Physical And Chemical Analysis Of Some Nigerian Gypsum Minerals For Application In Manufacturing, Construction And Allied Industries.

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ABSTRACT: The present work investigates the physical and chemical characteristics of some local gypsum minerals and to develop an appropriate process technology for their exploitation, refining and utilization in Nigeria. Though a number of gypsum deposits have been found in Nigeria, three varieties were studied in this work. The gypsum samples from lgbokotor and Ibeshe villages in ogun state were observed via a manual pitting method while the third variety was procured from Potiskum in Borno state. The raw gypsum were beneficiated to remove obvious physical impurities and air-dried. In this experiment, the raw gypsum were analysed to determine their chemical constituents using the conventional wet silicate technique. The six major significant constituents, Carbon Dioxide (CO_2); Calcium Oxide (CaO); Magnesium Oxide (MgO); Sulphur Trioxide (SO_3); Ferrous Oxide (Fe_20_3) and combined matter (Loss-on-ignition) were determined. Using an electrical Kiln with digital control, a calcinations sequence of 160°c — 30'c (temperature) range against 60minutes — 300minutes (time) *was* used during the heat treatment procedure. The results of the experiment showed that the optimum water — plaster ratio was 3:2 while the setting or hardening time was between 3.0— 8.0 minutes. Other physical properties such as the density, colour, particle size were found to be in agreement with literature. Consequent upon the investigation reported here, an adaptive refining process technology for Nigerian Gypsum mineral has been developed and the process development and description are presented.

Keywords: Calcination, Dehydration, Rehydration, Setting, Accelerator, Plaster of Paris (P.O.P) and Refining.

1. INTRODUCTION

Geologically, the gypsum mineral is formed from super saturated acqueous solutions in shallow seas which evaporated and deposited carbonates, then sulphates and chlorides among others in order of increasing solubility, Lotze [1]. It is naturally occurring as a soft rock in association with limestone, silica, clays and a variety of soluble salts as impurities. Scientifically, Le Voisier [2] is known to have started research into the principles of gypsum technology.

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He showed that the change from gypsum to plaster of paris (P.O.P) during calcinations was due to the absorption of water and that the hardening of plaster after mixing with water was due to the absorption of water to reform the original compound. In 1883, Le Chatelier [3] proved that plaster of paris was a definite **hydrate, $CaS0_4.2H_20$** and has shown that when water is added to the calcined plaster, a solution is obtained from which the hydrated sulphate soon crystallizes out, thus allowing more of the plaster to be dissolved. The alternate solution and crystallization continues until the whole plaster becomes hydrated. The most unique property of the gypsum mineral is its ability to lose its water of hydration at elevated temperatures and then recombine with appropriate amount of water at low temperature to form the original hardened dehydrate. Thus, the two properties is described as the gypsum phenomena while the processes involved in achieving the dehydration and rehydration procedures leading to the wide range of industrial applications is known as the Gypsum Technology, [4] and [5]. The **$CaSO_4.H_20$** system has five phases and four is known to exist at room temperature thus, calcium sulphate dehydrate, calcium sulphate hemihydrate, anhydrite III and anhydrite II while the fifth, referred to as anhydrite I exist only above 1180^{o}C [6]. The product of gypsum dehydration (calcinations), the calcium sulphate hemihydrate, **$CaSO_4.1/2H_20$,** commercially referred as plaster of paris (P.O.P), occurs in two different forms of α and β phases. The mechanism of plaster setting or hardening has been well reported. The work by Le Chatelier (1883), established the theory of recystallization in which the calcium sulphate, 11-hemihydrate, in water, first form a saturated solution. In 1926 Baykoft [9] proposed the colloid theory which state that the hydration proceeds via a colloidal intermediate through the formation of an absorption process between the calcium sulphate hemihydrate and water with little success. The recrystallization theory agrees with the widest conclusion which describes the hardening process as the result of the calcium sulphate dehydrate forming needles which intergrow into an interlocking network.

