

The Effect of Alkaline Additives on the Operating Conditions of Kaolinitic Polymerization

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Abstract

Jordanian kaolinite polymerization has been studied to determine the effect of using different alkaline materials such as NaOH, KOH, and Ca(OH)₂, and sand as a filler on the compressive strength, durability under wet and dry conditions, and water absorption of the produced specimens. The studied parameters were mixing proportions such as; alkaline materials to kaolinite ratios, filler to kaolinite ratios, water to kaolinite ratios, and filler particles size. The polymerization step was carried out by mixing the kaolinitic and the filler with the alkaline solution in the case of NaOH and KOH before molding the specimens. In the case of Ca(OH)₂, the kaolinitic, fillers, and powder of Ca(OH)₂ were dry mixed together before water is added. The moulding step was carried out using a stainless steel mould at a pressure of about 15 MPa, the curing temperature was 80°C. Twenty four hours was the curing time for specimens containing NaOH or KOH, while 7 days was the curing time for Ca(OH)₂ specimens. The obtained results indicate that using NaOH as an alkaline material has the best compressive strength of 45.45 MPa under dry condition, 20.38 MPa under wet condition, and the lowest water absorption of 9.41 %. The composition of the specimens with optimum values consist of 42.37 % kaolinite, 5.93 % NaOH, 42.37 % sand, and 9.32 % water, with sand particles size of (180-355) µm. The specimens of NaOH and sand as a filler showed highest durability under dry cycling condition compared with KOH specimens. While the specimens of KOH and sand as a filler showed highest durability under immersion, and wet cycling condition.

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Keywords: Kaolinite; geopolymerization; alkaline; durability; compressive strength

1. Introduction

One of the basic demands for development of any society is the availability of construction materials. These materials are mainly obtained from cement industry. Cement manufacturing is one of the largest carbon emitting industrial sectors with carbon dioxide emitted as a result of both fuel combustion and process-related emissions. The production of one tone of cement generates 0.55 tons of chemical CO₂ and 0.40 tons as combustion product [1], also consumes high energy. High cost of energy and transportation has made it an expensive industry. Therefore it becomes necessary to look for other sources of construction materials that consider both economical and environmental factors. These new materials should be characterized by low production cost, low energy consumption, and low CO₂ emission. Soil is the most common building material world-wide, because it is available almost everywhere on earth. The use of soil as a construction material is a need for improvement to achieve stable, reliable, and high quality materials.

Mineral polymerization techniques offer possibilities for soil improvement. It refers to reactions between alkalis and minerals such as kaolinite which will be hardened and transformed into useful construction materials. Kaolinite is the main clay minerals that have been widely used as a raw material in several industrial sectors such as paper,

ceramics, paint and cement. The Natural Resources Authority (NRA) of Jordan has published a geological survey indicating the availability of kaolinite in commercial amounts so that it can become a main economic resource to the industrial sector [2]. This encourages researchers to study the use of the Jordanian kaolin as a raw material to produce mineral polymers that are suitable for use as construction materials.

The objectives of this work described here are to evaluate the suitability of using different alkaline additives such as NaOH, KOH and Ca(OH)₂ with silica sand as a filler in Jordanian kaolinite polymerization to produce mineral polymers that are suitable for use as construction materials, and to determine some physical and mechanical properties of the produced specimens with respect to filler particles size and mix proportions, such as alkaline material to kaolinite ratio, filler to kaolinite ratio, and water to kaolinite ratio. The property determination will cover compressive strength, durability under wet and dry conditions and water absorption degree.

2. Theoretical background

Earth soil is the oldest and most widely used building material in the world, because it is cheap and abundant. The use of soil as a constructional material is limited by low strength, volume stability and durability, so there is a need for soil improvement. Alkaline addition is a

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chemical technique for the soil improvement refers to the addition of alkali hydroxide or silicate to the soil. Davidovits [3] defined the chemical reactions of aluminate and silicate species that originate from the dissolution of silicon and aluminum containing materials under highly alkaline conditions as geopolymerization process that yield geopolymer which is an amorphous to semi-crystalline three dimensional alumino-silicate polymer produced at temperature lower than 100°C.

Various sources of silicon and aluminum are used as raw material for geopolymerization process such as kaolinite and bentonite. The common activators are NaOH, Na₂SO₄, Na₂CO₃, K₂CO₃, KOH, and K₂SO₄ [4]. Xu and Deventer [5] used fifteen natural Al-Si materials in geopolymerization with sodium and potassium hydroxide, the potassium hydroxide based geopolymer showed highest strength. Borbosa et al. [6] synthesized geopolymer of metakaolinite and sodium silicate with a maximum compressive strength of 48.1 MPa. Cioffi et al. [7] reported that geopolymers of kaoline with potassium silicate have better strength than geopolymer of kaolin with sodium silicate. Wang et al. [8] concluded that strength of metakaolinite and sodium hydroxide geopolymer increased with increasing of sodium hydroxide content.

Gogo [9] has evaluated the stabilization of soil from Ghana for building and construction purposes by mixing and curing the soil with various additives; Na₂CO₃, KOH, NaOH, Ca(OH)₂ and a mixture of K₂CO₃ and Ca(OH)₂. Soil stabilized with the K₂CO₃ and Ca(OH)₂ system as a solid additive have shown the best resistance to failure in the dry state while the NaOH system has shown the best resistance to failure in the wet state. A study by Al shaaer [10] evaluated the quality and suitability of different kaolinitic soils from Jordan for construction purposes using inorganic polymerization technique. The results showed that red brownish portion of Jordanian soil produce stable and good quality construction materials with compressive strength of 22 MPa and 50 MPa under wet and dry conditions, while gray portion of Jordanian soil has low quality to produce construction materials since it could not attain an acceptable compressive strength after immersion or cycling process. Linh [11] concluded that kaolinitic soil from Vietnam can be stabilized successfully with NaOH and sand to form strong and durable construction material. Fai [12] studied the possibilities of using the inorganic polymerization technique for the kaolinitic soils from Cameroon with NaOH for the aim of making them more suitable for construction purposes.

Esaifan [13] study was to evaluate the quality and suitability of kaolinitic soils from the southern Jordan desert to use it in the future work as a raw material for build up an in-situ water harvesting system after applying the technology of inorganic polymerization technique using NaOH. Khoury et al. [14] studied the production of a new construction material using kaolinitic clay, sand, sodium, potassium and calcium hydroxide by mineral polymerization.

3. Experimental

3.1. Materials:

In the present study Jordanian Hiswa kaolinite, supplied by the Public Mining Company (Jordan), was used as a raw material. It has a particle size less than 425 µm and a density of 2.8 g/cm³. Analytical grade of sodium hydroxide NaOH was used as an alkaline additive in the form of pellets, it was obtained from Merck with ≥ 99 % purity and a density of 2.1 g/cm³. Analytical grade of potassium hydroxide KOH was used as an alkaline additive in the form of pellets, it was obtained from Scharlau with ≥ 85 % purity and a density of 2.0 g/cm³. Synthesis grade of calcium hydroxide Ca(OH)₂ was used as an alkaline additive in the form of powder, it was obtained from Scharlau with 90 % purity and a density of 2.2 g/cm³. Jordanian silica sand, supplied by the International Silica Industries Company (Jordan), was used as a filler, with particle size of 45 <d ≤ 500 µm and a density of 2.1 g/cm³. Distilled water was used in the preparation of sodium, potassium hydroxide solutions or was added to calcium hydroxide-kaolinite-silica sand mixture.

