

A study on the compressive strength and microstructure characteristic of alkali-activated metakaolin cement

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ABSTRACT

The main purpose of this study was to investigate the compressive strength and microstructure characteristic of alkali-activated metakaolin cement (AAMC). In accordance with this purpose, besides the pure metakaolin activation five other mixtures were designed by substitution of different OPC ratios instead of metakaolin (MK) from 0 to 25 (5, 10, 15, 20, 25% OPC). AAMC was activated with sodium silicate (Na_2SiO_3) of modulus $M_s = \text{SiO}_2/\text{Na}_2\text{O} = 3.1$ and NaOH solutions (32% of NaOH, 68% of water by mass). The ratio of liquid/solid (L/S) was kept constant at 0.65. All specimens were cured at 70 °C for 72 hours then kept in room conditions until the days that experiments were performed. Compressive strength and UPV experiment tests were carried out on all specimens at different curing periods of 3,14,28 and 90 days. In addition, the microstructure of 28 days alkali-activated metakaolin cements were analyzed with scanning electron microscope (SEM). The results showed that AAMC specimens reached the desired strength and major part of the final strength was gained at the end of 3 days of curing.

Keywords: Alkali activated cement; Geopolymer; Metakaolin, microstructure, UPV

1. INTRODUCTION

Alkali activated cements are inorganic polymers generated from activation of solid aluminosilicate materials such as blast furnace slag, fly ash or metakaolin to form a new class of three-dimensionally network of alkali aluminosilicate [1-3]. Studies on the improvement of alkali activated binder as an alternative to conventional cement has been considerably increased in the last years. The reasons of this increase are many advantages of alkali activated cements over conventional cement such as lower CO₂ emission and development of desired strength and structural properties [4]. The use of high cement content in the construction sector is neither economically nor environmentally acceptable. It is known that about 7 % of global CO₂ emissions originate from Portland cement production. The decrease of Portland cement production percentage will reduce the degradation of raw materials and also reduce the rate of CO₂ released to the atmosphere which will affect the hazard of global warming [5,6]. Supplementary materials such as fly ash, ground granulated blast furnace slag, silica fume, and metakaolin are used as partial replacements to decrease the consumption of Portland cement [3,5]. Nevertheless when these supplementary materials are used more than a certain amount the mechanical properties of the cement are greatly reduced, particularly at early ages [7]. Alkaline-activated cements and/or geopolymers which are investigated in our study can produce up to 100% of replacement of these materials with Portland cement.

Metakaolin is the low-calcium material one of the most commonly used in alkaline cement. The main reaction product of activation of materials such as kaolin comprising primarily aluminum and silicon is N-A-S-H (geopolymer) gel which is three dimensional inorganic alkaline polymers [8]. Fernandez-Jiménez et al. explain N-A-S-H gel formation as follows. When the aluminosilicate source is contacted with the alkali solution, it is dissolved in many species, primarily silica and alumina monomers. Dimers are formed by these monomers interaction then interact with the monomers to form the trimers, tetramers and so on [9].

Very few research has been done on AAMC [10]. Rashad et al. [11] studied compressive strength of

alkali-activated metakaolin replaced with quartz powder at different levels ranging from 0 to 30% with an increment of 5%, by weight. They reported that the mixture produced from fully metakaolin exhibited high compressive strength at age of seven days. They also reported that 28 and 365 days compressive strength was only 2.22% and 3.4% higher, respectively, in comparison with 7 days. In a study conducted by Wianglor et al. [10] OPC was used to replace part of metakaolin at 5, 10, 15, 20, 30% by mass of binder.

Specimens were cured at $23 \pm 2^\circ\text{C}$ (55% RH) and 60°C (95% RH) conditions. With the increase of OPC replacement and curing temperature compressive strength of AAMC increased. At 60°C (95% RH) curing condition matrices appeared denser than when cured at $23 \pm 2^\circ\text{C}$ (55% RH). Yunsheng et al. [12] studied effect of slag replacement on the mechanical properties of MK/slag alkali activated mortars. Water/binder ratios were kept constant as 0.35 for all mixtures. The all specimens were cured at 20°C and 100% relative humidity for 28 days. Replacement of slag increased both compressive and flexural strength.

The highest compressive and flexural strengths were obtained in the mixture containing 50% slag and they are 64.1 MPa and 8.01 MPa, respectively. Buchwald et al. [13] studied the performance of Alkali-activated metakaolin-slag blends. They used NaOH as alkali activator. It is reported that the blend containing 100 % slag reached the highest strength values while blend containing 100 % metakaolin showed a lowest strength. The blends containing metakaolin/slag (0.5/0.5) and (0.25/0.75) reached similar strength values. Burciaga-Diaz and Escalante-Garcia [14] compared the performance of alkali activated slag/metakaolin cement pastes. They prepared mixes of slag-metakaolin weight proportions of 100-0, 50-50 and 0-100. They also prepared a blended ordinary Portland cement as control. The alkaline activators used composed of blends of sodium silicate. Different water/solid ratios were used in each mix. They kept all specimens at 20 C and 80% relative humidity for 24 h then at 60 C in dry conditions for 48 h. The one with 100 % BFS reached the highest strength of 92 MPa. The mixtures 50-50, %100 MK, and the control reached 58, 41 and 53 MPa respectively. There are some other studies on kaolin based alkali activated materials [15-17].