2. EXPERIMENTAL PROCEDURE

Suitable quality Igbokotor gypsum, weighing about 5.0kg was transferred into a suitable container for beneficiation. The material was washed with water using a wire brush to remove the surface impurities such as clay, sand and allowed to air-dry. The cleaned sample was crushed in a hammer mill to reduce the rocky lumps to between 0.1mm — 25mm aggregate sizes. After crushing, the material was transferred to Kiln/furnace and calcined at a pre-determined temperature and time. The gypsum process technology which is based on heat treatment, involves a series of chemical reactions which occur to transform gypsum to the calcium sulphate hemihydrate called plaster of Paris (P.O.P) according to the following expressions:

Dehydration

$CaSO_4.2H_2O \longrightarrow CaSO_4.1/2H_2O + 3/8H_2O$ ……………(1)

(Gypsum)

$CaSO_4.2H_2O \longrightarrow CaSO_4 + 2H_2O$ ……………....(2)

$CaSO_4.1/2H2O + 3/8H_2O \longrightarrow CaSO_4.2H_20 + Heat$ …………………(3)

$CaSO_4.2H_2O \longrightarrow CaSO_4.2H_2O + Heat$ ……………..... (4)

TABLES 3.0 CALCINATION TEMPERARURE – WEIGHT LOSS ANALYSIS

IBESHE						IGBOKOTOR						POTISKUM					
Specimen Code	Calcination Temp/°C	Calcination Time (Mins)	Weight Loss (%)	Colour	Result	Specimen Code	Calcination Temp/°C	Calcination Time (Mins)	Weight Loss (%)	Colour	Result	Specimen Code	Calcination Temp/°C	Calcination Time (Mins)	Weight Loss (%)	Colour	Result
IBESH 1	160	60	4.77	Off White	Poor pop	IGBO 1	160	60	10.75	White	70%	POT 1	160	60	7.5	Very White	Good pop
IBESH 2	160	120	7.25	Off White	Poor pop	IGBO 2	160	120	17.18	White	Plaster	POT 2	160	120	9.26	Very White	V. G pop
IBESH 3	160	180	10.80	White	Good pop	IGBO 3	160	180	24.69	Very White	Good Plaster	POT 3	160	180	12.70	Very White	V. G pop
IBESH 4	160	240	14.52	White	Good pop	IGBO 4	160	240	27.44	Very White	V. G Plaster	POT 4	160	240	14.40	Very White	V. G pop
IBESH 5	160	300	18.07	White	Good pop	IGBO 5	160	300	33.06	Very White	V. G Plaster	POT 5	160	300	18.55	Very White	V. G pop
IBESH 6	170	60	8.60	White	Poor pop	IGBO 6	170	60	14.56	White	pop	POT 6	170	60	8.80	Very White	V. G pop
IBESH 7	170	120	12.45	White	Poor pop	IGBO 7	170	120	21.18	Very White	V. G pop	POT 7	170	120	11.50	Very White	V. G pop
IBESH 8	170	180	17.40	Good White	Good pop	IGBO 8	170	180	26.90	Very White	V. G pop	POT 8	170	180	13.81	Very White	V. G pop
IBESH 9	170	240	21.61	Good White	Good pop	IGBO 9	170	240	33.05	Very White	V. G Pop	POT 9	170	240	17.25	Very White	V. G pop
IBESH 10	170	300	27.26	Good White	Good Pop	IGBO 10	170	300	37.55	Very White	V. G Pop	POT 10	170	300	22.72	Very White	V. G pop
IBESH 11	180	60	11.24	White	Poor pop	IGBO 11	180	60	18.43	White	Good pop	POT 11	180	60	10.90	very White	V. G pop
IBESH 12	180	120	14.80	White	Poor pop	IGBO 12	180	120	23.68	White	V. G pop	POT 12	180	120	13.41	Very White	V. G pop
IBESH 13	180	180	18.57	Good White	Good pop	IGBO 13	180	180	28.72	Very White	V. G pop	POT 13	180	180	16.60	Very White	V. G pop
IBESH 14	180	240	22.65	Good White	Good Pop	IGBO 14	180	240	35.12	Very White	V. G pop	POT 14	180	240	19.24	Very White	V. G pop
IBESH 15	180	300	26.90	Good white	Good pop	IGBO 15	180	300	39.79	Very White	V. G Pop	POT 15	180	300	21.42	Very White	V. G pop

