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PERFORMANCE ASSESSMENT OF GEOPOLYMER BUILDING BRICKS INCORPORATING SANITARY WARE SLUDGE WASTE

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ABSTRACT

The sludge waste from a sanitary ware production line was used in the manufacturing of geopolymer bricks. The sludge was first dried and then fired at 800ºC. The fired powder was characterized using XRF, XRD, Particle size analysis. Caustic soda and slaked lime were used at percentages of 1% and 6 – 10% respectively. The waste was mixed with the caustic materials in molds whereby geopolymerization took place to produce the required bricks. After four weeks of curing, the produced bricks were tested for their physic-mechanical properties It was found that the compressive strength increased with the degree of geopolymerization and that the properties of the obtained bricks generally conformed to ASTM C 62 / 2017 standard for mixes consisting of 0.5% NaOH, 6% Ca(OH)2 and 45% water. These results were confirmed by SEM imaging.

Keywords: sanitary ware sludge; causitc soda; slaked lime, geopolymer bricks.

1. INTRODUCTION

Traditional solid waste management by landfilling causes deleterious effects on the surrounding environment through several kinds of pollution in addition to the effort and cost needed to dispose of these wastes. This problem is often alleviated by using industrial wastes as raw materials in some industries. Researches in that field have focused on preserve the environment by utilizing diverse types of wastes in friendly and economical ways. In particular, wastes such as blast furnace slag, red mud, fly ash, recycled aggregates, etc. have been often used in the construction industry [1–3].

Geopolymers are categorized as being a type of inorganic polymer that can be prepared at temperatures slightly above room temperature [4]. They are produced by the reaction between a pozzolanic material referred to as "binder" with an alkaline solution (activator). The development of strength in that case is totally different than in the case of ordinary pozzolanic cements. While the development strength of these later type of cements mainly depends on the presence of calcium oxide to form calcium-silicate-hydrates (CSH), it relies in geo-polymers, on the poly-condensation of the pozzolanic binding material in the presence of the activating alkaline solution [5].

Standard pozzolanic binders for geopolymers are bagasse [6], metakaolin [7, 8], fly ash from coal combustion [9, 10], the residue obtained upon incinerating of municipal solid waste [11], glass wastes [12–14], slag waste from metallurgical industries [15–17], etc. In this respect, the carbon dioxide emissions associated with the processes used in the production of geopolymers are much lower than those emitted from the manufacture of OPC [18, 19].

The final properties of geopolymer building components depend to a great extent on the type of raw materials used and the preparation conditions, in particular the curing temperature and curing time. In this connection, Elimbi et al. [20] prepared metakaolin by calcining kaolin at temperatures ranging from 500ºC to 700ºC and concluded that increasing the calcination temperature caused the compressive strength of the produced geopolymer bricks to increase. However, when calcination was carried out at higher temperatures, the strength started to drop. The effect of varying the type of binder was reviewed by Srinivasan et al. [21] with particular emphasis on the merits of using fly ash. On the other hand, Hardijito [22] using a fly ash binder found out that increasing the curing temperature from 30 to 90ºC, caused the compressive strength to increase from 35 MPa to 65 MPa. Also, the effect of varying the binder to activator ratio played a role in assessing the final properties of the geopolymer product, as reported by Rao et al. [23]. These authors used a mixture of blast furnace slag and fly ash as binding material and a mixture of sodium hydroxide and silicate as activator. They concluded that using an activator to binder ratio of 0.4 resulted in the highest compressive strength of the prepared concrete. The use of different alkaline activators has also been researched by several authors. Amin et al. [24] using ground clay bricks powder as binder, replaced most of the costly NaOH by lime water (Ca(OH)$_2$) to obtain geopolymer bricks abiding by ASTM standards. Also, the effect of sodium silicate to NaOH ratio on the properties of fly ash geopolymers was researched by Morsy et al. [25] who obtained a maximum compressive strength at a 1:1 ratio. On the other hand, insulating geopolymer bricks were prepared by El Naggar et al. [26] using clay bricks powder waste binder and slaked lime activator. Porous bricks were obtained from

the reaction of aluminium waste trimmings with the caustic activator whereby values of thermal conductivity as low as 0.26 $W.m^{-1}K^{-1}$ could be obtained.

Traditionally, geopolymers have been prepared by mixing meta-kaolin or any other binder with a strong caustic solution in concentrations reaching 14M [27]. In the current paper, most of sodium hydroxide was substituted by the much less costly calcium hydroxide. Moreover, a priceless waste, namely, the sludge produced from sanitary ware manufacturing, was utilized that helps minimizing pollution and offers an economic and environmentally friendly method for producing building bricks.

# 2. EXPERIMENTAL WORK

2.1. Raw Materials for Bricks

The raw materials used consist of sanitary ware, dried sludge waste, and alkali activators. Sanitary ware sludge is the waste produced during the manufacturing process of ceramic sanitary ware. Slaked lime and caustic soda were used as activators.

Dried sanitary ware waste was analyzed by X-ray fluorescence (XRF) using a wavelength Dispersive (WD-XRF) Sequential Spectrometer for chemical composition, while the phases constituting the raw materials were determined by means of X-ray diffraction (XRD).

Thermal analysis of the dried waste was carried out in air using simultaneous Thermogravimetry – Differential Thermogravimetry (TG–DTG). The heating rate was maintained at $10°C.min^{-1}$. The sample was ground to 200 mesh (74 µm).

The particle size distribution (PSD) of the as-received waste was determined using a Laser Particle Size Analyzer [28], while the densities of the powder waste were determined using the standard Pycnometer method (Density flask) [29].

