



Preparation of Geopolymer Bricks from Alum Waste

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Abstract

Geopolymers are conventionally prepared by the reaction between a pozzolanic binder and an alkaline activator. In the present work, the waste from alum industry, commonly known as “dealuminated kaolin” (DK) was used as binder and sodium hydroxide solutions (with molarities ranging from 2 to 14 mole.L⁻¹) as activator. The waste powder was first dried, finely ground and characterized by X – Ray fluorescence (XRF) and X – Ray Diffraction (XRD) analyses as well as SEM micrography. This was then mixed with the alkaline activator in a mass ratio of DK to NaOH solution equals 0.7 and molded in 50 mm steel molds. The effect of curing time and molarity of the alkaline solution on the properties of the produced bricks were assessed. It was observed that the degree of polymerization after 28 days generally increases with the increase of NaOH solution up to 8–10 M to reach about 90% after which the effect of NaOH concentration is less pronounced. Water absorption was found to drop considerably following the increase in NaOH concentration from 4 M to 14 M. The results also showed that the 28 days compressive strength reaches the minimum allowable value of strength of building bricks (8.7 MPa according to ASTM C 62) starting from NaOH concentrations of about 12 M.

"Keywords: Dealuminated Kaolin; Geopolymer; Bricks; Alum; Waste"

1. Introduction

Although the term “brick” was originally restricted to shaped units made of clay, it has now broadened to include cement based and geopolymer bricks. These latter types are normally shaped by pressing or casting while clay bricks are either formed by pressing or stiff mud extrusion before being dried and fired [1–4].

The production of geopolymer bricks, on the other hand, was originally based on the pioneering work of Davidóvits [5]. The preparation process is based on the effect of an alkaline substance (activator) on a pozzolanic material (binder). The reactions result in the formation of the polymeric bonds Si – O – Al – O with the following formula: $M_n[-(SiO_2)_z - AlO_2]_n \cdot xH_2O$ [6].

Classical choices for both ingredients were caustic soda and meta-kaolin respectively. In this connection, Hamdi et al. [7] presented a comprehensive review regarding the different types of cheap clays that can be used as fillers. In that review, they compared between

the properties of geopolymers obtained from each type of clay.

Economic production of geopolymer bricks was subsequently carried out by using waste materials as fillers or activators. Youssef et al. [8] used clay bricks refuse as source of meta-kaolin and NaOH as activator. They determined that a 5% reduction in the cost of the geopolymer bricks obtained was realized. Using the same approach, El Naggar et al. [9] prepared geopolymer bricks using waste lime (Ca(OH)₂) instead of the much more expensive NaOH as activator. The possibility of using iron slag as binding material was reported by Ionescu et al. [10] who reviewed the previous studies carried out on the different types of slag. They concluded that heat treatment of mixes at about 100 °C was necessary to improve the strength characteristics of the produced geopolymer components. On the other hand, the use of fly ash as binder has been widely investigated and several review articles were published on the subject.

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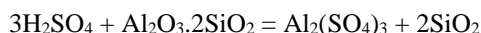
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In particular, Jiang et al. [11] compared between the properties of geopolymers obtained on using different types of fly ash as binders. Also, in a comprehensive work, Amran et al. [12] reviewed the effect of different types of activators used in conjunction with fly ash binders on the properties of the formed bricks. They concluded that NaOH, KOH and Na_2SiO_3 are the most suited activators ensuring long term durability of the bricks. On the other hand, the effect of using iron slag on the mechanism of polymerization of fly ash geopolymers and was studied by Nath and Kumar [13]. They proved that the formation of gel phases like calcium silicate hydrate contribute to increasing the strength of the formed geopolymers. Another common binder is silica fume, which is a very fine amorphous form of silica produced as by-product from the silicon or ferrosilicon industry [14]. This material has been used by several associated with iron slag [15] or with fly ash [16,17]. In all cases the presence of silica fume contributed to improving the mechanical properties of the obtained geopolymer.

On the other hand, dealuminated kaolin (DK) is a waste by-product formed by the action of sulfuric acid on meta-kaolin in the manufacturing process of alum. The basic reaction is:



This waste is particularly problematic because of its acidic nature and the large amounts generated which are normally landfilled with negative effect on aquifers.

The use of dealuminated kaolin as cement replacement materials was introduced by Abdelaziz et al. [18]. Their study showed that the use of as received (DK) as in cement paste accelerates the setting time while the lime-treated samples lead to its retardation. While the use of conventional strength enhancers such as lime or silica fume normally results in decreased flowability, the addition of DK hardly affected the flow characteristics of cement pastes. These authors found out that the use of up to 10% cement replacement by DK resulted in an improvement in tensile and in 8 weeks compressive strengths.