ANALYSIS OF THE PLASTER OF PARIS PRODUCT

TABLE 6.0 CHEMICAL ANALYSIS RESULT

S/No	Gypsum Plaster	CaO	SO_3	MgO	Fe,O_3	L. 0. I	C/ H_20
I.	IGBOKOTOR(POP)	29.70	43.50	0.30	0.21	7.80	9.30
2.	1BESHE (POP)	32.40	45.80	0.26	0.15	6.20	10.60
3.	POT1SKUM (POP)	36.10	47.30	0.33	0.09	4.30	12.40

TABLE 7.0 PHYSICAL PARAMETERS

S/N.	Gypsum Plaster	Density g/cm3	Strength MOR N/mm^2	Colour (%)	Sieve % Res. (120)	Initial Setting Time(sec)	Final Setting Time(mins)
I.	IGBOKOTOR (POP)	2.59	3.76		3.20	30 - 80	7 - 30
2.	1BESHE (POP)	2.60	2.98	85+	3.50	30 - 70	6-30
3.	POTISKUM **(POP)**	2.62	4.71	90+	3.17	30 - 60	5 - 35
4	**PROCESS** Calcination Temperature / Time - 160 - 200°C/ 3 - 4 hours						
5	Water: Plaster ratio - 70: 30						
6	Particle size - >75 pm (70 - 90%/ 200mest.)						

TABLE 1.0 CHEMICAL AND PHYSICAL ANALYSIS

S/No.	GYPSUM DEPOSIT (RAW)	DENSITY	COLOUR	MONS Hardness	Type	
1.	IGBOKOTOR-OGUN	2.29	Translucent Grey	3.2	Gypsum Spar	
2.	**IBESHE -** OGUN	2.29	Translucent White	3.2	Gypsum Alaba	
3.	POTISKUM	2.30	White	3.3	Gypsum Rock	
S/No.	**WT.** %	CaO	SO_3	MgO	Fe_2O_3	L. 0. I
1.	IGBOKOTOR	29.50	40.20	0.18	025	0.74
2.	**IBESHE**	29.80	42.78	0.24	0.19	0.67
3.	POTISKUM	33.60	45.30	0.15	0.10	0.56

METHODOLOGY CALCINATION SEQUENCE: TEMPERATURE Vs TIME

TABLE 2.0 CALCINATION SEQUENCE

S/N	TEMPERATURE AT.	CALCINATION TIME (mins)				
I.	160	60	120	180	240	300
2.	170	60	120	180	240	300
3.	180	60	120	180	240	300
4.	190	60	120	180	240	300
5.	200	60	120	180	240	300
6.	220	60	120	180	240	300
7.	250	60	120	180	240	300
8.	300	60	120	180	240	300