Slaked lime was prepared using the waste product from acetylene production following the reaction:

$$CaC_2 + 2H_2O = C_2H_2 + Ca(OH)_2$$
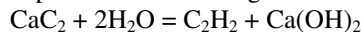
The powder obtained possessed a median size (D_{50}) of 1 mm and a minim size of about 147 µm. The purity of the powder was assessed by comparing its calcium oxide content to the corresponding stoichiometric percentage. It was found to exceed 98%.

Caustic soda was purchased as flakes (MERCK Chemicals) and was stated to contain above 99.9% NaOH.

2.2. Preparation of Geopolymer Building Bricks

The dehydroxylation of kaolin to metakaolin was carried out at 800°C following Plátček et al. [31]. Slaked lime solutions were prepared at concentrations of 6 to 10% as well as dilute solutions of caustic soda containing up to 1% NaOH following the work of Amin et al. [32], Khater et al. [33] and Youssef et al. [34]. The solid binder was then mixed with the solution for half an hour followed by molding in cubic steel molds (50 mm × 50 mm × 50 mm).

After being left overnight in molds, the specimens were removed and kept for 28 days at (25 ± 1) °C.

Following the determination of the most suitable recipe that gave a maximum strength, commercial size brick samples were prepared for testing following ASTM C67 [35]. Curing of these samples was carried out by subjecting them to temperatures reaching 100°C for time periods up to 90 days.

2.3. Testing and Measurement

This step included determination of the degree of geo-polymerization of brick samples having the selected composition after 28 days curing at different temperatures, following the method described by Nikolić et al. [36].

The physic-mechanical properties of the cured samples were determined as function of their composition. These included sintering parameters such as water absorption and porosity besides their compressive strength. Each tested sample consisted of three specimens and the mean value reported.

The compressive strength of commercial size bricks of the chosen recipe (120 mm × 250 mm × 60 mm) was determined as function of curing conditions.

Scanning electron microscope (SEM) test was conducted for some selected specimens. The SEM apparatus used was a Quanta FEG 250 scanning electron microscope with a maximum resolution of 500000×.

3. RESULTS and DISCUSSIONS

3.1. Characterization of Raw Materials

3.1.1 Mineralogical analysis of raw materials

The crystalline phases of the waste powder used were investigated using XRD (Figure-1). The main phases appearing are: kaolinite $Al_2(Si_2O_5)(OH)_4$, quartz (SiO_2), and albite feldspar ($NaAlSi_3O_8$).
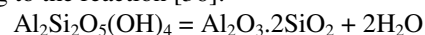
3.1.2 Chemical analysis of raw materials

Table-1 shows the average chemical composition of the dried sanitary ware sludge waste obtained from sanitary ware industry.

3.1.3 Thermal analysis of raw materials

Figure-2 illustrates the TGA–DTA chart for the dried sanitary ware sludge waste.

At temperatures below 200°C moisture is lost, accounts for about 1.3% of the total weight. The first DTG peak appearing at about 362°C normally corresponds to the devolatilization of organic impurities present in the sludge waste, while the main weight drop associated with consecutive DTG peaks starting at about 500°C and ending at about 790°C corresponds to the dehydroxylation of kaolinite resulting in the formation of meta-kaolin according to the reaction [36]:
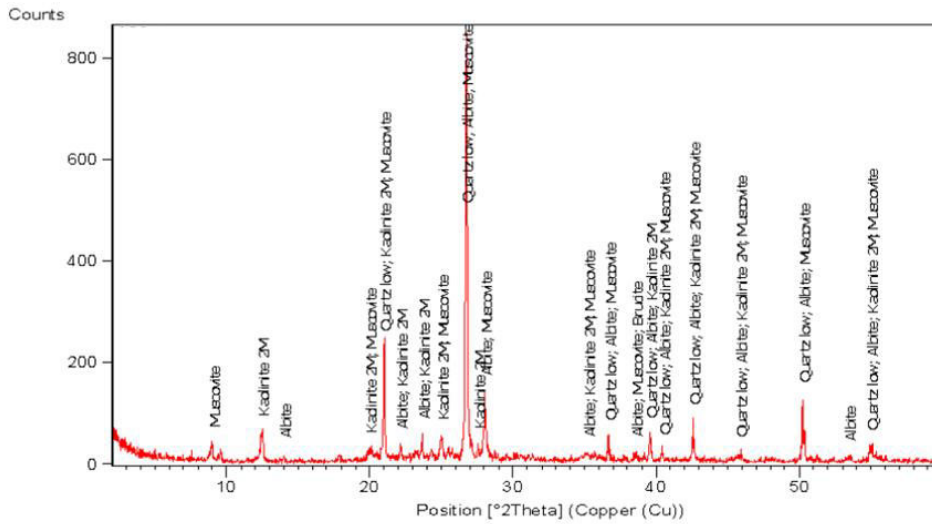
$$Al_2Si_2O_5(OH)_4 = Al_2O_3.2SiO_2 + 2H_2O$$

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Figure-1. XRD pattern for sanitary ware waste.

