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Ministry of Higher Education and Scientific Research
University of Technology
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PRODUCTION AND PROPERTIES OF GEOPOLYMER ATTAPULGITE BRICKS

A Thesis Submitted to the Building and Construction Engineering Department of the University of Technology in Partial Fulfillment of the Requirements for the Degree of Master of Science in Building Materials Engineering

Submitted by

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بسم الله الرحمن الرحيم

﴿ قَالُواْ سُبْحَنَكَ لاَ عِلْمَ لَنَا إِلاَّ مَا عَلَّمْتَنَا إِنَّكَ أَنتَ الْعَلِيمُ الْحَكِيمُ ﴿32﴾ ﴾

صدق الله العظيم

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Abstract

This study concludes investigation of the possibility of clay bricks product by geopolymerization process. Prepared mixes of locally Attapulgite clay were grinding to less than 250 µm with different concentration of NaOH solution (i.e., 4, 6 and 8 Mole), and formed using iron mold by applied (13-14) kN of load.

Brick samples were prepared in two groups; group 1 which contained 270 samples that subjected to curing process at (80, 100, and 120 °C) for 24 hr, while group 2, 270 samples, also was subjected to burning process at (400, 500, and 600 °C) for 3hr. In addition, two reference mixes with 60 samples (0% of NaOH), were prepared, namely (Ref.1) which were burned at optimum temperature of (500 °C), whereas (Ref.2) were burned at normal burning temperature for production of clay bricks, i.e.1000 °C.

Shape and dimension, compressive strength, water absorption, and efflorescence tests were carried out according to Iraqi standard No. 24 for 1988. Simultaneously, longitudinal shrinkage, bulk density, and modulus of rupture tests were performed according to British standard No. 84 for 19734. In addition, microstructure tests (SEM and EDS) were performed for brick samples of optimum mixes, and Ref.1 set.

It was concluded that the optimum percentage of NaOH and the preferred burning temperature were 4 M and 500 °C, respectively. Where, the bricks obtained with these parameters was have (13.9 MPa) compressive strength, which exceeded twice times the compressive strength value of reference mix (Ref.1); i.e. (3.9 MPa).These parameters gave bricks with engineering properties within (class B) requirements of Iraq’s standard for clay building bricks.

The improvements were up to 256 %, 12.66 % and 80.95 %, for compressive strength, water absorption, and modulus of rupture, respectively. The reducing in burning temperatures reach to about 50 %, whereas the compressive strength of brick that product at 500 °C was equivalent to 90 % of that reference mix (Ref.2) strength’s, which was burned at 1000 °C.

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CHAPTER ONE

Introduction

INTRODUCTION

1.1 General

Clay is one of the oldest building materials on earth among other ancient, naturally materials occurring due to geologic effects, such as stone and organic materials like wood. Over half of the world's population, especially in less developing countries, are still live or work in buildings constructed with clay as an essential part of their load-bearing structure. Many construction techniques are exist for building construction, some of these are traditional as rammed earth, cob and adobe [1, 2]. Others, are more modern, such as extruded and pressed bricks[3].Production of conventional brick involves burning process that generate atmospheric pollution[4], so for the environmental issues the manufactures in high demand to provide materials and building products that are more environmental friendly[5] and economically by reducing firing temperature of brick. At the same time, this leads to reduce fuel amount.

1.2 History of clay bricks

The regions that locate in modern-day (Iraq country) was originally inhabited by the people who fabricated the arch, column, wheel, algebra, geometry, astronomy, and potter's wheel. Nevertheless, since 11,500 years ago, during the *Pre-Pottery Neolithic* period, the people there were lived in very simply standard, their buildings without structures, made of mud brick. They lived in upper Mesopotamia and the Levantine, in the Fertile Crescent area. Later and at south, Sumer land and its people (The Sumerians), they were the first who developed the written means of communication; they wrote on clay tablets by hand which is represent the evolution from pictograms to cuneiform**.** Literature was important to them, and their work recognize more

easily in the today on the *Epic of Gilgamesh*. Sumerians were pantheistic; their temple (the ziggurat) was a climbing of mud bricks (Plate 1-1). Babylon and Assyria were also other famous Mesopotamian places [6].

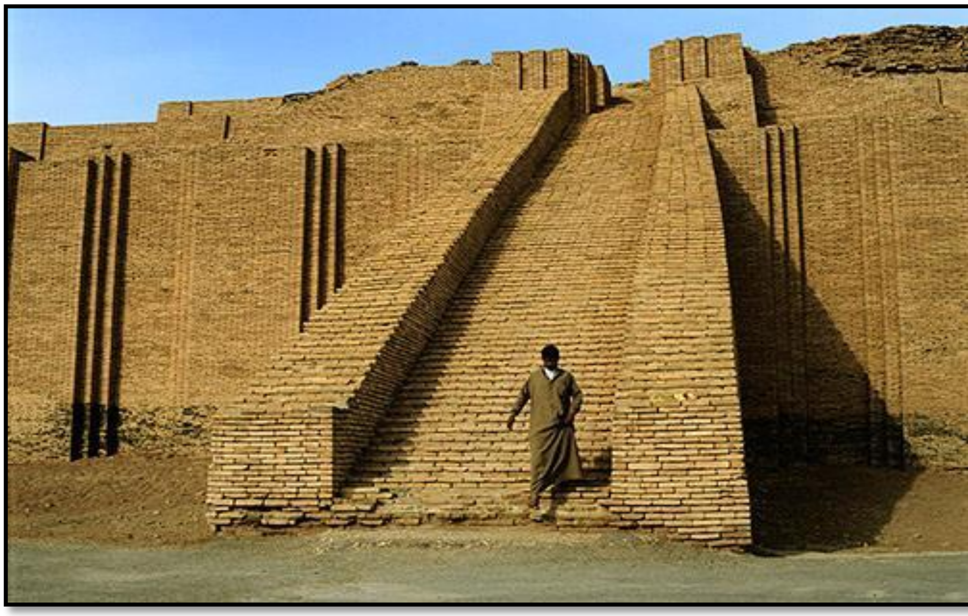


Plate (1-1): The Ziggurat "which made of mud bricks"[6].

Hammurabi and his rule, series of laws, are between Babylon's patrimonial. It can note that some of the foundations of Babylonian templates were mud brick only; in other places were mud brick with burn clay faces and somewhat with stone pieces in faces. Regardless of the Babylonians were the first people who burned the clay, there were found other index to use of clay for daily works; as Axe heads of clay, weaponry in the form of sling bolts and bullets, also nail-shaped objects made of clay, thought to be used as pestles or as a tool for tanning. In addition, according to Peter Roger Stuart Moorey's (Ancient in Materials and industries of Mesopotamian); The Archaeological Evidence. "It makes perfect sense that the clay would be utilizing for such objects in areas that rich in clay, chiefly with obscurity of

others building materials". The table below (Table 1-1) shows the chemical composition for clay and sherds of Babylon and Kish civilizations [(6)].

Table 1-1: Chemical composition for Babylon and Kish civilizations clays[(6)].

	Clay[a]	Sherds[b]	Brick[c]	Bodies[d]
SiO_2			47–52	50[e]
Al_2O_3			14.3–17.7	15–16
Fe_2O_3	5.3–6.2	6.0–7.4	5.7–7.7	7–9
CaO	13.0–17.2	15.6 (Kish sherds)	16.3–17.5 (21.8)	16–18
MgO			5.9–7.1	5–6
Na_2O	1.2–2.1	1.8–2.3	1.7–2.4	1.5–2
K_2O	1.4–2.6	1.5–1.9 (1.1)	1.7–2.2	1

[a] Neutron activation analysis. Brookhaven National Laboratory. Babylon Clays VZM 189–192. Kish Clay VZM 183.
[b] Neutron activation analysis. Brookhaven National Laboratory. Babylon Sherds DBBB 1–6 (CaO not determined): see Ref. 9. p. 74. Kish sherds VZM 184, 185.
[c] Electron beam microprobe analysis, Smithsonian Institution. Five glazed bricks from the Ishtar Gate at Babylon.
[d] Isoprobe and optical emission spectrometry, Research Laboratory for Archaeometry and the History of Art, Oxford, England. (Ref. 8. p. 31. Table 1.)
[e] Estimated by difference.
Source: Matson 1985: 65.

Brick manufactures were the main industries in the Mesopotamian, especially in the south, were the wood supply was small and with absence of stones. Clay was the most available materials in Sumer's and it was mixed with sand, water, mud, and organic material husks or straw. At most, it was sun-dried as there were few fuels to burning. Pottery and brick productions were going concerns in these days and occupies large part of the economy [(6)].

The Sumerians utilizing clay even in sickles manufactures. Throughout former Mesopotamia, one can find old town sections made of mud brick structures (predominating enclosed by a mud brick walls). As well, brick manufacture continues to be ongoing interest. The example of mud brick walls in Al Hillah-Iraq (Plate 1-2) the residue of Babylon civilization [(6)].

Plate (1-2): Mud brick wall in Al Hillah, Iraq [6].

Davidovits [7] had discover and developed the geopolymer at end 1970s; which is one type of three-dimensional CaO-free aluminosilicate binder. Geopolymer is a ceramic material that produced by alkali activation of raw materials of aluminosilicate, which are in their turn, transformed by reaction product in geopolymerization process in a high environment of (pH) and comparatively low curing temperature. He used kaolinite in his studies on geopolymers. Then, others aluminosilicate source like calcined clays, fly ash, and slag were investigated. Using these materials in production of geopolymer brick can support construction sustainability and also contributes to the development of the construction industry in terms of producing more efficient materials that are more environmental friendly.

In this research, will be investigate the effectiveness using locally materials (Attapulgite clay) in the production of clay brick as a geopolymeric materials by geopolymerization process.

Attapulgite is a fibrous clay mineral; as a kind of crystalloid hydrous Magnesium - Aluminum silicate mineral, which has chemical absorbed water in its structure. Attapulgite form at the surface of the earth with low temperature of clay environments , hence it is classified as clay. The structural linkage of (tetrahedral – octahedral) strips forms the needles of Attapulgite. The chemical form of Attapulgite introduced by Carrol in1970 (8) as:

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

In general, Attapulgite resides in the western desert and in Al-Najaf region (Plate 1-3). The form of Attapulgite varying in thickness and extent in the Akashat region (Safra and Traifawi) since (55 - 60) million years(9).

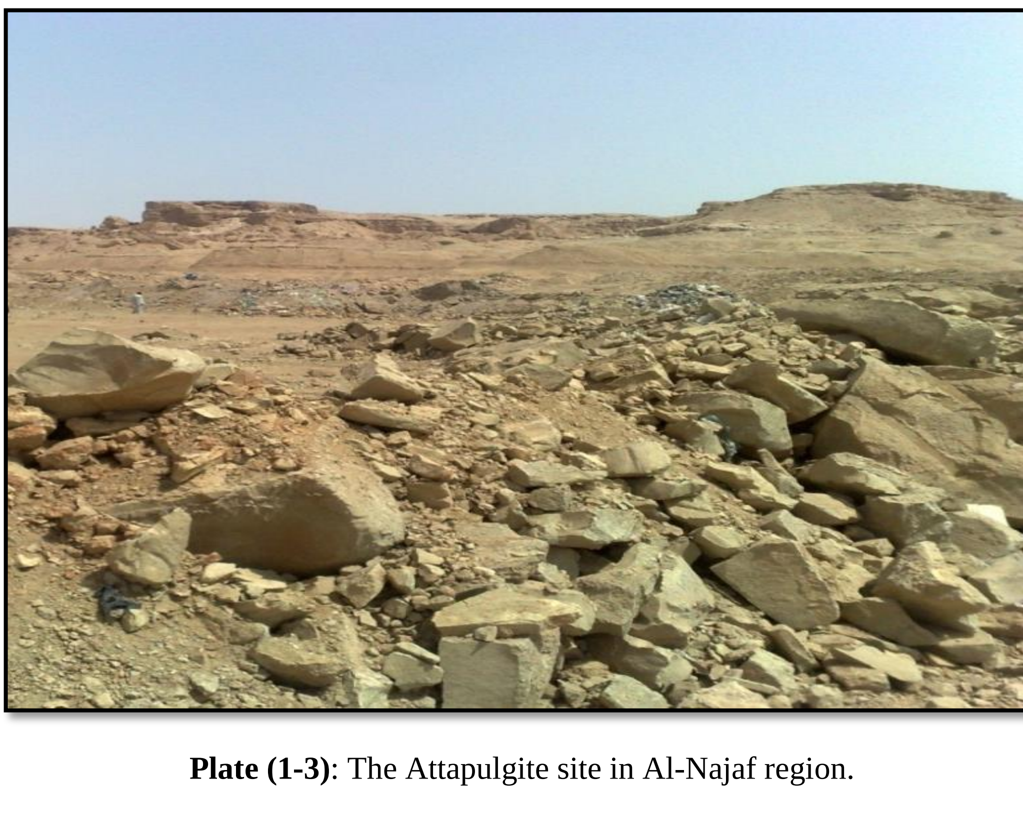


Plate (1-3): The Attapulgite site in Al-Najaf region.

1.3 Research Significance

The main significance of this research is to reduce the manufacture bricks cost, by recline burning temperature of clay brick to minimum. Subsequently, decreasing fuel amount, which demand for burning process. In addition, environmental issues is considered, through reduce atmosphere pollution, in the same way, by reducing burning temperature of clay brick.

1.4 Objective

The main goal of this research is to produce bricks (based on locally Attapulgite clay) which have acceptable properties accordance to the Iraqi standards with less degree of burning temperature compared with normal burning temperature for production clay bricks.

1.5 Thesis Layouts

The work presented in this research is given in five chapters:

Chapter One presented the introduction.

Chapter Two reviews the previous researches on the bricks type, main materials, and technique that introduce in its manufacture.

Chapter Three includes the raw materials, its preparation process for brick manufacture, trial mixes, mix proportioning, methods of testing and other experimental details.

Chapter Four explain the results of the experimental work and their discussion and interpretation.

Chapter Five exhibit the conclusions of the research and recommendations for further researches.

CHAPTER TWO

Literature review

LITERATURE REVIEW

2.1 General

The traditional method for bricks manufacturing is to produce a clay–sand mixture, then burn the mix in a comparatively high-temperature furnace [10]. Environmental issue is not taken in this manner of production's method. Obviously, the world is becoming more environmentally conscious, currently. Whereas, varieties of other waste materials have been used elsewhere for brick's manufacture. Furthermore, clay brick manufacture needs high degree of burning temperature to gain acceptable mechanical properties, which consequently increases required fuel to complete burning process, as subsequent, make it sometime, not economical.

2.2 Brick and sustainability

Sustainable buildings are planned in a manner that utilizes available resources efficiently, in a responsible way, balancing environmental, societal and economic impacts, to meet the design purposes of today while considering future effects. They should be resilient to withstand the effects of nature with minimum harm. Sustainable buildings are designed to be energy efficient, water efficient and resource efficient. Through their design, they address the well-begin of the occupants by providing thermal and acoustic comfort, indoor air quality and appealing aesthetics. They also see the impact of their construction, functioning and maintenance of the environment, and the environmental impact of their constituent materials. Most significantly, a sustainably designed building comes up to all these prospects through its full life cycle, including its operation and maintenance [6].

Mbereyaho et al. [11] searched for an alternative resolution to the high consumption of valley clay as main material during brick production. By providing and supplying burned earthen bricks, which will, firstly, serve as "Environmental friendly " in fact that it will enhance local technical skills and depend on available local raw materials. Secondly, will be" Economical based "in fact that it will be marketable within local context at reasonable cost, and finally the product will offer sufficient compressive strength to be confirmed as an engineering material reflecting sustainability in modern buildings. The primary aim of their work was to find out an alternative material to the ordinary burnt valley clay brick by replacing them as raw material, basing on comparative analysis of compressive strength characteristics between finishing mud brick and offers significant required strength. They based on soil sampling from different regions, these samples were examined and results analysis for identification of the type of soils presented. Then the soils that used in bricks preparation for which strength characteristics were finally identified. They concluded from the results that the mud brick could be used for some simple buildings in which their strength characteristics could depend on the character of the soils used.

2.3 Materials used in manufactures and methodology

2.3.1 Fly ash

Fly ash is fine glass powder obtained from the gases of fired coal during the electric production, it is micron-sized earth elements made primarily from silica, alumina, and iron. During the firing of coal, the products formed are classified into two types; fly ash and bottom ash.

The residue that is fused into particles is termed as (bottom ash), and the part of the ash, which is entrained in the combustion gas away the boiler is (Fly ash). Fly ash is collected either through electrostatic or mechanical collectors [12].

2.3.1.1 Physical Properties of Fly ash

Shape; Fly ash has spherical shape particles with (150 µm - 1mm) in size [12].

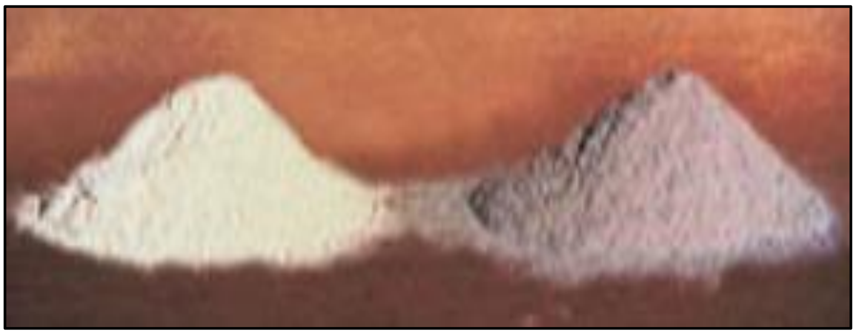


Plate (2-1): Fly ash color and finesse [12].

Color; Fly ash derives its color mainly from carbon and iron elements. High carbon content brings the gray or black colors, while high iron content gives tin colored ash (Plate 2-1).

Fineness; It is expressed in fly ash in terms of specific surface area, which changes from $2*10^7$ to $5*10^7$ m^2/kg. Higher fineness of fly ash required high dosage of air entraining admixture. The large amount of surface energy is utilized to combine with lime.

Chen et al. **[13]**, studied geopolymer brick manufacture by using bottom ash from circulating fluidized bed combustion (CFBC). The original diameters of the bottom ash were from about 2 mm to 2 cm, with 95% retention on a 45µm sieve. This ash was then ball-milled for 75 min, which reduced the percentage of retention on the 45µm sieve to 7%. The main

components of the CFBC bottom ash were (SiO_2 and Al_2O_3, > 85 % of the total).

The alkaline solutions used were sodium silicate solution (SiO_2/Na_2O modulus (molar ratio) = 1.2, 1.5, and 2.0), sodium hydroxide solution (10, and 5 M), potassium hydroxide solution (10, and 5 M), and lithium hydroxide solution (5 M). The mass ratio between liquid and solid was (0.3) for all mixtures. First, the bottom ash and alkaline solution mixed by a cement mixer for at least 4 min (2 min at 62 rpm and 2 min at 125 rpm). Afterwards, the mixtures were put into brick molds (4×4×8 cm). Then, an iron block (4×4×8cm) placed on top of the mix and a 300kN pressing machine used to press the mix from its top via the iron block. The pressure that used was 60 kN for 10 seconds. After pressing, the mold blocked up for about 4 cm from the bottom, and the pressing machine using again to press the brick samples out of the mold. Finally, the brick samples stored at 40 $^{\circ}$C and 100% humidity.

The compressive strength was conducted after 3, 7, 14, and 28 days. The compressive strength of the bricks under certain conditions reached up to 28 MPa. Lithium hydroxide (LiOH) solution was to be a very effective alkaline activator as was the more than typical NaOH. The geopolymerization reactions occurred during the setting of the bricks, and an amorphous aluminosilicate ‘gel’ phase was the main reaction. Alongside the ‘gel’, a small amount of crystalline minerals could be found by SEM when the material was prepared using pure alkaline solution.

S.D. Muduli et al.[(14)], undertook a research on the effect of NaOH concentration in fly ash building brick production. Geopolymerization technology was used with wastes materials such as fly ash in manufactures of high strength bricks.

In this work, sodium was used as alkaline activator consisting of anions of Cl- and $SO_4^{=}$ of different concentrations with presence of water. The mix consisted 87% fly ash and alkaline activator which used by maintaining $Na_2O/(Al_2O_3+SiO_2)$ ratio from 0.025 to 0.078 to product building materials that have compressive strength of 6 to 48 MPa.