3.2. Determination of materials properties:

3.2.1. Plastic limit determination:

The consistency of kaolinite depends on the water content. Using water content in the mixing process equals to the plastic limit was proposed to improve the quality of produced polymer. Plastic limit is defined as the water content below which the soil is nonplastic [15]. The plastic limit was determined for kaolinite sample after dried at 105 °C for 24 h to remove all moisture content using Shel Lab oven (model CE3). The kaolinite sample is mixed with water, then the mixture was divided into several smaller samples. These smaller samples were rolled between the fingers into a thread with a diameter of about 3 mm without breaking or sticking to the fingers until its water content is reduced to a point at which the thread just crumbles and is no longer is able to be rerolled. The wet threads were put into a weighed can and the weight of wet threads plus the can was recorded. Using KERN EG/EW electronic balance (model 3000-2M) the weights of the threads before and after drying at 105 °C for 24 hr were recorded, the difference between the two weights is the plastic limit. A value of 21.9 ml of water was obtained for the kaolinite sample used in the present work.

3.2.2. Chemical and mineral analyses:

X-Ray Fluorescence (XRF) analysis was performed to identify the chemical composition of kaolinite, while X-Ray diffraction (XRD) analysis was performed to identify the major mineralogical components of samples. The XRF and XRD analyses were carried out at the Natural Resources Authority laboratories using X ray fluorescence spectrometer, and Philips X ray diffractometer (model PW 3040 with Co K_α radiation) respectively. Table 1 shows the chemical composition of the kaolinite used in the present study that was identified using XRF analysis.

Table 1: Chemical composition of the raw materials used in the present study.

Material	SiO ₂ %	TiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	MnO %	MgO %	CaO %	Na ₂ O %	K ₂ O %	P ₂ O ₅ %	SO ₃ %	L.O.I %
Kaolinite	48.94	0.86	25.17	7.53	0.01	0.21	0.68	0.21	1.37	0.15	2.94	11.89

The XRD diffractions of materials were obtained as graphical output in the form of (2θ) angle diffraction within 2 to 60. These data were analyzed for the reflection angle and was compared to the tables of interplanar spacing (d-value) to identify possible peaks. Figure 1 shows an X-ray diffraction of a representative sample of kaolinite. The most abundant minerals are kaolinite and quartz, there are trace amounts of muscovite and hematite.

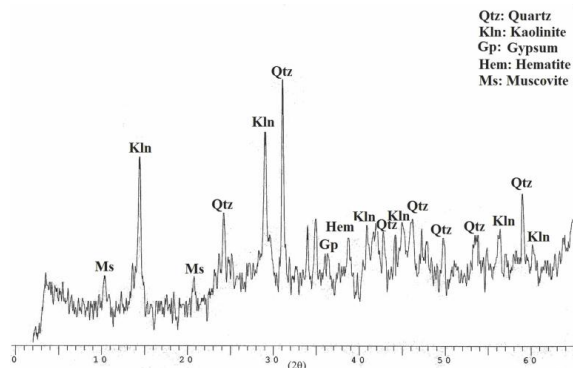


Figure 1: X-ray diffraction of kaolinite.

3.3. Polymerization of kaolinite:

3.3.1. Polymerization mixtures:

Throughout this study the components of a mixture were prepared by considering the kaolinite ratio to be 1, while the ratios of the other components are presented with respect to this ratio. For example, using silica sand as twice as kaolinite by weight, then we consider the silica sand to kaolinite ratio to be 2.

3.3.1.1. Polymerization of kaolinite using NaOH and silica sand.

Fourteen batches of kaolinite, NaOH, silica sand and distilled water were prepared. Eleven batches NaOH1 to NaOH11 (Table 2) were prepared with silica sand size range of (45 < d ≤ 500) μm. Three batches of (NaOH4) (Table 2) were prepared by using the silica sand size range as (45 < d ≤ 180), (180 < d ≤ 355) and (355 < d ≤ 500) μm.

Table 2: Ratio, mass fraction and weight of batch components for kaolinite polymerization with NaOH and silica sand.

Batch Name	Kaolinite			NaOH			Silica Sand				Water		
	R _i	X _i	W _i (g)	R _i	X _i	W _i (g)	R _i	X _i	W _i (g)	Particles size	R _i	X _i	W _i (g)
NaOH 1	1	41.67	250	0.18	7.5	45	1	41.67	250	45 < d ≤ 500	0.22	9.17	55
NaOH 2	1	42.02	252.1	0.16	6.72	40.34	1	42.02	252.1	45 < d ≤ 500	0.22	9.24	55.46
NaOH 3	1	42.19	253.16	0.15	6.33	37.97	1	42.19	253.16	45 < d ≤ 500	0.22	9.28	55.7
NaOH 4	1	42.37	254.24	0.14	5.93	35.59	1	42.37	254.24	45 < d ≤ 500	0.22	9.32	55.93
NaOH 5	1	42.74	256.41	0.12	5.13	30.77	1	42.74	256.41	45 < d ≤ 500	0.22	9.4	56.41
NaOH 6	1	43.1	258.62	0.1	4.31	25.86	1	43.1	258.62	45 < d ≤ 500	0.22	9.48	56.9
NaOH 7	1	34.97	209.79	0.14	4.9	29.37	1.5	52.45	314.69	45 < d ≤ 500	0.22	7.69	46.15
NaOH 8	1	38.31	229.89	0.14	5.36	32.18	1.25	47.89	287.36	45 < d ≤ 500	0.22	8.43	50.57
NaOH 9	1	47.39	284.36	0.14	6.64	39.81	0.75	35.55	213.27	45 < d ≤ 500	0.22	10.43	62.56
NaOH 10	1	42.92	257.51	0.14	6.01	36.05	1	42.92	257.51	45 < d ≤ 500	0.19	8.15	48.93
NaOH 11	1	41.84	251.05	0.14	5.86	35.15	1	41.84	251.05	45 < d ≤ 500	0.25	10.46	62.76
NaOH4-1	1	42.37	254.24	0.14	5.93	35.59	1	42.37	254.24	45 < d ≤ 180	0.22	9.32	55.93
NaOH4-2	1	42.37	254.24	0.14	5.93	35.59	1	42.37	254.24	180 < d ≤ 355	0.22	9.32	55.93
NaOH4-3	1	42.37	254.24	0.14	5.93	35.59	1	42.37	254.24	355 < d ≤ 500	0.22	9.32	55.93

3.3.1.2. Polymerization of kaolinite using KOH and silica sand:

Twelve batches of kaolinite, KOH, silica sand and distilled water were prepared. Ten batches KOH1 to

KOH10 (Table 3) were prepared with silica sand size range of (45 < d ≤ 500) μm. Three batches of (KOH2) (Table 3) were prepared by using the silica sand size range as (45 < d ≤ 180), (180 < d ≤ 355) and (355 < d ≤ 500) μm.

Table 3: Ratio, mass fraction and weight of batch components for kaolinite polymerization with KOH and silica sand.

Batch Name	Kaolinite			KOH			Silica Sand				Water		
	R _i	X _i	W _i (g)	R _i	X _i	W _i (g)	R _i	X _i	W _i (g)	Particles size	R _i	X _i	W _i (g)
KOH ₁	1	40.49	242.91	0.25	10.12	60.73	1	40.49	242.91	45 < d ≤ 500	0.22	8.91	53.44
KOH ₂	1	41.67	250	0.18	7.5	45	1	41.67	250	45 < d ≤ 500	0.22	9.17	55
KOH ₃	1	42.02	252.1	0.16	6.72	40.34	1	42.02	252.1	45 < d ≤ 500	0.22	9.24	55.46
KOH ₄	1	42.37	254.24	0.14	5.93	35.59	1	42.37	254.24	45 < d ≤ 500	0.22	9.32	55.93
KOH ₅	1	42.74	256.41	0.12	5.13	30.77	1	42.74	256.41	45 < d ≤ 500	0.22	9.4	56.41
KOH ₆	1	34.48	206.9	0.18	6.21	37.24	1.5	51.72	310.34	45 < d ≤ 500	0.22	7.59	45.52
KOH ₇	1	37.74	226.42	0.18	6.79	40.75	1.25	47.17	283.02	45 < d ≤ 500	0.22	8.3	49.81
KOH ₈	1	46.51	279.07	0.18	8.37	50.23	0.75	34.88	209.3	45 < d ≤ 500	0.22	10.23	61.4
KOH ₉	1	42.19	253.16	0.18	7.59	45.57	1	42.19	253.16	45 < d ≤ 500	0.19	8.02	48.1
KOH ₁₀	1	41.15	246.91	0.18	7.41	44.44	1	41.15	246.91	45 < d ≤ 500	0.25	10.29	61.73
KOH ₂	1	41.67	250	0.18	7.5	45	1	41.67	250	45 < d ≤ 180	0.22	9.17	55
KOH ₂	1	41.67	250	0.18	7.5	45	1	41.67	250	180 < d ≤ 355	0.22	9.17	55
KOH ₂	1	41.67	250	0.18	7.5	45	1	41.67	250	355 < d ≤ 500	0.22	9.17	55

Ca(OH)₂5 (Table 4) were prepared with silica sand size range of (180 < d ≤ 355) μm.