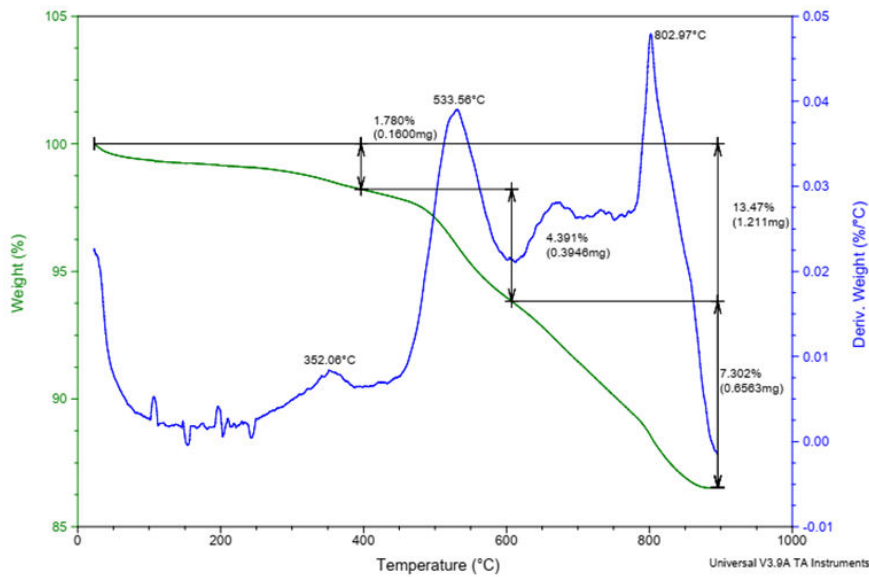**Figure-2.** TGA pattern for dried sanitary ware waste.

Table-1. Chemical analysis of sanitary ware waste.

Constituents	Wt. (%)
SiO_2	61.80
Al_2O_3	19.94
$Fe_2O_3^{tot.}$	2.07
TiO_2	0.37
MgO	0.18
MnO	<0.01
CaO	1.23
Na_2O	1.01
K_2O	1.46
P_2O_5	0.02
Trace elements	0.18
L.O.I	11.6
Total	**99.87**

The weight loss associated with that step amounts to about 9.3%. The final weight loss corresponding to the peak appearing at about 803°C mainly corresponds to the calcination of calcium carbonate present in the raw material and corresponds to a weight loss of about 2.1%. The overall loss in weight excluding the initial moisture loss amounts to about 12.2%, in fair agreement with the value of the loss on ignition obtained from XRF chemical analysis.

3.1.4 Particle size distribution of fired material

Figure-3 illustrates the particle size distribution of the fired sanitary ware waste. The vertical axis represents the percentage passing from any particular screen opening. This figure shows that the meta-kaolin obtained on firing consists of very fine particles of median particle size $(D_{50}) \approx 23\mu m$.
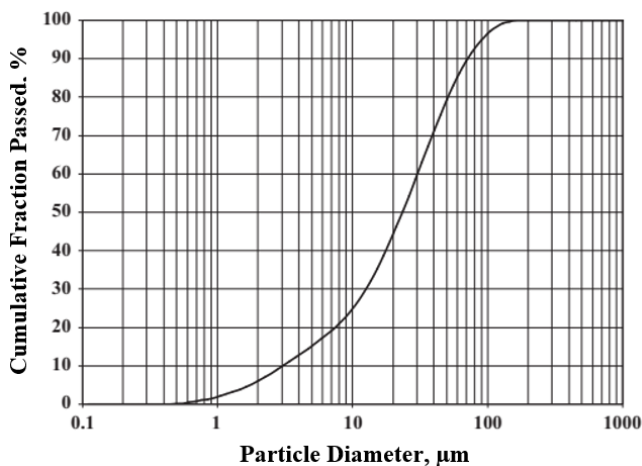
Figure-3. Percent passing against particle size of meta-kaolin used.

3.1.5 Powder density of raw material

The particle density of the powder obtained on firing was identified using the pycnometer method according following ASTM B 311 [30] taking the average value of three readings. The powder density = 2.42 g.cm^{-3}.

3.2. Effect of Percentage Caustic Solutions on the Properties of Geopolymer Bricks

3.2.1 Bulk density (ρ_B)

Figure-4 represents the effect of varying the percentages of both hydroxide and water on the bulk density of the obtained samples. A maximum bulk density is achieved at a NaOH level of about 0.5%, decreasing with increasing Ca(OH)$_2$ addition. The impact of varying the percentage NaOH on the variation in bulk density overcomes that of Ca(OH)$_2$. This figure shows that increasing the NaOH level over 0.5% tends to stabilize or even to slightly lower the bulk density.

This is due to the fact that the formed geopolymer possesses a lower density than the original meta-kaolin raw material as it lies within the range 1.36 – 1.95 g.cm^{-3} [31, 32, 37]. The effect of Ca(OH)$_2$ percentage is less marked, while the effect of percent water used for mixing is irregular. This was assessed by determining the individual correlation coefficients between the three parameters and bulk density. It appears that the percentage NaOH used has the highest effect on bulk density, followed by that of Ca(OH)$_2$, while the effect of water is negligible (Table-2).

3.2.2 Cold and boiling water absorption

The effect of the composition of the activator solution on cold water and boiling water absorption is illustrated in Figure-5. It appears that the percent CWA goes through a minimum value at about 0.5% NaOH, except when 10% Ca(OH)$_2$ was used where water absorption slightly decreased. The level of NaOH at which the absorption is minimal coincides with that at

which a maximum bulk density was observed, which is presumably related to a minimum value of porosity. Varying the amount of water added did not appreciably affect cold water absorption. The observed increase in water absorption associated with increasing NaOH levels over 0.5% can be attributed to the increase in geopolymerization that increases the porosity of the body owing to decreased density [31, 32, 37].

As observed from the figures, the behavior observed for the variation of percent BWA with the activator composition does not differ much from that of percent CWA. In general, BWO tends to rather decrease with as the percent NaOH was raised.