The pozzolanic activity of alum waste (DK) and calcined kaolin (CK) was investigated using cement kiln dust (CKD) or hydrated lime (CH) as activators by Abo El-Enein et al. [19]. A set of combinations of both activators and binders was prepared and the chemically combined water as well as free calcium

oxide were determined using Differential Scanning Calorimetry (DSC), X-Ray diffraction (XRD) and Fourier Transform Infra-Red Analysis (FTIR). The higher pozzolanic character of DK was explained as being due to the lack of free lime compared to other binders such as calcined kaolin and silica fume.

Dealuminated kaolin was also used for the manufacture of forsterite type refractories (Mg_2SiO_4). Mixtures of dead burnt magnesite and alum waste were sintered and showed excellent refractoriness [20]. They were suggested to be used in the clinkering zone of cement rotary kilns owing to their basic character besides excellent refractory properties.

On the other hand, Mostafa et al. [21] found out that using dealuminated kaolin increased the rate of strength development when used to partially substitute cement. In that respect, the rate of strength increase was higher than when silica fume was used at the same substitution levels.

A study by Bendary et al. [22] investigated the possibility of using dealuminated kaolin as cementitious material in the building industry. Sand was substituted by percentages of 0% to 10% of DK and the flow properties, initial and final setting and 28 days compressive strength determined as function of percent substitution. The results revealed that it was possible to substitute sand by 7.5% DK without impairing the properties of the cement mortar.

A recent research by Abdelmawla et al. [23] was carried out for the preparation of geopolymer cubes from two wastes, the first being dealuminated kaolin waste (DK) and the second water treatment sludge (WTS). They studied the effect of varying the ratio of DK / WTS used, and the amount and concentration of NaOH alkaline solution on the compressive strength of cubes. Their results showed that the optimum conditions providing maximum strength are a $\text{Na}_2\text{O} / \text{SiO}_2$ ratio of 0.56 and WTS / DK ratio of 30:70, 60 g NaOH with a concentration of 10 N and curing at 70 °C for 72 hours. The compressive strength of WTS / DK geopolymer reached 22 MPa after 28 days. However, these authors failed to indicate the relative amount of caustic soda used to that of DK, which made the assessment of the economics of the process questionable.

The aim of this study is to prepare geopolymer bricks suitable for use as building material by using the dealuminated kaolin waste produced from alum industry as the pozzolanic binding material through the reaction with sodium hydroxide activator (NaOH).

Physical and mechanical properties of these bricks will be investigated as function of caustic soda molarity and curing time.

2. Experimental Work

2.1. Raw materials

Two main raw materials are used in this work

- Dealuminated kaolin (DK), a waste from the alum industry that was kindly supplied by the alum factory of “El-Nasr Company for Intermediate Chemicals (NCIC)”, Egypt. This waste was dried at 100 °C and finely ground.
- Pure sodium hydroxide pellets, supplied by “El-Sharq El-Awsat Company for chemicals”, Egypt.

2.2. Characterization of raw materials

The following tests were performed on the waste powder:

Chemical Analysis of Raw Material (XRF) analyses were run on an AXIOS, analytical 2005, Wavelength Dispersive (WD-XRF) Sequential Spectrometer. This analysis technique allows for the quantitative determination of the chemical species constituting a material. It relies on irradiating the specimen by an X-ray beam, causing this specimen, in turns, to emit X-rays of his own. The wave lengths of the emitted rays are characteristic for each component [24].

The raw materials mineralogical composition was determined by X-ray diffraction (XRD) Bruker D8 advanced computerized X-ray diffractometer apparatus with monochromatic $\text{CuK}\alpha$ radiation, operated at 40 kV and 40 mA. In that method of analysis, cathode rays are allowed to impinge against a copper target causing the emission of X rays. These rays are allowed to collide with the tested material which causes them to diffract at specific angles for each phase. This way, the phases present in the material can be identified [24].

Scan Electron Microscopy (SEM) was used to assess the morphology of the fired samples. A JEOL-JSM 6510 apparatus (zoom magnification power up to 300,000) was used to generate SEM images for some selected fired specimens. These images are formed when an electron beam is made to interact with the surface of the material. This causes the emission of secondary electrons, X rays and light emissions which carry information about the morphology of the

material. This is converted as images appearing on a TV screen [24].