3.3.1.3. Polymerization of kaolinite using Ca(OH)₂ and silica sand:

Five batches of kaolinite, Ca(OH)₂, silica sand and distilled water were prepared. These batches Ca(OH)₂1 to

Table 4: Ratio, mass fraction and weight of batch components for kaolinite polymerization with Ca(OH)₂ and silica sand.

Batch Name	Kaolinite			Ca(OH) ₂			Sand				Water		
	R _i	X _i	W _i (g)	R _i	X _i	W _i (g)	R _i	X _i	W _i (g)	Particles size	R _i	X _i	W _i (g)
Ca(OH) ₂ 1	1	40.49	242.91	0.25	10.12	60.73	1	40.49	242.91	180 < d ≤ 355	0.22	8.91	53.44
Ca(OH) ₂ 2	1	42.37	254.24	0.14	5.93	35.59	1	42.37	254.24	180 < d ≤ 355	0.22	9.32	55.93
Ca(OH) ₂ 3	1	43.1	258.62	0.1	4.31	25.86	1	43.1	258.62	180 < d ≤ 355	0.22	9.48	56.9
Ca(OH) ₂ 4	1	43.86	263.16	0.06	2.63	15.79	1	43.86	263.16	180 < d ≤ 355	0.22	9.65	57.89
Ca(OH) ₂ 5	1	44.64	267.86	0.02	0.89	5.36	1	44.64	267.86	180 < d ≤ 355	0.22	9.82	58.93

3.3.2. Specimens Preparation:

3.3.2.1. Components Preparation:

The components of each mixture; the kaolinite, alkaline additives, silica sand and distilled water were accurately weighed. Several batches of specimens were prepared with different materials and mass fractions with a total weight of 600 g. The mass fraction of each component was calculated according to the following equation:

$$x_i = \left(\frac{R_i}{\sum_{i=1}^n R_i} \right) * 100\% \quad (1)$$

Where x_i is the mass fraction of component i, R_i is the ratio of component i to kaolinite and n is the number of components.

The weight of each component was calculated according to the following equation:

$$W_i = \left(\frac{x_i}{100} \right) * 600 \quad (2)$$

Where W_i is the weight of component i (g) and x_i is the mass fraction of component i.

3.3.2.2. Solutions preparation:

Sodium and potassium hydroxide solutions were prepared by adding a weighed amount of distilled water to a weighed amount of hydroxide pellets in plastic bottles; the bottles were closed to avoid evaporation then were shaken. As these solutions become hot, they were placed in a water bath to allow the solution cool down.

3.3.2.3. Constituents mixing:

Mixing of the weighed kaolinite, silica sand, alkaline and distilled water was done using a Kitchen Aid Artisan mixer (model 5KSM150PS). Two methods were used to carry on the mixing step; liquid method where kaolinite and silica sand were mixed at speed 1 for 2 minute then solutions of sodium or potassium hydroxides were added to the mixture before mixing was continued at speed 2 for 10 minutes and solid method where kaolinite, silica sand and calcium hydroxide in a powder form were mixed at speed 1 for 2 minutes then distilled water was added to the dry mixture before mixing was continued at speed 2 for 10 minutes. At several time intervals of 4 minutes mixing was stopped to remove any materials stuck on the rotating shaft or on the body of the mixing bowl.

3.3.2.4. Specimens moulding:

The mixture of each batch with total weight of 600 g was divided into ten equal specimens by weight (60 g). The specimens were immediately moulded after weighing to avoid both drying and decrease of the workability of the mixture. They were moulded using a stainless steel cylindrical mould with dimensions of 50 mm length and 26 mm diameter under a pressure of 15 MPa using a manually SHOP hydraulic press (model GARG-501-9). Then the moulded specimens were extruded using CARVER laboratory press (model C), before they were weighted and given numbers.



Figure 2: Extruded and numbered samples.

3.3.2.5. Specimens curing:

The extruded specimens were cured by placing them for 24 hours in BINDER oven (model ED 115) at a temperature of 80°C. In the case of Ca(OH)₂ curing was done under enclosed curing condition (closed in sealed container) at 80°C for 7 days in the oven, this variation in curing conditions was recommended to avoid the premature dehydration of the specimens and formation of CaCO₃ by absorbing CO₂ from atmosphere, which had a negative effect on the development of strength [10]. After this period of time the specimens were removed from the oven and they were allowed to cool down under laboratory conditions. The cured specimens were then weighed and the dimensions (length and diameter) were measured using KERN EG/EW electronic balance (model 3000-2M) and OXFORD digital caliper (model 331-3120K) respectively.

3.3.2.6. Pre-test treatment:

Specimens of each batch were variously treated; they were divided into four groups. The first group consists of 3 specimens where they were dried in SHEL lab oven (model CE3) at 40 °C for 7 days then their density, uniaxial compressive strength and durability under dry conditions were measured. The second group consists of 3 specimens where they were immersed in plastic containers filled with tap water for 7 days and after that their water absorption, their uniaxial compressive strength and durability under wet conditions were measured. The third group consists of 2 specimens where they were subjected to five cycles of drying and wetting, that means specimens were immersed in tap water for one day and then dried in an oven at a temperature of 40 °C for a day; this was repeated five times, before they were immersed in water for another day and after that uniaxial compressive strength and durability were measured. Finally the fourth group consists of 2 specimens, which were subjected to five cycles of drying and wetting, that means specimens were immersed one day and then dried in an oven at a temperature of 40 °C for a day; this was repeated five times before they were put in an oven at a temperature of 40 °C for another day before determining their uniaxial compressive strength and durability [10].

3.3.3. Testing of specimens:

3.3.3.1. Determination of water absorption:

Water absorption is the property of a material to be saturated with water [16]. It was determined for specimens soaked by total submergence in tap water for 7 days using the following equation [9, 10]:

$$A\% = \left(\frac{W_w - W_d}{W_d} \right) * 100\% \quad (3)$$

Where A% is the percentage of water absorbed by the specimen, W_w is the weight of the immersed specimen after drying with a paper towel (g), W_d is the weight of the specimen after curing at 80 °C (g).

3.3.3.2. Uniaxial compression test:

Uniaxial compression strength is a mechanical property which refers to the capacity of a material to withstand axially directed pushing forces. It is that value of uniaxial compressive stress reached when the material fails completely. The uniaxial compression test was performed using the CONTROLS CBR testing machine (model SBA-5T). The uniaxial compressive strength was determined [9, 10] using the following equation:

$$S = \frac{L}{A} \quad (4)$$

Where S is the uniaxial Compressive strength (N/m² or Pa), L is the maximum load applied before failure (N), A is the cross section area of the specimens (m²).

Compressive strength was measured for specimens treated in different ways. S1 is the dry compressive strength of specimens that dried for 7 days at a temperature of 40 °C, S2 is the immersed specimens compressive

strength of specimens that immersed for 7 days in a tap water, S4 is the cycling dry compressive strength of the specimens that immersed in tap water for one day and then dried in an oven at a temperature of 40 °C for a day; this was repeated five times, then they were dried in an oven at a temperature of 40 °C for one day. S3 is the wet cycling compressive strength of the specimens that immersed in tap water for one day and then dried in an oven at a temperature of 40 °C for a day, this was repeated five times, then they were immersed in water for one day.