The process of manufacture followed the conventional method. The raw materials with a suitable mix design were mixed in a pan mixer in the presence of the alkaline activator and water. The mixture cast by a compaction pressure of 20 ton to form standard size for brick (230 × 110 × 75 mm). The cast products were cured at 20°C, and under normal atmospheric condition (35°C) for comparison and for calibration purpose. It was found that the response process of geopolymerization depends on the type of raw materials and the concentration of alkaline activator. Also, they found that the compressive strength increases with the increment of $Na_2O/(Al_2O_3+SiO_2)$ ratio, which was dependent on the concentration of sodium hydroxide.

Rishi et al. [(15)], studied geopolymers that have synthesized coal fly ash (CFA) with KOH and Na_2SiO_3 as activators. Some parameters such as alkali concentration, amount of Na_2SiO_3, and curing time have been varied in order to improve the quality of geopolymer product. The geopolymerization process was carried out using (3-8) M KOH solutions, Na_2SiO_3/CFA mass ratio of 0.25-2.00, and curing time variation from 6 to 28 days. The curing temperature fixed at 40°C in all the cases.

The testing results demonstrated that the compressive strength increased at the beginning and then decreased with raise potassium hydroxide concentration. The maximum compressive strength was 6.62 MPa, which obtained with CFA and 7 M KOH solution. If Na_2SiO_3 amount was changed,

compressive strength increased and then decreased with raise concentration of Na_2SiO_3. The maximum compressive strength was 28.1 MPa, which obtained with 1.75 mass ratio of (Na_2SiO_3/CFA). The compressive strength was found to increase with increasing curing time, suggesting that curing for a longer period of time at low temperature is preferable for the synthesis geopolymer of higher compressive strength. The compressive strength was found to increase upwards to 41.9 MPa with increase curing time to 28 days.

Ferone et al.[16], carried out their investigation on weathered coal fly ash which was used in polycondensation processes for production of ceramic bricks by geopolymer–based at low temperature. The wet fly ash brought from power plant located in the Southern Italy (Brindisi city). (Table 2-1) shows all parameters of study those categories in 6 systems.

Table (2-1): Parameters of system tested [16].

Sys tem	Dry ash, g	Wet ash, g	Sodium silicate solution, g	NaOH solution, g	Solid NaOH, g	H_2O, g	SiO_2/Na_2O molar ratio	H_2O/(total solids) w/w ratio
1.1	1000	-	200	250 (14 M)	-	-	0.61	0.28
1.2	-	1205	200	250 (14 M)	-	-	0.61	0.45
1.3	-	1205	168	-	82	-	0.61	0.28
2.1	1000	-	244	306 (10 M)	-	-	0.76	0.31
2.2	-	1205	244	306 (10 M)	-	-	0.76	0.49
2.3	-	1205	244	-	92	9	0.76	0.31

A Hobart mixer was used to homogenize the mixtures. Afterwards, the mixes were cast in series of cylindrical molds of size (3 heights x 6 diameter) cm. The molded samples were cured for 8 days, according to four different curing systems as follows:

(a) 8 days' of curing at room temperature. (b) 1 day at room temperature, 1 day at 60 °C and 6 days at room temperature. (c) 1 day at room temperature, 2 days at 60 °C and 5 days at room temperature. (d) 1 day at room temperature, 7 days at 60 °C.

It was concluded that, when weathered fly ash was used after drying, the products performances were passable for the manufacture of ceramic brick based geopolymer at low temperature. However, weathered fly ash was applied wet (as received) without altering the additions of activating solutions; the polycondensation process suffered from high water content and weakness mechanical performances for produced brick. From the other hand, when fly ash was used in wet state and mix design takes into account its water content, an improvement in performances was obtained, whereas the results was like to those offered by fly ash at dry state. Lastly, it was observed that the curing conditions effect on the results very strongly. Where, curing at 60 °C, even if for 24 h only out of 7 days, has a very strongly influence on the mechanical strength properties that increased as much as 3.5 times.

2.3.2 Mine tailings

Mine tailings, also called mine dumps, slimes, leach residues or slickens are the wasted materials which appear throughout separating the valuable minerals of economic interest from the gangue or wastes of an ore. Mine tailings are produced during ore processing stage which include commination, concentration, upgrading, and leaching [(17)], Fig. (2-1) shows its production stage.

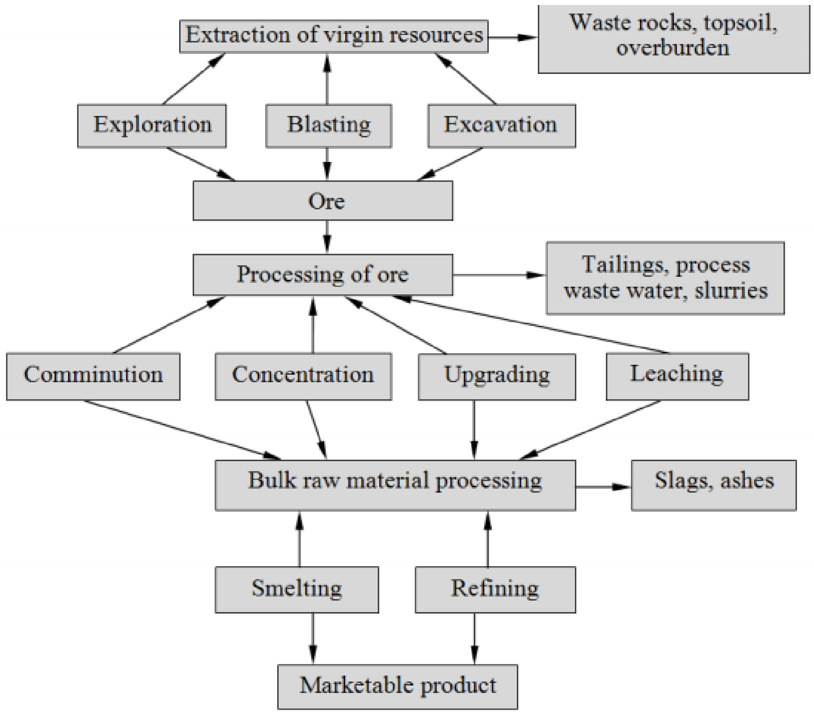


Figure (2-1): Mining processes and waste generation.

Das et al. [18], described a sustainable mode of handling iron ore tailings by applied them into a new product as wall tiles and ceramic floor for construction applications. The iron ore tailings was contained a high percentage of Si, thus they concluded that iron ore tailings up to 40% by weight could be considered for utilizing as a raw materials for ceramic product's in wall tiles and floor due to these percentages of silica content. The ceramic products from these materials were found to be superior in terms of scratch hardness and strength.

The new products of the iron ore tailings, also, have other main properties of the conventional raw materials. The application of iron ore tailings in the ceramic tiles production was economical in comparison with the traditional clay material for ceramic tiles production. Ahmari & Zhang confirmed the possibilities of making economy-friendly bricks that

conformed to the ASTM requirements using geopolymerization technology by selecting suitable preparation conditions [19].

Ahmari and Zhang [19], studied durability and leaching of mine tailings (MT)-based geopolymer bricks using unconfined compression strength (UCS), water absorption, weight loss, and concentration of heavy metals after immersion in solutions (pH = 4 and 7) for variants time periods.

The MT mixed with NaOH solution, firstly. The NaOH solution was prepared by adding NaOH flakes to water and stirring for about 5-minutes. Because of heat generation, the solution was allowed to cool at room temperatures before it using. The sodium hydroxide solution was added to the MT and mixed for 10 min to ensure homogeneity of mixture. The consistency of mixture changed from semi-dry to semi-paste as water content raised (from 8 to 18%). Then, the mixture placed in Harvard miniature compaction cylindrical molds with (33.4 mm diameter and 72.5 mm height) with little compaction. The compacted specimens were compressed by use (loading machine) with different loading rates to ensure that the duration to reach the specified forming pressure was about 10 min for all the specimens. After compression, the specimens de-molded and left uncovered in an oven for curing until tests. Depend on the results they concluded the following:

1. UCS of the products show a decrease substantially after immersion in solutions of pH = 4 and 7 for 4 months, in contrast the water absorption was still relatively small, and weight loss was smaller than for Portland cement. The decreases in UCS were mainly because the high initial (Si/Al) ratio and incompletion of the geopolymerization process of MT.

2. The heavy metals were effectively immobilized in the MT- bricks. This effect might be due to the involving of heavy metals in the geopolymer network or physical packaging.

3. The reaction/diffusion model can be used satisfactorily to describe the leaching of heavy metals in (MT) - bricks. Its means that the solubility or reaction rate was an important factor that controlling the leaching conducts.

2.3.3 Ground Granulated Blast Furnace Slag

Ground Granulated Blast Furnace Slag (GGBFS) is a by-product from the iron industries. BFS is identified as the (non-metallic product), it is essentially compounded from calcium silicates and other bases that are developed in a molten condition simultaneously with iron in a blast furnace (7). In production of iron, the blast furnaces filled with iron ore, fluxing agents, and coke. When the iron ore, that is consist of iron oxides, silica, and alumina, companied with the fluxing agents, the slag is molten and results the iron. The molten slag then goes through a particular process which depending on what type of slag it will become. Air-cooled slag has a rough finish and larger surface area, when compared to natural aggregates has the same volume, however, promising binding with Portland cement as well as asphalt mixtures can be expected. GGBFS is produces when molten slag is quenched rapidly using water jets, which produces a granular glassy aggregate.

Mainly slag made of 95% of silica, alumina, calcium oxide, and magnesia. Others as manganese, iron, sulfur, and few percentages of other elements represent 5%. The concentrations of elements changes slightly due to where and how slag was produced (20).

Venugopal et al.[21], focused in their study on the sustainable technology to save the natural and scarce materials. They used Class-F fly ash, which was procured from Raichur thermal power plant (RTPP). Commercially available ground granulated blast furnace slag (GGBFS) was used as binder along with F-fly ash. Manufacture sand also used as fine aggregate.

Alkaline activator solution was prepared using sodium hydroxide flakes and sodium silicate solutions. These chemicals were dissolved in specified amount with tap water. The ratio of sodium hydroxide to sodium silicate was equal to 1.5. GGBFS and Fly ash were mixed in the proportion of 20:80 in dry condition. Later, fine aggregate was added and mixed gain until homogeneous mix was obtained. Alkaline solution was added and mixed to get wet mix mortar. The ratio of fluid–to-binder was 0.2, and the ratio of total binder to aggregate was 1:1. This mortar mix was used to prepare bricks and blocks, specified amount of mortar was placed into the mold and static compaction was applied to mold of the block/brick. The bricks/blocks were kept in open air for curing until the age of testing.

Based on the results of test, they concluded that geopolymer masonry blocks performed better in contrast to normal blocks. The water absorption was found less than 10% by its weight. Hence, the increase in molarity decreased the water absorption, due to increase in density of the alkaline solution. Also, they concluded that the geopolymer blocks can be recommended for the construction of masonry structures.

2.3.4 Attapulgite clay

Attapulgite or Palygoriskite are two names for one mineral. The former is the earlier, which was used by Shevchen Kove in 1862[9]. Bradley introduced the name of Attapulgite in 1940 for the same mineral Attapulgus, Georgia [22].

Mouhamadou et al. [23], investigated the possibility of manufacturing Brick from Attapulgite clay at low temperature by geopolymerization process. They used Attapulgite, which is contained, in Allou Kagne clay, (Table2-2) includes the chemical composition of such clay.

Table (2-2): Chemical composition of Allou Kagne Attapulgite clay(wt%)[23].

SiO_2	CaO	Al_2O_3	Fe_2O_3	MgO	K_2O	Na_2O	TiO_2	MnO	P_2O_5	LOI
44.9	9.65	4.2	2.43	10.3	0.3	0.06	0.22	0.01	0.54	27.39

Clay grounded to approximately 250 µm, Attapulgite mining waste was mixed with different alkali concentrations (4, 8, and 12 M) of NaOH to form thick paste of clay and statically compacted (~10 MPa) in (2.5 ×5.0 cm) cylinders mold. The samples cured at 40°C and 60 % RH for long-term storage (1 week–3 months) and at 120°C, and 0 % RH for short-term storage of varying time periods (6, 12 and 24 h).

In short-term tests (i.e., bricks kept at 120°C and 0 %RH during 6, 12 and 24 h), compressive strength increased very rapidly. The maximum strength (27.1MPa) was obtained after 24 h with 12 M concentration. For all concentrations of NaOH, the strength obtained after 24 h were greatest; (14.6 MPa for 4 M, 24.7 MPa for 8 M and 27.1 MPa for 12 M). The lowest strength (4.5 MPa) was obtained with 8 M concentration after 6 h of curing.

In long-term tests, bricks kept at 40 °C and 60 % RH during 7, 14, 28, 60, and 90 days. The strength seems to be independent of time. For all periods, strength obtained with 8 M concentration was the largest (16.7 MPa). For this concentration, strength of 15.8 MPa was obtained after only 7 days of conservation. The lowest strength (6.3 MPa) was obtained with 12 M concentration after 3 months of curing. For most of the alkali-activated clay which have been tested in similar conditions (concentration of NaOH, humidity, curing temperature),the strengths in long-term were usually the highest. For the Attapulgite clay studied by Bassir et al., the strength in short-term was greater.

2.3.5 Other materials

2.3.5.1 Marine clay

Tamizi et al.[(24)], investigated the Kuala Perlis marine clay properties, to examine its possibilities to use as a pozzolanic materials. This material has good amount of aluminum and silicon, which expected to enhance the geopolymer brick properties.

Marine Clay used in this research obtained from along seashore in Kuala Perlis Malaysia, at different depth from 0.3 to 1 m. The brick samples were dried up in the oven at 105°C for 48 h, then crushed and sieved on sieve 315 µm. The combination of Na_2Si_3O solution and NaOH solution was used as alkaline liquid. To prepare (NaOH) solution, the sodium hydroxide powder (with purity of 99%) distilled in water. The NaOH solution was prepared with 12 M concentration and left to cool down at room temperature.

The dry marine clay mixed homogenously with (alkaline solution) to product composition paste in fixed ratio of 1:2, and the (Na_2Si_3O /NaOH)

ratio was 2.5 by mass. The pastes were mixed with fine aggregate in ratio 1:3 and molded in the mold has dimensions according to BS 3921. Then, the samples cured at 70 °C for 24 h and left at room temperature until reach the testing times.

Results indicated that the chemical composition of oxides component, which were (SiO_3), (Al_2O_3) and (Fe_2O_3) are conformed to the ASTM C618-13 was proved that the average composition of 70% of the weight Kuala Perlis marine clay which mixed with appropriate alkaline activator solution, was gain the compressive strength in early day. However, using the marine clay composition, improved compressive strength due to suitability of its composition with previous pozzolanic materials.

2.3.5.2 Niemenike clay

Mouhamadou et al. [(25)], tested the use of Niemenike clay with variance concentration of NaOH, in order to make a synthesis paste of clay. The mix compressed with manual hydraulic press until water get out the samples. Before the clay had been used, it need to grinded for about less than 250 µm. Then the sample cured with two temperatures (40 and 120 °C) for different periods.

Table (2-3): Chemical composition of Niemenike clay[(25)].

Component	$Si0_2$	Al_2O_3	K_2O	TiO_2	Fe_2O_3	MnO_2	ZrO_2	LOI
Malaysia Kaolin [%]	54.0	31.7	6.05	1.41	4.89	0.11	0.10	1.74
Niemenike Clay [%]	57.0	25.6	0.8	0.48	4.78	0.03	-	11.01

After testing, he concluded that the compressive strength for clay that activated with 12M of NaOH solution and subjected to 12 h of curing can

reach its compressive strength of 13.4 MPa, which was twice the strength that obtained from natural clay (Malaysia clay), (6.1 MPa).

2.3.5.3 Rice husk ash

Sultana et al. [26], studied the effects of rice husk ash and fly ash on properties of red clay collected from Naogaon district of Bangladesh. Different percentages of rice husk ash (RHA) and fly ash (5%, 10%, and 15%) were thoroughly mixed with clay to analyses various physical and chemical properties of clay followed by heat treatment at 800°C to 1100°C. The samples were testes for compressive strength, linear shrinkage, water absorption, porosity, and bulk density.

XRD analysis indicated that the clay sample was mainly illite type. Water absorption and porosity increased with increasing percentage of ashes (rice husk ash and fly ash). The water absorption for different mixes ranged from 6 to 10%, that may be appropriate for tiles and ceramic products. Fly ash and RHA used with 15% to enhance somewhat clay properties. The optimum firing temperature was found 1050°C for all samples of study. XRD test for clay with fly ash and rice husk ash that fired to 1050°C, exhibit the presence of feldspar and quartz as mainly phase, and hematite and cristobalite phase as secondly phase. This red clay deposits reinforced with variance suitable amount of rice husk ash and fly ash that used for various applications at low temperatures in building, construction and industry uses.

Agbede et al. [27] investigated the influence of rice husk ash (RHA) on the burned clay bricks properties. Mix percent (2 - 10 %) of RHA with Ibaji clay. Atterberg limits, specific gravity, compressive strength, and water absorption tests were performed for each mixture. X-ray diffraction pattern on

Ibaji soil and geochemical tests were carried out for rice husk ash. The results of X-ray test showed the presence of kaolinite as main composition phase in Ibaji soil.

From the results, it was found that the plasticity index decreased systematically and reached to minimum value when using 6 % of RHA. The compressive strength and water absorption still at a maximum value of strength 18.64 N/mm^2, and a minimum value of 14.8N/mm^2, respectively, when using 2 % of RHA. Also, using 2 % RHA have a significant effect to improve the Ibaji fired clay brick properties.

2.3.5.4 Sewage Treatment Plant Sludge

Sandeep et al .[(28)] studied recycling the waste products like Sewage Treatment Plant (STP) Sludge and Fly ash by incorporating them into clay bricks. It is a practical solution for problems like cost expenditure on waste management and its effect on environmental. The STP sludge and Fly ash are extremely close to clay brick in chemical composition so; it could be a potential substitute for clay brick. The sludge generated in most of the treatment systems around the world is discharge into the nearest watercourse. Among all disposal options, the use of sludge in producing constructional elements was considered to be the most economically and environmentally sound option. The results showed that the most successful composition of brick sludge; Fly ash was 80:20, Crushing strength = 6.4 N/mm^2, Water absorption = 19.4 %. Also, the produced bricks successfully passed the hardness and soundness test, while manufacturing Cost = 0.64.

Cusido et al.[(29)] studied the recycling sewage sludge that produced during manufacturing of paper. The plants need the incorporation and/or

alternative recovery solutions to reduce the quantities of waste generation, like this use in the cement industry or in soil remediation.

A practice substitution is the use of that waste, i.e. paper sludge, as raw material in the clay bricks production or ceramic product's. They tried to incorporate sewage sludge from different sources into ceramic materials, unfortunately with little success in the result for most types of sludge. Nevertheless, those experiments carried out using as additive sludge from the paper industry succeeded in producing a material suitable for the red ceramic industry. Duple mixes of clay and paper sludge at different formulations and parameters were produced, while their physical and chemical properties were examined too. The results showed that the Increment in percentage of paper sludge in the clay mixture had improvement properties of the material regarding its acoustic and thermal insulation. From the other side, it reduced its mechanical strength. However, this weakness of the material was compensates by enhancing ductility. Regarding leaching of metals, the ceramics have no environmental restrictions due to their use as building material, as the obtained results were virtually distinguish to those for the 100% pure clay samples.

They concluded that the volatile organic compound (VOC) emissions during firing of the new ceramic material did not exhibit any particular problem. The experience obtained after more than 10 years of industrial production for this new mixed ceramic product was also analyzed. This analysis allowed confirmation that clay brick production with incorporation of paper sludge waste is a practical solution from a technical point of view.

2.4 Bricks formation methods (Molding)

The first step in the forming process is produced homogeneous plastic clay mass. Usually, this achieve by adding water to the clay in a pug mill (mixing chamber with one or more revolving shafts with blade extensions). After pugging, the plastic clay mass is ready to form. There are three principal processes for forming brick namely, stiff-mud, soft-mud, and dry-press.

2.4.1 Stiff-Mud or (extrusion process) (6)

In this process, water in the range of (10 – 15 %) is mixed with the clay to produce plasticity. After pugging, the tempered clay goes through a de-airing chamber that maintains a vacuum of (375 to 725 mm) mercury. De-airing removes air holes and bubbles, giving the clay better workability and plasticity, which in its turn result in greater strength (6).