Screen analysis is used to sort granular solids to definite size fractions. A laboratory sieve set consists of a series of sieves of standard openings placed one above the other in decreasing opening sizes from top to bottom. A pan is placed beneath the bottom screen to collect the fine dust remaining. The granular solid is poured onto the surface of the top screen and the set shaken for about one hour. The amounts retained on each sieve are then determined and the percent by weight of each is calculated. The standard sieving procedure described by [ASTM 276–13] was used [25].

Determination of the powder true density [ASTM B 311] was used [26]. This was varied out using the conventional density bottle technique.

2.3. Preparation of building brick specimens

Dried dealuminated kaolin was first ground to pass mesh 20 sieve (0.814 mm). Different concentrations of the alkaline sodium hydroxide solution are prepared (from 2 M to 14 M). At each concentration, the alkaline solution was added to the DK waste in a mass ratio of DK solid to NaOH solution equals 0.7 and thoroughly mixed. After mixing, the mixture was poured in a rectangular mold divided into three partitions. Each partition consisted of a cube of dimensions $50 \times 50 \times 50 \text{ mm}^3$. The mold was then put on a shaker for around 10 minutes to remove any trapped air that and to ensure complete homogeneity of the mixture. The mixtures were left in molds till drying then removed and left to cure at room temperature and under normal humidity conditions. The compressive strength, water absorption and degree of geopolymerization are then tested on specimens after 7, 14, 21, 28 days. Specimens' preparation and testing were carried out in the Chemical Engineering Department, Cairo University. Samples after demolding are shown in Fig. 1.



Fig. 1: Bricks specimens after demolding

2.4. Testing of building brick specimens

The following tests will be performed to study the effect of change of sodium hydroxide and curing time on brick properties:

- ❖ Determination of the degree of geopolymerization
- ❖ Determination of bulk density [27].
- ❖ Water absorption & Saturation coefficient
- ❖ Determination of Compressive Strength [ASTM C 67]. The machine used in the test of type (SHIMADZU, UH-500kNIR) [28]

3. Results and Discussion

3.1. Characterization of raw materials

The XRF analysis of alum waste is shown in Table (1). It reveals the predominance of silica in a proportion exceeding 70%. The presence of alumina is due to a minor amount of unreacted kaolinite and aluminum sulfate. This is emphasized by the presence of slightly more than 5% sulfate ion. As the sum of silica and alumina content exceeds 70% the material can be classified as pozzolanic in nature [29].

The amount of silica present in unreacted metakaolin can be deduced from that of free silica. This was determined using the Trostbl method [30] and found to equal 66.4%, meaning that only about 5.5% of silica was in combined form.

Mineralogical analysis of alum waste shown in Fig. 2 displays main peaks of quartz with minor peaks of titanium oxide (anatase). No peaks for amorphous metakaolin appeared, as expected.

The morphology of the used powder was assessed by scan electron Microscope (SEM) micrographs, one of which illustrating the presence of large quartz grains to which adhered smaller particles, is shown in Fig. 3.

The particle size distribution of the powder was determined by the standard sieves. The cumulative analysis is presented in Fig. 4 and reveals that the median particle size $D_{50} = 0.21$ mm.

Table (1): Chemical (XRF) analysis of alum waste

Constituents	(%)
SiO ₂	71.86
TiO ₂	2.95
Al ₂ O ₃	7.48
Fe ₂ O ₃ ^{tot.}	0.51
MgO	0.08
CaO	0.31
Na ₂ O	0.11
K ₂ O	0.08
P ₂ O ₅	0.05
SO ₃	5.28
Cl	0.02
ZrO ₂	0.2
L.O.I	10.98
Total	99.91