FIG. 1 GRAPH OF IBESHE P.O.P

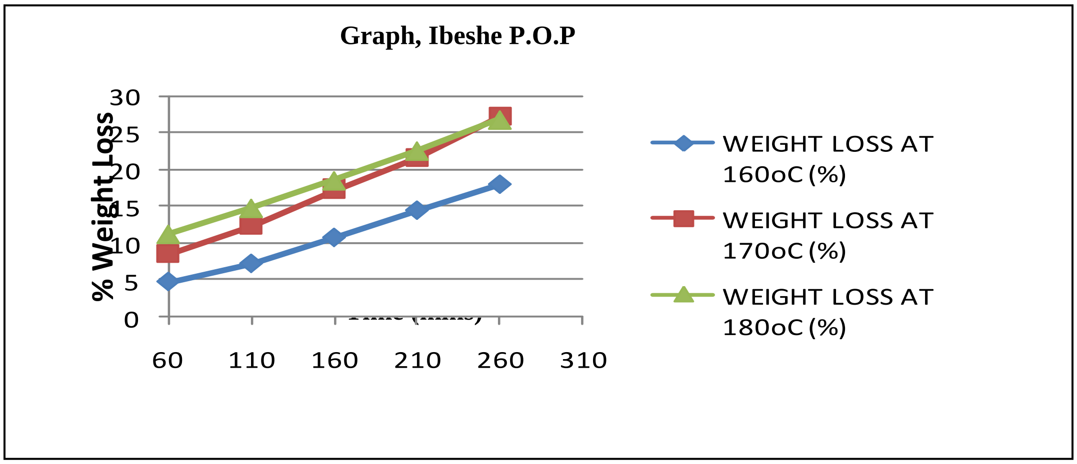


FIG. 2 GRAPH OF IGBOKOTOR P.O.P

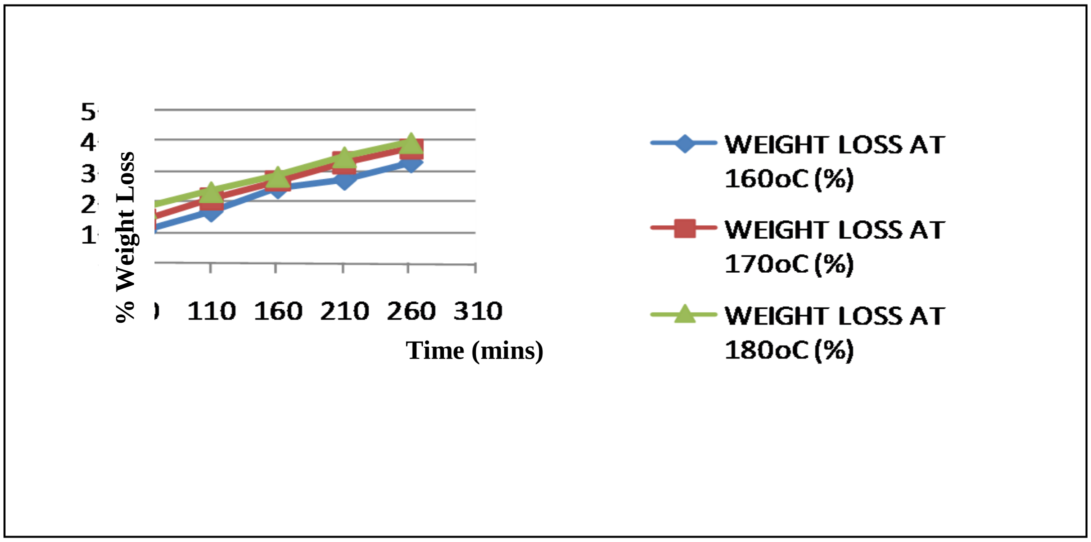
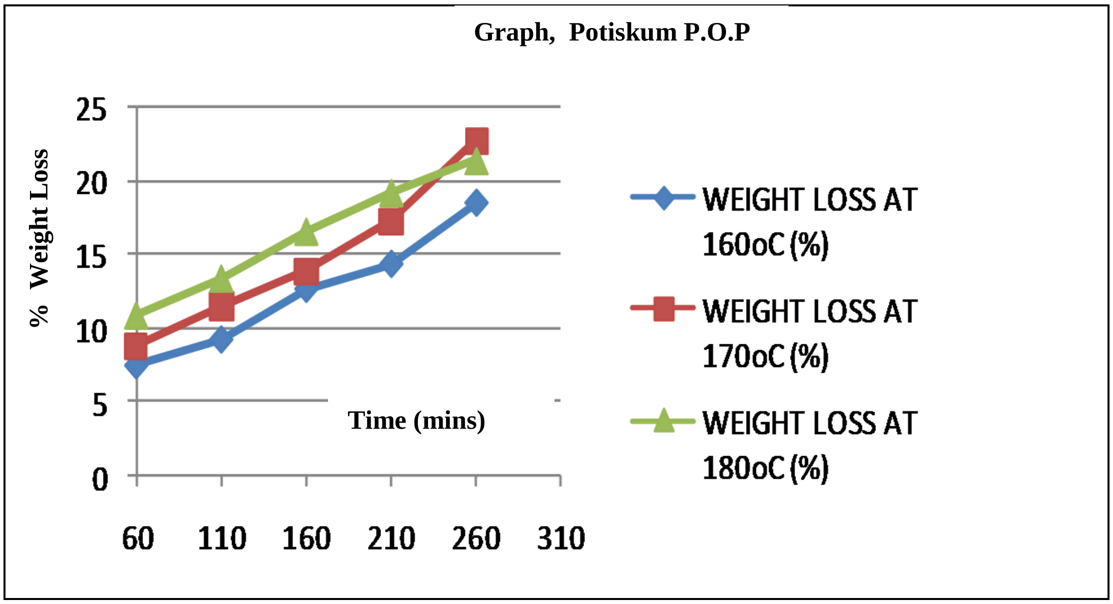


