

Effect of curing temperature and particle size distribution on unconfined compressive strength of raw and treated fly ash-lime modified phosphogypsum waste

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Abstract. Voluminous quantity of phosphogypsum waste is generated by phosphoric acid manufacturing industry. Due to limited technologies available to treat the waste and render it useful, this waste is either landfilled or discharged into the sea in a form of slurry. It is critical that the focus shifts from landfilling or disposal into the sea as this result into environment contamination. This study was carried out to determine applications for raw and treated waste phosphogypsum in building and construction and determine the best conditions to produce the final product. Two significant, readily available waste materials namely phosphogypsum and fly ash were used and blended with hydrated lime. Conditions that yielded best strength from a mixture of phosphogypsum-lime-fly ash specimens were at elevated temperatures. The strengths obtained at the temperature of 80 °C were above the permissible strength for building masonry bricks of 3.5 MPa with unconfined compressive strength up to 4.8 MPa. At lower temperature of 40 °C the optimum strength obtained was at the minimal phosphogypsum content of 30%. The optimum strength was achieved at the PG content of 50% for raw PG and 60% for treated phosphogypsum at elevated temperatures. The strengths obtained for the treated phosphogypsum were lower than the minimum permissible load bearing strength due to the non-uniform particles distribution and the presence of impurities and forces of adhesion between citric acid and phosphogypsum. Increasing the curing temperature also reduced the radionuclides initially present in the raw PG without any prior treatment.

1 Introduction

Utilization of solid waste is extremely significant to the sustainable development of the economy and society due to the depletion of raw resources. Phosphogypsum (PG) and fly ash (FA) are the wastes produced in large quantities in South Africa. The quantities of this waste occupy notably large land and discharged to the environment without any prior treatment, and result in bringing on heavy air, soil and water pollution [1]. PG stockpiles with a quantity of 2 million tonnes are located at the now non-operational plant of AECL in Chloorkop and 5 million tonnes with an increase by 240 000 tonnes per annum at the Potchefstroom plant [2]. PG is a by-product of the manufacture of phosphoric acid by a wet chemical process and normally have a $\text{CaSO}_4 \cdot \text{H}_2\text{O}$ content higher than 95%. Studies have been conducted to make PG into binders and wall bricks or partitions [3-6].

Around 6.5 billion m^3 of road base courses are constructed per annum in China. Although a few applications of fly ash, Portland-fly ash cement, clay—fly ash bricks, sand—lime bricks, etc., have been developed enormous quantity of this waste is still unutilized [6]. These volumes of unutilized industrial wastes can be reused in combination of phosphogypsum, fly ash and

lime, for the building and construction industry [6]. Kumar investigated the use of gypsum-fly ash-lime material to make bricks and hollow blocks of adequate strength, an economical alternative to burnt clay hollow bricks, burnt clay bricks and concrete hollow blocks [6]. Mashifana et al. (2018), studied the geotechnical properties and application of lime modified phosphogypsum [7]. The results obtained showed that, the stabilization of mild acid treated phosphogypsum with lime-fly ash significantly improved the unconfined compressive strengths, liquid limit and plastic limit of the material [7]. In this study a new type of material from the mix design of PG-Lime-FA which yielded the strengths above the minimum permissible strength for building brick were produced and the best conditions to produce these material were investigated.

2 Experimental details

Phosphogypsum was collected from Phalaborwa mine in South Africa. To determine the optimum mix design that yielded the highest unconfined compressive strength (UCS) and the optimum curing temperature, material designated Raw 1 was produced by blending 30% phosphogypsum (PG), 50% fly ash (FA) and 20%

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hydrated Lime (L). Mix design designated Raw 2 was the mixture of 40% PG, 40% FA and 20% L. The third mix, Raw 3, contained 50% PG, 30% FA and 20% L. The last mix prepared, designated Raw 4 was made up of 60% PG, 20% FA and 20% L. Only waste PG and FA were varied in the mix designs and hydrated lime as a commercial product was kept constant at a proportion of 20%. The test specimens were prepared by blending the raw phosphogypsum, raw fly ash, lime powder and water at optimum water content. Standard proctor compaction tests were conducted on the produced mix designs, to determine the maximum dry density (MDD) and optimum moisture content (OMC) for the unconfined compressive tests.