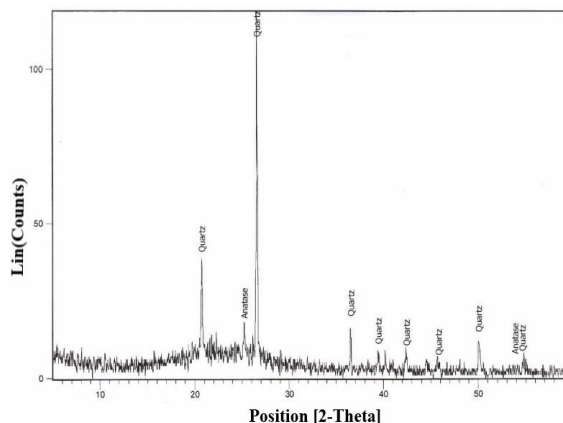


Fig. 2: XRD pattern of alum waste

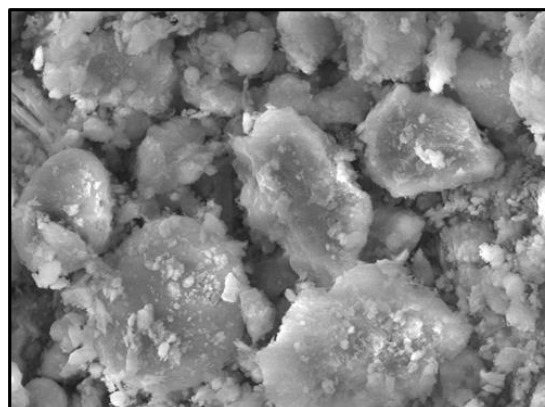


Fig. 3: SEM micrograph of the as-received waste powder (4000×)

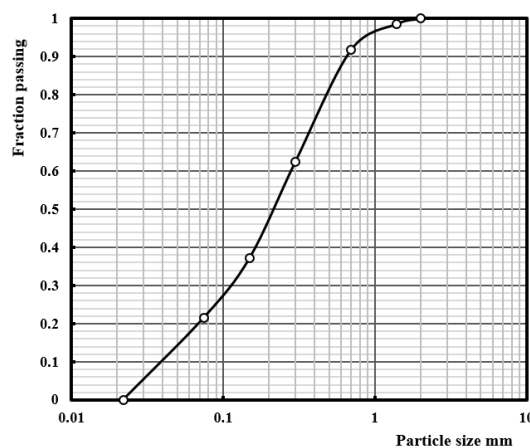


Fig. 4: Particle size distribution of waste alum powder

True density of waste alum powder was determined using the density bottle and found to equal 2.8 g/cm³.

3.2. Physical properties of the prepared geopolymer samples

Degree of geopolymerization: The method used in determining the degree of geopolymerization was explained by Nikolić et al. [31]. In Fig. 5, it appears that the effect of curing time shows strongly at earlier periods but fails to seriously affect the degree of geopolymerization as of 14 days curing. Also, the molarity of sodium hydroxide solution has a strong bearing on the degree of geopolymerization up to about 8 to 10 M., after which its effect is less pronounced.

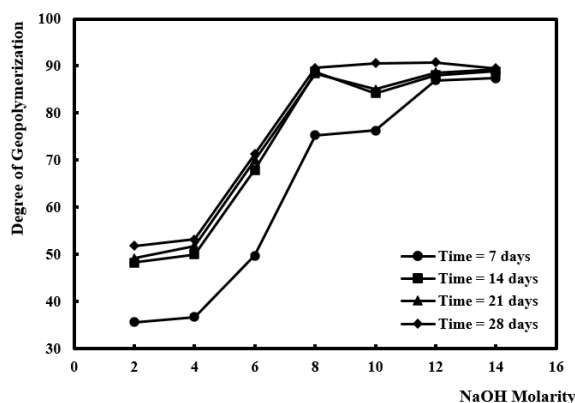


Fig. 5: Effect of NaOH molarity and curing time on the degree of geopolymerization

To assess the relative contribution of the two parameters (NaOH molarity and curing time) on the degree of geopolymerization, the correlation Table (2) was set up. It shows that the much stronger dependence of the degree of geopolymerization (D.G.P.) on curing time rather than on NaOH molarity, as evidenced by their respective correlation coefficients.

Table (2): Correlation table for the degree of geopolymerization

	Molarity	Time	D.G.P.
Molarity	1		
Time	0	1	
D.G.P.	0.880	0.235	1

It was possible to establish a linear empirical correlation between the degree of geopolymerization (DGP) and both NaOH molarity (M) and curing time (t days) in the following form, with a reasonable correlation coefficient = 0.9:

$$DGP = 40,132 + 3.652M + 0.264t \quad (1)$$

The correspondence between the observed and calculated values of degree of geopolymerization using that expression is presented in Fig. 6.