The stability and durability of produced specimens was evaluated by calculating the differences between the compressive strength under different conditions. L1 is the loss of compressive strength due to immersion process. L2 and L3 is the loss of compressive strength due to cycling process under dry and wet conditions. The loss of compressive strength was calculated by using equations ((5)-(7)) [10].

$$L1 = \left(1 - \frac{S_2}{S_1}\right) * 100\% \quad (5)$$

$$L2 = \left(1 - \frac{S_3}{S_1}\right) * 100\% \quad (6)$$

$$L3 = \left(1 - \frac{S_4}{S_1}\right) * 100\% \quad (7)$$

3.3.3.3. Chemical and mineral analyses:

XRF analysis was performed to identify the chemical composition of the produced specimens, and XRD analysis was employed to identify the major mineralogical components of the produced specimens. The analysis was carried out at the NRA laboratories for those produced specimens that give optimum compressive strength.

4. Results and Discussion

4.1. Effect of NaOH and silica sand on kaolinite polymerization:

4.1.1. Effect of varying the ratio of NaOH to kaolinite:

In order to study the effect of sodium hydroxide content on the produced specimens. Several NaOH to kaolinite ratios of 0.18, 0.16, 0.15, 0.14, 0.12, and 0.10 were used with silica sand of particles size range of ($45 < d \leq 500$) μm . All the produced specimens were stable, no swelling or shrinking was observed. Figure 3 shows the water absorption as a function of NaOH to kaolinite ratio. Water absorption decreases with increasing NaOH to kaolinite ratio from 10.28 to 9.44 %. This may be attributed to the fact that higher alkali content in the mixture gives better reactivity, since NaOH having an affinity for water and readily dissolving in water, then more Al and Si species dissolve, resulting in denser microstructure, which means fewer surfaces for water to absorb on [17].

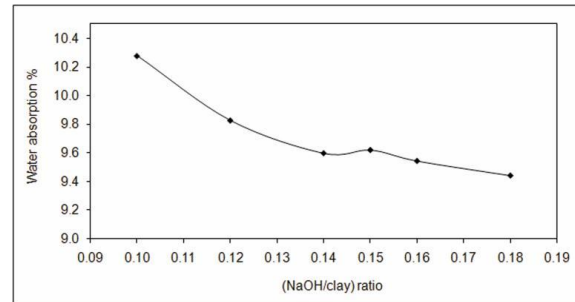


Figure 3: Water absorption as a function of NaOH to kaolinite ratio.

Figure 4 shows the variation of the specimens compressive strength as a function of NaOH to kaolinite ratio. A maximum compressive strength of 44.01 MPa was obtained for dry specimens (S1) containing NaOH to kaolinite ratio of 0.14. Similarly, the compressive strength of the immersed specimens (S2), the compressive strength of the dry cycling specimens (S3), and the compressive strength of the wet cycling specimens (S4) show a maximum value of 18.68, 39.08, and 20.12 MPa respectively. As a result NaOH to kaolinite ratio of 0.14 was considered as the optimum ratio which was used for all the next stages.

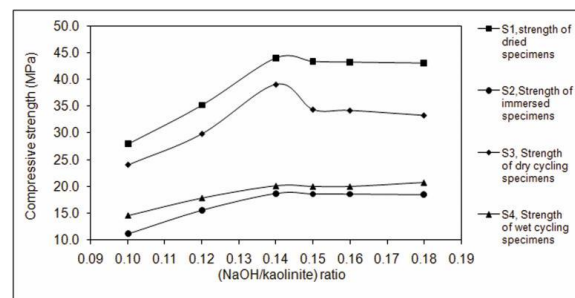


Figure 4: Variation of the specimen's compressive strength as a function of NaOH to kaolinite ratio.

The compressive strength of the specimens under different conditions increases with the increase of NaOH to kaolinite ratio from 0.10 up to 0.14 and then the compressive strength starts to decrease for specimens prepared with NaOH to kaolinite ratio greater than 0.14. This is probably due to the fact that with increasing the NaOH content affects the dissolution of Al and Si from kaolinite and more monomers will liberate into the solution ready for polymerization. Increasing the amount of sodium hydroxide within the specimens can be beneficial until certain limit. Beyond which excess sodium content can form Na_2CO_3 as a result of CO_2 absorption from the atmosphere may disrupt the polymerization process [6]. This salt will dissolve in water and even in the presence of the humidity. Salt dissolution causes a reduction in the strength and stability of the end products [13].

It is noticed that the loss of compressive strength values varied from 55.80 to 60.12 % due to immersion in water (L1), from 11.20 to 22.73 % due to cycling under dry condition (L2), and from 47.71 to 54.28 % due to cycling under wet condition (L3).

4.1.2. Effect of varying the ratio of silica sand to kaolinite:

The use of silica sand as a filler material in kaolinite polymerization process has two advantages; the first is to make the mixing process easier by reducing the cohesion between particles, and the second is to give strength to the end product by improving the grain packing within the specimens [13]. In order to study the effect of the silica sand content of particles size range of (45-500) μm on the produced specimens, four batches were prepared by varying the ratio of the silica sand to kaolinite as 1, 1.50, 1.25, and 0.75.

Figure 5 shows the water absorption as a function of silica sand to kaolinite ratio. Water absorption decreases from 9.85 to 9.60 % by increasing silica sand to kaolinite ratio from 0.75 to 1, then increases from 9.60 to 9.97 % by increasing silica sand to kaolinite ratio from 1 to 1.5. This indicates that increasing silica sand to kaolinite ratio from 0.75 to 1 causes much better grain packing, and thus a dense and strong structure with a reduction in water absorption, then any increment in the silica sand content to a constant kaolinite content will produce cohesionless grain packing with an increase of water absorption.

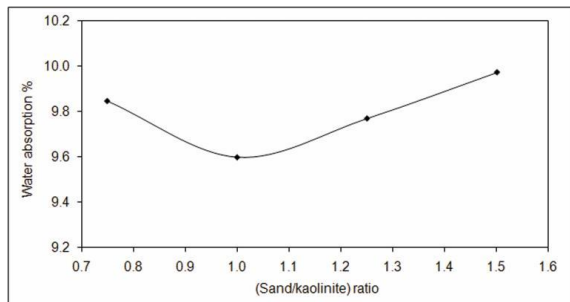


Figure 5: Variation of water absorption with the variation of silica sand to kaolinite ratio.

Figure 6 shows the variation of the specimens compressive strength as a function of silica sand to kaolinite ratio. A maximum compressive strength of 44.01 MPa was obtained for the dried specimens (S1) with silica sand to kaolinite ratio of 1. Similarly, maximum values of 18.68, 39.08, and 20.12 MPa were obtained for immersed (S2), dry cycling (S3), and wet cycling specimens (S4), respectively with a silica sand to kaolinite ratio of 1. As a result this silica sand to kaolinite ratio is considered as the optimum ratio used for all the next stages.

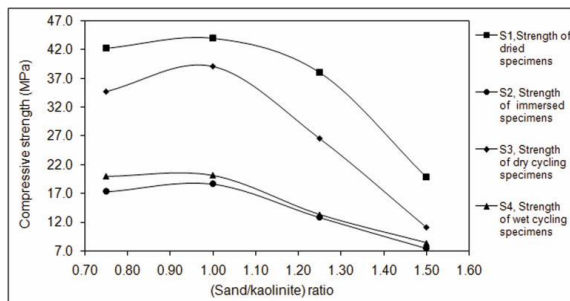


Figure 6: Variation of the specimen's compressive strength as a function of silica sand to kaolinite ratio.