The brick specimen was cast in a 100mm x 100mm mould and cured at different temperatures of 40 °C and 80 °C, for 4 days. The unconfined compressive strength for the different mix designs was determined. The paste produced was characterised using X-ray diffraction (XRD), Energy dispersive X-Ray spectroscopy (EDS), X-ray fluorescence (XRF) and Scanning electron microscope (SEM) to determine the chemical composition and the morphology of the final produced material. PG was treated with citric acid, oxalic acid, sodium bicarbonate and sodium bicarbonate and the best reagent that reduced the radionuclides in the material was found to be citric acid. Citric acid was used as a leaching reagent to treat the material at room temperature, leaching for 24 hours. The slurry was then filtered, dried in the oven for 24 hours at 80 °C and then analysed for chemical composition and morphology by XRD, XRD and SEM. The final product obtained was blended with FA and lime with the same proportion as the raw material and followed the same procedure.

3 Results and discussion

3.1. Elemental composition of raw material

The elemental compositions of the materials utilized to prepare the mix designs for raw and treated phosphogypsum, fly-ash and lime are presented in Table 1. The raw phosphogypsum was laden mainly with sulphur trioxide and calcium oxide and consisted of radionuclides. After the chemical treatment majority of the contaminants that hinder some of the processes in civil engineering were reduced, thus radionuclides, phosphorus and fluorides. Radionuclides are not desirable in any building material as they are radioactive and can contaminate the environment and affect people health. The hydrated lime utilized was dominated with calcium oxide and fly ash consisted of constituent such as silica, calcium oxide, ferrous iron and titanium oxide.

Table 1. Elemental analysis of the raw material (wt%).

Component (%)	Raw PG	L	Treated PG	FA
F	1.0633			
Al ₂ O ₃	0.2276	0.279	0.091	28
SiO ₂	1.3702	0.503	0.857	47.9
P ₂ O ₅	1.2839	0.0288	0.719	0.725
SO ₃	51.0129	0.191	54.4	0.606
CaO	43.6526	73.4	42.75	5.13
TiO ₂				2.47
Fe ₂ O ₃	0.1214	0.225	0.048	4.83
Total Radionuclides	0.6198		0.405	

3.2. The effect of curing temperature on the UCS

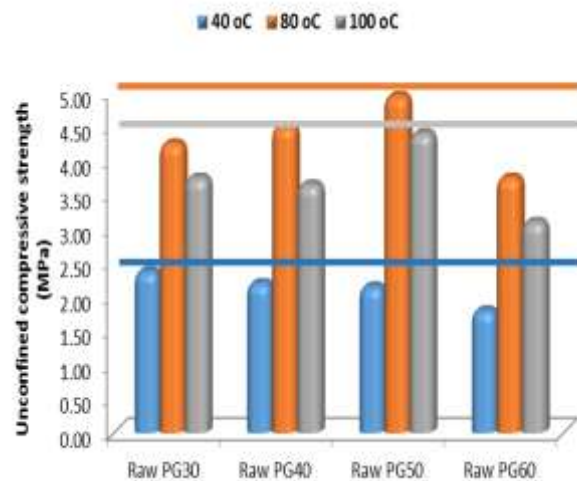


Figure 1(a). UCS of mix designs at lower and elevated temperatures for raw PG.

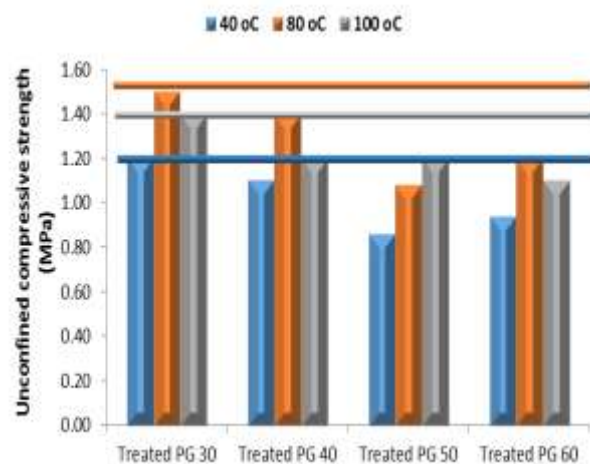


Figure 1(b). UCS of mix designs at lower and elevated temperatures for raw PG.