Next, the clay is extruded through a die to produce a column of clay. As the clay column leaves the die, textures or surface coatings may be applied (Textures, Coatings, and Glazes). An automatic cutter then slices through the column of clay to create the individual brick. Cutter spacing and die sizes must carefully calculated to compensate for normal shrinkage that occurs during drying and firing (6).

Ahmed A. (30), studied the effect of adding Attapulgite clay to normal clay brick as additive material, and investigated effect on the burning temperature, which required gaining brick the acceptable mechanical properties. Al-Nahrawan soils was used, which lie in Baghdad city, as basis material, while Attapulgite from Al-Najaf city was introduced. Attapulgite clay was used with rates of (10, 20, and 30 %) as a partial substitute by weight of the clay. Then, it was mixed with the original soil to form a

homogeneous mixture by adding water with specified ratios. Samples of bricks made in the laboratory by extrusion method can be seen in (Plate2-2).

Plate (2-2): Extrusion process [30].

After drying process is done the samples, it was burned with different degrees (750, 800, 850, 900, 950, and 1000 °C) for each percentage of Attapulgite additive.

Based on this investigation, it was concluded that the effect of using Attapulgite clays as a partial substitute of (10, 20, and 30 %) by weight of the clay, having the following effects:

a- Negative impact on drying shrinkage, whereas a positive effect on bulk density after drying.

b- Positive impact on compressive strength after drying.

c- Reduction in (longitudinal shrinkage, bulk density and efflorescence) for different degrees of burning.

d- Increasing in (compressive strength, water absorption, and modulus of rupture) for different degrees of burning.

2.4.2 Soft-Mud Process

The soft-mud process is particularly suitable for clays that containing high amount of water to be extruded by the stiff-mud process. Clays are mixed with (20 – 30 %) of water, and then formed into brick molds. To prevent clay from sticking, the molds are lubricated with either sand or water to produce “sand-struck” or “water-struck” brick. Brick may be produced in this manner by hand or by machine (6).

Francis et al. (31), studied a new methodology for producing bricks in a cost-effective and environmentally friendly manner, by using the tailings which are produced from iron ore mines in Western Australia (WA). The particle-size distribution of the mine tailings as supplied was contained about (6.50 %) fines, which used after limited grinding. The tailings, also, comprised up to (70.5 %) sandy and silty-sized molecules. The mine tailings screened through a sieve mesh size of (212µm). A mold having a size of (11.5* 5.5 *3.8 cm, length*width*high) was used to make the bricks in the laboratory. For activating the mixture, 20% of 12 M NaOH solution was added (14). In addition, Na_2O_3Si solution added to the mine tailings and the mixture stirred using a laboratory concrete mixer for about 10 min, until homogeneity of the mixture was gain.

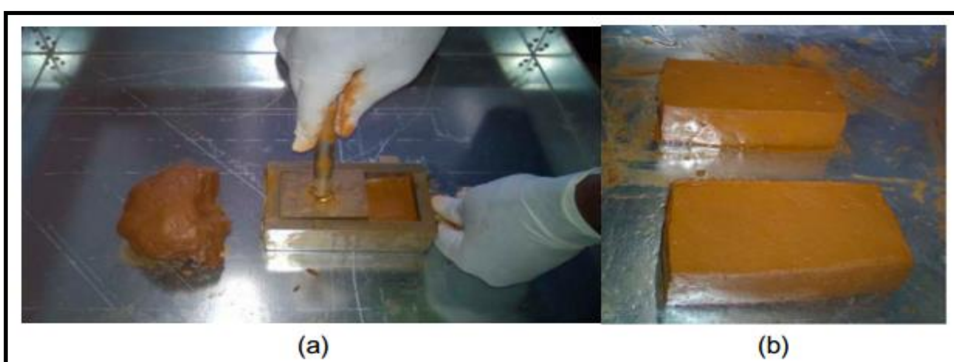


Plate (2-3): a. The brick molding by tamper b. Prepared bricks (fresh state).

The interior surfaces of molds were oiled to reduce friction and allow easy release from the mold after casting. The brick was cast by manual pressure with the assist of a temper (Plate 2-3 a). Later on each brick was removed from its mold, then left to sit for 24 h at room temperature (Plate 2-3 b). After soaking, the samples placed in the oven and cured according to the curing schemes. The conclusions of this study were:

1. The strength of the product's bricks was greatly affected by the curing temperature, the compressive strength increased as the curing temperature increased to a certain optimum point of 80 °C. Than the compressive strength started to droop with further increase in temperature.

2. The optimum parameters were Na_2O_3Si solution content (31%), curing temperature of (80 °C). Furthermore, the curing time for 1-day resulted in an optimum compressive strength of (19.18 MPa), 3-days curing provided a compressive strength of (34.0 MPa), and 7-days curing resulted in an optimum compressive strength of (50.35 MPa). Moreover, when submitted to further curing above 7-days, the strength started to wane.

2.4.3 Dry-Press Process

This process is particularly suited to clays of very low plasticity. Clay is mixed with a minimal amount of water (up to 10 percent), then pressed into steel molds under pressures from (3.4 to 10.3 MPa) using hydraulic or compressed air rams(6).

Hasson et al. (32) studied the replay method distinguished (material quality produced, summarizing costs or time, keeping environment from pollution and persistence to earth vibrations). They suggested the use of local basic material to produce bricks by replacing the manufactured bricks

method, and making physical tests by (measuring the content of humidity and measuring the granular grading on it). They prepared four types of mixtures, with ratios of sand to clay of (0% -100 %), (30 % - 70 %), (50 % - 50 %), and (70 % - 30 %). They used cement in different ratios, namely; (6 %, 9 %, 12 %, and 15 %) by weight of dry mixture. The formation process was made by semi dry pressing with humidity content from (5%-7.5%) depended on the type of pressed mixture. Product models recognized as high efficiency based on results of compressive strength.

Faheem et al. **[33]**, carried out their investigation on the production process of the clay- bricks based geopolymer from the mixing stage even curing stage by using a geopolymer brick making machine. The kaolin used in this work was obtained from Associated Kaolin Industries (AKI). (Table 2-4) shows the chemical composition of the used kaolin.

Table (2-4): Chemical composition of the kaolin [33].

Component	SiO_2	Al_2O_3	K_2O	TiO_2	Fe_2O_3	MnO_2	ZrO_2	LOI
Kaolin [%]	54.0	31.7	6.05	1.41	4.89	0.11	0.10	1.74

As for fine aggregate, quarry dust was applied, because it is usually employed in a production of brick masonry. For the ratio of sand to kaolin, the range is approximately 6 to 10. Equally, for the ratio of kaolin to alkaline solution is just about 0.5 to 1.5. The ratio for Na_2O_3Si to NaOH is ranged from 0.2 to 0.4.

Plate (2-4): Geopolymer brick making machine [33].

The manufacture process for kaolin-based geopolymer brick was not the same of that conventional clay bricks process, because this method was included geopolymerization process in brick's production. The production process done by using the geopolymer brick making machine as shown in (Plate 2-4).

The main conclusion of this research showed the possibility of using kaolin-based geopolymer brick as an alternative construction material instead of commercial brick in the market. The production procedure of geopolymer brick was simple, which help to create high brick quantities to satisfy the demand of the building industry. A unique possibility for mass utilization of kaolin could be employed as a geopolymer brick. However, the properties of the kaolin based geopolymer brick is still need to be considered in term of compressive strength, its resistance to the environment and so on before can be used as building material.

2.6 Concluding Remarks

With return to foregoing review, it can be notice that there are considerable amounts of research that deals with geopolymer brick manufacture by use fly ash, slag, rice husk ash…etc. However, there is a lack of data concerning the investigations about geopolymer brick containing locally Attapulgite clay.

In addition to the most of available work in Iraq dealing with clay brick was devoted to study the effect of additives various material as (ground glass, tires, industrial waste, cement…etc.) to enhance mechanical properties of clay bricks such as (strength, absorption and unit weigh), or to reduce its burning temperature degree. In this study, it will investigate new type of clay brick, which depends "geopolymerization or alkali activated bricks process based on Iraqi Attapulgite clay".

CHAPTER THREE

Experimental work

EXPERIMENTAL WORK

3.1 General

This chapter describes the process of producing geopolymer brick based on locally Attapulgite clay, also includes determination of the suitable burning temperature of Attapulgite clay brick based geopolymerization or alkali-activated process. The materials used in the experimental work, the methods adopted in the preparation and testing of bricks are presented too.

Properties of clay brick such as compressive strength, water absorption, efflorescence, shape and dimensions, longitudinal shrinkage, bulk density, modulus of rupture, and microstructure tests are studied.

In addition, this chapter provides information about mixing trials and mixing procedure.

3.2 Experimental program

Three different parameters are adopted in this study, which are hydroxide sodium concentration (i.e. 4, 6, and 8 mole (M)), temperatures of curing (i.e. 80,100, and 120 °C) and temperatures of burning (i.e. 400, 500, and 600 °C).

The brick samples dimensions are (75x38x25 mm), they were selected based on the available quantity of material that prepared for this research, and devices according to British requirements No.84 for 1974 [34]. Fig. (3-1) shows the details of the experimental program.

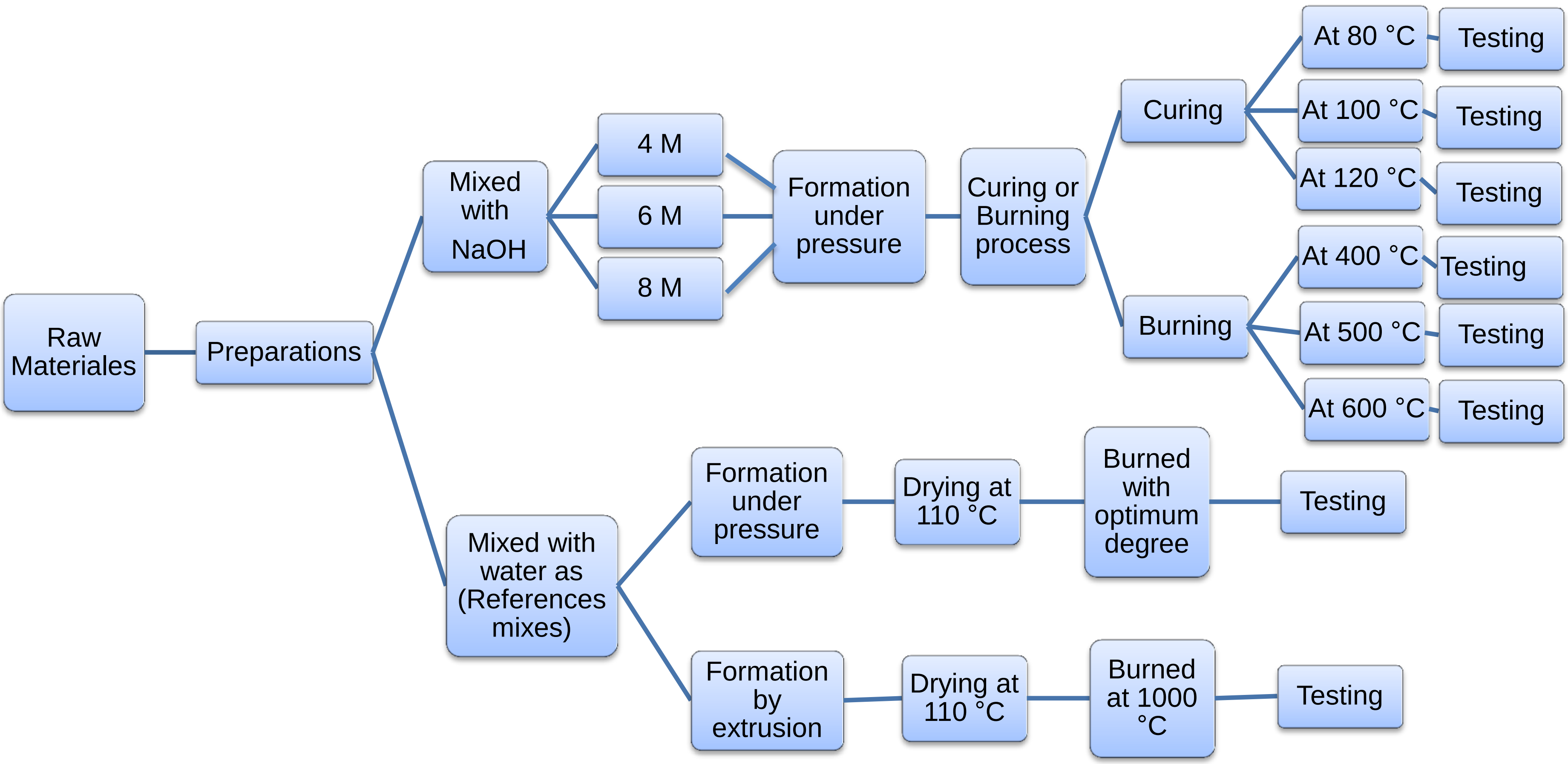


Figure (3-1): Experimental program.

3.3 Materials and their preparation

3.3.1 Attapulgite clay

In Al-Najaf and Karbala provinces, Attapulgite dominates clay mineral Mudstones of the Injana formations (Plate 3-1). Injana formations (late Miocene –Pliocene) are exposed in Al-Najaf region (Tar Al-Najaf), as bluish green and gray clay stone, which have about (0.5 m) thick with plants remains [35].

Plate (3-1): The Attapulgite site in Al-Najaf region formation Injana[35].

Attapulgite clay brought from (Al-Najaf province), and dried in sunlight or in drying furnaces until completely dry state. Then, crumbled it and before clattering used, it grinded to less 250 μm. Grinding achieved by use balls mill (Plate 3-2) for about 2 hr with 12 balls (450 gm each one). The time and number of ball examined by trail until reach the target fineness. After grinding process it finish, the ground clay passed on sieve 300 μm. After that, the chemical analysis carried out on Attapulgite grinds.

Plate (3-2): Balls mill.

3.3.1.1 Chemical analysis of Attapulgite clay

The main oxides of (Attapulgite clay) are listed in Table (3-1).The chemical component of the soil gives an important indicator of the nature and properties of the bricks factory from this soil [36].

**Table (3-1): ***Chemical analysis of 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**

* Chemical analysis of Attapulgite after grinding was performed at the Geological Survey and Mining state Company.

3.3.1.2 Physical properties of Attapulgite clay

The main of physical properties for Attapulgite clay, which are specific gravity equal to 2.34 and its color was bluish green.

3.3.1.3 Grain size distribution for Attapulgite clay

Finally, wet sieving and hydrometer method (Plate 3-3), were used accordance (ASTM D 422) [37], to classify soil for accuracy components.

Plate (3-3): Hydrometer method.

Results of grain size distribution for (Attapulgite clay) shown in Fig. (3-2) and Table (3-2).

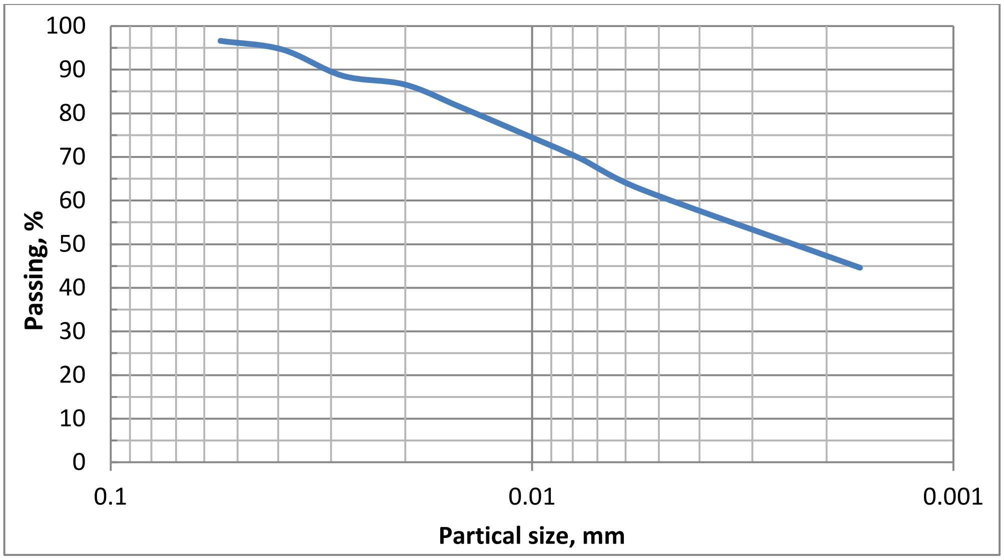


Figure (3-2): Particle size distribution of Attapulgite clay.

Table (3-2): Classification of Soil according ASTM standard D422-63 [37].

Grain Size (mm)	Content in soil (%)
≤ 0.002	47.5
0.002 – 0.02	39
≥0.02	13.5

Result above distinguished that the soil has high fineness, thereby surface area is high accordingly, which can generate good activity in geopolymerization process to produce geopolymer bricks.

3.3.2 Water

There is no much limitation for used water, except that the water must be not severely polluted. Normal tap water has been used for mixing, preparing and test of samples.

3.3.3 Hydroxide sodium solution

Commercial type of NaOH flakes or pellets with purity of 98% was provided from the locally market. The NaOH solution was prepared by dissolving its flakes in tap water at different concentrations as required. The (NaOH) solution left for about one day before use, as recommended [21,38]. To evaluate (NaOH) solution with different molarity (4, 6, and 8 M), the molarity formula (Eq. 3-1) is used, and the weight of NaOH flakes can calculate as shown below:

$$[\boldsymbol{M} * \boldsymbol{V} = \boldsymbol{W_t} / \boldsymbol{M_w}] \quad \text{……..………………………………… Eq. (3-1)}$$

Where; M=molarity in mole/liter, V= volume of solution in liter,

W_t=weight of solution in grams, Mw = molecular weight of solute gram/mole (which equal to 40 for NaOH).

For example, if it is required 1 liter of 8M (NaOH) solution, the formula is become; [8 x 1=W_t / 40], then Wt= 320 g of NaOH flakes for making 1 liter of solution.

In order to measure the quantity of the water added, a 1000 ml volumetric flask and a 1000 ml cylinder needed for this job, besides safety cautions like costume and safety gloves. A 1000 g of water is weighted in cylinder and a 320g of NaOH flakes is weighted in volumetric flask. Then, a 500g of water is add as a start from the cylinder to the volumetric flask, stir until complete dissolving of NaOH solids. Adding water is continued with stirring until reach the 1000 ml marks of the volumetric flask. The remaining water is measured in the cylinder to determine the added water, which should

be about 900ml. Therefore, 320 g of NaOH flakes needs 900 ml of water to prepare 1 liter of 8M NaOH solution.

3.4 Preparation and formation brick samples

3.4.1 Mix trail samples

3.4.1.1 Extruding method

At beginning (30 bricks sample) were made as a reference mix (Attapulgite clay with water) at normal burning temperature for production clay bricks, which is about (1000°C).

Plate (3-4): Laboratory Muds Extrusion Device.

3.4.1.1.1 Plasticity Testing

To find the optimum quantity of water which required for formation and get formable clay paste with less losses of strain or formable, Pfefferkorn test [34] (Plate 3-5) was used. This test is recommended when laboratory machine for clay brick production (laboratory muds extrusion device) is used.

Plate (3-5): Pfefferkorn test.

In Pfefferkorn test, a plate having defined weight (1192) g is dropped from a height of (186) mm on to a measured cylinder having a diameter of 33 mm and height (h_0) of 40 mm, h_1 (the height of cylinder after buckling) is measured. However, the water content which allows a compression ratio (h_0/h_1= 3) is considered to be the plasticity number according to the Pfefferkorn method [34]. This is determined by making many clay pastes sample's with different water contents, and drawn interpolation between them and then choose the water content value corresponding to plasticity number equal to 3.

To fined coefficient of elasticity for mixing were determined in Pfefferkorn method, making four clay pastes mixes with different water content, and the results represents in Fig. (3-3).