The main objective of this study is to explore the compressive strength of alkali-activated metakaolin cement (AAMC) by destructive and non-destructive methods and also the microstructure was assessed using SEM and EDX. For this objective, six mixtures were used, which were substituted MK with OPC and a mixture was used as a control. Strength properties were assessed by compressive strength and UPV measurement at 3, 14, 28, and 90 days. Water absorption of AAMC specimens were tested at 28 days of curing period for durability property.

2. MATERIALS AND METHODS

2.1 Aluminosilicate precursors

The raw material (precursors) used in the study was metakaolin (MK). Particle size distributions d_{50} , d_{90} and d_{98} were $4.22 \mu\text{m}$, $21.43 \mu\text{m}$ and $37.84 \mu\text{m}$ respectively. Chemical composition of metakaolin precursor is shown in Table 1.

Table 1: Chemical composition of Metakaolin

Chemical composition (%)	MK
SiO ₂	50.62
Al ₂ O ₃	45.7
Fe ₂ O ₃	0.31
CaO	0.2
MgO	0.32
Na ₂ O+K ₂ O	0.32
Na ₂ O+0.658K ₂ O	-

2.2. Portland Cement

An ordinary Portland cement (CEM I 42.5N) was used to replace metakaolin at different rates. Chemical and physical properties of Portland cement are shown in Table 2.

Table 2: Properties of Portland cement

Chemical composition (%)	OPC
SiO ₂	21.12
Al ₂ O ₃	5.62
Fe ₂ O ₃	3.24
CaO	62.94
SO ₃	2.3
Loss in ignition	3.52
<u>Physical Properties</u>	
Specific Gravity (g/cm ³)	3.1
Specific Surface Area (cm ² /g)	3490

2.3 Alkali activators

A mixture of sodium hydroxide solution and sodium silicate solution was used to activate aluminosilicate precursor. Sodium silicate (Na₂SiO₃) solution used in this study is commercially available. The ratio of SiO₂ to Na₂O by mass(Ms) in the sodium silicate solution was 3.1. Chemical composition of sodium hydroxide solution used is NaOH –32%, Water –68% by mass. Alkali activators were used as 65% by weight of the total binder.

2.4 Superplasticizer

Modified polycarboxylate-based polymer type superplasticizer (SP) was used in this study. The specific weight of SP used was about 1.1 g/cm³ and pH was in the range of 3–7. SP was used as two percent by weight of the total binder.

2.5 Preparation of mixtures and experimental techniques

To investigate the compressive strength and microstructure characteristic, six mixtures were designed in this study which is shown in Table 3. The ratio of liquid/solid (L/S) and superplasticizer percentage of all mixtures were kept constant to compare the compressive strengths to each other. In our study, liquid refers total alkali solution and solid refers total binder. All pastes were blended with the aid of a mixer for about three minutes. After mixing pastes were cast into 50*50*50 mm³ cubic molds and vibrated for one min. with the vibrating table. Molds were completely covered with film made of polyethylene[1]. After these steps, all mixtures were cured at 70 °C in an oven for 72 h. After 72 h cured at 70 °C, the specimens were demoulded and kept in room temperature for different curing periods. Ultrasonic Pulse Velocity(UPV) measurements and compressive strength experiments were performed after 3, 14, 28 and 90 days of curing periods of all specimens. Also the microstructure properties were observed using scanning electron microscopy(SEM).

Table 3: Mix proportions

Formulation	Replacement(wt%)		Ms	Na ₂ SO ₃ /NaOH(byweight)	L/S	SP(%)
	MK	PC				
MK	100	-	3.1	3	0.65	2
MKC05	95	5	3.1	3	0.65	2
MKC10	90	10	3.1	3	0.65	2
MKC15	85	15	3.1	3	0.65	2
MKC20	80	20	3.1	3	0.65	2
MKC25	75	25	3.1	3	0.65	2

2.6 Compressive strength test

Compressive strength test were carried out according to ASTM C 39[2]. The average of three specimens was used at these experiments. Compressive strength was determined at the age of 3, 14, 28 and 90 days for aging effect of AAMC.

2.7 Ultrasonic pulse velocity test (UPV)

The UPV measurements, a non-destructive test to learn the quality of concrete in terms of velocity, was carried out according to ASTM C597[3]. UPV measurements were conducted on cube samples of 50x50x50 mm³ at different days of curing. A relationship is established between the UPV values and the compressive strength of the concrete. There is no direct relationship between the UPV values and the compressive strength but there is a certain relationship between the UPV values and the density of the concrete i.e. the higher UPV value the higher density. And the higher density means higher compressive strength[4].