The UCS obtained for raw PG at different temperatures is presented in Figure 1(a). The strength of

the composites Raw PG30, Raw PG 40, Raw PG 50 and Raw PG 60 increased with the increase in curing temperature. The highest strength is evident in composite with the concentration of 20% L, 50% PG and 30% FA at the temperature of 80 °C. At lower temperatures UCS was the highest at PG 30. At 80 °C, the UCS almost doubled and is the highest at PG 50. Although there was a slight decrease in the UCS from 80 °C – 100 °C, the UCS at 100 °C is higher than the lowest temperature investigated and the highest UCS is also observed at PG 50. For raw PG the optimum curing temperature that yielded the highest UCS of 4.98 MPa is 80 °C for the composite containing 50% PG. The optimum strength obtained at PG 50 is more than the minimum permissible burnt masonry clay according to The South African standard (SANS 227, 2007) which requires a minimum of 3.5 MPa [8]. Therefore the material can be used in a load bearing wall. Increasing the content of PG resulted in increased UCS, a trend clearly visible when curing at 80 °C. For lower temperatures, the UCS decreased with increasing PG content.

The UCS results obtained for treated PG, the specimen cast at the respective MDD and OMC and cured at lower and elevated temperatures of 40 °C, 80 °C and 100 °C are shown in Figure 1(b). The highest strength is observed at the minimal PG content of 30% for all the curing temperatures. The PG content of 50 % yielded the lowest strength and a clear observation of a decrease in UCS with the increasing PG content is evident, the opposite of what was obtained for raw PG. Similar observation occurred with the elevated temperature, the UCS obtained at 80 °C and 100 °C is higher than the UCS at 40 °C.

The highest strength obtained is 1.5 MPa at 80 °C. Therefore, curing temperature has a direct effect on the unconfined compressive strength of PG-Lime-Fly Ash composite for both treated and untreated PG.

The unconfined compressive strengths for the treated PG specimen is lower than the strength for the raw PG, this can be attributed to the particles disintegration with each other in the presence of citric acid, due to morphological change. Ionization carboxylic groups giving rise to the highest anionic charge density exist in citric acid, resulting in maximizing citric adsorption on the surface of phosphogypsum; this was proven by the increase in the surface area of the treated PG as presented in Table 3.

The two oxygen ions in carboxylic group of citric acid are exactly the same at 4.0 Å and the forces of adhesion in gypsum crystals are higher in magnitude [9]. Lanzón and García-Ruiz (2012), found that the treatment of gypsum with citric acid significantly reduces the compressive strength of gypsum [10]. The measurement of the material hardness also revealed a decrease with citric acid treated gypsum. Researchers reported that gypsum crystal habit determines the physical properties and compactly of the material. In study when citric was used to treat PG, the material showed a reduction in UCS and became less rigid due to the lower degree of interlocking [11-14]. These results are similar to those reported by [10].

Lime combined with fly ash reacted with silica and alumina pozzolans and formed strong cementitious matrix that characterizes a lime-stabilized layer. To study the mechanism that has occurred when raw PG and treated PG were stabilised with lime and fly ash and subjected to elevated temperatures and to understand what influenced and contributed to higher strength for the raw PG composite and lower strength for treated PG composites, a thorough study was conducted on the mineralogy of the composites to know the hydration products formed and on the particle size distribution. The obtained composites after strength determination were analysed and the results are presented in the following sections.

Only comparison between the lower temperatures of 40 °C and only 80 °C, were investigated further as the optimum elevated curing temperature that yielded the highest UCS is 80 °C and no further tests on curing temperature of 100 °C were conducted. The standardized documents reviewed by Delgado and Guerrero (2007) indicate that compressive strength standards range between 1.3 and 2.1 MPa for use in non-load bearing walls [15].