On the other hand, the percentage Ca(OH)$_2$ addition doesn't seem to play any crucial role in assessing the amount of water absorbed, whether on a cold or boiling basis.

Table-2. Correlation coefficients for bulk density.

Parameter	Correlation Coeff.
% NaOH	0.623
% Ca(OH)$_2$	− 0.412
% Water	− 0.163

3.2.3 Saturation coefficient (SC)

Saturation coefficient is simply calculated by the ratio between cold and boiling water absorption. This parameter is indicative of pore size. Large pores allow air bubbles to overcome surface tension and lodge themselves into these pores. In that case, the difference between the readings of CWA and BWA will not be appreciable and the values of SC will be close to unity. Small pores, on the other hand, by preventing access to air bubbles will result in much lower values of SC [38]. The current results indicate that the values of SC remain practically unaffected by sludge addition, ranging from 0.76 to 0.86.

3.2.4 Apparent porosity (P)

The results of variation of percent apparent porosity with percent caustic solutions and water expectedly matched with those of percent BWA.

Figure-6 shows a plot between the values of apparent porosity and those of bulk density determined under different conditions of caustic levels and waste percentages. They reveal an almost linear relationship between these two parameters.
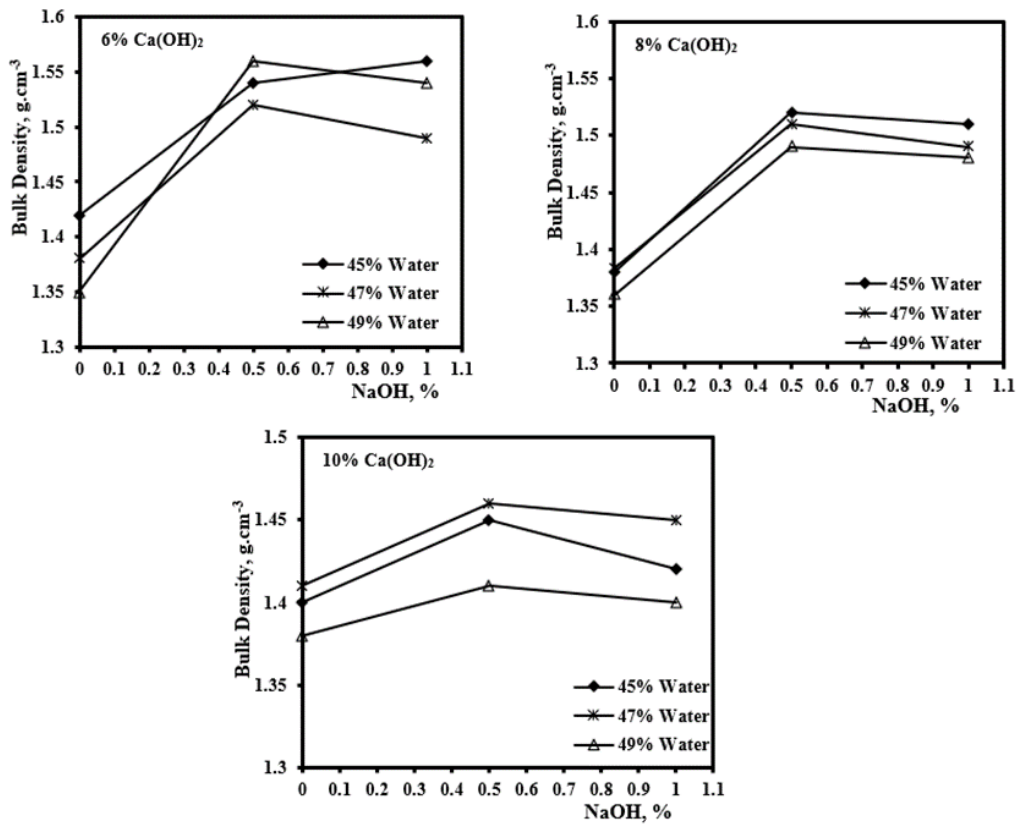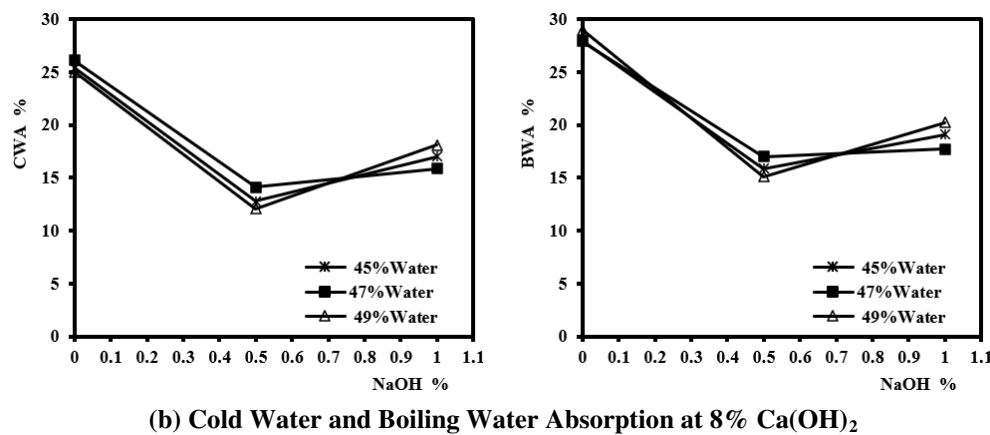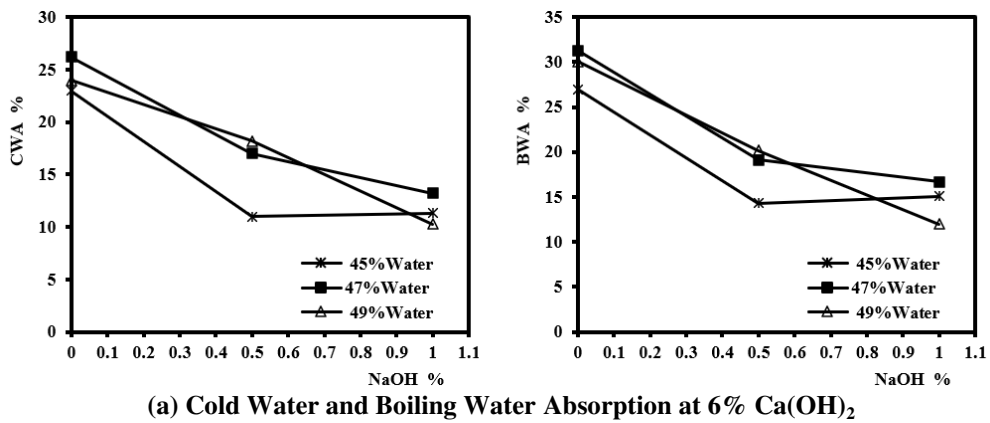
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Figure-4. Effect of percent NaOH and water on bulk density at different percent $Ca(OH)_2$.