One possible explanation for the increase of the specimens compressive strength as a result of increment of silica sand to kaolinite ratio from 0.75 up to 1 is the improvement of the grain packing of the specimens by reduction of pores, and thus a dense and strong structure is

formed. Beyond the silica sand to kaolinite ratio of 1, an observable reduction in the specimens compressive strength due to the cohesionless grain packing with increment of pores and less is geopolymerization [18].

The loss of compressive strength values range from 57.56 to 66.30 % due to immersion process (L1), from 11.204 to 44.028 % due to cycling under dry condition (L2), and from 52.81 to 65.04 % due to cycling under wet condition (L3).

4.1.3. Effect of varying the ratio of water to kaolinite:

To reveal the effect of water content on the produced specimens, three batches were prepared using ratio of water to kaolinite of 0.19, 0.22 and 0.25, respectively. It is observed that using water to kaolinite ratio of 0.19 showed low workability. The mixture was cohesionless, the compaction and extruding of the specimens from the mould was very difficult. The workability of the mixture was improved by increasing the water to kaolinite ratio to 0.22, but again, the workability of mixing becomes very low by using 0.25 water to kaolinite ratio because at this ratio the excess amount of water made cohesive mixture, difficult to compact and to extrude. Figure 7 shows the water absorption as a function of water to kaolinite ratio. The percentage of absorbed water decreased from 9.98 to 7.99 % as the ratio of water to kaolinite increased. This may be referred to the tendency of mineral polymerization mixtures to form larger crystals when the total amount of water in the reaction mixture is increased. This increment in the water content will lead to a decrease in the specific surface area for each specimens so decrease in the water absorption [18].

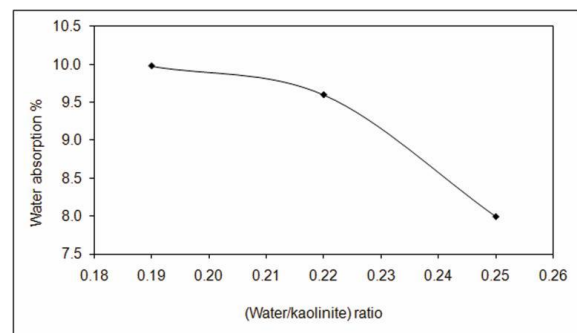


Figure 7: Water absorption as a function of water to kaolinite ratio.

Figure 8 shows the variation of the specimens compressive strength with the variation of water to kaolinite ratio. A maximum compressive strength value of 44.01 MPa was obtained for the dried specimens (S1) with water to kaolinite ratio of 0.22. Similarly maximum values of 18.68, 39.08, and 20.12 MPa were obtained for immersed (S2), dry cycling (S3), and wet cycling specimens (S4), respectively. As a result water to kaolinite ratio of 0.22 was considered as the optimum ratio and used in all the next stages.

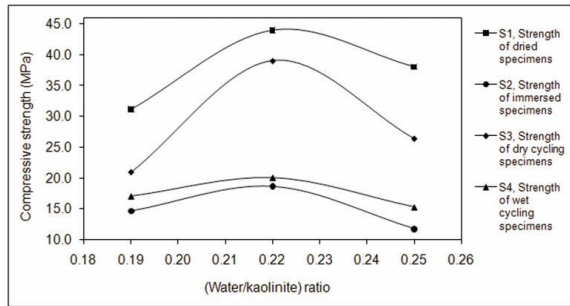


Figure 8: Variation of the specimen's compressive strength with the variation of water to kaolinite ratio.

Increasing the water to kaolinite ratio above 0.22 causes a reduction in the compressive strength. This may be attributed to two possibilities; the first is the evaporation of the water leaving large voids with fragile microstructure with low compressive strength for the end products. The second possibility is that the dilution effect of excess water that lowers the hydroxide ion concentration in the solution, which could decrease the dissolution of Al and Si from kaolinite during the polymerization process [6, 13].

For these samples the loss of compressive strength values varied from 52.88 to 69.08 % due to immersion process (L1), from 11.20 to 32.61 % due to cycling under dry condition (L2), and from 45.13 to 59.81 % due to cycling under wet condition (L3).

4.1.4. Effect of varying silica sand particles size:

To investigate the effect of silica sand particles size range on the produced specimens, three batches were prepared by varying the silica sand particles size range as ($45 < d \leq 180$), ($180 < d \leq 355$), and ($355 < d \leq 500$) μm . The absorbed water by the specimens as a function of silica sand particles size is shown in Figure 9. The absorbed water decreased from 9.72 % to 8.86 % as the silica sand particles size increased. This may be referred to the reduction of surface area as the particles size increase.

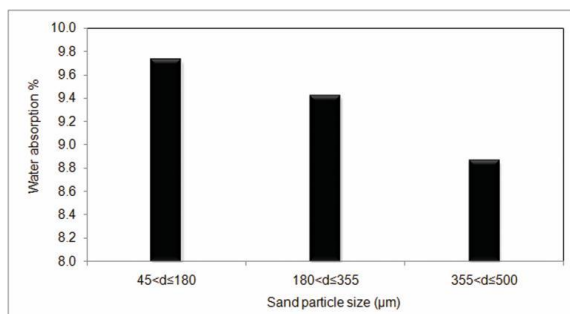


Figure 9: The absorbed water by the specimens as a function of silica sand particles size.

The specimen's compressive strength as a function of silica sand particles size is presented in Figure 10. A maximum compressive strength value of 45.45 MPa was obtained for the dried specimens (S1) for specimens produced with silica sand particles size range of ($180 < d \leq 355$) μm . Similarly, the immersed (S2), the dry cycling (S3), and the wet cycling specimens (S4) show maximum values of 20.38, 40.38, and 22.72 MPa, respectively.

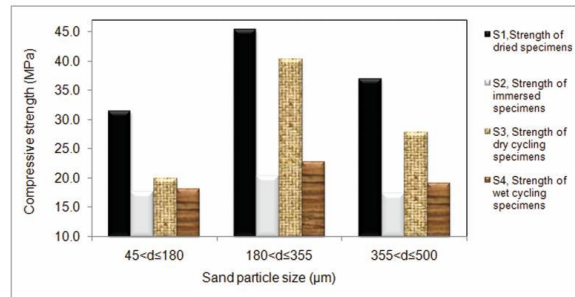


Figure 10: The specimen's compressive strength as a function of silica sand particles size.

The addition of NaOH to the polymerize kaolinite produced specimens with improved strength compared to reference specimens (kaolinite, NaOH, silica sand, and water ratios are 1, 0, 1, and 0.22 respectively) which have (S1) value of 2.53 MPa, and disintegrated completely immediately after immersed in water. The compressive strength of the dried specimens (S1), the immersed (S2), show maximum values of 45.45, and 20.38 MPa, respectively, and a water absorption of 9.41 % were obtained for specimens produced with kaolinite: NaOH: silica sand: and water ratios fixed at 1: 0.14: 1: and 0.22, respectively, and silica sand particles silica sand range of ($180 < d \leq 355$) μm .

The loss of compressive strength values varied from 43.44 to 55.16 % due to immersion process (L1), from 11.17 to 24.75 % due to cycling under dry condition (L2), and from 42.39 to 50.01 % due to cycling under wet condition (L3).

4.2. Effect of KOH and silica sand on kaolinite polymerization:

4.2.1. Effect of varying the ratio of KOH to kaolinite:

To study the effect of potassium hydroxide content on the produced specimens, five batches were prepared by varying the ratio of KOH to kaolinite as 0.25, 0.18, 0.16, 0.14, and 0.12, the silica sand particles size range was fixed at ($45 < d \leq 500$) μm . Using KOH to kaolinite ratio less than 0.12 produced unstable specimens in water, this may be referred to the low percentage of KOH to polymerize kaolinite. All produced specimens were stable. The variation of water absorption as a function of KOH to kaolinite ratio is presented in Figure 11. Water absorption decreased from 10.29 to 8.80 % as the KOH to kaolinite ratio increased from 0.12 to 0.25 %. This may be attributed to the fact that higher alkali content in the mixture gives better reactivity resulting in denser microstructure, which means fewer surfaces for water absorption [17].