In 2011, Deboucha and Hashim's study suggest that in practice typical compressive strength in up to 1 storey construction range from 1 – 4MPa [16]. Some of the national standards prescribing minimum compressive strength of solid clay and concrete masonry units, where the minimums are given as 8.6, 5 and 3.5 MPa respectively are reported [17-19]. The raw PG composites attained strengths more than the minimum permissible by SANS 1215 [19]. For chemically stabilised pavement material following 97% modified AASHTO, the required minimum and maximum unconfined compressive strengths for C1-C4 material are as follows; 6-12 MPa, 3-6 MPa, 1.5-3 MPa and 0.75-1.5 MPa [20].

3.2 Mineralogy analysis of the composite (raw PG) at elevated temperatures

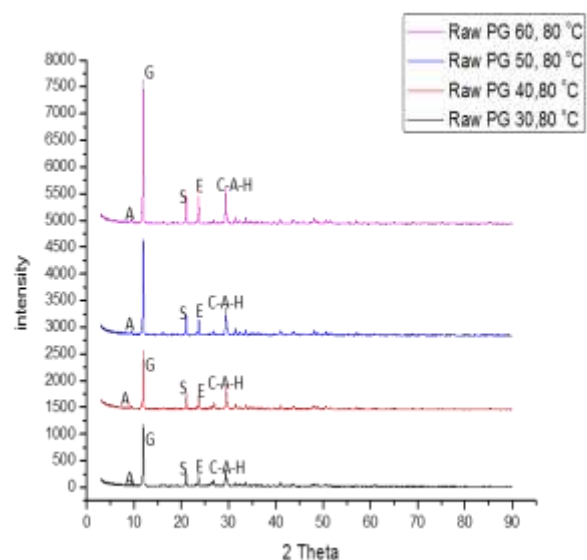


Figure 2(a). XRD analyses of raw PG composites at elevated temperature (80 °C), (G-gypsum, A-silimanite, E- ettringite, C-A-H-calcium aluminium sulphate, S-hypothetical silica).

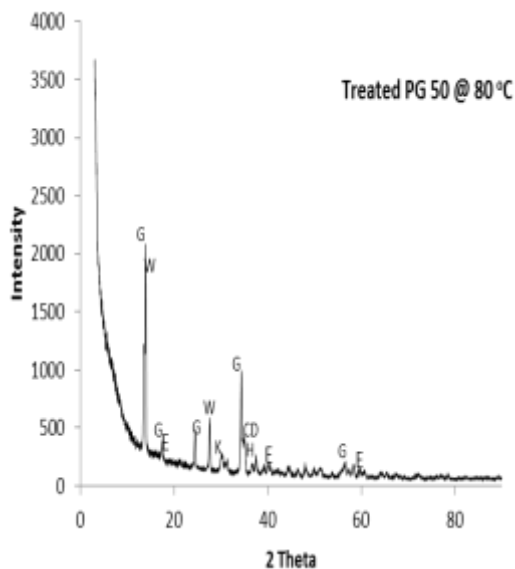


Figure 2(b). XRD analyses of treated PG 50 composite at elevated temperature (80 °C).

The gypsum content is increasing with the increase in PG content for composites cured at 80 °C, Figure 1(a). A significant increase of gypsum is observed in the PG composites cured at elevated temperature, ranging from 81% to 94%. At raw PG 50, the constituents obtained with their respective contribution are those of gypsum, $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$ (93.69%), calcium aluminium sulphate, $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$ (4.64%), hypothetical silica, SiO_2 (0.41%), calcium iron oxide, CaFeO_3 (0.09%) and hedenbergite calcium, $\text{CaFeSi}_2\text{O}_6$ (1.17%). The predominant constituents in the composites produced at lower and elevated temperatures are that of gypsum and silica. The highest unconfined compressive strength was achieved in a composite containing 20% lime, 50% PG and 30% FA, when the material was cured at 80 °C. The results also indicate that with increasing temperature to 80 °C, gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) initially present in the material, was transformed to $\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$, indicating a decrease in water absorption, due to the voids and pores in the matrix being filled by the new constituents that were formed. There were new hydration products formed at PG 50 namely; calcium aluminium sulphate $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$, silimanite, Al_2SiO_5 , calcium iron oxide, CaFeO_3 , hedenbergite calcium, $\text{CaFeSi}_2\text{O}_6$. A hydrated calcium sulphate has a distinct powder diffraction pattern, with a different structure and content.