FIG. 3 GRAPH OF POTISKUM P.O.P



3. RESULTS AND DISCUSSION

The calcinations or dehydration processes carried out on the three varieties of gypsum were successful and good quality β hemihydrate calcium sulphate, commercially called plaster of Paris, were successfully produced. The quality of the plaster product compared favourably with imported brands. The whiteness, finess and reheological properties with water matched available standard. The water plaster ratio was found to be at optimum of 3:2 while the setting or hardening time was found between the range 0.30 — 7.0 minutes, initial setting time to 7.0 — 30 minutes, final setting times. The hardeness was found to be at the optimum between 7.0 — 10 minutes setting time. The densities, porosity and other physical characteristics were found to be in agreement with available standard. The school writing chalk products that were produced from the plaster of pans were of very good quality and when compared with samples from the market, it matched some of the best quality. To assess the material yield and shrinkage characteristics, the percentage weight loss during calcinations or heat-treatment were plotted against calcinations time. As shown in Figures 1.0, weight loss increases with increasing calcinations time. This is due to the loss of combined water and carboneous matter. The setting phenomenon of plaster of pans is known to revolve around the needle or rod like shapes of the particles which re-arrange itself during setting or hardening to provide an inter-locking structure of the particles. A microscopic view of the hardening of plaster when mixed with water is considered in terms of the following reaction:

$$\mathbf{CaSO_4.1/2H_20 + 3/2H_20 \longrightarrow CaSO_4.2H_20}$$

(Hemihydrate) + Water ⟶ Set Plaster

By simple chemistry, based on the molecular weight of $\mathbf{CaSO_4.2H_2}$0 (172), approximately 27 grams of $\mathbf{H_20}$ is given off during calcinations process to achieve the calcined calcium sulphate hemihydrate, $\mathbf{CaSO_4.1/2H_20}$ **(145).** When appropriate amount of water is added, approximating to the $\mathbf{3/2H_20}$ or 27 grams that was driven off, a good and consistent blend of plaster/water slurry is obtained for product development. Several factors are known to influence the properties of the $\mathbf{CaSO_4.2H_2}$0 system. The work carried out in this study confirms that the varieties of gypsum found in Igbokotor, Ibeshe, and Potiskum are of good variety. Though the Potiskum gypsum was found to be of the highest purity, the three samples met the basic physical characteristics for a good quality plaster. A blend of two or all the three varieties produced a very high quality plaster product. The study offers good potentials for the development of cottage and small scale plaster production enterprise across the country to generate employment, economic empowerment and conserve the nation's foreign exchange on the importation of plaster of Paris and other plaster based products.

4. CONCLUSION

The varieties of gypsum minerals investigated in this study (Potiskum. Borno State, Igbokotor and Ibeshe, Ogun State, Nigeria) have been found to be suitable for the production of quality Calcium Sulphate **Hemihydrate** $\mathbf{(CaSO_4.1/2H_20)}$ which is termed Plaster Of Paris (P.O.P). The ability to control the time of rehydration rather precisely within 4.0 minutes to 8.0 hours by addition of retarders or accelerators offers the huge and diverse nature of industrial applications in medicine, art, ceramics, education, building and construction among others.

ACKNOWLEDGEMENTS

This research has been carried out with Financial Support from the Federal Institute of Industrial Research, Oshodi, Lagos State, Nigeria.

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