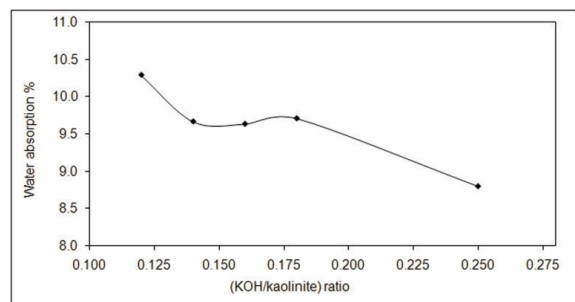


Figure 11: Variation of water absorption as a function of KOH to kaolinite ratio.

Figure 12 shows the specimens compressive strength as a function of KOH to kaolinite ratio. The specimens compressive strength increased by increasing the KOH to kaolinite ratio, a maximum compressive strength of 26.00 MPa was obtained for the dried specimens (S1) using KOH to kaolinite ratio of 0.25. Similarly, the immersed (S2), the dry cycling (S3), and the wet cycling specimens (S4) show maximum values of 10.87, 15.09, and 13.58 MPa, respectively.

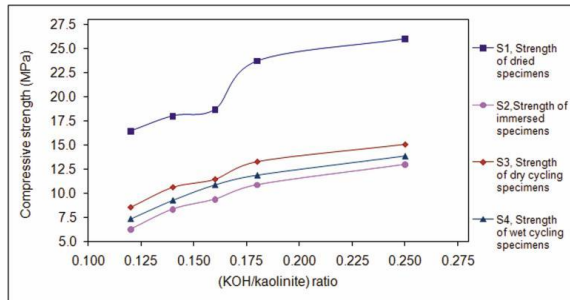


Figure 12: The specimen's compressive strength as a function of KOH to kaolinite ratio.

The compressive strength of the specimens under different conditions increased as the KOH to kaolinite ratio increased. This is probably due to the fact that increasing the KOH content will result in more dissolution of Al and Si from kaolinite and more monomers liberated into the solution ready for polymerization [6].

The tests show that the loss of compressive strength values varied from 38.68 to 48.10 % due to immersion process (L1), from 11.20 to 32.61 % due to cycling under dry condition (L2), and from 41.79 to 55.20 % due to cycling under wet condition (L3).

4.2.2. Effect of varying the ratio of silica sand to kaolinite:

In order to study the effect of silica sand content of particles size range of ($45 < d \leq 500$) μm on the produced specimens, four batches were prepared by varying the ratio of silica sand to kaolinite of 1, 1.50, 1.25, and 0.75.

The absorbed water percentage as a function of silica sand to kaolinite ratio is shown in Figure 13. Water absorption decreased from 10.86 to 9.71 % by increasing the silica sand to kaolinite ratio from 0.75 to 1, and then increased from 9.71 to 10.63 % by increasing the silica sand to kaolinite ratio from 1 to 1.5. This indicates that increasing the silica sand to kaolinite ratio from 0.75 to 1 caused much better grain packing, and thus a dense and strong structure which reduced the amount of absorbed water, increasing the silica sand to kaolinite ratio above 1 produced specimens with cohesionless grain packing, which absorbed more water as a result of the poor grain packing.

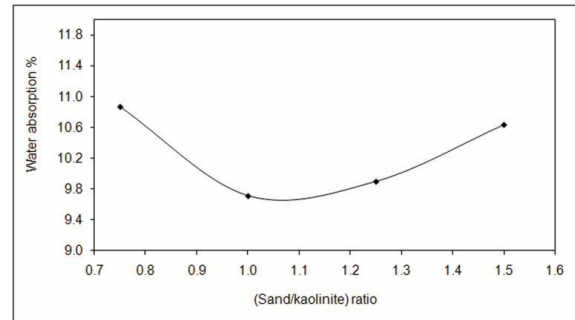


Figure 13: The percentage of absorbed water as a function of silica sand to kaolinite ratio.

Figure 14 presents the variation of the specimens compressive strength with the variation of the silica sand to kaolinite ratio. A maximum compressive strength (S1) of 23.71 MPa was obtained for the dried specimens with silica sand to kaolinite ratio of 1. Similarly, (S2), (S3), and (S4) show maximum values of 10.87, 13.29, and 11.18 MPa respectively using silica sand to kaolinite ratio of 1. Silica sand to kaolinite ratio of 1 was considered as optimum ratio and used in all the next stage.

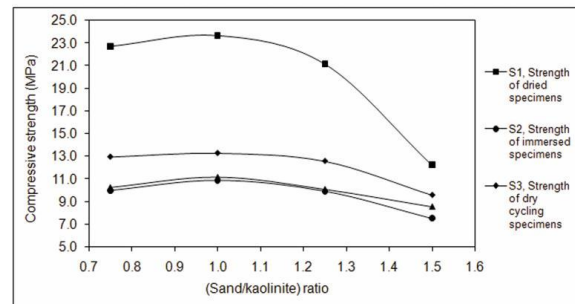


Figure 14: Variation of the specimen's compressive strength with the variation of silica sand to kaolinite ratio.

One possible explanation for the increase in the specimens compressive strength as a result of silica sand to kaolinite ratio increment from 0.75 up to 1 is the improvement achieved in the grain packing of the specimens by reduction of the pores number. Then, as the silica sand to kaolinite ratio increased from 1 up to 1.5 a reduction in the specimens compressive strength was observed due to the cohesionless grain packing and increment of pores number [18].

The variation of the loss of compressive strength values was from 38.71 to 56.10 % due to immersion process (L1), from 21.83 to 43.92 % due to cycling under dry condition (L2), and from 29.99 to 54.81 % due to cycling under wet condition (L3).

4.2.3. The Effect of varying the ratio of water to kaolinite

Three batches were prepared to investigate the effect of water content on the produced specimens. The absorbed water by the specimens as a function of water to kaolinite ratio is presented in Figure 15. The absorbed water decreased from 10.29 % to 8.60 % as the water to kaolinite ratio increased from 0.18 to 0.25. This may be explained by the tendency of mineral polymerization mixtures to form larger crystals when the total amount of water in the reaction mixture, this lead to a decrease in the specific surface area for each specimens and hence a decrease in the amount of absorbed water [19].

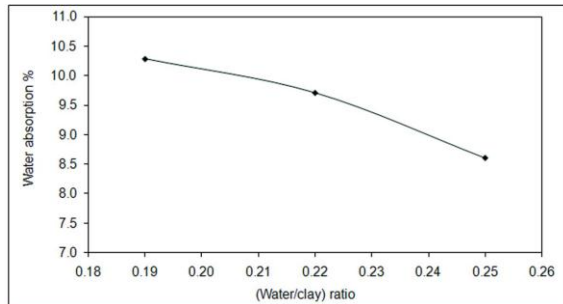


Figure 15: The absorbed water by the specimens as a function of water to kaolinite ratio.

The variation of the specimens compressive strength with the variation of water to kaolinite ratio is shown in Figure 16. A maximum compressive strength value of 23.707 MPa was obtained for the dried specimens (S1) for specimens prepared with water to kaolinite ratio of 0.22. Similarly, the immersed (S2), dry cycling (S3), and wet cycling (S4) show maximum compressive strength values of 10.87, 13.29, and 11.18 MPa respectively at the same water to kaolinite ratio of 0.22. Water to kaolinite ratio of 0.22 was considered as the optimum ratio and used in all the next stages.

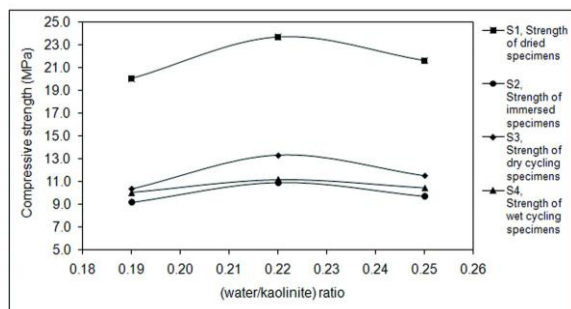


Figure 16: Variation of the specimen's compressive strength with variation of water to kaolinite ratio.

