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ALKALI ACTIVATED BRICK AT LOW TEMPERATURES BASED ON IRAQI ATTAPULGITE

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ABSTRACT
In this work, alkali activated brick have been synthesized from Iraqi Attapulgite clay using NaOH solution as activators. Prepared 300 brick samples have dimensions (75 x 38x 25 mm) by applied static press of (13-14 kN). In this study, two parameters with 270 samples have all, concentration of NaOH (4, 6 and 8 M) and varies curing temperatures (80, 100 and 120°C) in order to minimize firing temperature of products brick. As well, 30 samples as reference, which do not have any NaOH and fired at 1000 °C. The prepared samples subject to (water absorption, compressive strength and efflorescence) tests according to Iraqi standards [13], and rupture strength according British standard [10], in addition to SEM and EDS test was carried out. The results showed the maximum compressive strength of 13.4 MPa was obtained with 4 M of NaOH and 100 °C of curing, and the water absorption enhancing by 56.9% at 120 °C, and the increasing in term of strength rupture was 48.15% at 4 M and 80 °C. Whereas, the samples were prepared with 6 and 8 M are not holds together when immersed in water to perform tests, furthermore all brick samples exhibits high efflorescence with thick layer of NaOH salts.

INTRODUCTION
The production of conventional clay bricks involving on firing process, which caused atmospheric pollution [1]. So and for environmental issues the needs a raises to provide more environment friendly products and more economy by reducing fuel consumption due to reduce fire temperature.

Recent years have seen a large development in a family of building materials. In the latest thirty years, new silica-aluminate inorganic polymers, named geopolymers by Davidovits in 1979 [2], have emerged as components of a new class from low-energy materials, which characterized by highly desirable mechanical and chemical properties. Such materials are now of great interest for a wide range of applications in several different sectors of engineering [3]. In the civil engineering fields, geopolymer-based materials are also referred to "alkali-activated cements" or "chemically-bonded ceramics" obtainable starting from raw materials with low (or zero) CaO content. Many researchers studies utilization of some materials as flay ash, ground granulated blast furnace (GGBF), rick husks ash and waste limestone [1]. In this work will study Iraqi Attapulgite clay.

Attapulgite is a fibrous clay mineral as type of crystalloid hydrous Magnesium - Aluminum silicate mineral and have absorbed water in its chemically structure. Attapulgite form at the earth surface with low temperature of clay environments, hence they are classified as clays [4], the Attapulgite needles are formed by the structural linkage of tetrahedral - octahedral strips. The chemical form of Attapulgite introduced by Carrol 1970 as [5]:

$$Si_8\,Mg_5\,O_{20}\,(OH)_2\,(OH_2)_4 .\ 4H_2O$$

In Karbala and Al-Najaf regions, Attapulgite dominates clay mineral Mudstones of the Injana formation, which is (late Miocene – Pliocene) are exposed in Al-Najaf region (Tar Al-Najaf) as bluish green and gray clay stone, 0.5 m thick with plants remains [6].

MATERIAL AND METHODOLOGY

Materials

The Attapulgite clay employed in this work was bring from (Tar Al-Najaf / Injana formation) located in Al Najaf city (Iraq country). Then it crumble and dried in sunlight or in drying oven until completely dry state, and before is clattering used, it needs to be ground to less than 250 µm [7]. That is by use balls mill (for about 2 hr.) and 12 balls used (with 450 gm weigh of each one). After grinding process it finish, passing the ground clay on sieve 300 µm before it use. After then the chemical analysis carried out on grinds Attapulgite (Table 1).

Table (1): Chemical analysis for Attapulgite clay.

Oxide Composition	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	SO_3	Na_2O	K_2O	L.O.I
Oxide Content, %	51.45	8.91	4.81	0.56	7.01	5.52	0.68	1.01	1.8	17.2

The Attapulgite clay particle size distribution was determined according to ASTM D442 [8], (fig. 1). The sodium hydroxide supplied from local market in solid flakes form with (98 %) of purity and clean tap water is used to preparation alkali activator solutions with different concentration (4, 6 and 8 M).