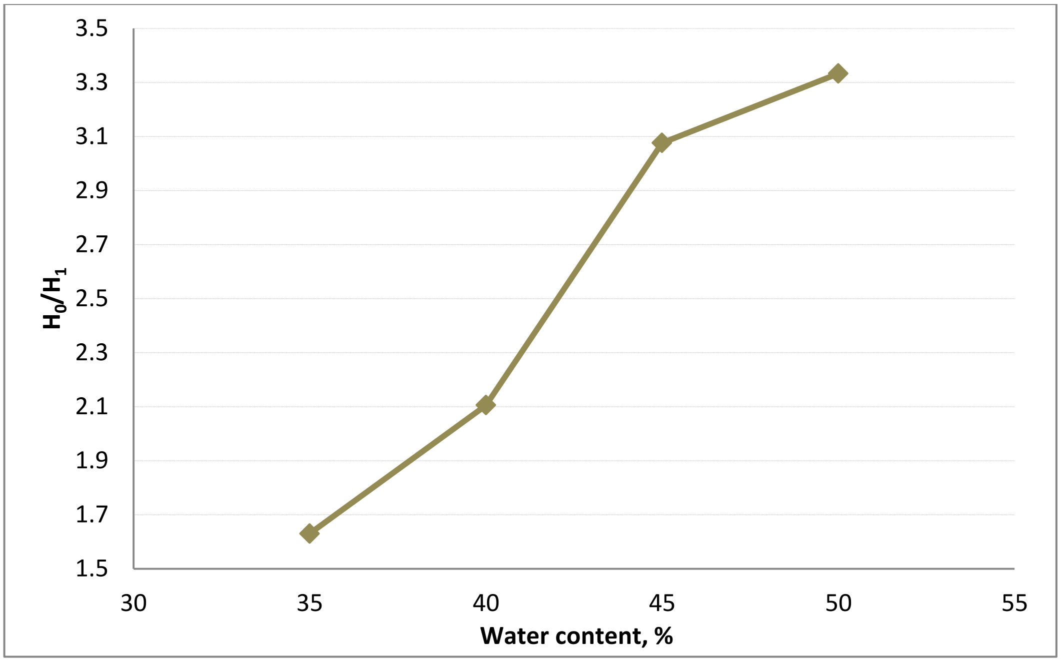


Figure (3-3): Reading of Pfefferkorn test*.

* This test carried out at “Building Researches Center” in “The Ministry of Housing and Reconstruction”.

Interpolation has been made between the values 2.105 and 3.076 to get the water content that corresponding to 3 (which is target number of Pfefferkorn test). The target water content was equal to (44.6 %). The result showed high coefficient of elasticity for soil, which is due to the clay’s content and high silica, this confirmed by the results of the chemical analysis for the soil which has been shown in (Table 3-1).

3.4.1.1.2 Formation of brick samples

Preparations start by weighting an appropriate quantity of soil with required water, then mixed together for about (10-15 min.) using cement mortars mixer until gets homogeneous consistency (Note: manual mixing could be needed). After that, the prepared clay paste delivered gradually into extrusion device mud (Plate 3-6).

Plate (3-6): Formation of brick samples for reference mixture (Ref.2).

After completing formation process for samples, they left for about one hour (Plate 3-7) for acquire their necessary durability to conduct the process of weighing and dimensions measurement.

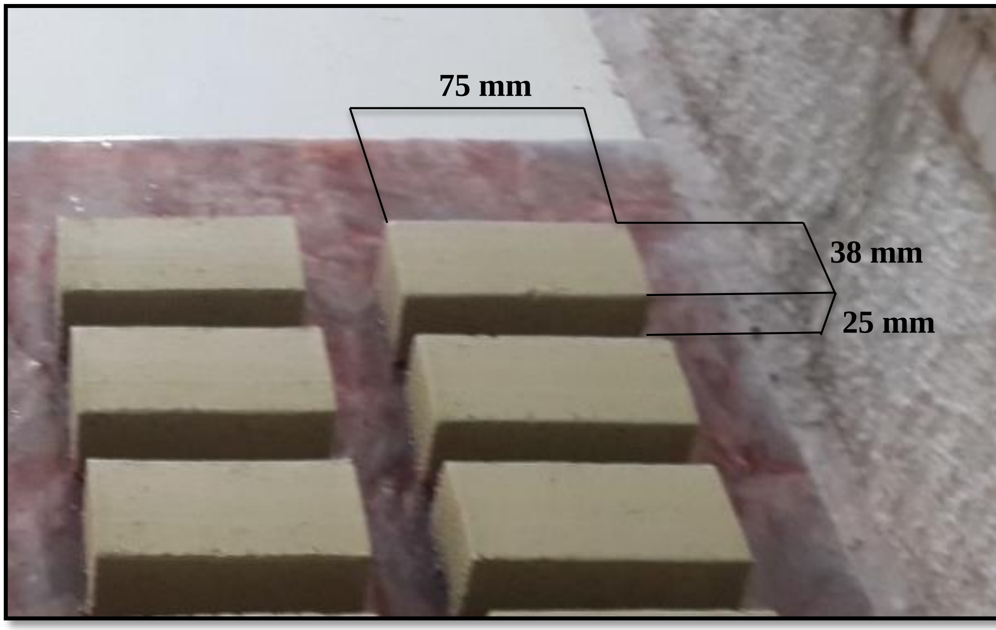


Plate (3-7): Brick samples of reference mix (Ref.2).

In contrast, when tried to mix and formation of Attapulgite clay with (8 M NaOH solution) as a geopolymer brick, the paste clay became very stiff (At same water content used for reference mix which mentioned earlier), and have cross cracks at extruding (Plate 3-8). It is worth noting that many different mixes with increasing in water content, reach to 35 % above reference mix and reducing in NaOH concentration until 4 M, but with no avail to enhance formation process of geopolymer bricks by this method.

Plate (3-8): Failure extruding.

3.4.1.2 Pressing method

To investigate the possibility of formation Attapulgite clay and NaOH solution by pressing method, cylinder samples of Attapulgite clay paste with different concentrations of NaOH solution were molded in dimensions of (diameter=30 x height=30) mm, with applied (10 kN) manual hydraulic press (Plate 3-9).

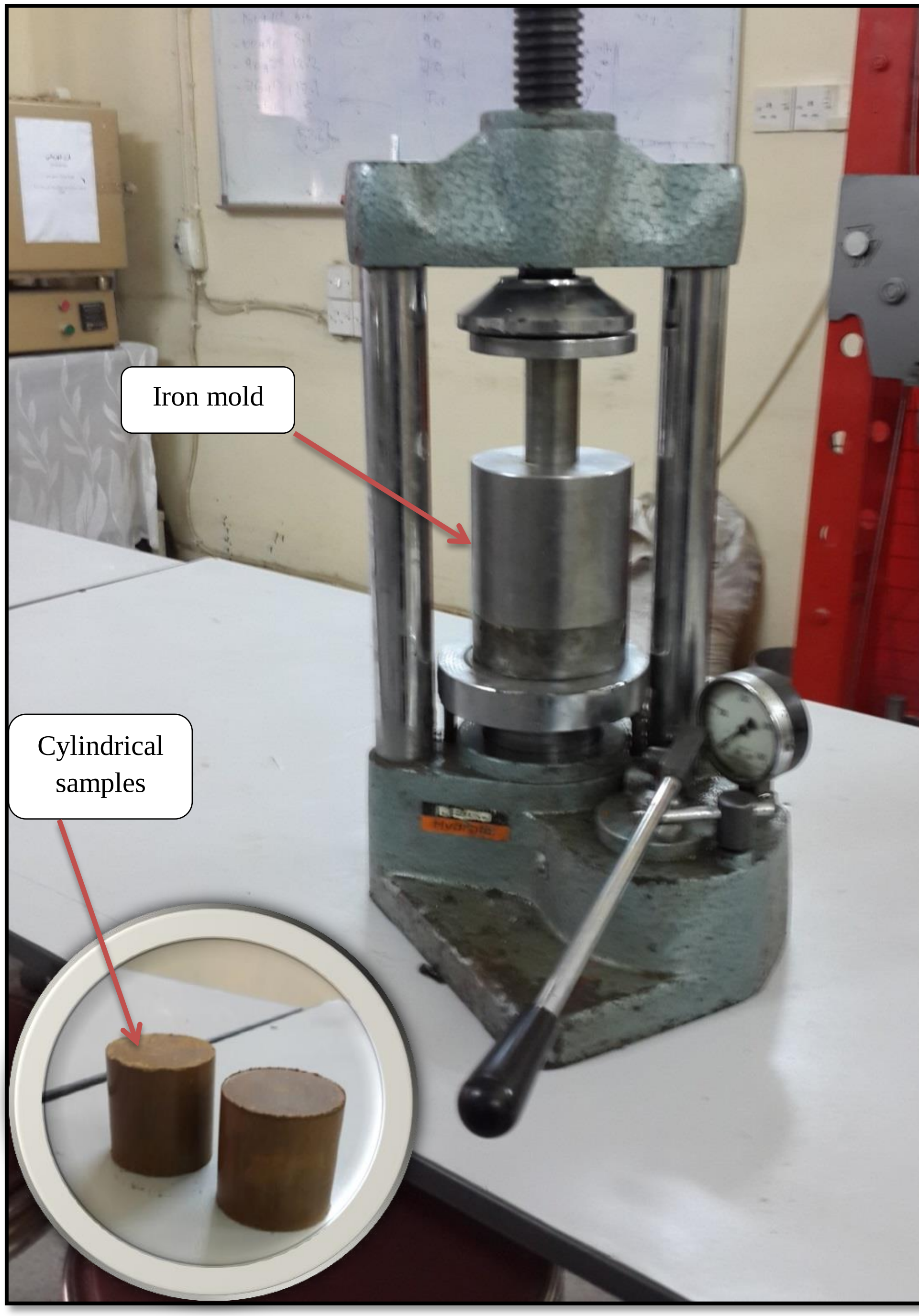


Plate (3-9): Trail mix formation by press*****.

* This trail mix carried out in Materials Engineering Department laboratories.

3.4.2 Formation of program work samples

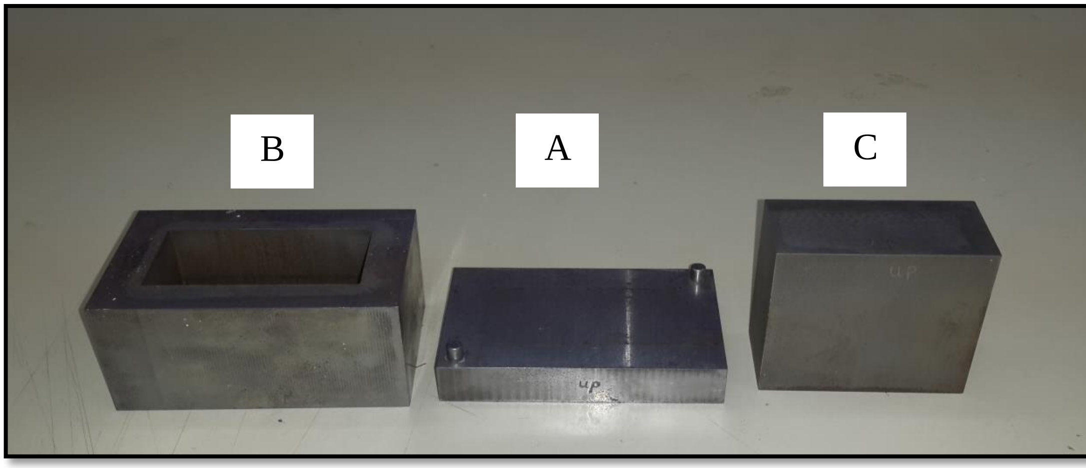
Due to succeed formation of bricks by press method, an Iron mold according to British standard No. 84 for 1974(34) for testing methods and manufacture laboratory brick samples was manufactured. This mold within the standard dimensions (75x38x25 mm). The iron mold consisted of three parts (Plate 3-10) as follows:

A) The base, which have parallelogram shape with (105 mm length, 68 mm width, and 15 mm in thickness).

B) The sides, which is surrounded the sample in mold during pressing process and gives required specific section. All the sides have 40 mm in height and 15 mm in thickness.

C) The piston (iron block) that press the clay paste in mold to have cubic shape with 75 mm in length, 38 mm in width and 65 mm in height.

This mold manufactured from strong metals to gain it durability and resist deforms or frictions that result from frequent use. In addition, the mold parts were products with high accuracy in dimensions by using electronic turning in one of the local factories.



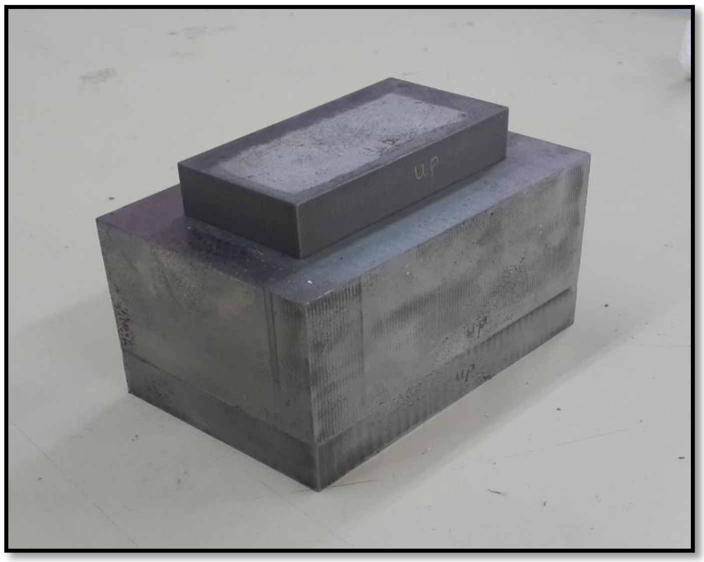


Plate (3-10): Brick sample mold.

The Attapulgite clay was mix with three concentrations of NaOH solution (4, 6, and 8 M). The sodium hydroxide solution slowly added to the Attapulgite clay and mixed for about10 min to gain homogeneous mixture. The consistency of mix has semi-dry state as the (solution/solids) ratio equal to 25%.

The mixtures were put into a brick mold (75×38×40mm) with hand compaction by using steel rod until the mold was full (Plate 3-11 A). Then, the iron block (75×38×65mm) placed on top of the mix and electric loading

machine pressed the mix from its top (Plate 3-11 B). The pressure used was (13-14) kN. This value of pressure was chose to simulate air pressure which applied by extrusion device in extruding method; depending on wet density of brick sample products by this manner, which was about (1.9 g/cm^3) for normal density clay bricks[30]. The formula used to find required pressure as following:

$$[\rho = W/V] \quad \text{Eq. (3-2)}$$

Where; ρ = wet density of brick which = 1.9 g/cm^3

V= volume of brick sample= 7.5x3.8x2.5=71.25 cm^3

W= required weight

After performed calculation, result required weight of binder equal to 136 grams. Many trials of various press value were carried out until reach to the required thickness of brick (i.e., 25 mm). After pressing, the mold blocked up for about 25 mm from the bottom, and the pressing machine used again to press the brick samples out of the mold (Plate 3-11 D). After compression, the specimens were de-molded (Plate 3-11 E) and allowed to sit overnight at room temperature [23] (Plate 3-11 F) with sealed vessels fitted with teflon before being cured at different temperatures (80, 100 and 120°C), or drying at (110°C). This “soaking time” is typically used to allow time for dissolution and for geopolymer precursors to form [39,40]. Then, uncovered specimens placed in an oven for drying or curing depending on work programs. (Plate 3-11) explains process steps of brick samples products.

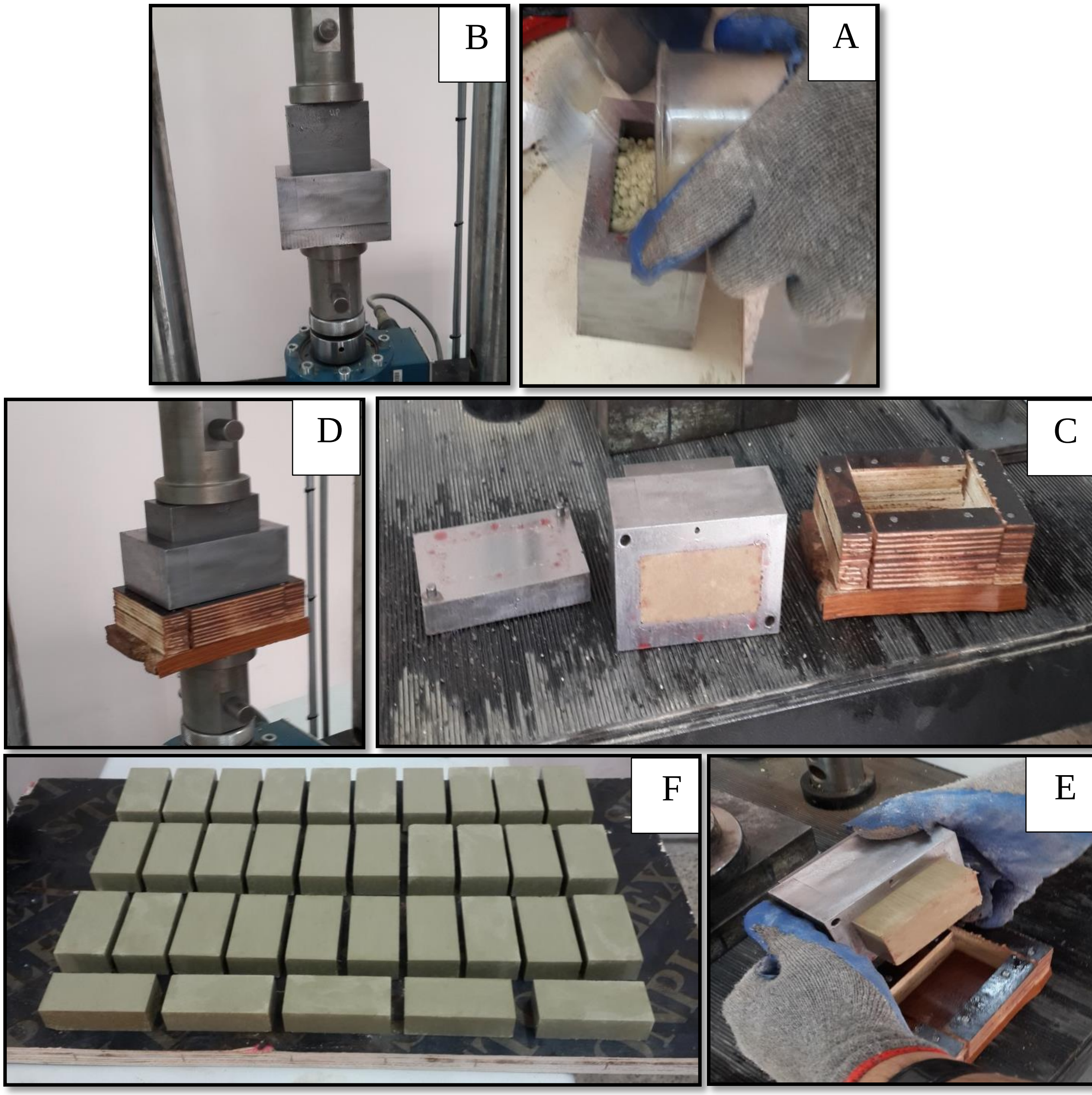


Plate (3-11): Preparation process for brick samples.

Twenty mixes were prepared with total of 600 samples (30 brick samples for each mix), (Plate 3-12). 18 mixes were content full parameters, Table (3-3) and Table (3-4). In addition, two mixes produced for comparison (Reference mixes).

Table (3-3): Group no.1, mixes were subject to curing only.

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

Table (3-4): Group no.2, mixes were subject to drying and burning.

Mix. No.	(NaOH) Concentration, M	Drying Temperature, °C	Burning Temperature, °C
10	4	110	400
11	6	110	400
12	8	110	400
13	4	110	500
14	6	110	500
15	8	110	500
16	4	110	600
17	6	110	600
18	8	110	600

Plate (3-12): Programs brick samples.

3.5 Curing and drying process

After soaking time for group 1 mixes samples, curing in drying furnace (Plate 3-13) achieved with different temperatures (80, 100, and 120 °C) at 24 hours. Then, samples were tested. Whereas, samples of mixes group 2 were dried in same furnace mentioned for curing of (100-110 °C) until reach to fixed weight of dried samples. Then, measured their dimensions and weight for dry state and became ready to burning.

Plate (3-13): Curing and drying furnace.

