the ability to withstand tensile stress (Stress due to pull as in metallic materials) (Soyinka, 2015). The test was carried out at the EEC civil laboratory using the Electric Hydraulic Pressure Testing Machine with the following observations;

The comparison between POPm and that of Fika (Kwarri) samples at 0.5m, 1.0m and 1.5m is presented in Figure 3.

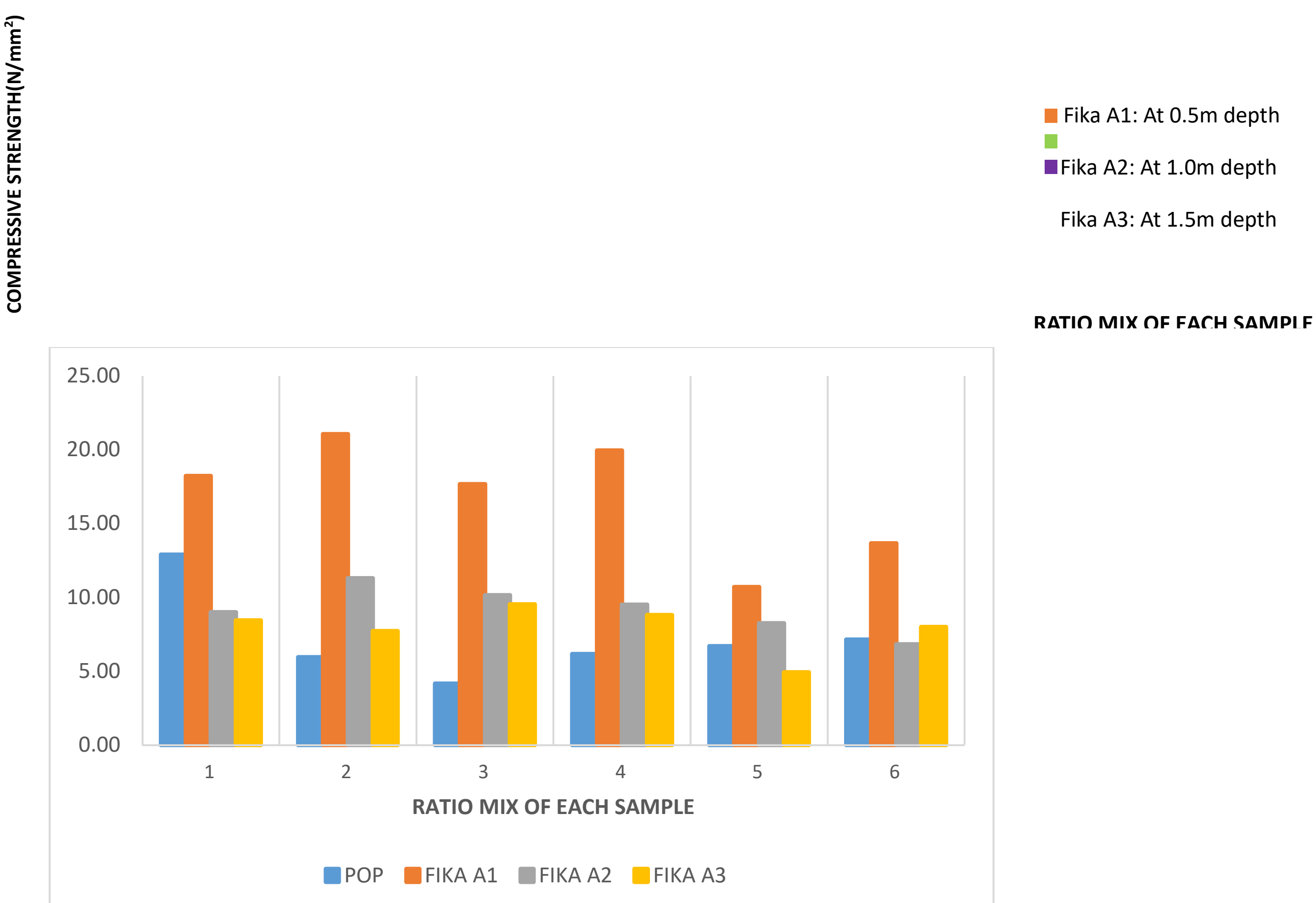

Figure 3: Comparison between compressive strengths of POPm and Fika (kwarri) POP

It was observed in Figure 3 that the compressive strength of POPm was lower than that of the three depths. The highest compressive strength of POPm was 12.88N/mm² at 100:80 and the lowest 4.16N/mm² at 100:100 ratio mix of POP to water respectively. The case was different for Fika (Kwarri) at 0.5m as it had the highest compressive strength as 21.04N/mm² at 100:90 and the least was 10.69N/mm² ratio mix of POP to water. At 1.0m layer of the sample, the compressive strength was low compared to the 0.5m layer the highest was 11.30N/mm² at 100:90 and the least 6.81N/mm² at 100:130 ratio mix of POP to water. The 1.5m layer however, had a lesser compressive strength as compared to the previous two layers, the highest was 9.51N/mm²and the least 4.92N/mm². It was observed from the compressive strength of POPm, and the three depths that the stress decreases with increase in the water volume. This observation agreed to Madu *et al*., (2016) and Soyinka, (2015) that increase in the volume of water reduces the compressive strength of POP.