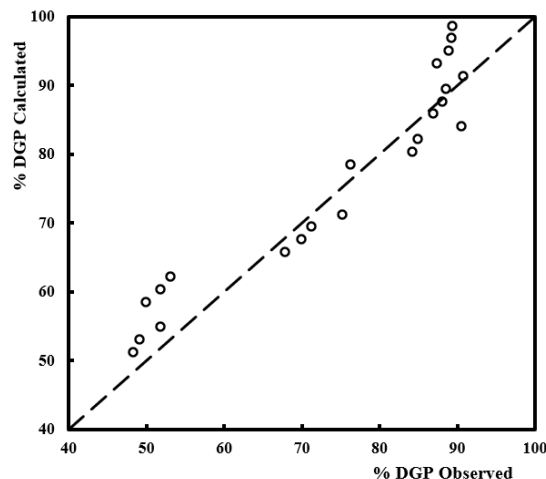


Fig. 6: Comparison between calculated and observed values of % DGP

Bulk density: The results of bulk density, presented in Fig. 7, point out at as strong dependence on NaOH molarity and a much lower effect of curing time. In general, it is clear that as the caustic soda molarity is increased, there is an increase in density that nearly stabilizes at a molarity of 12. On the other hand, the produced geopolymer, having a lower true density than the original material caused a lowering in bulk density on prolonged curing. According to Aredes et al. [32], the average specific gravity of kaolinite based geopolymers is about 2.24, a figure lower than that of the original aluminum waste. On the other hand, the correlation of Table (3) corroborates these findings as it shows an extremely elevated dependence of bulk density on NaOH molarity and a weak negative variation with curing time.

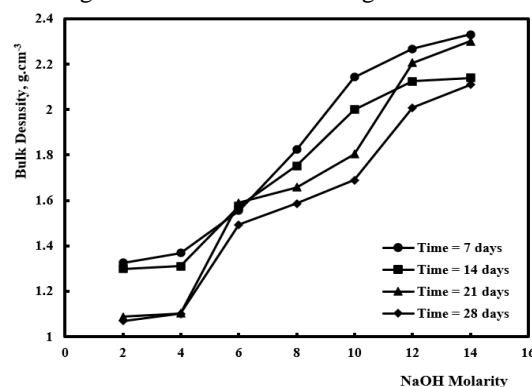


Fig. 7: Effect of NaOH molarity and curing time on the bulk density

Table (3): Correlation table for bulk density

	Molarity	Time	D.G.P.
Molarity	1		
Time	0	1	
D.G.P.	0.949	- 0.229	1

Here also, it was possible to correlate the bulk density to the molarity of NaOH and curing time to obtain the following expression with a correlation coefficient = 0.976. The corresponding comparison between calculated and observed values of bulk density is shown in Fig. 8.

$$\rho_B = 1.187 + 0.0937M - 0.0127t \quad (2)$$

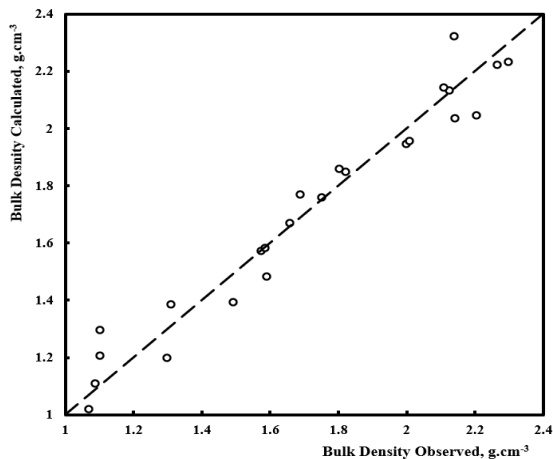


Fig. 8: Comparison between calculated and observed values of Bulk density

Water absorption: The percent water absorption was determined for both cold and boiling absorption, after the termination of the 28 days of the curing period. The effect of NaOH molarity is shown in Fig. 9. It appears from that figure that there is a common trend to both curves consisting of a decrease in water absorption following increased caustic soda molarity. This is due to the increase in bulk density that accompanies more concentrated NaOH solutions, resulting in lower porosities.

To assess that point, the values of boiling water absorption obtained after 28 days curing for all NaOH molarities were plotted against the bulk density. A decreasing relation was obtained as clear from Fig. 10, emphasizing the reduction in water absorption as the porosity is reduced resulting in increased bulk density.