Curing consumed the hydrated lime by the pozzolanic reactions, as the result calcium silicates, aluminates, and alumina silicates the durable constructional material compounds were precipitated during the curing process. The results obtained agree with the study conducted by [21]. The study conducted by Plowman and Caprera (1984) reveals that lime addition activates the alumina and silica phases and the final material contained silimanite and hypothetical silica with higher proportion than the raw material [22]. The hydration product in a poriferous PG-lime-FA specimen result into particles

integration with each other. 50% (1:1.5) lime to fly ash content to 50% PG content yielded the highest strength. Thus indicating that at PG 30 and PG 40, fly ash is in excess and lime is not sufficient for complete pozzolanic reaction to take place.

When the fly ash -lime ration to phosphogypsum is reduced from 70:30 to 50:50, maximum strength is achieved, showing that the optimum pozzolanic reaction between lime-fly ash which contributes to strength development takes place at fly ash content of 30%. Below 30% fly ash content, lime content to fly ash might be in excess for the pozzolanic reaction to occur for the stabilization of phosphogypsum, hence the drop in strength from PG 50 to PG 60. Reddy and Gourav (2011) reported similar result when they investigated the strength of lime-fly ash, compacted using different curing techniques and gypsum additive [23]. There was a decrease in the composite strength attributed to lime: fly ash ratio. The highest peak on the raw PG composite cured at elevated temperature ranging from 1180 cps to 2710 cps at 2θ is associated to gypsum.

The XRD analysis of the treated PG 50 at elevated temperature of 80 °C is shown in Figure 2(b). The identified constituents are Gypsum (G), $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ (32.35%), calcium aluminium sulphate, $\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12} \cdot 26\text{H}_2\text{O}$ (33.18), wollastonite (W), CaSiO_3 (17.93%), hedenbergite (H), $\text{CaFeSi}_2\text{O}_6$, (1.48%) and keatite (K), SiO_2 (15.01%). The gypsum content in the treated PG composite is less pure than PG in the raw composite and less pure than the PG before curing. The new hydration products formed are, calcium aluminium sulphate, wollastonite and hedenbergite. The XRD result agrees with the XRF as the constituents in the treated PG mineralogy are associated with silicate, iron and calcium, the chemicals also present in high weight % as reported using XRF. There was a reduction in the gypsum content as compared to the treated PG prior curing, a reduction of 65% showing that during the curing process, gypsum was consumed. Due to the addition of fly ash, an increase of 87% silicate oxide is observed, a compound which has silicate as the predominant compound. No radionuclides were detected in the treated PG composite. In 2010, Huang and Lin on their investigation on phosphogypsum-steel slag-granulated blast-furnace slag-limestone cement, reported an increase in peaks of ettringite and a decrease in that of PG due to the consumption of PG during hydration and formation of more ettringite [24].

3.3 pH and relative density of the raw and treated PG

Figure 3 shows the specific gravity of the raw PG and treated PG composites measured using two techniques, a pycnometer.

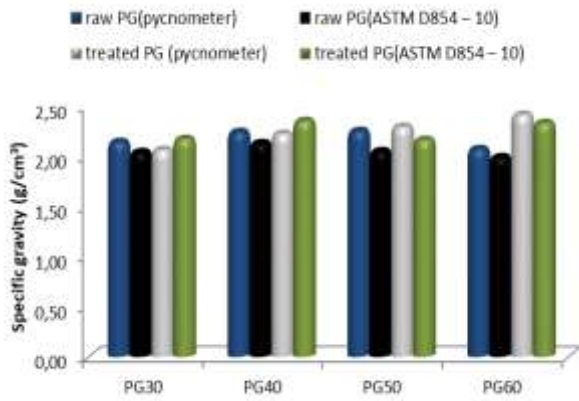


Figure 3. SG of raw and treated PG composites

The results show that in all the composites the density for treated PG composites is higher than that of the raw PG composites. Therefore, the specific gravity did not play a major role in contributing to the high strength obtained for the raw PG composites and the lower strengths for the treated PG material. Relative density is the most representative measure of the degree of compactness of soil [25]. The relative densities of soils fall between 2.5 for clays, 2.65 for silica beach sand and above 2.65 for red/brown sand. These materials when compacted could be up to 1600 kg/m³ and the material of