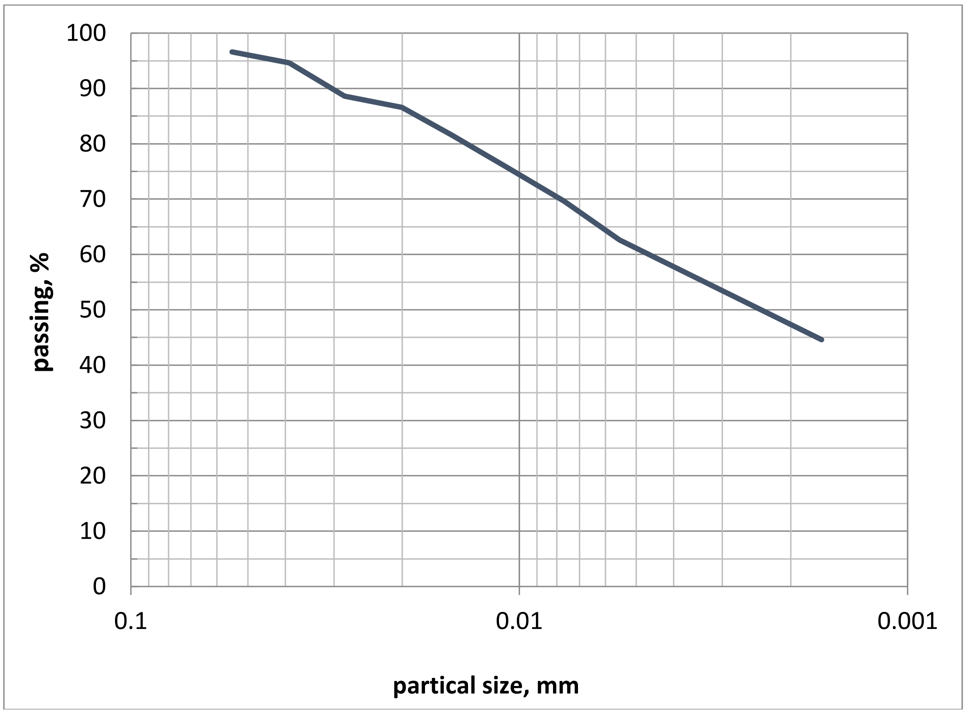


Figure (1): Particle size distribution of attapulgite

Samples preparation

The mixtures are mixed by mortar mixer for about 10 min to gets its homogeneity, and the ratio of NaOH to solid clay is used 0.25. After complete mixing process, the mixtures put into a brick mould, that have interior dimensions (75 x 38 x 25 mm) with using hand compaction by steel rod until the mold was full. Then an iron block placed on top of the mix and electric loading machine applied with (13-14 kN) of press from its top. After compression process is done, the brick samples were de-molded and allowed to sit overnight at room temperature with sealed vessels fitted with teflon before being cured at different temperatures (80, 100 and 120°C). This "soaking" is used to allow time for dissolution and for geopolymer precursors to forms [7, 9, 10]. Then placed uncovered in oven for 24 hour of curing. Nine mixes were prepared with 30 brick samples for each one, content all parameters (Table 2), and one reference mix that fired at normal firing clay brick, which is 1000 °C.

Table (2): Mixes with parameters.

Mix. No.	(NaOH) Concentration, M	Curing Temperature, °C
1	4	80
2	6	80
3	8	80
4	4	100
5	6	100
6	8	100
7	4	120
8	6	120
9	8	120

RESULT AND DISCUSSION

Reference samples

The results test of reference brick samples are listed in table (3).

Table (3): Results tests of reference samples.

Property	Compressive strength, MPa	Water absorption, %	Efflorescence	Modulus of rupture, MPa
Magnitude	14.9	25.9	Non	4.88

Rupture strength

From results (fig.2), found the use 6 and 4 M of NaOH will increased rupture strength of bricks by 157.4% and 48.15% at 80 °C of curing temperature respectively, and also at 100 °C the increment in rupture strength was 107.5%, 29.71% and 25.8% for 6,4 and 8 M of NaOH respectively. Whereas the strength is decreased at 120 °C of curing for all concentrated of NaOH. This gain in rupture strength is reducing the losses of bricks due to transformation during construction works, and will diminish cracks that result from simply settlement of soil.