3.6 Burning process

After completing drying process for samples group 2, they burned in digital burn furnace (Plate 3-14) at different temperatures (400,500, and 600 °C) with rate of rise 4°C per min until reach required degree. The furnace was set at this degree for 1 hour as soaking time. After completed all time of burn, the furnace was waited to cool and open to extract samples and prepared to test.

Plate (3-14): Digital burning furnace.

3.7 Reference mixes

There are two mixes were made for comparison in this study, table (3-5) listed their condition. First mix (Ref.1), was comprised Attapulgite clay and water. Such mix have the same conditions of preparation and formation, but burned with optimum mix that have best mechanical properties for clay brick after testing. From this mix, we can easley notes the response of brick to geopolymerization process and effect of NaOH on Attapulgite clay bricks.

Second mix (Ref.2) was conducted for comparison between method of manufacture brick mentioned in this study (forming by press) and that used as popular manufacture method (extruding method). This mix was comprised Attapulgite clay with same fineness in Ref.1. However, its samples were

formed by extruding method, while burning achieved at normal burning temperature for production clay bricks, i.e., (1000 °C).

Table (3-5): Reference mixes.

Mix. No.	Clay type	Burning Temperature, °C	Formation method
Ref.1	Attapulgite	1000	Extruding
Ref.2	Attapulgite	500	Pressing

3.8 Testing of brick samples

All tests carried out according to Iraqi's standards No.24 for 1988 (41), except those tests of density, shrinkage and modulus of rupture which done according to British standard No. 84 for 1974 for testing bricks (34). Every test result represented the average of 10 brick samples for each mixes; beyond dimensions measurement test, which was performed on twenty-four samples.

3.8.1 Dimensions measure test

24 samples of brick were used to determine the mean dimensions. Samples were collected and aligned behind straight line and on leveled surface. Then, length, width and thickness of the samples measured as one unit by use long ruler (41). The measured dimension were divided by 24 to get the average length, width and thickness.

3.8.2 Shape tests

a- **Concavity;** ruler was placed longitudinally on surface of brick sample that subjected to measure its concavity, where the place that gives a bigger distance from leveling is determined. The distance between

mass surface of the sample and ruler to nearest 1 mm was measured and recorded as concavity in surface [41].

b- Convexity; the brick placed on leveled tablet, where its convexity surface contact with tablet, the distance between the leveled tablet and every angle of four brick angles was measured and the mean value was took as a convexity [41].

3.8.3 Water Absorption

Samples drying were achieved by, placed them in drying furnace for not less than (36 hr) until fixedness of weight under a temperature of (110-115) ^{o}C. Samples then weighed after cooling at room temperature, and the samples were sunken in water container with room temperature for 24 (±1) hr. Later they left from container and wiped with wet piece of cloth then weighted during one minute of rising. The water absorption percentage determined as follows [41]:

Water absorption = [(Ws – Wd) / Wd] x 100 ……………..…Eq. (3-3)

Where;

Ws : Weight of sample after 24 hr sinking in water (g).

Wd : Weight of sample at dry state, before sinking in water (g).

Calculated of water absorption was done to the nearness 0.1 %.

3.8.4 Compressive strength

Samples are tested according to placing feature in building, whereas the two loading faces to nearest (1 mm) are calculated and the smaller area between them is use to determine the strength.

Samples were sunken in water continuously for (24 hr), and then sample was left from water, wiped its surfaces with wet piece of cloth and

then placed between two sheets of thin wood [41]. The applied loading rate equal to 0.6 MPa/min (This rate was adopted depended on previous research) [30], until failure of brick sample and the compressive strength value determined as following:

$$\mathbf{Cs} = {}^{\boldsymbol{p}}/_{\boldsymbol{A}} \quad \ldots\ldots\ldots\ldots\ldots\ldots \text{Eq. (3-4)}$$

Where;

Cs= compressive strength (MPa).

P=applied load at failure (N).

A=loaded area (mm^2).

This test carried out by using 20T (W 210) Hydraulic Compression Test Materials Machine, (Plate 3-15).

Plate (3-15): Compressive strength test machine.

3.8.5 Efflorescence

Samples placed on ends (minimum surface area of brick sample) in flat vessel containing distilled water with depth (7.5 mm). Samples then left at room temperature for seven days with adding dropped water due to evaporation or absorption to maintain its level, after those samples let to drying at same vessel and room temperature and notes the efflorescence after three day of water drying (41).

3.8.6 Shrinkage

The shrinkage of clay when dried and burned is an important property in the clay brick's production, since it controls the size of the finished product when the size of brick in the wet state is fixed. Burning shrinkage often used as a check on the adequacy of burning process. Shrinkage may be measured linearly or volumetrically. However, for speed and practical implications, the linear measurement is preferred (34).

Plate (3-16): Measurement shrinkage by digital vernier.

The brick samples are carefully measured, immediately after making. It dried at 110 °C to constant weight, cooled in a desiccator and measured again by digital Vernier to nearness 0.1mm (Plate 3-14). The linear drying shrinkage calculated as follows [34]:

$$L.S)_d \% = [(Lw - Ld) / Lw] \times 100\% \quad \text{Eq. (3-5)}$$

Where;

L.S : Longitudinal shrinkage

L w : Length of sample before drying (mm)

L d : Length of sample after drying (mm)

Burning shrinkage measurement normally follows that of drying shrinkage and use the same test piece that used in the wet state. The dry piece is burned in kiln, and then cooled, the latest stage of cooling being in a desiccator. The bricks dimensions are again measured by vernier. The linear drying shrinkage calculated as follows [34]:

$$L.S)_b \% = [(H d - H f) / H d] \times 100 \quad \text{Eq. (3-6)}$$

Where:

L.S : Longitudinal Shrinkage.

H d : Sample length before burning (mm).

H f : Sample length after burning (mm).

3.8.7 Bulk Density

Samples' three dimensions and weighting were measured by using balance with accuracy of (0.1g), after all (formation stage, drying stage and burning stage) calculate their bulk density as follows [34]:

a- Bulk density of samples after drying

B.D) $_d$ **= W d / V d** ……………………………………….Eq. (3- 7)

Where:

B.D)$_d$: Bulk density after drying process (g / cm ^{3}).

W d : Sample's weight after drying (g).

V d : Sample's volume after drying (cm^3).

b- Bulk density of samples after burning

B.D)$_b$ = W f / V f ……………………………………..……Eq. (3- 8)

Where:

B.D)$_f$: Bulk density after burning process (g / cm ^{3}).

W f : Sample's weight after burning (g).

V f : Sample's volume after burning (cm^3).

3.8.8 Modulus of rupture

The modulus of rupture is calculated from the force required to break a bar-shaped or rod test-piece supported at two points near the ends, the force being applied equidistant from the points of support. The point of load application and the two supports should be arcs of cylinders at least 6mm diameter [34].

The test-pieces of the same size as those used for another tests (water absorption, efflorescence tests, etc…) can be used.

The test-piece is place in a suitable attachment to the testing machine, so that the supports are symmetrically placed near the ends of the test-piece and perpendicular to the cylindrical test-piece axis (Fig. 3-4).

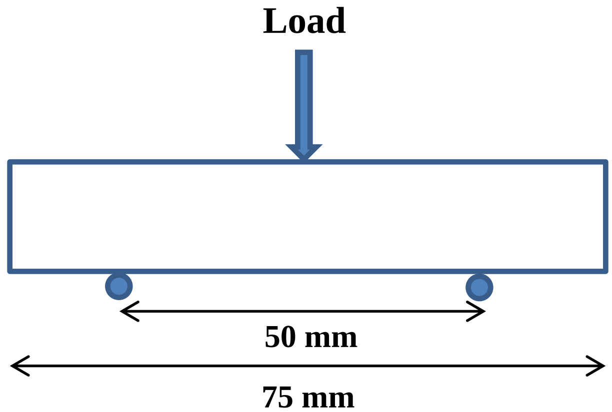


Figure (3-4): Details for samples under modulus of rupture test

The load is applied perpendicular to the plane containing the edges of the supports and the test-piece. The measurements are carried out by vernier calipers to 0.01 mm and the mean of the four readings taken. If bars used with rectangular cross-section, the appropriate formula is [34]**:**

$$\boldsymbol{M.O.R} = \frac{\boldsymbol{3PL}}{\boldsymbol{2BD^2}} \quad \text{.................. ....Eq. (3-9)}$$

Where:

M.O.R: Modulus of rupture (N/mm^2)

P: The breaking force (N)

L: The distance between supports (mm)

B: The breadth (mm)

D: The depth of the cross section (mm)

3.8.9 Microstructure test

3.8.9.1 Scanning Electron Micrograph (SEM)

SEM has become a very important tool that describes an image for studying the materials microstructure. SEM consists of electron gun with tungsten filament for liberate electrons when heated at high temperature. High potential (1-30 kV), were subjected to the electrons results in acceleration them, then focused and scanned the sample. The received image from the surface is review on the computer screen. To conduct this test, a suitable volume sample required to prepare. This test carried out by using tested cubic specimens trimmed to produce block cube samples with dimension of (5x 5 x 5) mm. (TESCAN-VEGA3 LM) apparatus(42) used in this test (Plate 3-17).

3.8.9.2 Energy Dispersive Spectroscopy Systems (EDS)

Energy Dispersive Spectroscopy (EDS) Analysis provides elemental and chemical analysis of a sample inside the SEM, TEM or FIB (43). In this test, used (AZtec) apparatus (Plate 3-17), a new and revolutionary material characterization system provides by Oxford Instruments that gather accurate data at the micro- and Nano scales. Aztec Energy EDS Micro analysis software characterizes material composition of samples inside a SEM or FIB. From high-speed acquisition, data analysis and reporting of single points, line scans and area mapping (17).

Plate (3-17): SEM and EDS test apparatus.

* This test carried out by Department Of Production Engineering And Metallurgy /University of Technology.

CHAPTER FOUR

Results and discussion

RESULTS AND DISCUSSION

4.1 General

This chapter presents the results and discusses some properties of geopolymer bricks, effect of different degrees for curing and burning, and variation in concentration of NaOH solution that used. The physical tests that performed after curing or burning are (shape and dimensions, longitudinal shrinkage, Bulk density, Compressive strength, Water absorption, Efflorescence, and Modulus of rupture). Also, microstructure tests SEM and EDS are performed. All these tests are performed to determine the properties of produce's bricks and its optimum degree of (burning – curing) temperature and prefect NaOH concentration.

4.2 Physical properties of brick

4.2.1 Shape and Dimensions

All dimensions of brick samples with all parameters (group 1 and group 2) exhibited high stability with sharp edges and straight surface (concavity or convexity not found) before and after curing or burning. This might be due to method of forming (press mud process), and using press in molding, furthermore activity role of NaOH solution might help for correlation between particles of soil.

4.2.2 Color

The brick samples color changed according their state, i.e. depending on their degree of expose to temperature of curing or firing, (Plate 4-1).

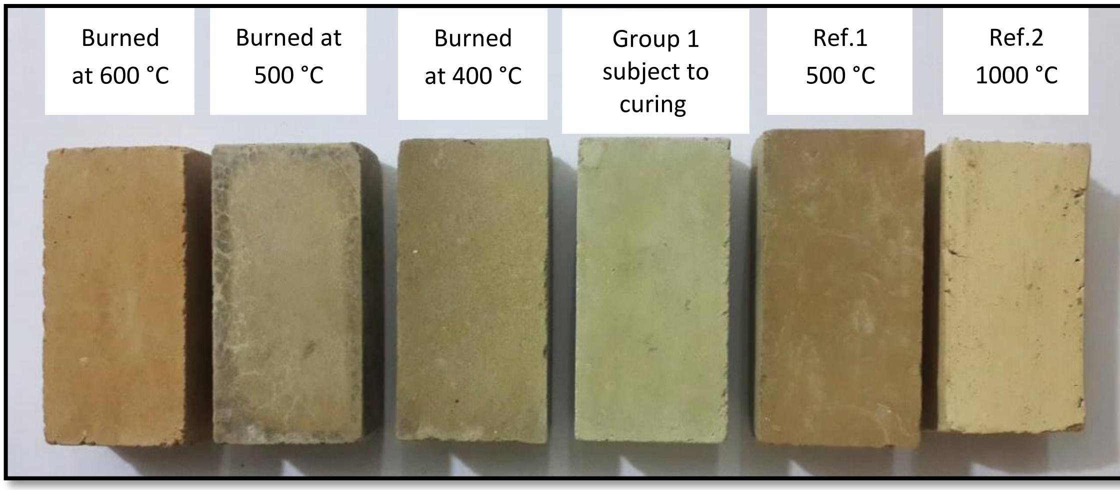


Plate (4-1): Variation of samples color with different (curing and burning) temperatures.

Where the following colors were noticed: light gray for all curing temperatures (80,100 and 120 °C), and ranged from gray to red at burning temperatures (400, 500 and 600 °C). Light brown for reference samples (Ref 1) that burned at 500 °C, and milk color for reference samples (Ref 2) which burned at 1000 °C. These changing in color could be back to transformation phases of brick's materials with variation of temperatures.

4.2.3 Water Absorption

Results of water absorption with various NaOH concentration and burning temperatures for brick samples group 2, present in Fig. (4-1).

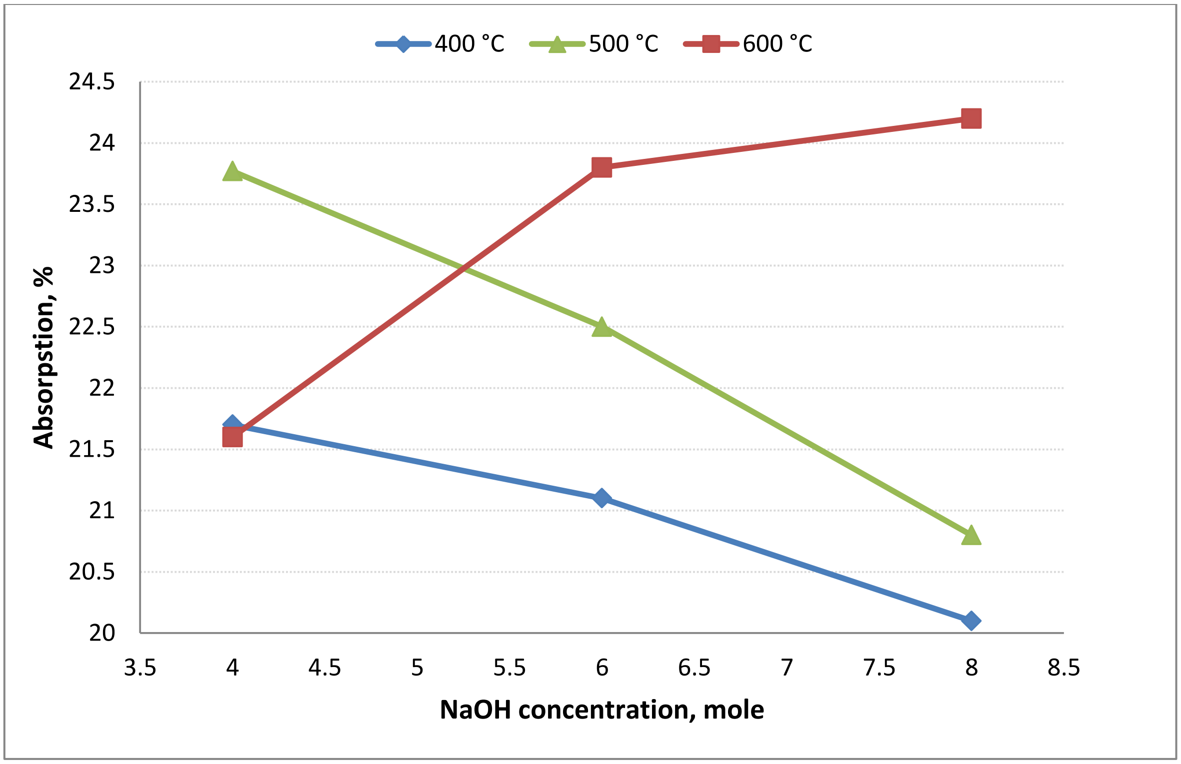


Figure (4-1): (Water absorption - NaOH concentration) relationship with different degree of burning temperatures for mixes group 2.

Fig.(4-1) above distinguishes that the raising in NaOH concentration solution reduces the water absorption of brick for each of 400 and 500 °C of burning, this agreed with Venugopal [21], counter to the samples which burned in 600 °C which exhibited increasing in water absorption with rising of NaOH concentration. In general, it is obvious an enhancing in the water absorption of bricks comparison with reference mix (Ref.1 and Ref.2) at all concentrations of NaOH and burning temperatures, by 32.83%, 26.54% and 23% for 400 °C with 8, 6 and 4 M respectively. also, by 28.36%, 18.66% and 12.65% for 500 °C with 8, 6 and 4 M respectively, and by 10.33%, 12.2% and 23.6% for 600 °C with 8, 6 and 4 M respectively. That is mean that the present of NaOH in bricks paste helped in fill pores, and when burning temperatures have increased (from 400 to 500 °C) this might led to evaporate part of this material and raised rate of water absorption. Moreover, an

increment of temperature to 600 °C drive out more amount of NaOH, which are commensurate with its concentration in existing originally brick and occupy space of within its total mass. So, the bricks which have high concentration of NaOH will contains more porosity for this degree (600 °C), subsequently increase its ability for water absorption.

4.2.4 Compressive strength

Results of compressive strength with variation of NaOH concentration and burning temperature for brick samples group 2 are represented in Fig. (4-2).

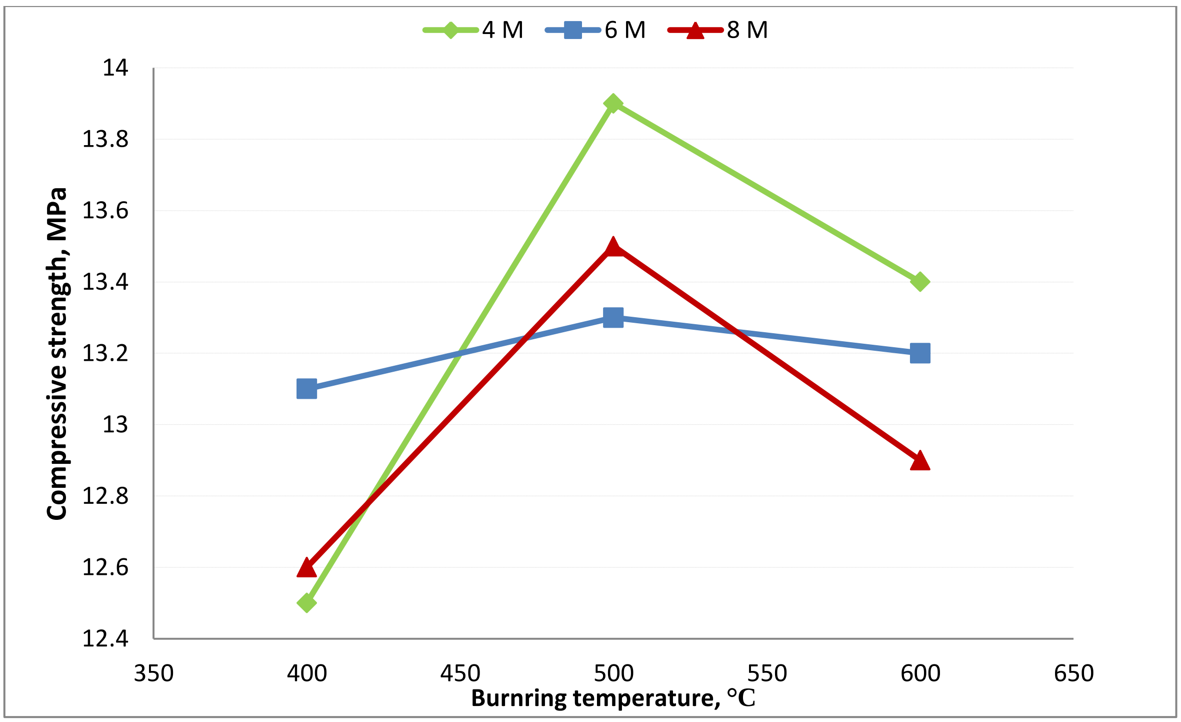


Figure (4-2): (compressive strength – firing temperature) relationship with different concentration of NaOH for mixes group 2.