3.4 Density Tests Results

The density test was carried at the EEC civil engineering laboratory.

Figure 4 compares POPm density to that of Fika (Kwarri) Samples at 0.5m, 1.0m and 1.5m depth.

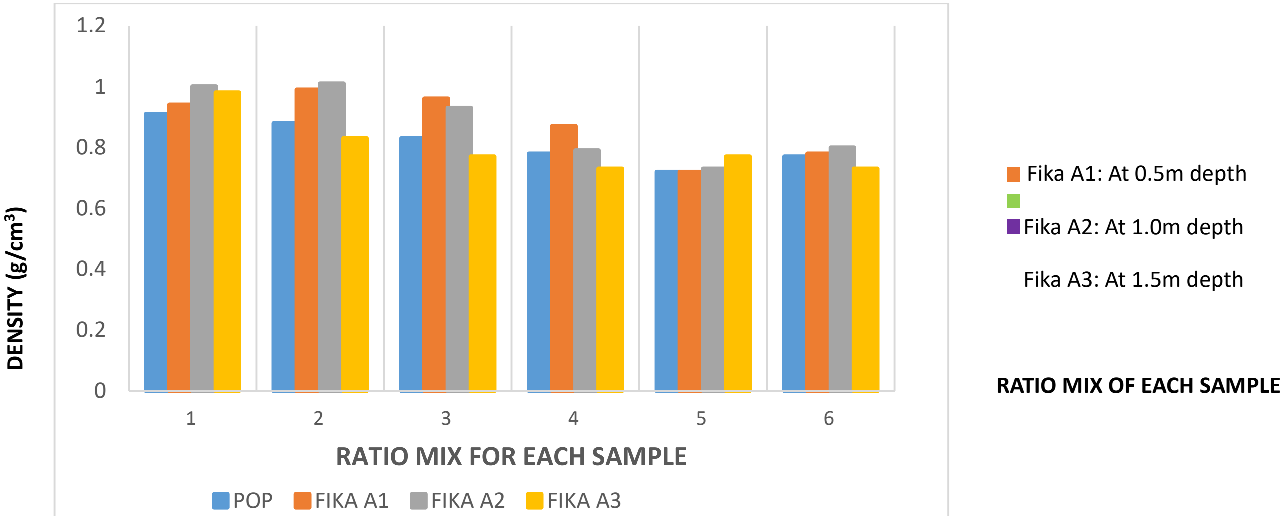

Figure 4: Comparison between densities of POPm and Fika(Kwarri) POP

From Figure 4, POPm had its density highest which was 0.91g/cm³ and lowest at 0.72g/cm³ at 100:80 and 100:120 ratio mix of POP to water. For Fika (Kwarri) sample at 0.5m depth, the highest density was 0.94g/cm³ and lowest 0.72g/cm³ at 100:80 and 100:120 ratio mix of POP to water respectively. The values at 1.0m depth was 1.01g/cm³ and 0.73g/cm³ highest and lowest at 100:90 and 100/120 ratio mix of POP to water respectively. At 1.5m depth, the highest was 0.98g/cm³ and lowest 0.73cm³ at 100:80 and 100:130 ratio mix of POP to water respectively. It can be observed that the decrease in density was similar to that of Soyinka, (2015) and Madu *et al.,* (2016) which showed a decrease in density as the volume of water increases per sample.

4.0 CONCLUSION

This study developed and characterized POP from gyp sum deposit in Fika (Kwarri) site. From the research the following conclusions were made;

i. Gypsum is readily available in deposit sites of Fika Local Government area of Yobe State which can be harnessed for the production of local POP.

ii. The gypsum collected were processed and characterized with all the samples showing excellent porosity of 51.81 at the average, 0% shrinkage, compressive strength of up to 21.04N/mm^2, density of 0.91g/cm^2 and 1.01g/cm^2.

iii. The properties obtained from the characterized POP revealed that in all the samples, as the depth of collection increases, the POP has differing properties in terms of its porosity/absorbency, density, shrinkage and compressive strength. However, the POP when processed, can replace the one obtainable in the market, due to the excellent characterized properties which surpassed the ones being used.

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