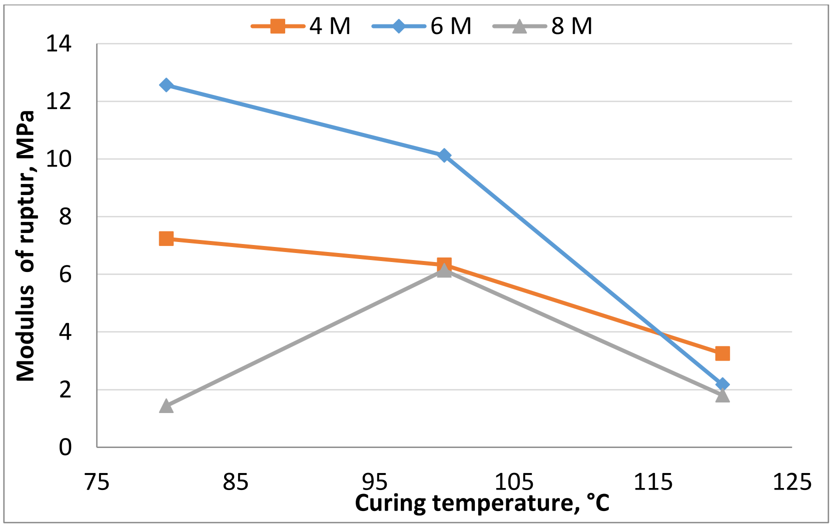


Figure (2): The relationship between modulus of rupture and curing temperature with different concentration of NaOH solution.

Water absorption

When immersion samples in water for 24 hr to carried out water absorption, we notes all the samples were prepared with 6 and 8 M are exposure to corrosion and have diagonally cracks, except that prepared with 4 M are withstand to water immersion and its tests result are representative in (fig.3).

we notes enhancing in water absorption of brick in comparison with reference samples at all curing temperatures 80, 100 and 120 °C by 56.9%, 47.15% and 46.32% respectively. Furthermore, the ability of brick samples to water absorption is increased with rising curing temperature form 80 to 100 °C and 100 to 120 °C, that is due to exit amount of unreacted (NaOH) way out by evaporation when subject to curing temperature, especially when increased from 80 to 100 °C, were increment in water absorption was 6.7 %. Then become less at 100 to 120, which was less than 1%.

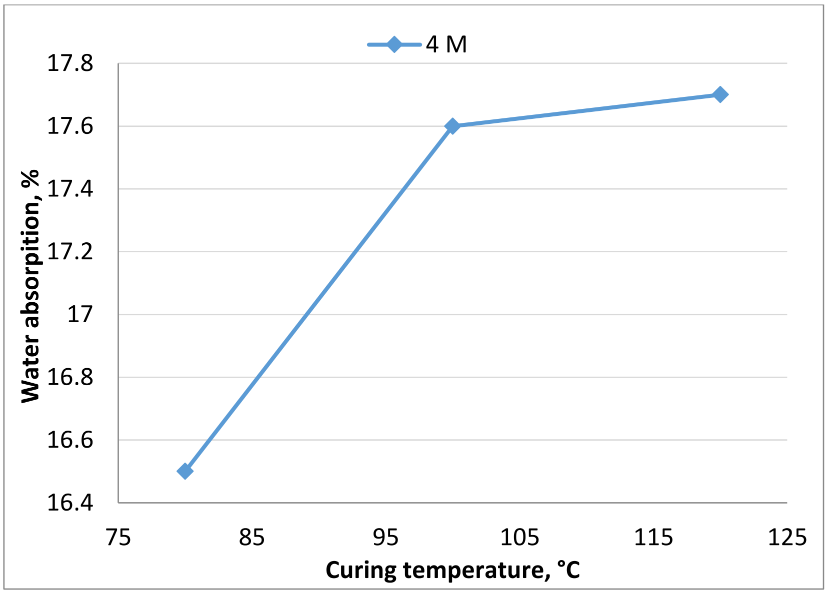


Figure (3): Relationship between water absorption and curing temperature.

Compressive strength

It's obvious from (fig.4) shown, the compressive strength was lowest that reference mix at all temperature of curing, were was 12.3 MPa at 80 °C then increased with raise curing temperature to reach 13.4 MPa at 100 °C after than decreased to 13.2 MPa at 120 °C. From that, distinguish the largest value for a product brick was at 100 °C is 12.4 MPa, which is equivalent to 90% of strength reference sample, which was 14.9 MPa.