Fig.(4-2) indicates that the compressive strength was directly proportional to burning temperature until 500 °C for two concentration 4 and 8 M of NaOH, that is agreed with Cordi-Géopolymère [44]. Where increasing

in burning temperature, compressive strength increase and get its maximum at 500 °C. This means raising in burning temperature will enhance the bonding molecular of brick, and overmuch effective of geopolymerization process. While any increase in temperature till 600 °C will give less rate of compressive strength. Whereas compressive strength for bricks have (6 M) of NaOH solution showed a few effected (± 0.75 %) about the average, this was with all burning temperatures (400,500 and 600) °C.

Moreover, the bricks with 4 M (NaOH) have largest compressive strength (13.9 MPa) compared with others molarity (6 and 8), which equal to 250%, in contrast with reference mix (Ref.1) and equivalent to 90% from compressive strength of (Ref.2), in addition their strength exceeded the others molarities at burning temperatures (500 and 600) °C. Nevertheless, when burned at 400 °C the compressive strength was the lowest value (12.5 MPa) compared with others. From the above we conclude the (4M) NaOH was the critical percentage of concentration in brick manufactures process, which affected steadily by burning temperature and more effective in gain compressive strength for bricks.

4.2.5 Water absorption and compressive strength for samples group 1

For brick samples group 1 results listed in (Table 4-1), we found that not all samples can perform (water absorption and compressive strength) test, because some of them did not stand up when immersion in water for 24 hours (according to test procedure) [41]. Either by cracking of faces, sides and edges or by crumbling its mass, and in some time both cases found (Plate 4-2).

Table (4-1): The results test for water absorption and compressive strength for brick samples group 1.

Mix. No.	(NaOH) concentration (M)	Curing temperature (°C)	Water absorption (%)	Compressive strength (MPa)
1	4	80	16.5	12.3
2	6	80	-	-
3	8	80	-	-
4	4	100	17.6	13.4
5	6	100	16.1	13.5
6	8	100	-	-
7	4	120	17.7	13.2
8	6	120	-	-
9	8	120	-	-

Plate (4-2): Cases of bricks sample group 1 after 24 hr immersion in water.

From results showed in Table (4-1) and Plate (4-2), it can conclude that four brick's mixes (No.1, 4, 5 and 7) only withstands when immersed in

water and can accomplish water absorption and compressive strength tests on them. When increases NaOH concentration above 4 M this will affect inversely on stability of brick against water exposure at all curing temperatures. Except of 100 °C, where expressed success in stand up by use of 6 M NaOH in brick mix. An addition the rising of curing temperature from 80 to 120 °C increased ability of water absorption of the bricks, and increased compressive strength up to (13.5 MPa) at 100°C, this consistent with Mei X. Peng(45), then decreased at 120 °C. That means 100 °C of curing is preferable temperature between others (80 and 120 °C), which facilitated more activity for geopolymerization process and maintain stability against water exposure.

4.2.6 Efflorescence

The results of efflorescence test for produced bricks with burning temperatures and different concentrations of (NaOH) for mixes group 2 listed in Table (4-2).

Table (4-2): Degree of efflorescence for brick samples group 2*.

NaOH concentration (M) / Burning temperature(°C)	4	6	8
400	Light	Moderate	Very intensive
500	Light	Moderate	Very intensive
600	Light	Moderate	Moderate

* Degree of efflorescence classified according to I.O.S no. (24) for 1988 (51).

From this results it is noted increased efflorescence amount, in general, by increasing (NaOH) concentration in sample, except that which

burned at 600 °C, where efflorescence amount still at same value (moderate) despite of changing NaOH concentrations (4,6 and 8 M). From that, it could conclude that the main factor that influenced on efflorescence is the NaOH concentration, in addition to raise burning temperature of the bricks. At 600 °C of burning, the efflorescence amount decreased for brick samples from (very intensive) to (moderate) for same concentrate of NaOH. The higher degree of burning helped to evaporation large amount of (NaOH) which originally exist in brick sample, subsequently minimized the remaining and deposited from it on brick in the form of efflorescence (plate 4-3).

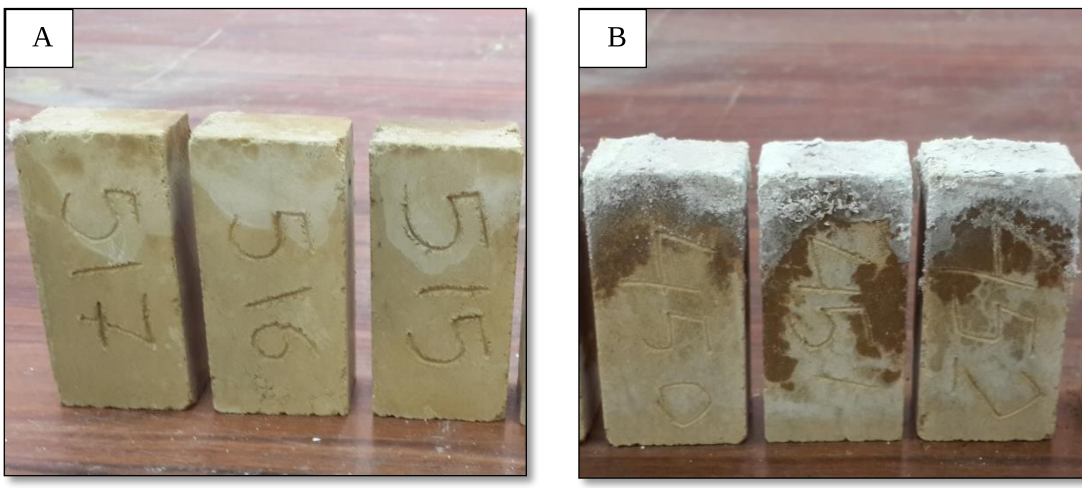


Plate (4-3): Degree of efflorescence for brick samples group 2. (A) Light (B) Moderate

On other side, the brick samples of group 1 didn't show any obvious result for degrees of efflorescence and exhibited shatter over upper part of sample when performed efflorescence test on them, as can be seen in (Plate 4-4).

Plate (4-4): Efflorescence test for brick samples group1.

It can conclude that the curing temperatures (80,100 and 120 °C) were not enough to product durable bricks against water exposure for long time (seven day or more, as efflorescence test procedure [41]). This may be a result of not fully completing or interaction between NaOH and alumni-silica materials (Attapulgite clay) at these degrees of temperature, counter to samples group 2.

4.2.7 Product's brick properties with I.O.S No.24 for 1988[41]

To classify produced bricks and know the conformity of their properties to specifications, compression with Iraqi standard for clay bricks [41] have been conducted (Table 4-3). However, due to the failure of samples bricks group 1 in efflorescence test, such compression for group 1 is excluded.

Table (4-3): Physical properties for brick samples group 2.

Description	NaOH concentration M	Compressive strength (MPa)	Water absorption (%)	Efflorescence
Mix no.10	4	12.5	21.7	Light
Mix no.11	6	**13.3**	**21.1**	**Moderate**
Mix no.12	8	12.6	20.1	Very
Mix no.13	4	**13.9**	**23.7**	**Light**
Mix no.14	6	**13.3**	**22.5**	**Moderate**
Mix no.15	8	13.5	20.8	Very
Mix no.16	4	**13.4**	**21.6**	**Light**
Mix no.17	6	**13.3**	**23.8**	**Moderate**
Mix no.18	8	12.9	24.2	Moderate
Limit Class B (clay brick), I.O.S No.24 for 1988[4]	-	≥ 13	≤ 24	Moderate

From the results under review in Table (4-3), five mixes of brick (mix No.11, 13, 14, 16, and 17) were responsive to requirements of class B clay bricks. In other words, all bricks were mixed with 4M of NaOH and burned at different temperature (400, 500 and 600 °C) were satisfied class B requirements. In addition, mix (No.14 and 17) which were mixed with 6M of NaOH and burned at (500 and 600°C) respectively, also achieves the mentioned requirements.

4.2.8 Longitudinal Shrinkage

The results of shrinkage for brick samples (group 1and group 2) represented in Figs. (4-3, and 4-4).

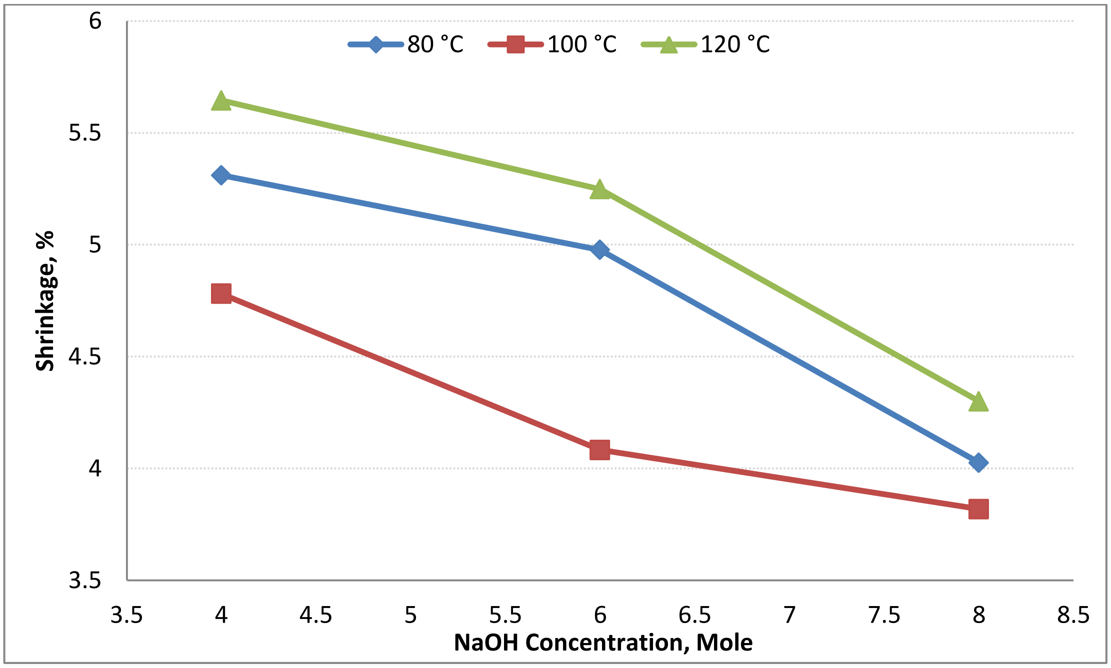


Figure (4-3): Longitudinal shrinkage for brick sample group 1.

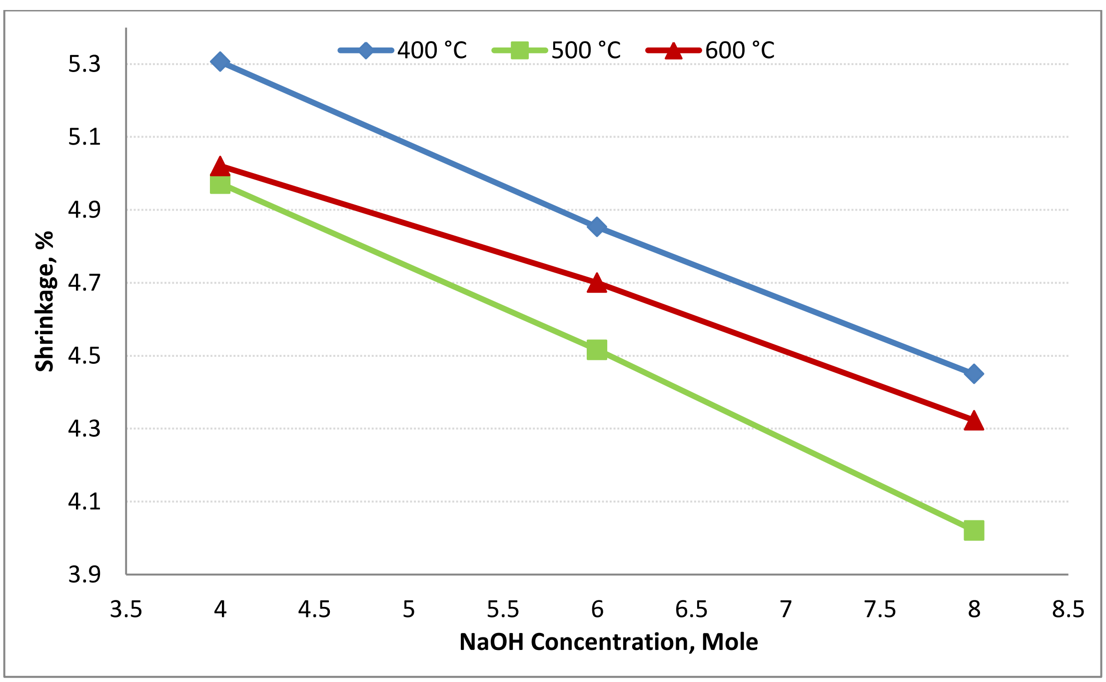


Figure (4-4): Longitudinal shrinkage for brick sample group 2.

From Figs (4-3) and (4-4), it can be noted that there is inversely relationship between shrinkage and NaOH concentration for all temperatures of (curing or burning). Where, increasing NaOH concentration caused decrease in shrinkage percentage. This is might be due to high NaOH percentage, lead to reduce the air pore in brick, which in its turn a result of evaporation of forming water by curing or burning temperatures, consequently, enhancing structural of sample. So, the raising of NaOH concentration in brick sample will restrict and reduce the shrinkage percentage.

On other hand, the increases of curing temperature lead to raise shrinkage percent regardless of NaOH concentration. Where, shrinkage of 120 °C more than 80 °C, that is a result of evaporating more amount of NaOH solution by ascent of temperature degree and leaves air pore, which helps to shrinkage. Whereas, (100 of curing and 500 of burning °C) have lowest shrinkage percent's and do not affect by change of temperature as other degrees that mentioned earlier. As a conclusion, the interaction at these degrees (100 and 500 °C) was completed and gave best results in term of shrinkage.

4.2.9 Bulk density

Obviously, from Fig. (4-5), there are direct correlations between density and NaOH concentration. Increase NaOH concentration leads to increase density, due to fill the air porosity with NaOH and makes the brick denser, that is result agree by Venugopal [21]. All values of density at 4M were less than 6 and 8 M. In addition, raise curing temperature helped in decreasing density with different degrees; where density of 120 °C was less than 80 °C,

and 100 °C have the lowest value of density. It can conclude that 100 °C was the preferable degree for curing to gives minimum density for bricks.

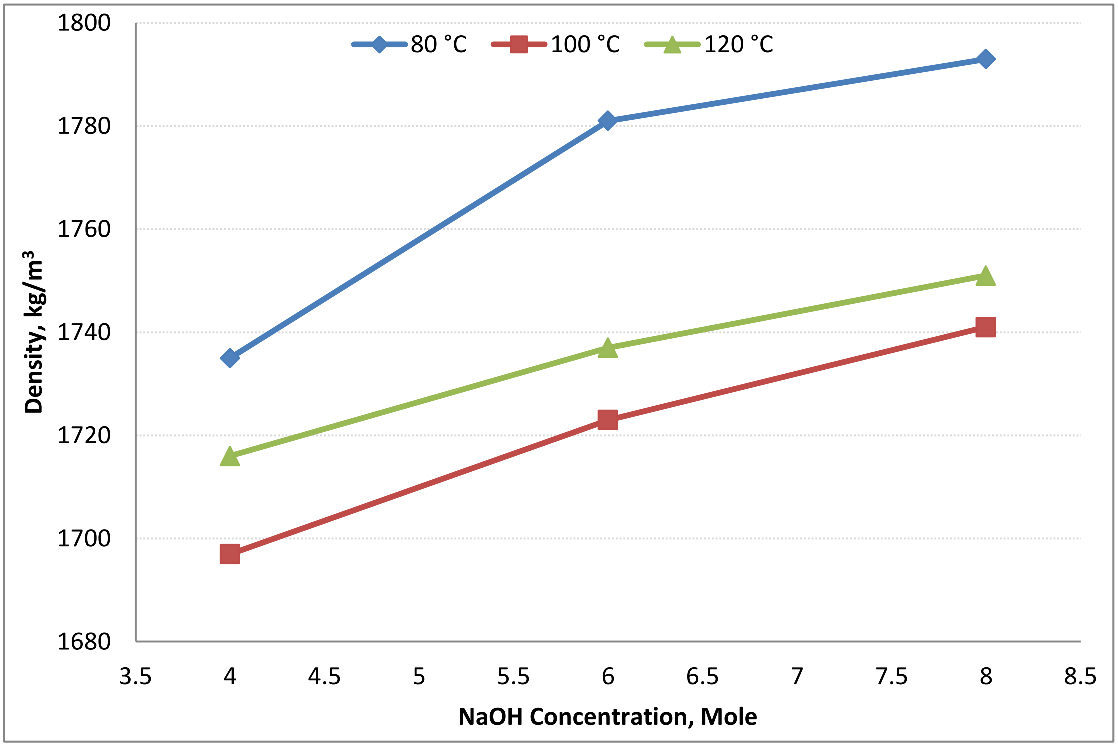


Figure (4-5): Relationship between density and NaOH concentration at different curing temperatures for brick samples group 1.

Also as in curing process at burning process, A direct correlation was found between density and NaOH concentration Fig. (4-6). In addition, increase NaOH concentration leads to increase density for same reasons. However, when bricks samples burned at 600 °C, the density have inversely relationship with concentration of NaOH. That mean, 600 °C was overmuch temperature for burn, which caused more evaporate amount of NaOH and left its space to fill with air, causing porosity in mass of bricks and reduce its density. Due to this relation of air pore with free spaces which originally occupied by NaOH, increase NaOH concentration in brick at this degree (600°C) of burning could make it less density.

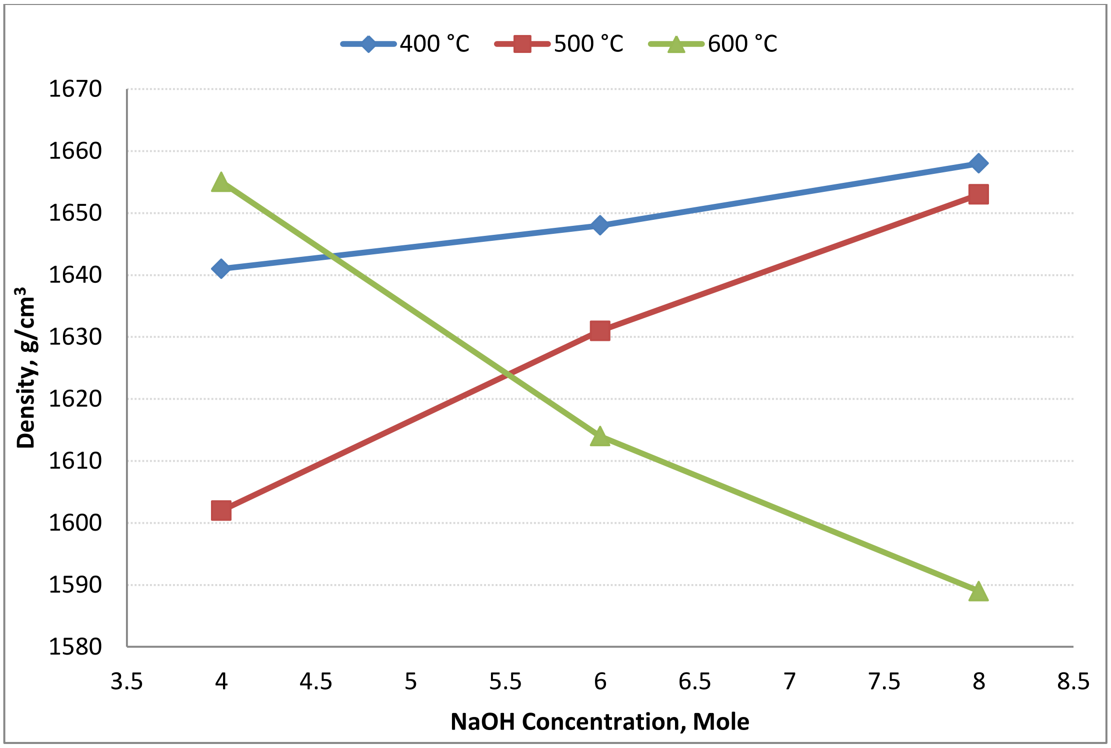


Figure (4-6): Relationship between density and NaOH concentration at different burning temperatures for brick samples group 2.