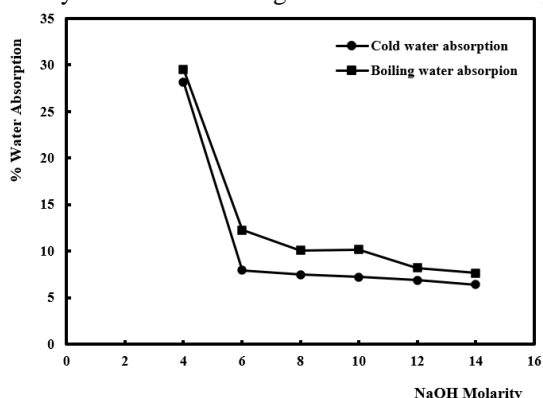


Fig. 9: Effect of NaOH molarity on water absorption after 28 days curing

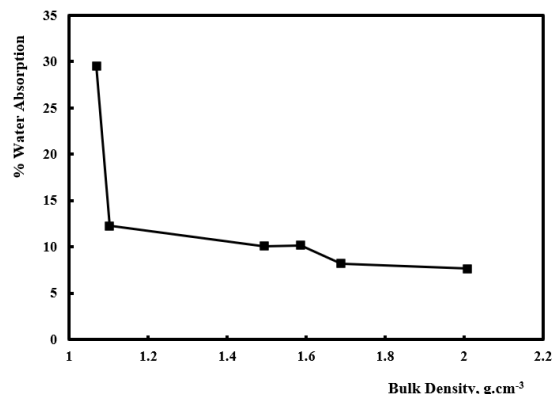


Fig. 10: Relation between percent boiling water absorption and bulk density

Saturation coefficient: This parameter indirectly reveals the size of the available pores. Large pores allowing access for cold water as well as boiling water will have saturation factors close to unity. This variable was only determined after 28 days.

The variation of saturation coefficient with sodium hydroxide concentration after 28 days curing reveals that at low concentration, the degree of geopolymerization being moderate, the available pores are relatively large in size. Their volume however tends to decrease on increased polymerization resulting in a sudden drop in saturation coefficient to a very low value (0.65). The slow increase in that parameter following the use of more concentrated activator solution has to do with the reduction in density following further formation of the geopolymer which results in the formation of relatively larger pores. Fig. 11 illustrates these effects. The saturation coefficient is seen to increase from 0.65 at 6 M NaOH to 0.84 at 14 M NaOH. This was emphasized by examining specimens of bricks prepared using 6 M and 14 M NaOH and cured for 28 days under SEM.

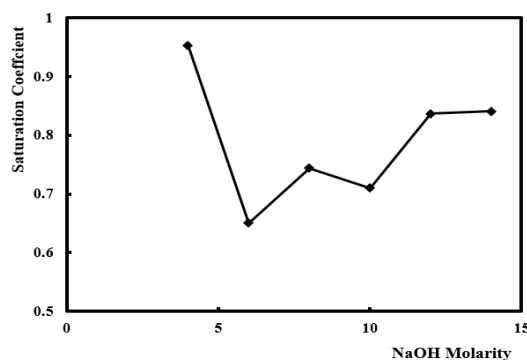


Fig. 11: Variation of saturation coefficient with NaOH molarity

SEM micrographs taken for bricks core on using 6 M and 14 M NaOH show that the pores in the latter

case are larger than in the former. Fig. 12(a), illustrates a micrograph taken at 800× magnification for a specimen prepared using 6 M NaOH and cured for 28 days, it is clear that the dimensions of pores range from about 8 μm to 18 μm. On the other hand, a micrograph taken for a specimen prepared with 14 M NaOH, shown in Fig. 12(b) reveals that the size of pores has increased to range from 13 μm to 72 μm.

Compressive strength: The most essential mechanical property for most types of bricks is their compressive strength. As geopolymerization goes on the material acquires more strength despite the slight volume expansion accompanying the formation of a new phase of lower density than the original material. The combined effect of curing time and NaOH normality is displayed by the set of curves in Fig. 13.

The logarithmic nature of the fitted curves suggests that it is possible to establish a correlation between σ and $\ln M$ and t , where σ is the compressive strength (MPa), M the molarity of NaOH and t the curing time (days). This correlation takes the following form with a determination coefficient $R^2 = 0.966$:

$$\sigma = -9.638 + 5.054 \ln M + 0.211t \quad (3)$$

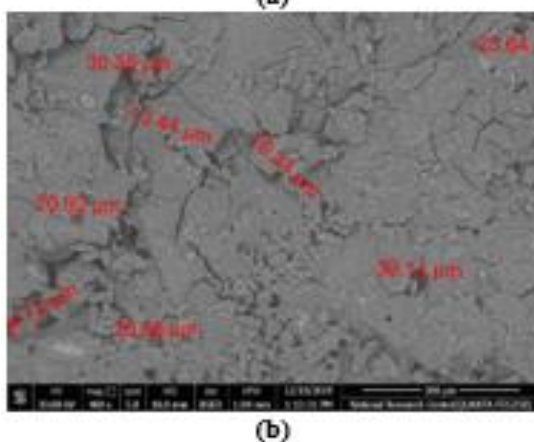
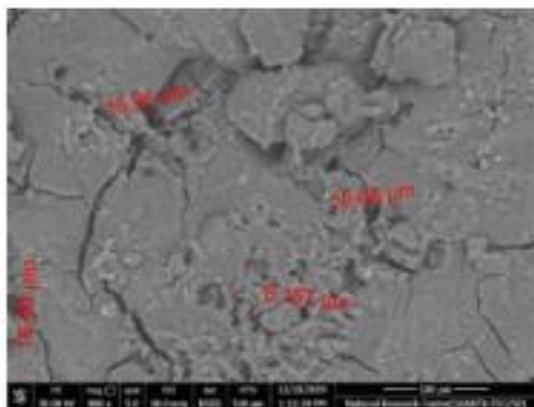


Fig. 12: Micrographs for samples: (a) 6 M NaOH, and (b) 14 M NaOH

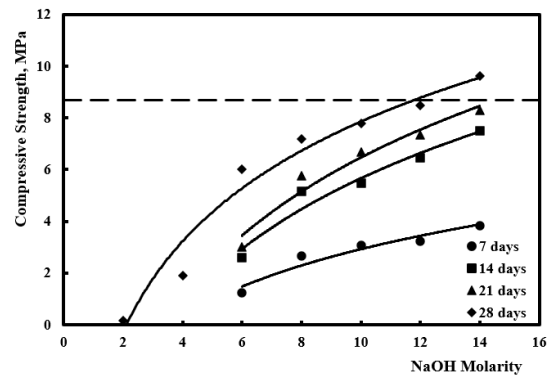


Fig. 13: Effect of NaOH molarity on compressive strength

The accuracy of that expression can be assessed by plotting the values of compressive strength as calculated from that equation against the observed values. Such plot is shown in Fig. 14 and reveals excellent concordance.

Another important fact is the expected correlation between compressive strength and degree of geopolymerization. The plot, illustrated in Fig. 15, was carried out for data obtained at all curing times. The correlation is parabolic with a correlation coefficient of 0.904.

$$\sigma = 7.931 - 0.302 (DGP) + 0.033 (DGP)^2 \quad (4)$$

The compressive strength curves increase in a logarithmic way with the increase of NaOH concentration and it was observed that the compressive strength reaches the minimum allowable standards for 28 days strength of building bricks under normal weathering conditions according to ASTM C 62 [33] which is 8.7 MPa at a concentration of about 12 M.

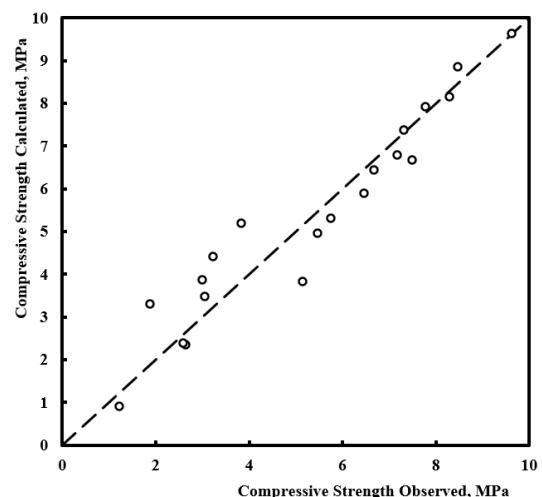


Fig. 14: Comparison between calculated and observed values of Compressive strength

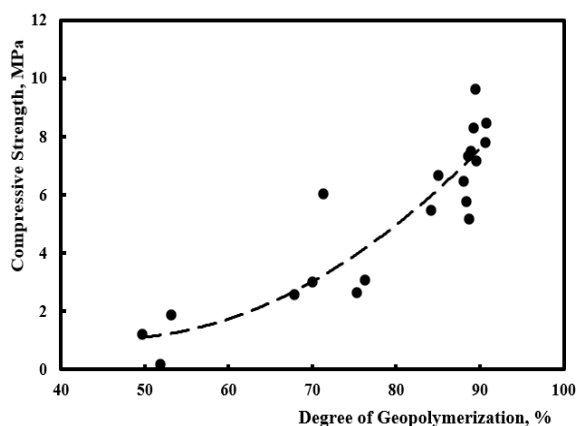


Fig. 15: Effect of degree of polymerization on compressive strength

4. Conclusion

Through this work, geopolymer bricks were prepared using a waste material as binder (Dealuminated kaolin waste from alum industry) while the activator was caustic soda solution. The aim of the work is to make use of that obnoxious waste to obtain building units. The main findings of the work were as follows:

- 1) The degree of polymerization is affected by both curing time and NaOH molarity. Increased curing time strongly affects the degree of polymerization at earlier periods but fails to seriously do so as of 14 days. On the other hand, the molarity of NaOH solution strongly affects this parameter up to 8–10 M after which its effect is less pronounced. The maximum value of the degree of geopolymerization was about 90%. It was reached after 14 days curing and using 8 M NaOH. Further increase in curing time or NaOH molarity did not seriously affect this value. The following equation was obtained that relates the degree of geopolymerization (%) to the molarity of NaOH (M) and curing time (t days):

$$DGP = 40.132 + 3.652M + 0.264t$$

- 2) The bulk density of prepared samples tended to slightly decrease with curing time and to increase by increasing NaOH molarity. Bulk density increases with the increase of NaOH concentration until it approximately stabilizes at 12 M concentration. The following equation relates the bulk density ($\text{g}\cdot\text{cm}^{-3}$) to both NaOH molarity (M) and curing time (t days):

$$\rho_B = 1.187 + 0.0937M - 0.0127t$$

- 3) It was also observed from the relation between water absorption and NaOH molarity, after a curing time of 28 days, that there is a common trend of decrease in water absorption, whether cold or boiling water absorption, with the increase of NaOH molarity. This is due to the

increase of bulk density with the increase of NaOH concentration resulting in lower porosity that causes lower water absorption.

- 4) The saturation coefficient is an indirect indicator of the size of available pores. A low saturation coefficient denotes the presence of small pores. Accordingly, the variation of saturation coefficient with sodium hydroxide concentration after 28 days curing reveals that at low concentration, the size of available pores was large since the saturation coefficient = 0.95. However, the pores volume tends to decrease on increased geopolymerization resulting in sudden drop in saturation coefficient to a very low value at 6 M concentration (0.65). As revealed by SEM micrographs, the pores volume tends to increase once more at higher NaOH concentrations, reaching 0.84 at 14 M NaOH.
- 5) The compressive strength (σ MPa) increases in a logarithmic way with NaOH molarity (M) and linearly with curing time (t days) as revealed by the following equation:

$$\sigma = -9.638 + 5.054 \ln M + 0.211t$$
- 6) It was observed that the compressive strength reaches the minimum allowable standards for 28 days strength of building bricks under normal weathering conditions according to ASTM C 62 (8.7 MPa) at a concentration of about 12 M. However, to ensure a reasonable margin of safety it is recommended to use a concentration of 14 M whereby the 28 days strength reaches 9.63 MPa.

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المخلص باللغة العربية

تحضير طوب جيوبوليميري من مخلف الشببة

يتم تحضير الأجسام الجيوبلمرية تقليدياً من خلال التفاعل بين مادة لاحمة ذات خواص بوزولانية ومحلول منشط قلوي. في هذا البحث تم استخدام مخلف صناعة الشبب والمعروف بالكاولين منزوع الألومينا كمادة رابطة ومحاليل هيدروكسيد صوديوم بتركيزات تراوحت ما بين 2 إلى 14 M كمنشط قلوي. تم تجفيف المخلف الصلب وطحنه إلى مسحوق ناعم، ثم تم توصيفه باستخدام الفحص بأشعة الحيود وأشعة التوهج السينية والميكروسكوب الإلكتروني الماسح. تم بعد ذلك خلط المسحوق مع محلول هيدروكسيد الصوديوم وصب النواتج في قوالب صلب مكعبة مقياس 50 مم. تم تتبع تأثير زمن المعالجة وتركيز المحلول القلوي على خواص الطوب المنتج، حيث إتضح أن درجة البلمرة تزداد بعد 28 يوماً بزيادة تركيز المحلول القلوي لتصل إلى 90% عند تركيز 8–10 M، أما إمتصاص الماء فحدث به انخفاض واضح بزيادة التركيز من 4 إلى 14 M، كما أوضحت النتائج أن مقاومة الإبتضاغ بعد 28 يوماً بلغت الحد الأدنى من متطلبات المواصفة القياسية ASTM C62 بدءاً من تركيز للمحلول القلوي = 12 M.