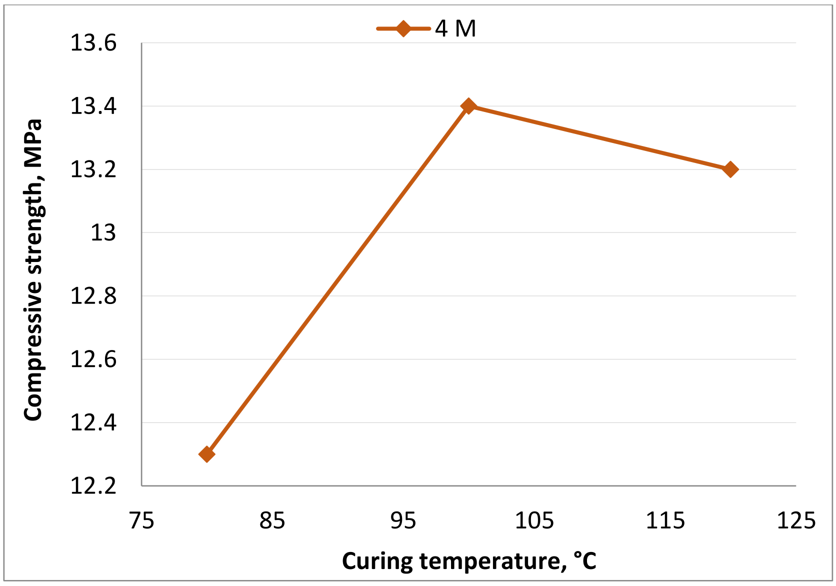


Figure (4): Relationship between compressive strength and curing temperature.

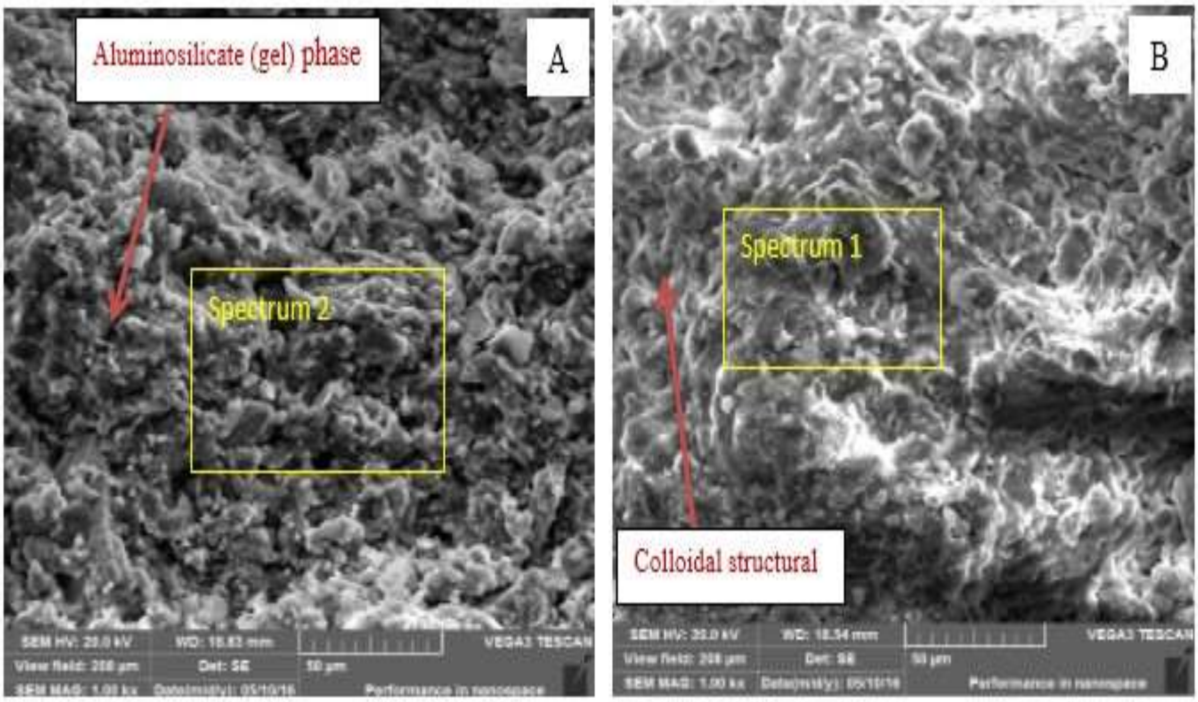
Efflorescence

When carriedout efflorescence test, by submerge lower part of brick sample in water for 7 day and left for 3 days to dry, was observed athick layer of NaOH in the salts form. In addition, erosion of upper parts of brick, and these erosion and salts increased with increase concentration of NaOH that used to prepare the sample.

Microstructure properties

Scanning electron micrograph (SEM)

TESCAN-VEGA3 LM apparatus used in this test [11], (fig.5-A) shows the electron images of the reaction products obtained from the original (Attapulgite clay) and water, were have colloidal structural. Compared with mix No.4 (fig.5-B), almost the particles of this mix are irregular, with very few spherical glass particles (presumably because of the lower firing temperature (100 °C)). The main product of geopolymerization is an amorphous aluminosilicate (gel) phase, which is composed of ($Na_2Al_4Si_9O_{25}$). In addition, the spherical particles increased in form and tended to conglomerate with found NaOH. Comparing between these two micrographs, it's clear that the products cured at 100 °C, which gave high values of compressive strength (13.4 MPa) shows much denser microstructures and more solidity than the reference samples, which did not have any amount of NaOH solution.