The compressive strength increased as the water to kaolinite ratio increased up to 0.22, then it decreased as the water to kaolinite ratio increased above 0.22. This may be explained by the evaporation of the water, which leaves large voids with fragile microstructure with low compressive strength for the end products. It also may be related to the dilution effect of excess water that lowering the hydroxide ion concentration in the solution, which could decrease the dissolution of Al and Si from kaolinite during the polymerization process [6, 13].

The loss of compressive strength for specimens prepared with the addition of KOH varied from 54.13 to 55.24 % due to immersion process (L1), from 43.92 to 48.39 % due to cycling under dry condition (L2), and from 50.00 to 52.82 % due to cycling under wet condition (L3).

4.2.4. The Effect of varying the ratio of water to kaolinite:

In order to study the effect of silica sand particles size range on the end product, three batches were prepared by varying the silica sand particles size range as ($45 < d \leq 180$), ($180 < d \leq 355$), and ($355 < d \leq 500$) μm .

The absorbed water by the specimens as a function of silica sand particles size is presented in Figure 17. Water absorption decreased from 10.82 to 9.28 % by increasing the silica sand particles size range from ($45 < d \leq 180$) to

($355 < d \leq 500$) μm . The reduction of the absorbed water may be attributed to the decrease of surface area as the particles size increase.

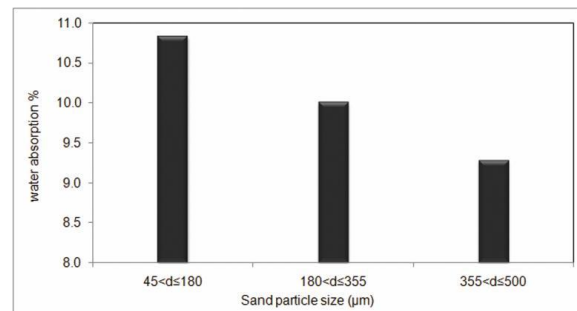


Figure 17: The absorbed water by the specimens as a function of silica sand particles size.

Figure 17 shows the variation of the specimens compressive strength with the variation of silica sand particles size. A maximum compressive strength value of 26.53 MPa was obtained for the dried specimens (S1) for specimens prepared with silica sand particles range of (180-355) μm . Similarly, the immersed (S2), dry cycling (S3), and wet cycling (S4) show maximum compressive strength values of 11.98, 14.21, and 12.04 MPa respectively at the same silica sand particles range of (180-355) μm . Silica sand particles range of (180-355) μm was considered as the optimum silica sand size range and used in all the next stages.

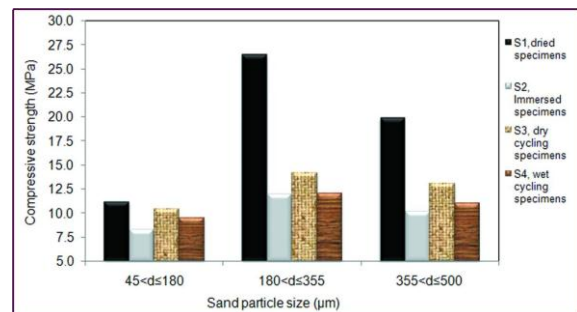


Figure 18: Variation of the specimen's compressive strength with the variation of silica sand particles size.

The loss of compressive strength values varied from 26.34 to 54.84 % due to immersion process (L1), from 7.38 to 46.44 % due to cycling under dry condition (L2), and from 8.79 to 15.77% due to cycling under wet condition (L3).

It should be noted that specimens that were prepared with the addition of NaOH exhibit significantly higher compressive strength values compared to the other specimens which were prepared with the addition of KOH, this may related to the fact that a higher extent of dissolution in NaOH than in KOH medium where Na^+ compared with K^+ enhances the dissolution of aluminum and silicate species [20]. As Na^+ and K^+ have the same electric charge, their different effects are a result of their different ionic sizes, the cation-anion pair interaction becomes less significant as the cation size increases. Cations with smaller sizes favour the ion-pair reaction with the smaller silicate monomers, dimers and trimers. Hence, it is expected that Na^+ with the smaller size will be more active in the reactions compared with K^+ , this should result in a higher extent of dissolution of minerals in the NaOH solution [5].

4.3. Effect of Ca(OH)₂ and silica sand on kaolinite polymerization:

To reveal the effect of calcium hydroxide content on the produced specimens, five batches were prepared by varying the ratio of Ca(OH)₂ to kaolinite as 0.25, 0.14, 0.10, 0.06, and 0.02, and silica sand of particles size range of (180 < d ≤ 355) μm. The produced specimens were stable and there were no observed swelling or shrinking.

The variation of water absorption as a function of Ca(OH)₂ to kaolinite ratio is shown in Figure 19. The absorbed water decreased from 18.00 to 12.80 % as the ratio of Ca(OH)₂ to kaolinite increased from 0.02 to 0.25. This may be attributed to the fact that higher alkali content in the mixture gives better reactivity resulting in denser microstructure, which means fewer surfaces for water absorption [5].

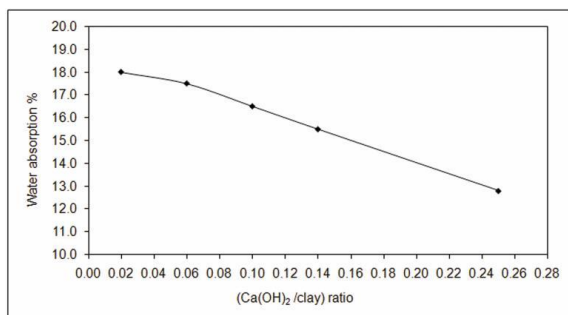


Figure 19: The variation of water absorption with the variation of Ca(OH)₂ to kaolinite ratio.

Figure 20 shows the variation of the specimens compressive strength with as a function of Ca(OH)₂ to kaolinite ratio. Compressive strength increases by increasing Ca(OH)₂ to kaolinite ratio. A maximum compressive strength value of 22.66 MPa was obtained for the dried specimens (S1) for specimens prepared with Ca(OH)₂ to kaolinite ratio of 0.25. Similarly, the immersed (S2), dry cycling (S3), and wet cycling (S4) show maximum compressive strength values of 7.89, 11.35, and 8.47 MPa respectively. The compressive strength of the specimens under different conditions increases with increasing the Ca(OH)₂ to kaolinite ratio. This is probably due to the fact that with increasing the Ca(OH)₂ content will lead to more dissolution of Al and Si from kaolinite and more monomers liberated into solution ready for polymerization [6].

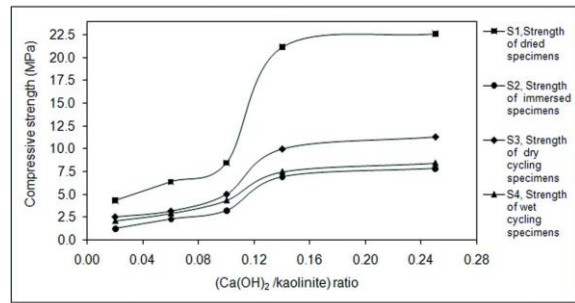


Figure 20: The variation of the specimens compressive strength with the variation of Ca(OH)₂ to kaolinite ratio.

Ca(OH)₂ specimens show the lowest compressive strengths values compared to that of NaOH and KOH specimens, and that was consistent with the low reactivity of Ca(OH)₂, the ageing of this specimens may increase the strength of it [9].

The loss of compressive strength values for specimens prepared with Ca(OH)₂ as the alkali material varied from 61.83 to 70.98 % due to immersion process (L1), from 41.47 to 52.69 % due to cycling under dry condition (L2), and from 48.36 to 64.54 % due to cycling under wet condition (L3).