(a) Cold Water and Boiling Water Absorption at 6% $Ca(OH)_2$

(b) Cold Water and Boiling Water Absorption at 8% $Ca(OH)_2$

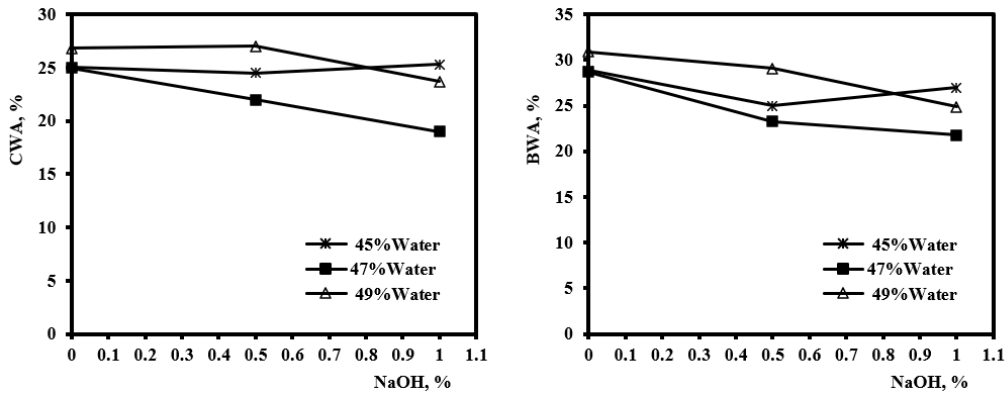**(c) Cold Water and Boiling Water Absorption at 10% Ca(OH)$_2$**

Figure-5. Effect of percent NaOH and water on both cold and boiling water absorption at different percent Ca(OH)$_2$.
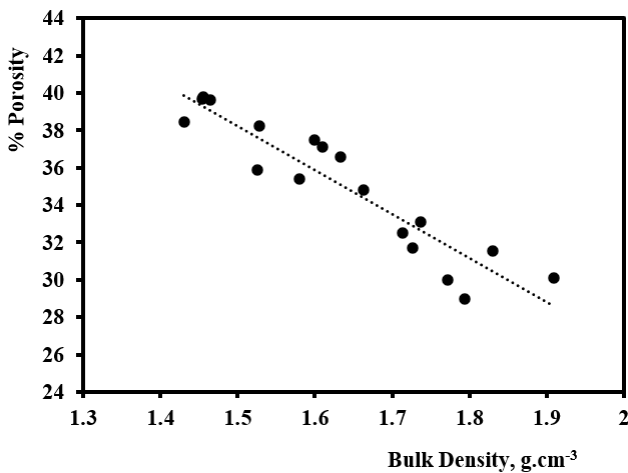

Figure-6. Relation between apparent porosity and bulk density.

3.2.5 Compressive strength

Compressive strength is actually the sole property specified by the latest specifications for bricks to be used in mild weather. A minimum compressive strength of 8 MPa is required for this type of brick as per ASTM C 62 [40]. The combined effect of all three parameters (% NaOH, % Ca(OH)$_2$ and % water) on the 28 days compressive strength of bricks is shown in Figure-7. As the level of polymerization increases, there is a corresponding increase in strength reaching a maximum value at a concentration of 0.5% NaOH is reached. Following this value, the strength tends to slightly decrease owing to an increase in porosity as revealed by the increase in water absorption (Figure-5). The effect of variations in slaked lime concentration on strength is negligible, presumably because of the low degree of ionization of Ca(OH)$_2$ in solution compared to that of NaOH. Finally, increasing the level of water used will increase porosity through evaporation, resulting in a decrease in strength.