4.2.10 Shrinkage and density with previous research

Ahmed A. [30] studied the effect of replacement 30 % of Attapulgite with normal clay brick on its properties; including (longitudinally shrinkage and density). Its results with different degrees of burning listed in Table (4-4). From these results, it is obviously found an enhancement in shrinkage by about (41.5 %) between average shrinkage for present and previous research. Moreover, the densities results were convergent, despite of gap between burning temperatures, the average of density was (1600 kg/m^3) at 500 °C for present research and (1610 kg/m^3) at 900 °C for previous.

Table (4-4): Shrinkage and density with previous research.

Brick samples for previous research[13] with 30 % attapulgite			Brick samples for present research with 4 M of NaOH		
Burning temperature (°C)	Shrinkage (%)	Density, (kg/m^3)	Curing temperature (°C)	Shrinkage (%)	Density (kg/m^3)
750	8.84	1690	80	5.31	1730
800	8.84	1590	100	4.78	1690
850	8.84	1630	120	5.65	1710
900	8.85	1610	400	5.31	1640
950	8.85	1560	500	4.98	1600
1000	8.85	1550	600	5.02	1650

4.2.10 Modulus of rupture

By looking to the failure pattern of tested brick samples (Plate 4-5), it can distinguish the failure was happen due to cross breaking.

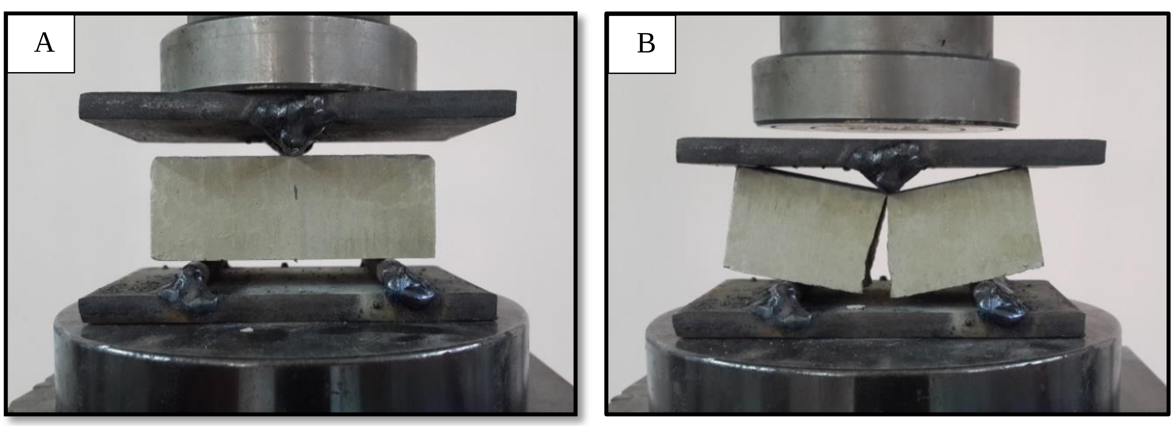


Plate (4-5): Failure pattern under modulus of rupture test, (A) before test (B) after test.

From the results in Fig.(4-7), which is obviously show the effect of NaOH solution in enhancing rupture strength for bricks on (Ref.1). It is found that the use of 6 and 4 M of NaOH increased modulus of rupture for bricks by 157.4% and 48.15% at 80 °C of curing temperature, respectively. Also, at 100 °C the increment in breaking 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 concentration 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.

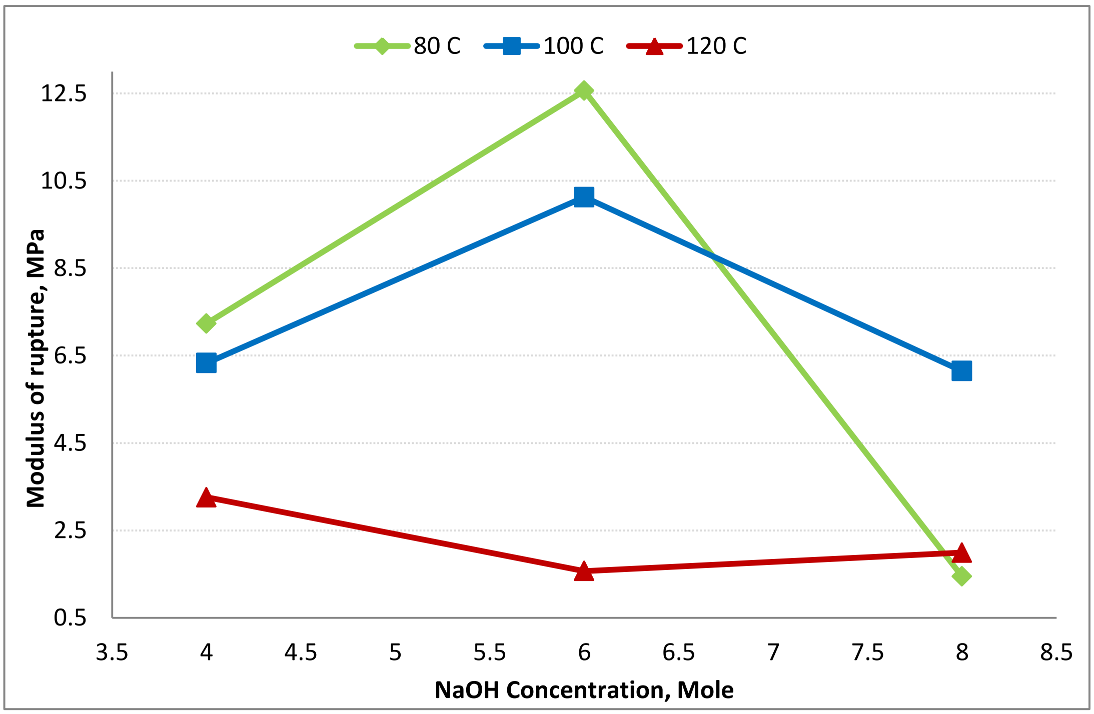


Figure (4-7): Relationship between modulus of rupture and NaOH concentration at different curing temperatures for brick samples group 1.

For brick sample's that subjected to burning, same behavior exhibited as in curing temperature, where, modulus of rupture decreased when raised burning temperatures Fig. (4-8). In addition, the samples which were burned at 400 °C have a manner in rupture strength similar to that for curing at (80

and 100 °C). Also, the burning at (500 and 600 °C) having consistent approach on modulus of rupture with variation of NaOH concentration, where the strength decreases with increase of concentration.

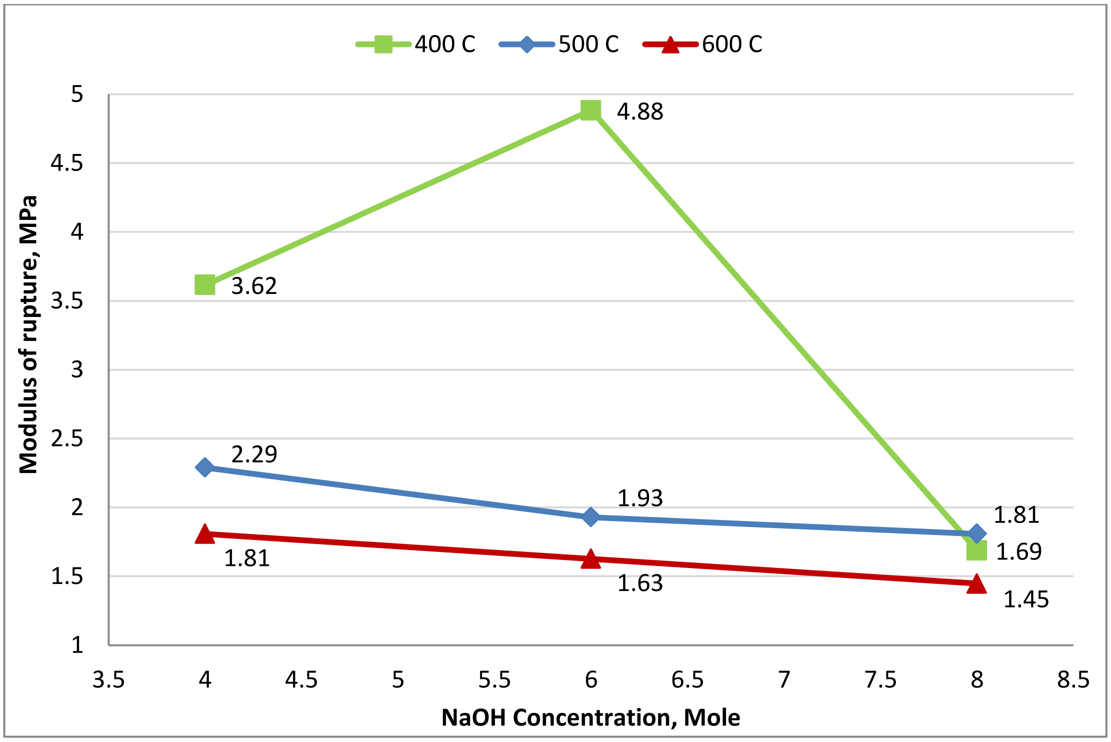


Figure (4-8): Relationship between modulus of rupture and NaOH concentration at different firing temperatures for brick samples group 2.

4.2.11 Reference mixes

To know effectiveness of NaOH and locally Attapulgite clay in geopolymerization process, and properties of produced brick a comparison is conducted with these which produced by normal method for clay bricks. Therefore, the tests results of reference mixes for (compressive strength, water absorption, efflorescence, shrinkage, density and modulus of rupture) are compared with produced geopolymer bricks properties, as listed in Table (4-5).

Table (4-5): Results test of reference mixes with group 1 and group2.

Mix No.	NaOH (M)	Burning or curing temperature (°C)	Compressive strength (MPa)	Absorption (%)	Efflorescence	Shrinkage (%)	Density (kg/m^3)	Flexural strength (MPa)
1	4	80	12.3	16.5	-	5.31	1735	7.23
2	6	80	-	-	-	4.977	1781	12.56
3	8	80	-	-	-	4.026	1793	1.44
4	4	100	13.4	17.6	-	4.781	1697	6.32
5	6	100	13.5	16.1	-	4.083	1723	10.12
6	8	100	-	-	-	3.818	1741	6.14
7	4	120	13.2	17.7	-	5.645	1716	3.25
8	6	120	-	-	-	5.248	1737	2.17
9	8	120	-	-	-	4.3	1731	1.808
10	4	400	12.5	21.7	Light	5.306	1641	3.61
11	6	400	13.3	21.1	Moderate	4.853	1648	4.88
12	8	400	12.6	20.1	Very intensive	3.846	1658	1.69
13	4	500	13.9	23.7	Light	4.972	1602	2.29
14	6	500	13.3	22.5	Moderate	4.516	1630	1.93
15	8	500	13.5	20.8	Very intensive	4.021	1653	1.8
16	4	600	13.4	21.6	Light	5.02	1655	1.8
17	6	600	13.3	23.8	Moderate	4.71	1614	1.63
18	8	600	12.9	24.2	Moderate	4.241	1589	1.44
Ref.1	-	500	3.9	26.7	Non	2.645	1523	1.26
Ref.2	-	1000	14.9	25.9	Non	11.797	1334	4.88

For all properties mentioned in Table (4-5), the produced bricks (geopolymer brick) express their superiority on reference mix (Ref.1). Except in effloresce property, where the reference mixes (Ref 1 and Ref 2) did not contain any effloresce, counter to other mixes (group1 and group 2), which their effloresce degree are ranged from light to very intensive. At the same time, the produced bricks expressed (with condition mix No.13) close properties to those of (Ref.2), which produced at twice times of burning temperature in contrast to geopolymer brick samples. Except in effloresce property, were reference mix (Ref.2) was best.

4.3 Microstructure properties

4.3.1 Scanning electron micrograph (SEM)

Plate (4-6) shows the electron images of the products reaction which obtained from the original (Attapulgite clay) and water; colloidal structural is noticed. Compared with (mix No.4) Plate (4-7), almost all the particles of this mix are irregular, with very few spherical glass particles (presumably because of the lower burning temperature (100 °C)). The main product of geopolymerization is an amorphous aluminosilicate (gel) phase, which is composed of ($Na_2Al_4Si_9O_{25}$). The spherical particles increased in form and tend to conglomerate with NaOH presence and raise the burning temperature; such reaction products can be found in Plates (4-7) and (4-8). Comparing these three micrographs, it is clearly noticed that the products which fired at 500 °C gave higher values of compressive strength (13.9 MPa) and showed much denser microstructures and more solidity and stability than the samples cured at 100 °C, in which little of unreacted particles were apparent, as can be seen in Plate (4-7).

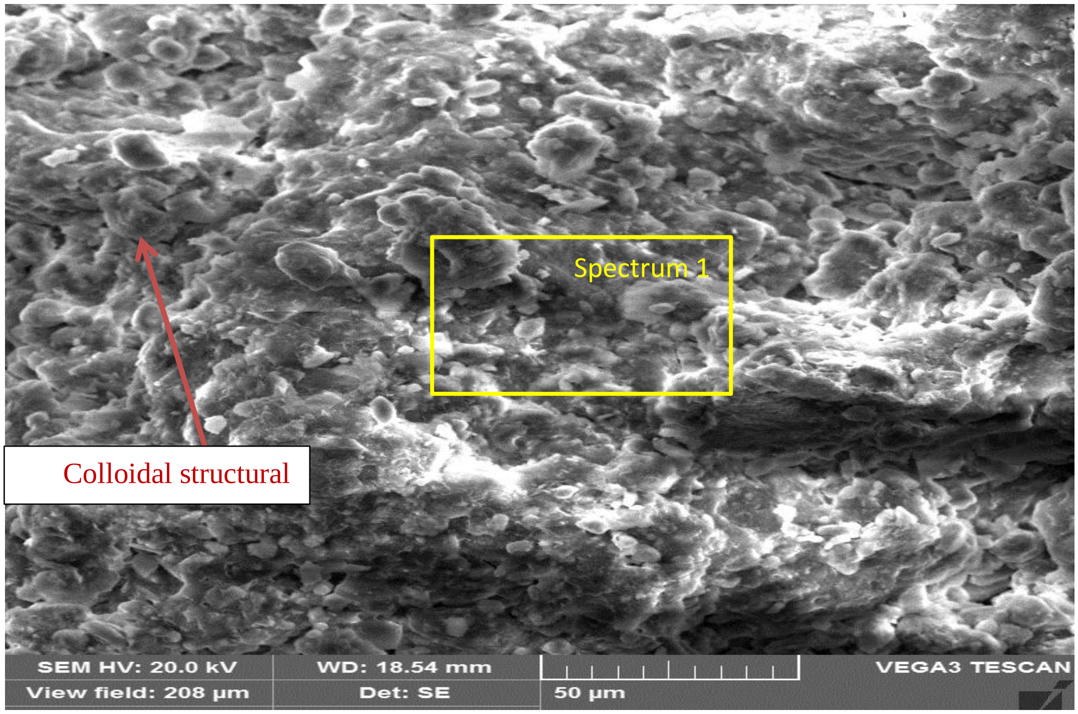


Plate (4-6) : SEM Image for (Ref 1) mixtures.

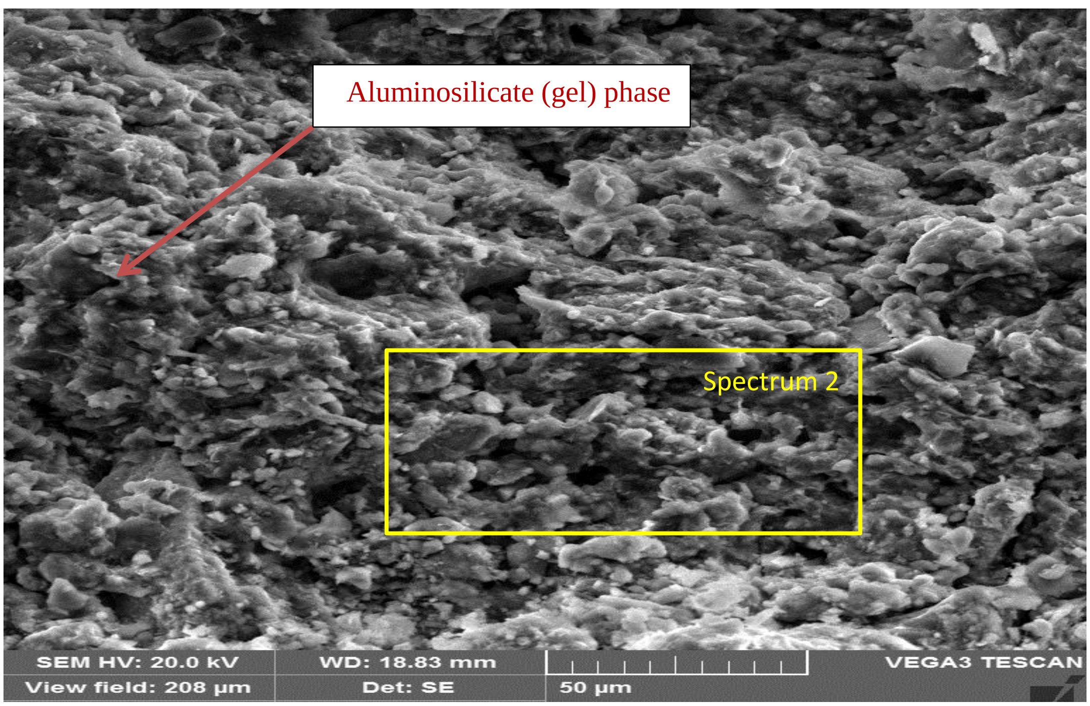


Plate (4-7) : SEM Image for (mix no.4).

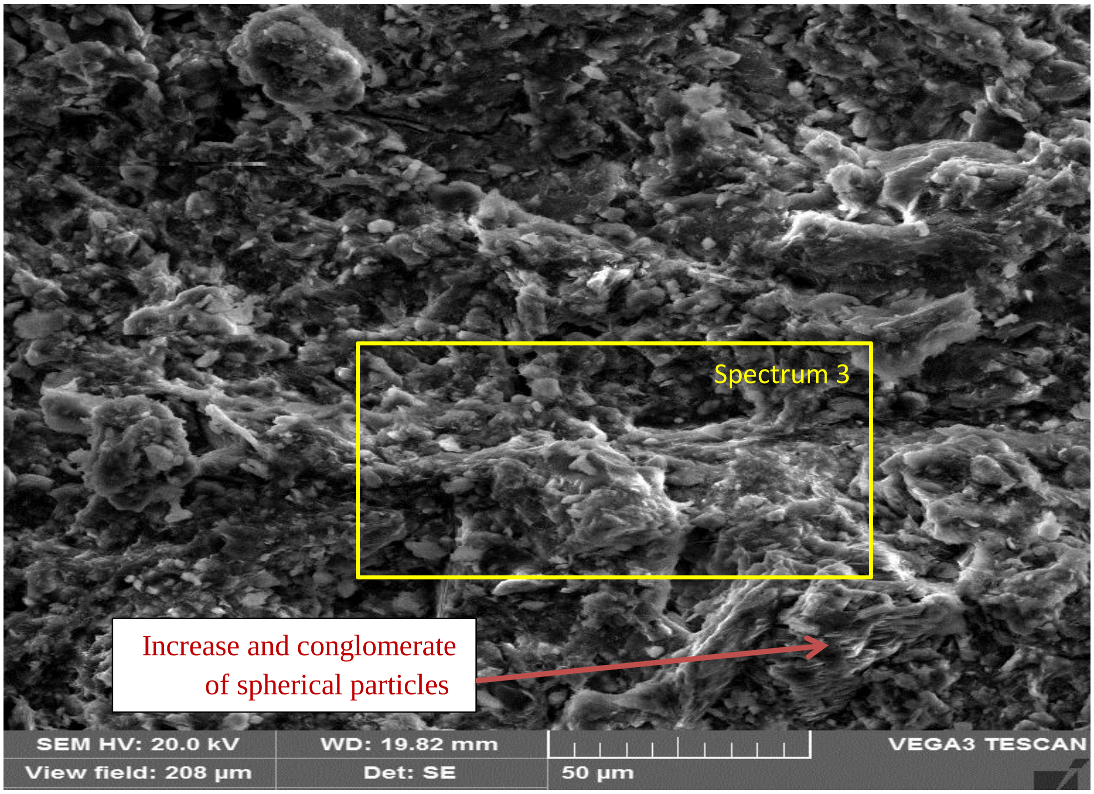


Plate (4-8) : SEM Image for (mix no.13).