such densities could mobilise UCS greater than 6 MPa in most cases with lime and fly ash. Thus the low relative density accounted directly for the low MDD, porous inter granular packing and low mobilized strength (UCS – bond stiffness of the binder and granular interlocking friction of fabric).

Both the raw and treated PG composites represents an alkaline materials. Although the raw PG and treated PG materials initially had an acidic pH of 4.17 and 2.31 respectively, blending the material with fly ash and lime and the development of composites, changed the pH to alkaline. This is due to the presence of hydroxide ion from lime used to stabilize PG which contributed to the alkalinity of the material. Lime stabilization causes a significant improvement in soil texture and structure by reducing plasticity and by providing pozzolanic strength gain [26]. Maintenance of high pH above 10 indicates the ability for the pozzolanic reaction to occur and continue over a long term promoting further strength gain [26].

3.4. Particle size distribution (PSD)

The elemental compositions of the materials utilized to prepare the mix designs for raw and treated phosphogypsum, fly-ash and lime are presented in Table

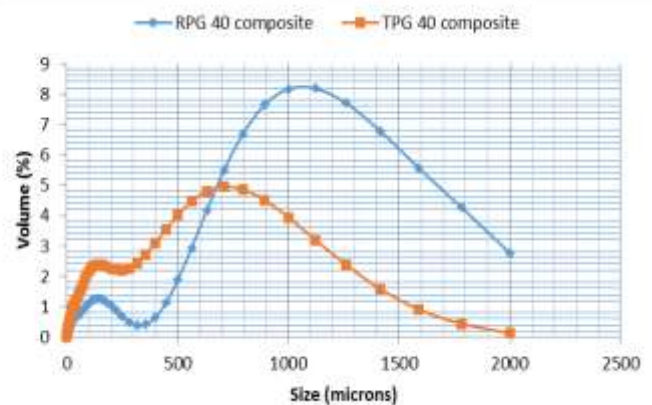
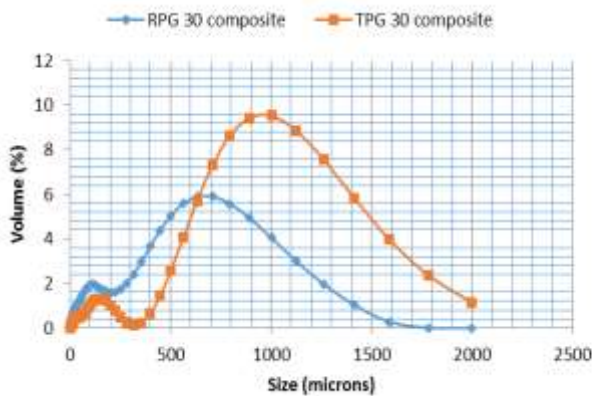


Figure 4. PSD for raw PG 30, 40 and treated PG 30, 40 at 80 °C.