According to Figure-7, the minimum strength, as required by ASTM C 62 [40] for normal duty bricks

(8.7 MPa) was only reached by samples of composition 0.5% NaOH, 6% Ca(OH)$_2$ and 45% water which produced a compressive strength of 9 MPa. This composition was therefore chosen for further investigation.
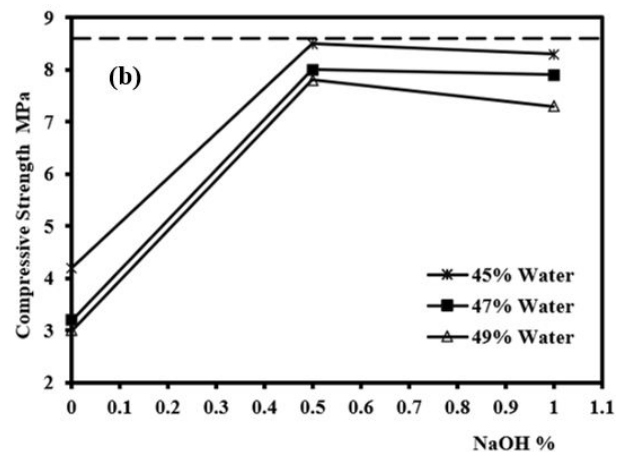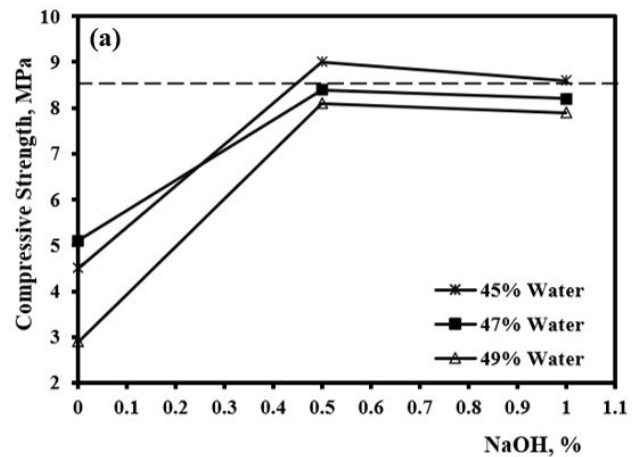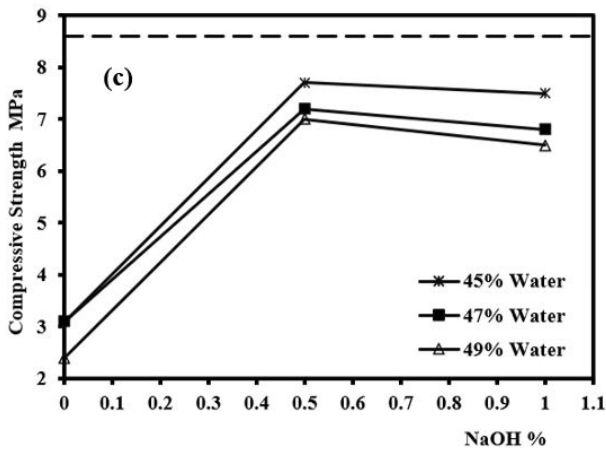
Figure-7. Effect of percent NaOH and water on compressive strength at different percent $Ca(OH)_2$: (a) at 6% $Ca(OH)_2$, (b) at 8% $Ca(OH)_2$, (c) at 10% $Ca(OH)_2$.

3.3. Effect of Curing Time and Temperature on the Properties of Commercial Size Geopolymer Bricks

As previously stated, ASTM C67 stipulates that curing must be carried out on commercial size bricks (120 mm × 250 mm × 60 mm). The mix exhibiting the highest compressive strength (Fired sanitary ware sludge waste blended with 45% water, 0.5% NaOH, and 6% $Ca(OH)_2$) was therefore cured for time periods for up to 90 days at different temperatures. The effect of curing temperature and time on the different properties of the bricks were investigated. Also, the degree of polymerization of samples cured for 28 days was followed as function of curing temperature.

3.3.1 Apparent porosity and bulk density

Figure-8 indicates that an increase in either curing time or temperature will have for effect to increase the apparent porosity and hence decrease the bulk density of tested bricks. This is presumably due to the fact that a higher curing temperature will increase the vapor pressure of water, leading to the creation of more porosity as vapor leaves off. A similar effect is observed on prolonging the curing period thus giving more time for vapor to diffuse through the body.

3.3.2 Cold and boiling water absorption

As expected, the values of water absorption (whether cold or boiling) will follow a similar pattern as those of porosity, except that the figures of BWA will be slightly higher than those of CWA as explained in section (3.2.3). After 90 days, the values of water absorption tend to stabilize as of 40ºC as the degree of geo-polymerization reaches its maximum value. For lower curing times, the process slows off at temperatures exceeding 40ºC, as evidenced by the decreasing slopes of the lines.
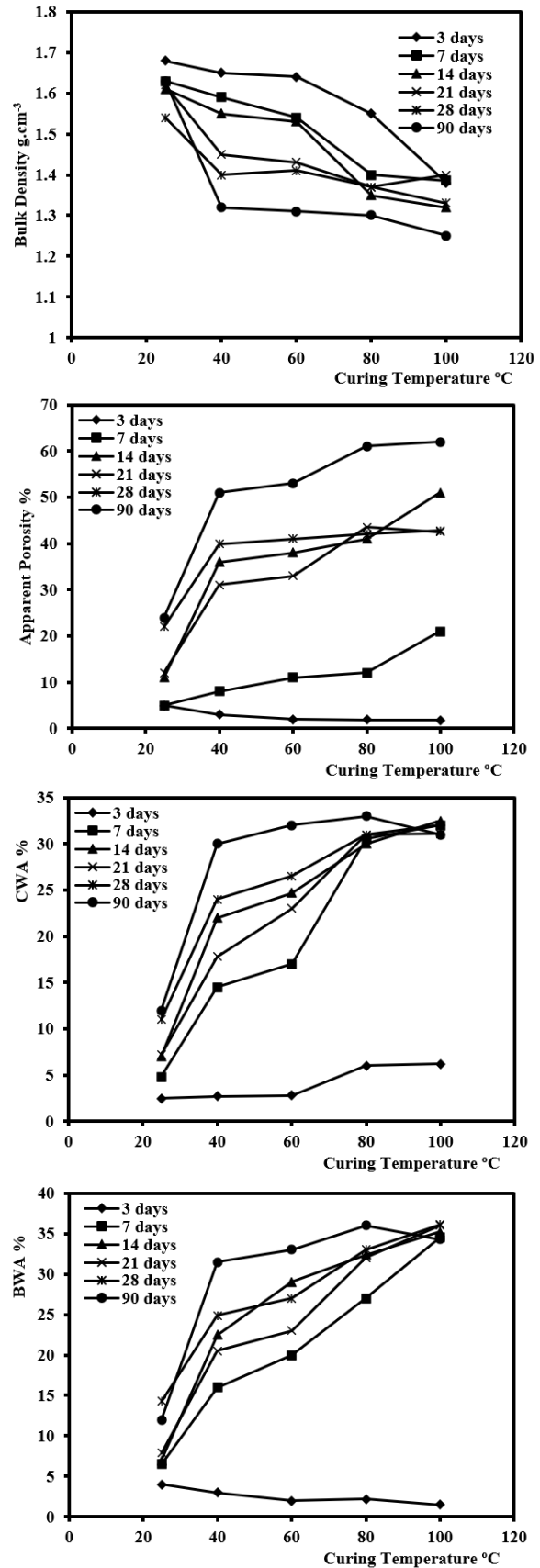