Figuer (5): SEM Image for (A) Sample with 4 M NaOH and100 °C of curing. (B)Sample without NaOH.

3.6.2 Energy Dispersive Spectroscopy Systems (EDS)

In this test, used (AZtec) apparatus[12], the elemental distribution of the crystalline acicular structures of sodium Hydroxide phases observed under EDX is given in (fig.6), which shows that the crystalline phases of acicular structure of Na-cross linked geopolymer bears and the elements like (Na, Al, and Si) correspond to the geopolymer phase. The micrographs study it is observed that the reaction of Attapulgite in alkaline activator consisting of anions of Cl− and $SO_4^{=}$ form crystalline grains. These grains minor to trace amounts of (Fe, Ti, Mg, Ca, and K) are mostly fibrous and elongated. These mineral phases with different cation and anion, substitution growing into amorphous to crystalline structures by dissolution and solidification. Assortment of the polymerized minerals helps in the build-up of the cementation property. Depending upon the bonding strength of the geopolymerization process is flexible to make high strength building bricks ranging compressive strength from 12.3 to 13.4 MPa.

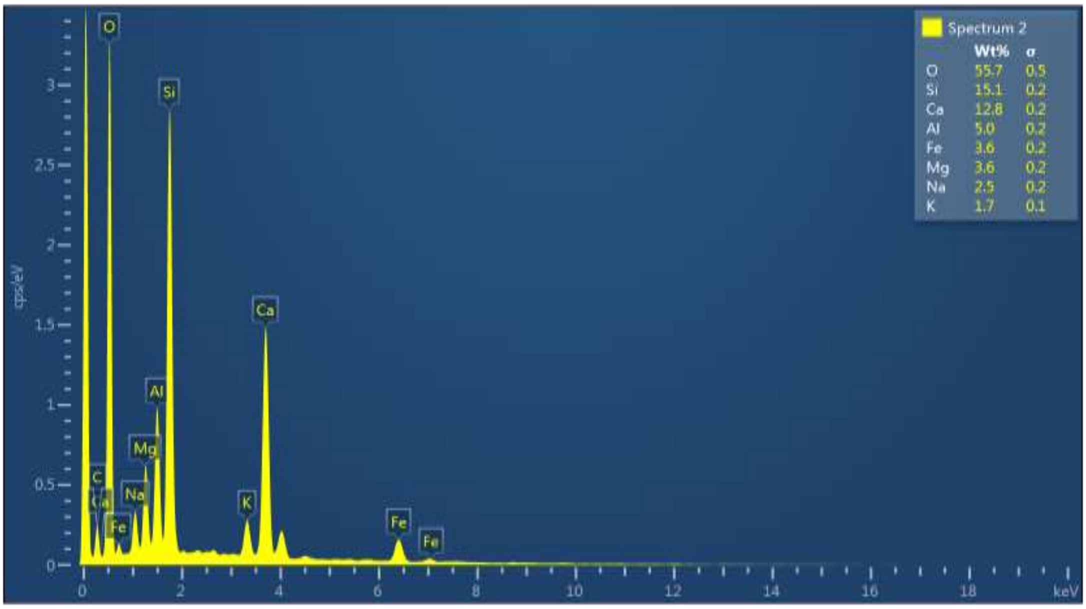


Figure (6): EDS image for mix no.4.

CONCLUSION

The use of NaOH solution in the brick has a significant impact on the final properties of Attapulgite clay brick. This strategy contributes to save energy, and at the same time to a reduction in the production costs and reduce pollution of atmospheric due to high firing temperature, which used in normal clay brick industry. Depending on Iraqi [13] and British [14] standard and, measurements on mechanical and microstructure properties of bricks have been carried out with good accuracy. Concluding that:

1) Possibility to product clay brick based Attapulgite by its activation with 4 M of NaOH solution at low curing temperatures (80 and 100 °C).
2) The largest value of compressive strength (13.4 MPa) was gets with optimum mix, that have parameters (4 M and 100 °C), were was equivalent to 90% of compressive strength for reference sample, which fired at 1000 °C. Besides the enhancing in water absorption property by 47.15 % and the increasing in rupture strength by 47.16.
3) Microstructure tests are showed enhancing in the internal structure of the brick when incorporated NaOH with Attapulgite clay, by make the brick more density, that's due to chemical interaction between them.
4) This type of brick are not resisting to moisture and water exposure, so its use needs preventing and protection from moisture and for indoor uses.

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