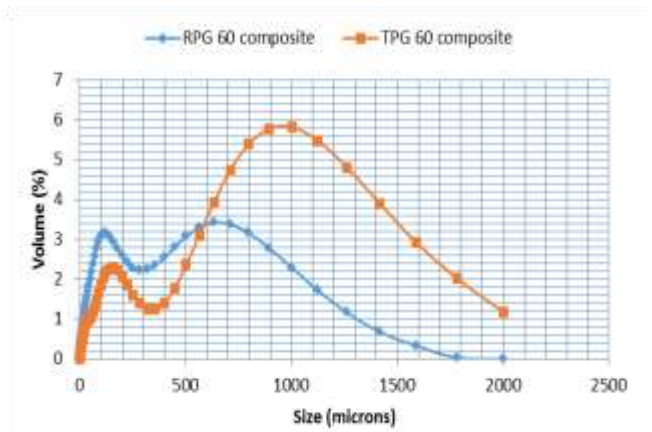
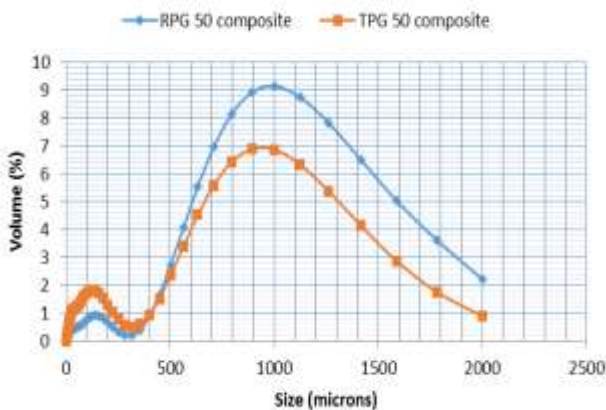


Figure 5. PSD for raw PG 50, 60 and treated PG 50, 60 cured at 80 °C.

There is a significant modal size shift in all the composites from the raw PG to treated PG. A bimodal

size shift to the right is observed with all the composites, indicating a change in the proportion of the larger sized particles of the PSD, thus enlargement of particles due to

conglomeration. Nelson (1983) presents a study indicating a strong dependence of strength on grain size and found out that a smaller grain size means greater strength; this is due to the grain contact models [27]. Well graded soils are generally denser than poorly graded or uniformly graded soils, and mobilized greater confined (biaxial or triaxial) and unconfined strength. This also provides explanation on the UCS results obtained.

3.5. Moments of Particle size distribution (PSD) Particle

The moments of PSD for the raw and treated composite is presented in Table 2 and Table 3.

Table 2. Moments of PSD of the raw PG composites.

Composite	0 th (#m ⁻³ x10 ¹³)	2 nd (m ² m ⁻³ x10 ³)	3 rd (m ³ .m ⁻³ x10 ⁻¹)
Raw PG	8.31	2.54	1.22
RPG 30	288.00	4.67	1.68
RPG 40	11.50	3.25	3.22
RPG 50	6.23	2.16	4.49
RPG 60	298.18	3.73	0.93

A drastic increase in the particles number for RPG 30 and RPG 60 composites is observed, this can be attributed to either formation of particles during the curing process or breakage and there was a slight increase in RPG40 composite. The number of particles in RPG decreased in RPG 50 composite and this is the composite where highest UCS was obtained. Therefore particles number plays a major role on the strength of the composites. This also proves that the RPG 50 was well compacted as the particles bonded together to form a large particle. The surface area for the composites RPG 30, 40, 60 increased. There was an increase in volume of the particles in RPG

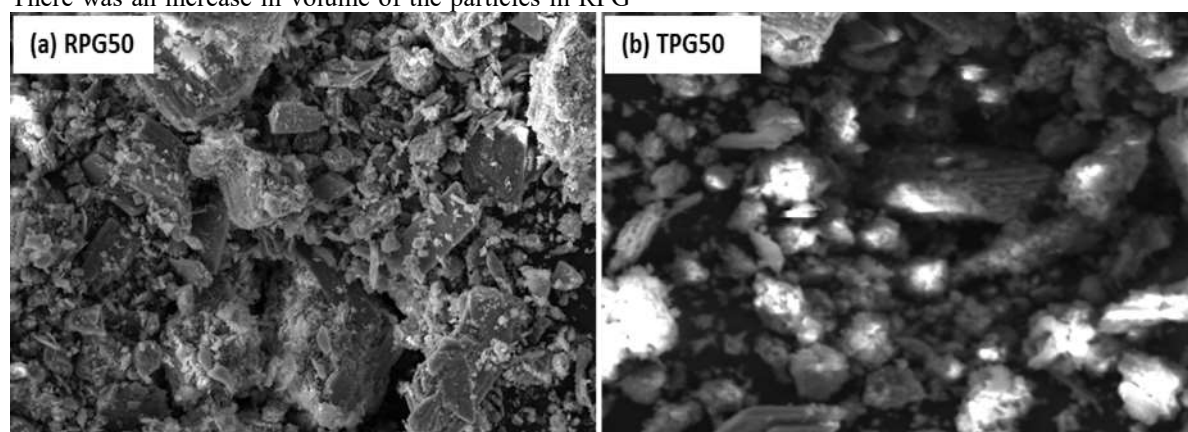


Figure 6. SEM micrograph of (a) raw PG 50 composite (b) treated PG 50 composite.