Figure-8. Effect of curing time and temperature on the physical properties of geopolymer bricks.

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3.3.3 Compressive strength

Brick samples cured at different time periods and at different temperatures were subjected to compressive loading. Figure-9 reveals that prolonged curing at any temperature will result in increased strength as polymerization proceeds. On the other hand, and in contradiction with the findings of several authors [42–45] an increase in temperature was associated with a decrease in strength. This is presumably caused by the increase in porosity following the evaporation of water enhanced by increased temperature resulting in the creation of more pores that cause the observed drop in strength. It also appears that the degree of polymerization of samples cured for 28 days decreases with increased temperature as the amount of available water for the process decreases (Figure-10). This could explain the behavior of the variation of strength with temperature.

The compressive strength of the samples was determined as a function of curing time and temperature for periods reaching 90 days. The effect of curing conditions is illustrated in Figure-9. It is clear that increasing the curing time has a beneficial effect on strength [41], while increasing the curing temperature tends to decrease the strength (except perhaps for 3 days samples). Actually, this contradicts most of the available literature which report that increased curing temperature enhances compressive strength. It is believed that the large increase in porosity following increased curing temperature could account for the observed drop in strength. This can be attributed to the decrease in liquid content in the prepared bricks associated with increased temperature [46–48]. The dotted line in Figure-9 represents the minimum allowable strength for normal duty bricks as stipulated by ASTM C 62 (8.7 MPa). It can be seen that it takes at least 28 days of curing at a maximum temperature of 40°C to exceed that Figure.

On the other hand, it can be seen from Figure-10 that the extent of geopolymerization linearly decreases with increased temperature, which readily accounts for the drop in compressive strength following increased curing temperature. This effect is emphasized in Figure-11 where it appears that there is an almost linearly increasing dependence of strength on the extent of geopolymerization.

3.4. SEM Results

Figure-12 illustrates a comparison between SEM micrographs of two specimens: the first was pre-pared using 0.5% NaOH and 6% $Ca(OH)_2$ while the second contained 1% NaOH and 10% $Ca(OH)_2$. Both were cured for 7 days at room temperature. While the first specimen showed no apparent porosity, the second one revealed the presence of some pores, in line with the findings related to water absorption, illustrated in Figure-5c.
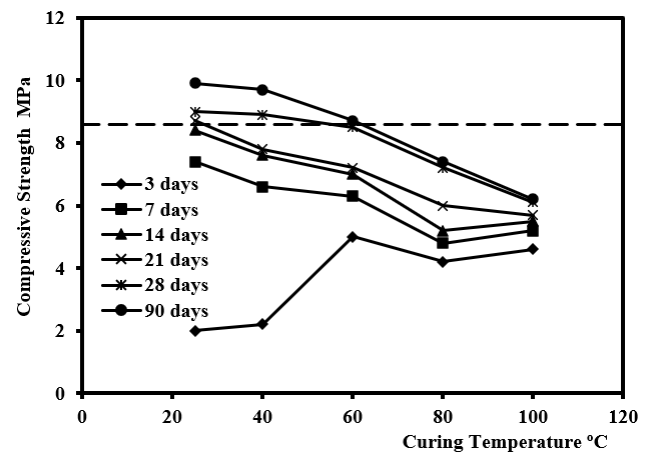

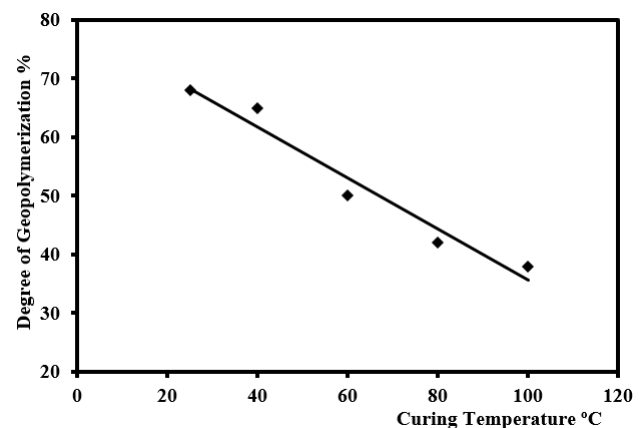**Figure-9.** Effect of curing time and temperature on the Compressive Strength of geopolymer bricks.