4.4. Specimens Characterization:

For all produced specimens in the present work, the reduction (loss) in the compressive strength on soaking maybe due to several factors; one of these factors is the hydration process of the produced specimens. The liberation of water they hold acts to reduce the binder strength. Another factor that may lead to the reduction is the loss of some additives in the water and this leaves pores which act to decrease the compressive strength [9].

4.4.1. Chemical and mineral analyses results:

Table 5 shows the chemical composition of the produced specimens that give the optimum compressive strength after drying for 7 days at a temperature of 40 °C. This data was obtained by X-ray Fluorescence XRF analysis. The data show that the major components are SiO₂ and Al₂O₃. Ca(OH)₂ specimens have the minimum SiO₂ and Al₂O₃ compared with other specimens, and the highest loss on ignition, this may be explained by the lowest kaolinite and sand weight when using highest Ca(OH)₂ to kaolinite ratio as shown in Table 5.

Table 5: Chemical composition of the produced specimens that produced optimum strength the present study.

Specimens	SiO ₂ %	TiO ₂ %	Al ₂ O ₃ %	Fe ₂ O ₃ %	MgO %	CaO %	Na ₂ O %	K ₂ O %	P ₂ O ₅ %	L.O.I %
NaOH4	75.28	0.54	11.93	4.73	0.29	0.81	4.76	0.71	0.08	2.37
KOH1	74.71	0.52	10.85	4.65	0.39	0.79	0.43	5.65	0.1	2.54
Ca(OH) ₂ 5	66.21	0.43	10.18	3.51	0.15	10.54	0.6	0.47	0.06	7.41

The results of the mineral identification by XRD for the produced specimens that give the optimum compressive strength after drying are shown in Figures from (21 to 23). The XRD patterns show that there are undissolvable ingredients of quartz and kaolinite remain in all the produced specimens. This referred to the partially dissolved of Si and Al by the alkali solution then forming

the units of geopolymers. There are also traces of undissolvable amounts of muscovite and hematite.

Figure 21 shows the X-ray diffractogram pattern of the NaOH4 specimens, it shows new peaks of sodalite (sodium aluminum silicate hydrate, Na_{5.7}Al_{5.7}Si_{10.3}O₃₂·12H₂O) compared to the original kaolinite XRD pattern that was presented in Figure 1. This material is a zeolite mineral which are hydrated aluminosilicates and consist of three-dimensional

frameworks of AlO_4 and SiO_4 tetrahedra linked to each other by sharing all oxygen, these are more stable and have higher strengths than the kaolinite sheets. Zeolite has porous structure, inner and outer charged surfaces, suitable for ion exchange [21].

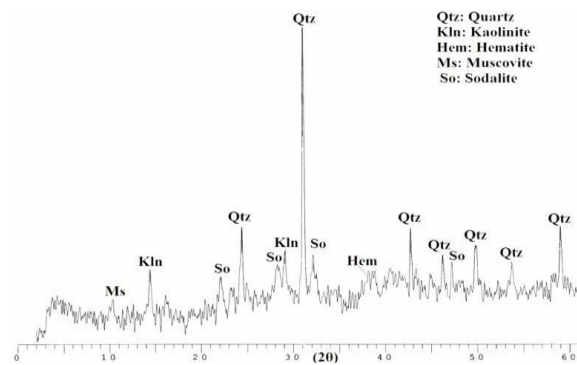


Figure 21: X-ray diffractogram of NaOH4 specimens.

Figure 22 shows X-ray diffractogram pattern of the KOH1 specimens, it shows new peaks of a zeolite mineral; phillipsite (potassium calcium aluminum silicate hydrate, $\text{KCaAl}_3\text{Si}_5\text{O}_{16}\cdot 6\text{H}_2\text{O}$) [3].

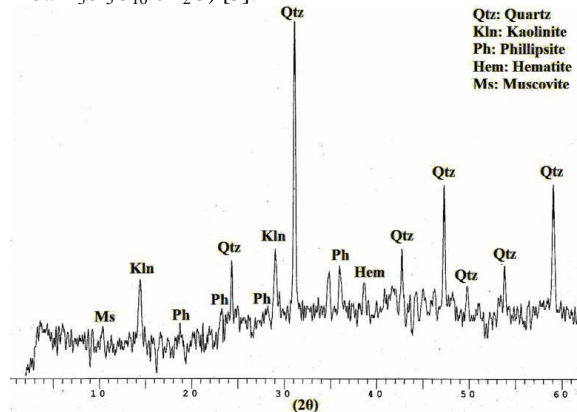


Figure 22: X-ray diffractogram of KOH1 specimens.

The X-ray diffractogram pattern of the $\text{Ca}(\text{OH})_2$ 5 specimens is shown in Figure 23, the figure shows new peaks of a [zeolite](#) mineral; gismondine (calcium aluminum silicate hydrate, $\text{CaAl}_2\text{Si}_2\text{O}_8\cdot 4\text{H}_2\text{O}$).

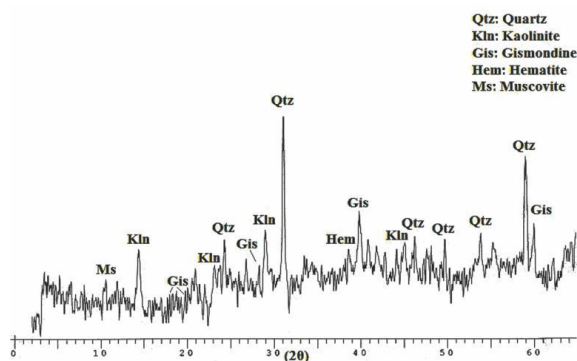


Figure 23: X-ray diffractogram of $\text{Ca}(\text{OH})_2$ 5 specimens.

5. Conclusion

- The optimum specimens were obtained using NaOH to kaolinite ratio of 0.14, water to kaolinite ratio of 0.22, and silica sand to kaolinite ratio of 1 with silica sand particle size of ($180 < d \leq 355$) μm . The values of water absorption, compressive strength under dry and wet condition was 9.41 %, 45.45, and 20.38 MPa respectively.
- Water absorption determination showed a decrease trend as alkaline content, and water content increased, on the other hand a minimum value was obtained when using silica sand to kaolinite ratio of 1.0.
- Compressive strength under different conditions showed a maximum value when using NaOH to kaolinite ratio of 0.14, on the other hand it showed increase trend by increasing KOH, and $\text{Ca}(\text{OH})_2$ content. A maximum value obtained when using silica sand to kaolinite ratio 1, and water to kaolinite ratio of 0.22.
- Variation of silica sand particle size shows an effect on the water absorption, and compressive strength of the produced specimens. This is a result of the changes in the grain packing of this material. Using silica sand particle size range of ($45 < d \leq 180$), ($180 < d \leq 355$), and ($355 < d \leq 500$) μm produce specimens of optimum compressive strength, with moderate density and water absorption.
- The acceptable specification for kaolinite based construction materials are hard to obtain. The standard requirement of 7 days compressive strength for ordinary Portland cement to be used in the general concrete construction has the specification limit of 12 MPa according to (ASTM C 150), while the ordinary Portland cement in Jordan have the 7 days dry compressive strength of 25 MPa. Comparison of this values with the obtained dry compressive strength values of the produced specimens in the present work show acceptable values using NaOH (45.45 MPa), and KOH (26.53 MPa) as alkaline additives with silica sand as a filler.
- Durability of produced specimens was evaluated by calculating the differences between the compressive strength under different conditions. The specimens of NaOH and silica sand as a filler showed the highest durability under dry cycling condition than KOH specimens. While the specimens of KOH and silica sand as a filler showed the highest durability under immersion, and wet cycling condition. The specimens of $\text{Ca}(\text{OH})_2$ and silica sand as a filler were showed the lowest durability.

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