A smooth surface is observed for the raw PG composites and unlike the treated PG surface which looks bristly. This might be a result of the new hydration products formed and covering the surface of the PG. Fairly large and stronger particles are clearly visible in the raw PG and the particles in treated PG seem to be more like flakes. The results obtained correlate very well

30, RPG 40 and RPG 50 composites, a decrease in RPG 60.

Table 3. Moments of PSD of the treated PG composites.

Composite	0 th (#m ⁻³ x10 ¹³)	2 nd (m ² m ⁻³ x10 ³)	3 rd (m ³ .m ⁻³)
Treated PG	19.01	2.96	0.13
TPG 30	9.71	2.74	0.33
TPG 40	218.30	3.38	0.12
TPG 50	273.86	4.00	0.15
TPG 60	283.25	3.93	0.13

For the treated PG specimen the highest UCS was obtained at TPG30, and the moments of PSD shows that this is the composite that yielded a significant decrease in the particle number, the same observation as RPG with the highest UCS. The decrease is attributed to aggregation. The number of particles for TPG 40, 50 and 60 composites increased drastically with the increase in the PG content, indicating that the existing particles broke into new smaller particles of varying sizes. The surface area increased for TPG 40, 50 and 60 composites. A decrease in the surface area for TPG 30 composite is attributed to aggregation, whereby smaller particles are bonded together to form a larger particle. The volume of particles remained almost the same for TPG 40, 50, 60 and increased in TPG 30 indicating that growth also played a role in size enlargement.

3.5. SEM image analysis

The moments of PSD for the raw and treated composite is presented in Table 2 and Table 3.

with the PSD analysis whereby a drastic decrease in number of particles is observed in raw PG 50 attributed to particle growth. The fine particles observed for the treated PG 50 correlate well with the PSD analysis that showed an increase in the number of particles in the treated PG composite due to breakage. This also explains the variation in strengths obtained for the raw and treated PG, thus treated PG is less strong and breaks easier as compared to raw PG resulting into the lower strengths.

The overall SEM analysis of raw and treated PG composites supports the particle size distribution analysis results obtained and proved that number of particles, surface area and volume of particles had a significant role in the higher unconfined compressive strengths. All the complexity of phosphogypsum originates from its crystal structure, which eventually influences its chemical behavior [28]. The precipitation of attringite as small crystals rather than large clumps was responsible for the gain in strength due to filling up of pores. The study conducted by James and Pandian (2014) postulated that more attringite with large clusters are formed when PG levels go beyond the optimal dosage, which may be responsible for the decrease in strength [29].

Conclusions

Stabilizing the raw PG with fly ash and lime and curing at elevated temperature resulted in the formation of new hydration products. The raw PG composite with the high percentage of hydration products, yielded the highest strength. Particle size, number of particles and the surface area also played a significant role on the UCS obtained. The TPG shows that at higher temperature (80 °C) UCS is improved and the final strength obtained are lower but within a range of 0.3 – 2 MPa and applicable to be used as backfill bearing material. From the moments of PSD for both the raw and treated PG composite, it is evident that particle size and particle surface area played a significant role on the unconfined compressive strengths obtained. The developed raw PG composites meet the minimum strengths requirements to be classified as a C2-C3 material and the treated PG composite falls under C4 material. The C2/C3 class material can be used as subbase material and C4 as subgrade material (TRH4, 1996). The obtained results also show that the geotechnical properties of the stabilized phosphogypsum, both the raw and treated by lime and fly ash, were significantly improved and in respect to strength the materials are suitable for base/sub-base materials in road construction.

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