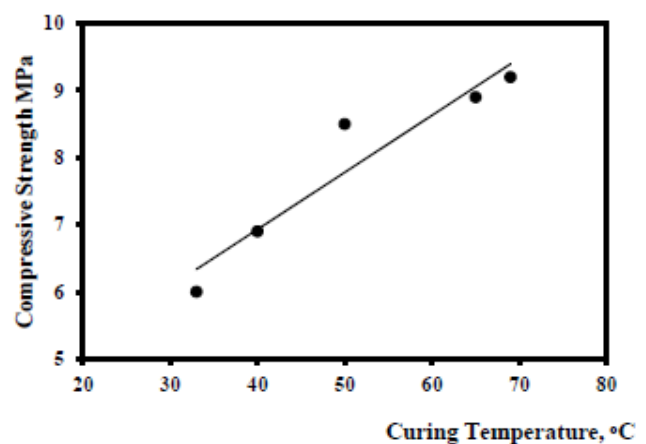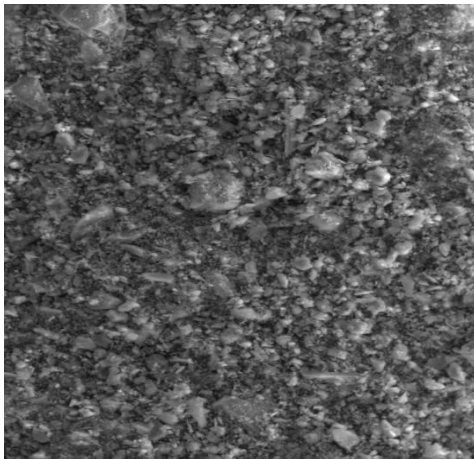**Figure-10.** Effect of curing temperature on the degree of geopolymerization.

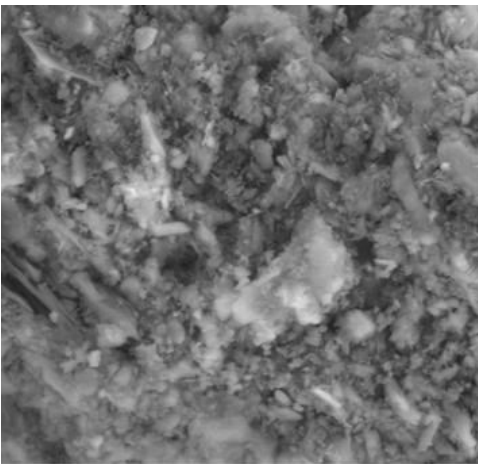**Figure-11.** Effect of the degree of geopolymerization on 28 days strength.

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(a)

(b)

Figure-12. SEM micrographs (150×) for two chosen samples:
(a): Sample with 0.5% NaOH, 6% $Ca(OH)_2$ and 45% water, cured at room temperature for 7 days;
(b): Sample with 1% NaOH, 6% $Ca(OH)_2$ and 45% water, cured at room temperature for 7 days.

3.5. Quality Assessment

As mentioned earlier, five geopolymer brick samples with dimensions $(120 \times 250 \times 60)$ mm^3 consisting of burnt sanitary sludge waste mixed with 45% water, 0.5% NaOH, and 6% $Ca(OH)_2$ were prepared. After curing at room temperature, they were tested according to ASTM C 62 [40] for quality assessment [30].

The bricks were found to have a minimum individual compressive strength of 8.9 MPa, a maximum BWA of 12%, and a saturation coefficient of 0.81, all of which are compatible with ASTM C 62 for mild weather normally loadable building bricks.

4. CONCLUSIONS

In the present study, a geopolymer body was prepared by calcining the dried sludge waste obtained from the residue of sanitary ware making with slaked lime and caustic soda.

First, the specimens consisted of $(50 \times 50 \times 50)$ mm^3 cubes that were found to exhibit minimum percent water absorption at 0.5% NaOH, corresponding to a maximum bulk density. This is since bulk density is a decreasing function of porosity and the associated water absorption.

The composition that exhibited a maximum 28 day compressive strength of about 9 MPa consisted of 6% $Ca(OH)_2$, 0.5% NaOH, and 45% water. This value of compressive strength exceeds the minimum requirements for normal duty building bricks. This was used to prepare commercial size bricks $(120 \times 250 \times 60)$ mm^3, the curves of water absorption of which generally showed an increase with both curing time and temperature.

It was also found that an increase in curing temperature caused evaporation of water thus decreasing the quantity available for the polymerization reaction. This was accompanied with a decrease in the extent of geopolymerization with a corresponding drop in strength.

Bricks with the aforementioned recipe cured at room temperature abided by the specifications of ASTM C 62 for normal duty building bricks and represent a reusability of a waste that is normally discharged to drain. The following Table summarizes the comparison between the obtained results and the ASTM requirements.

Table-3. Comparison of the properties of prepared bricks standards.

Property	Prepared brick	Normal duty	Medium duty
Compressive strength, MPa	8.89	> 8.7	> 15.2
Cold water absorption, %	13.6	No upper limit	No upper limit
Boiling water absorption, %	12.0	No upper limit	22
Saturation coefficient, %	0.81	No upper limit	< 0.88
Bulk density, kg/m^3	≈ 1300	No recommended value	No recommended value

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