4.3.2 Energy Dispersive Spectroscopy Systems (EDS)

The elemental distribution for the structures of crystalline acicular of NaOH phase's shows under EDS is presented in Figs. (4-9, 4-10, and 4-11). They show that the crystalline phases for acicular structure of Na cross-linked geopolymer are bear and element as (Al, Si and Na) which listed in Table (4-6) due to the geopolymer phase. The micrographs image confirmed that the interaction of Attapulgite in alkaline activator consisting of anions of Cl− and $SO_4^{=}$ form crystalline grains. These secondary grains to little amounts of (Mg, K, Fe, Ca, and Ti) are especially fibrous and elongated. These phases for minerals with variation of cation and anion exchange growing into amorphous to crystalline structures by disintegration and stabilization. Assortment of the polymerized minerals that helps in the reinforcement of the cementation property. Based on the strength of bonding for the geopolymerization process

which is elastic, make high strength building brick's that have compressive strength ranging from 11.2 to 13.9 MPa.

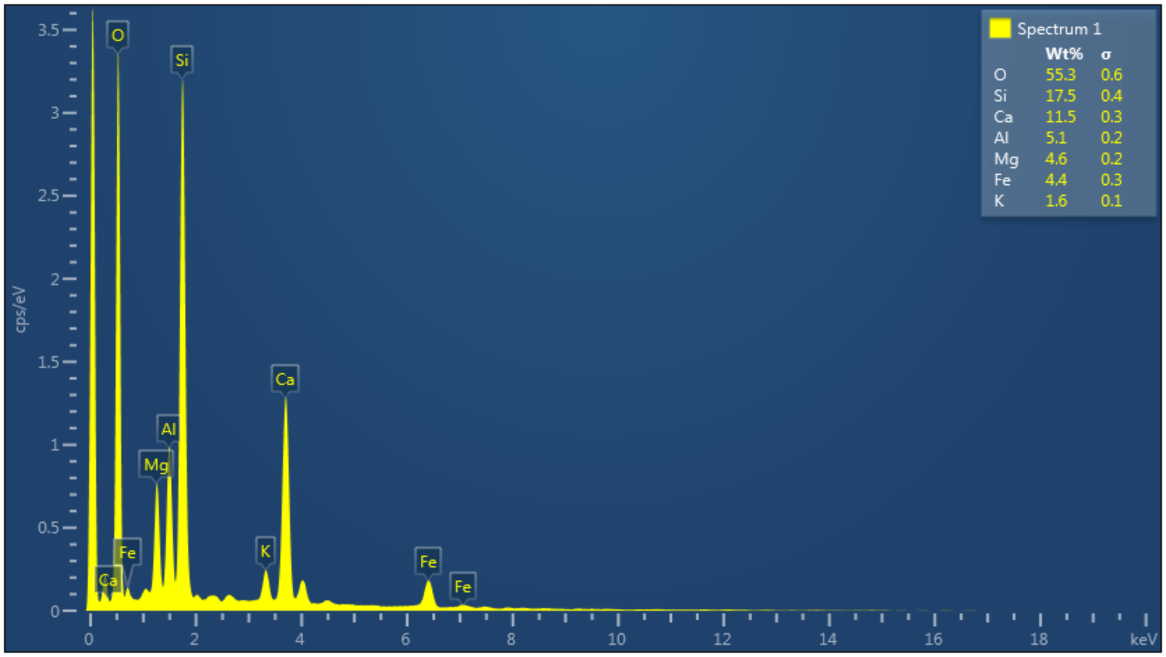


Figure (4-9) : EDS analysis showing elemental distribution of (Ref 1) mixtures.

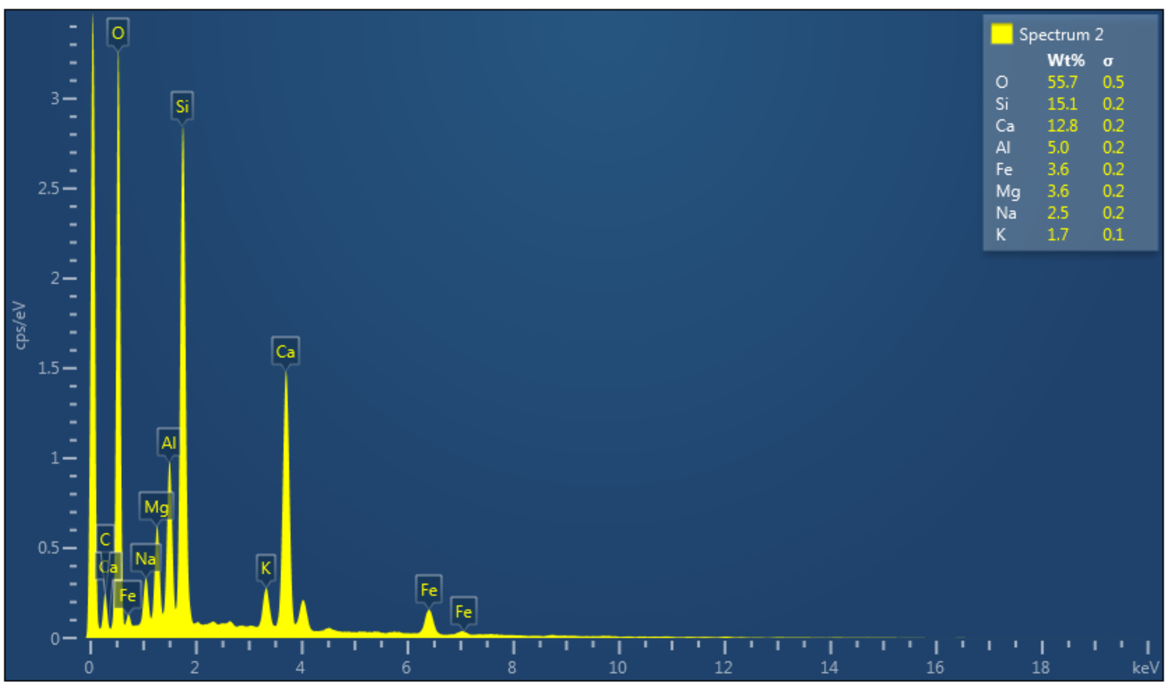


Figure (4-10) : EDS analysis showing elemental distribution for (mix no. 4).

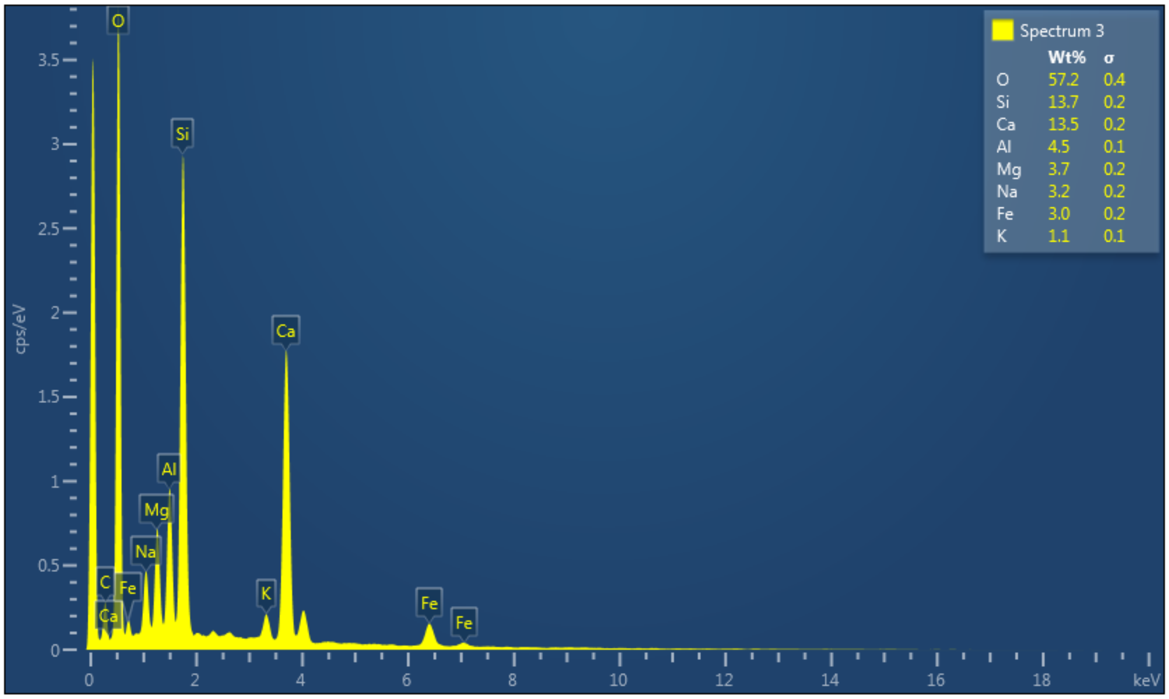


Figure (4-11) : EDS analysis showing elemental distribution for (mix no. 13).

Table (4-6): Elemental analysis for mix No.(4 and 13) and reference mix (Ref 1).

Element	Weight for (Ref. 1) (%)	Weight for mix (No. 4) (%)	Weight for mix (No. 13) (%)
O	55.3	55.7	57.2
Si	17.5	15.1	13.7
Ca	11.5	12.8	13.5
Al	5.1	5.0	4.5
Mg	4.6	3.6	3.7
Fe	4.4	3.6	3.2
K	1.6	2.5	3.0
Na	-	1.7	1.1

CHAPTER FIVE

Conclusions and recommendations

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Based on the experimental and analytical investigations of this research, the following concluding remarks are drawn:

1. It is possible to product geopolymer brick based on Iraqi Attapulgite clay at low burning temperature. Were the optimum mix was having the parameters (4 M of NaOH and 500 °C of burning temperature), which product bricks with properties conformity to class B clay bricks according I.O.S No. 24 for 1988.
2. The bricks which subject to curing temperature only (80, 100 and 120 °C), are not withstand to moisture and water exposure.
3. Higher concentration of (NaOH) solution at any term dose not responsible for higher compressive strength, and when using (8 M or above), the resulting alkaline solution will be danger and not safe on workers. In additions, the increase (NaOH) concentration will increases amount of effloresce on brick surface. From other hand, enhances water absorption property for bricks at any temperature of curing or burning.
4. Increasing burning temperature (until 500 °C) will enhancing stability of brick against moisture and water exposure, in addition increased strength property and reducing effloresce of geopolymer brick.
5. A 600 °C of burning did not gain increase in strength, on the contrary its raised rates of water absorption for bricks.
6. The compressive strength for optimum mix (13.9 MPa) exceeded 250 % compared with reference mix (Ref.1), whereas it has strength value

close to this of reference mix (Ref.2), which is (14.9 MPa) that burned at twice times firing temperature (i.e., 1000 °C).

7. The water absorption property for optimum mix brick's enhanced with percentage 23 % compared with reference mix (Ref.1).
8. Reducing about 50 % in consumption of fuel could achieve, when product geopolymer brick based on Iraqi Attapulgite instead of traditional clay bricks.
9. The bricks which subject to curing temperature only (80, 100 and 120 °C), are not withstand to moisture and water exposure.

5.2 Recommendations

Based on present research, the following suggestions are recommended for future researches:

1. Study uses another percentage of NaOH solution (less than 4 M).

2. Subject another temperature of firing which is between 120 to 400 °C.

3. Variation in press load value (use many press value above and below 13 kN) and investigate its effect on properties.

4. Use different type of alkaline solution (sodium silicate, Potassium Hydroxide).

5. Investigate the variation-n in curing period (3, 7 and 28 day).

6. Study the durability of this type of bricks.

7. Research about the possibility of using with real and ordinary brick dimensions (240 x 115 x 75 mm).

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Appendix A

Cost analysis

A simplified cost analysis based on Iraqi conditions, by ignoring the cost of the Attapulgite as considers available raw material, has been makes for a full-size brick as detailed below:

Mass of Attapulgite ground in each cycle in the ball mill = 10000 g

Time taken to grind 10000 g of Attapulgite = 120 min

Required size of Attapulgite clay needed = 250μm

Density of brick required = 1.9 g/cm3

Internal size of the standard mold: Length = 24 cm, width = 11.5 cm and height = 7.5 cm

Amount of the mixture of Attapulgite clay and sodium hydroxide required to produce one standard brick = $24 \times 11.5 \times 7.5 \times 1.9 = 3933$ g

The amount of the sodium hydroxide solution required to make one standard brick due to (solution/clay) ratio=0.25= $(25/100) \times 3933$ g = 983.25 g

The amount of solid flaks of (4 M) NaOH=130g

Amount of Attapulgite clay required to make one standard brick = 3933 g−983.25 g = 2949.75 g

Amount of Attapulgite clay coarser than 250μm available in the tailings = 13.5% of 2949.75 g = 0.13 x 2949.75 = 398.216 g

Amount of Attapulgite clay finer than 250μm obtained after grinding of the tailings = 2949.75 −398.216 = 2551.533 g

The milling time for producing one standard brick = (2551.533/10000) × 120 = 30.61 min or 0.51 h

The laboratory ball mill used has a power rating of 3 Amp. or 0.66 kW. The current electricity tariff in Iraq set as k1/k2 for industry uses is 155 (IQD) per unit (kW/h) [46]. Therefore, the cost of running the ball mill for the 0.51 h to make one standard brick = (0.66 × 0.51 × 155) = 52.173 IQD.

The hydroxide sodium solution was obtained from local market, and the price 25000 IQD per 25000 g. In this work, 25% of the activator amount used to make one brick from the Attapulgite clay. Therefore, the cost of the hydroxide sodium solution to make one standard brick = (130/1000) × 1000 =130 IQD

The power rating for the oven used is 5 Amp. or 1.0 kW. Therefore, the cost for running the oven for 1 h = 1 × 155 = 155 IQD

The oven has a capacity of 64000 cm3. Since one standard brick has a volume of 24 × 11.5 × 7.5 = 2070 cm3, the total number of bricks that the oven can take at the same time = (64000/2070) ≈ 30 bricks

The oven can take 30 bricks at a time at the same cost per hour. Thus, the cost of oven drying for one standard brick for 1 h = (1/30) × 155 = 5.16 IQD

Thus, the cost of oven drying for one standard brick for 3 h=3×5.16 = 15.5 IQD

From the above considerations, it can be estimated that the cost of producing one geopolymer brick from Attapulgite clay, with 3 hr of firing to obtain the strength of 13.9 MPa, is (52.173 + 130 + 15.5) = 197.673 IQD. While the cost of one Iraqi normal brick as handmade with the standard dimensions of (24 × 11. × 7.5 cm) is = 250 IQD (depends on its quality). The geopolymer brick will cost less by (250−197.673)/ 250 × 100 = 20.93%.

We must take in our mind this cost analysis was proximity and the cost of production one unit of geopolymer brick in reality much less than. Because the manufacture cost for any product in plant is different than laboratory. From the other hand, here calculated cost depends use electric power for firing of bricks, whereas in the practices side the oil fuel is used, and due to minimize firing temperature from 1000 °C to 500 °C that is lead to reduce time of firing and fuel used to about half and eventually production cost is reduce.

Appendix B

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Appendix C

وزارة التخطيط

الجهاز المركزي للتقييس والسيطرة النوعية

قسم الملكية الصناعية / براءات الاختراع والنماذج الصناعية

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عراقي

وصل استلام طلب براءة اختراع

رقم الطلب ٢٤٩/٢٠١٦

تاريخ الاستلام ٦/٦/٢٠١٦

اسم مقدم الطلب احمد طه سلمان

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عنوان الاختراع انتاج الطابوق الجيوبوليمري من مواد محلية

ع / مسجل براءة الاختراع والنماذج الصناعية

ملاحظة :

١ ـ عملا بأحكام الفقرة ٣ من المادة الثانية من نظام براءة الاختراع والنماذج الصناعية رقم ٢١ لسنة ١٩٧٠

٢ ـ لايعتبر هذا الوصل دلالة على قبول الطلب وصدور الشهادة.

Appendix D

- Technical specification for SEM apparatus [42].

Electron Optics	Electron Gun	Tungsten heated cathode /optionally LaB_6	
	Resolution	High Vacuum Mode (SE)	3 nm at 30 kV / 2 nm at 30 kV
			8 nm at 3 kV / 5 nm at 3 kV
		Low Vacuum Mode (BSE, LVSTD)	3.5 nm at 30 kV/2.5 nm at 30 kV
	Maximum Field of View	24 mm at WD 30 mm	
	Accelerating Voltage	200 V to 30 kV	
	Probe Current	1 pA to 2 µA	
Scanning	Scanning Speed	From 20 ns to 10 ms per pixel adjustable in steps or continuously	
Vacuum system	Gun Vacuum	for LaB6: < $3x10^{-5}$ Pa	
	Column Vacuum:	< $9x10^{-3}$ Pa	

الخلاصة

في هذه الدراسة تم بحث تصنيع طابوق طيني بعملية الجيوبوليمرايزيشن (geopolymerization). أعد خليط من تربة الاتابلكايت المحلية والمطحونة لنعومة 250 مايكرون مع محلول هيدروكسيد الصوديوم وبتراكيز مختلفة (4، 6 و8 مولاري). شكلت النماذج بأستخدام قالب حديدي وبتسليط قوة مقدارها (13-14) كيلو نيوتن. نماذج الطابوق اعدت على شكل مجموعتين، (المجموعة 1) تضمنت 270 نموذج والتي خضعت لعملية المعالجة عند درجات (80، 100 و120 درجة سيليزية) ولمدة 24 ساعة، بينما (المجموعة 2) احتوت ايضا 270 نموذج والتي خضعت لعمليات الحرق عند درجات (400، 500 و600 درجة سيليزية) ولمدة 3 ساعات. بالأضافة الى عمل خلطتين مرجعيتين من 60 نموذج ونسبة(0 %) من محلول NaOH ، حيث سميت بـ(الخلطة المرجعية Ref.1) والتي حرقت عند درجة الحرارة المثلى(500 درجة سيليزية) والمستحصلة من الدراسة، في حين (الخلطة المرجعية Ref.2) حرقت عند درجة الحرق الاعتيادية لأنتاج الطابوق الطيني والتي هي 1000 درجة سيليزية.

أجريت فحوصات (الشكل والابعاد ، مقاومة الانضغاط ، أمتصاص الماء و التزهر) طبقا للمواصفة القياسية العراقية رقم 24 لسنة 1988 و فحوصات (الانكماش الطولي ، الكثافة الكلية ومقاومة الكسر) طبقا للمواصفة البريطانية ، بالاضافة الى اجراء فحص البنية الهيكلية (SEM و EDS) لنماذج طابوق الخلطات المثلى والخلطة المرجعية (Ref.1).

استنتج من هذه الدراسة بأنه افضل نسبة من NaOH كانت 4 مولاري و 500 درجة سيليزية هي درجة الحرارة المفضلة ، حيث ان الطابوق المستحصل وفق هذه المتغيرات اظهر مقاومة انضغاط 13.9 ميكاباسكال ، والتي تزيد على الضعف من مقاومة انضغاط الخلطة المرجعية (Ref.1) والتي تساوي 3.9 ميكاباسكال. هذه المتغيرات أعطت طابوق بخصائص هندسية جيدة وضمن متطلبات (صنف ب) من المواصفة القياسية العراقية لطابوق البناء الطيني.

التحسينات بلغت 254 % ، 12.66 % و 80.95 % لمقاومة الانضغاط ، أمتصاص الماء و مقاومة الكسر، وعلى التوالي. التقليل بدرجات حرارة الحرق وصل حوالي 50 % ، حيث ان

مقاومة انضغاط الطابوق المنتج عند 500 درجة سيليزية كانت مكافئة الى 90 % من مقاومة أنضغاط الخلطة المرجعية(Ref.2) والمحروقة عند 1000 درجة سيليزية.

جمهورية العراق

وزارة التعليم العالي والبحث العلمي

الجامعة التكنولوجية

قسم هندسة البناء والانشاءات

أنتاج وخصائص طابوق الاتبلكايت الجيوبوليمري

رسالة مقدمة الى

قسم هندسة البناء والانشاءات في الجامعة التكنولوجية وهي جزء من متطلبات نيل درجة الماجستير في علوم هندسة البناء والانشاءات بتخصص مواد البناء

من قبل

أحمد طه سلمان

(بكالوريوس في الهندسة المدنية 2012)

بإشراف

الاستاذ المساعد الدكتور وليد عبد الرزاق عباس القيسي

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