2.8 Water absorption tests

Water absorption were performed on the specimens according to ASTM C642[5] end of 28 days of curing period. The weight of saturated and oven-dried specimens was measured to determine the water absorption value. The measured values were calculated using Eqs. (1) The results were obtained by the average of three samples. Methods for measuring water absorption were used successfully in a number of studies [6, 7].

$$Absorbtion(\%) = \frac{W_a - W_d}{W_d} * 100 \quad (1)$$

Where absorption is water absorption of AAMC paste(%), W_a is weight in air of saturated specimens(g) and W_d is weight of dried specimen(g).

3. RESULTS AND DISCUSSION

3.1 Compressive strength results

Fig. 1 shows 3, 14, 28 and 90 days of compressive strengths results of alkali-activated metakaolin cement pastes. As can be seen, significant increases in compressive strength were observed with the replacement of OPC with metakaolin at different ratios. This increase has begun with the replacement of OPC with 5% metakaolin which is the lowest rate of replacement. At the end of 28 days, the increases in the compressive strength of the MKC05, MKC10, MKC15, MKC20, MKC25 mixtures compared to the mixture produced entirely of metakaolin was 33.3, 38.5, 41.2, 47.9, 54% respectively. The compressive strength gaining were higher at the end of the third day compared to day of 28. When the compressive strengths of blends containing OPC and blend produced with entirely kaolin are compared it is seen that the difference between the compressive strengths is generally reduced with the increase of the curing period. Compressive strength gaining are based on formation of C-S-H gels resulting from OPC hydration, besides formation of N-A-S-H ($\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$) gels resulting from geopolymerization [8].

A research conducted by Garcia-Lodeiro has revealed that these two products don't develop singularly as two separate gels, they interact so structural and compositional change occurs in the process[9]. The compressive strength of all mixtures increased with the curing period. Compressive strengths of MK mixture at 14, 28 and 90 days were 4.9, 15.6 and 16.6 higher compared to day of 3, respectively. Compared to third day, compressive strength of MKC25 mixture at 14, 28 and 90 days were 9.2, 10.8 and 11.4% higher, respectively. Rate of compressive strength increases of mixtures containing OPC were similar to each other. When it comes to the difference between the initial and final compressive strengths of all mixtures, the compressive strength for MK, MKC05, MKC10, MKC15, MKC20 and MKC25 mixtures increased 16.6, 11.6, 6.9, 8.9, 11.7 and 11.4%, respectively.

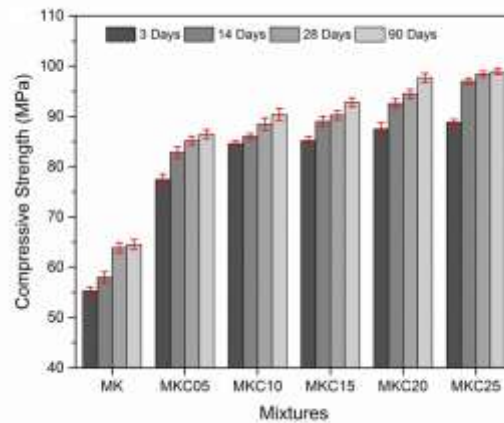


Figure 1: Compressive strength of AACM specimens at different ages.

In Fig. 1, when compared to final strength that AACM specimens gained it is seen that the compressive strength of the samples obtained from all the mixtures on the 3rd day is very significant. The blend which is completely produced with kaolin has already achieved 83% of its final strength at the end of 3 days curing period. It can be seen how high the early age compressive strength of alkali activated metakaolin based cements are when compared to the strength of normal concretes obtained in 3 days. In a study conducted by Wang [10] on compressive strength development of cement mortar it is reported that the 3 day compressive strength of normal concrete produced entirely from Portland cement is 58.9% of the 90 day compressive strength. In another study [11] metakaolin is used as mineral additive the result show that the 3 day compressive strength is about 40% of the 28 day compressive strength. In these types of cements produced by the activation of metakaolin with alkali activators the 3 day strength of the MK mixture was 86.4% of the 28 day strength. The compressive strength results show that these AACM pastes produced at these mixing ratios achieved relatively high early strength as well as the desired final strength. Fig. 2 shows the specimens from all blends subjected to the compressive strength and other experiments. As OPC replacement increased the color of specimens changed from cream to gray. The fact that the specimens have a smooth surface indicates good workability when fresh.



Figure 2: Hardened MK, MKC05, MKC10, MKC15, MKC20, MKC25 specimens, respectively

3.2 UPV results

UPV results of AACM specimens are shown in Fig. 3. The results of the UPV, a non-destructive test method, supported the compressive strength results. At the end of the 28-day curing period, UPV values were measured as 2956, 3247, 3356, 3356, 3472 and 3472 (m/s) for MK, MKC05, MKC10, MKC15, MKC20 and MKC25 specimens, respectively. It is seen that UPV values tend to increase as OPC content increase. these increase rates are more evident at age of 90. The UPV value measured at the end of 3 days of curing time of the specimens containing 100% metakaolin was 2874 (m/s) whereas this value was 3056 (m/s) after 90 days of curing period increasing 6 %. The difference between the 3 and 90 days of curing period of the MKC05, MKC10, MKC15, MKC20 and MKC25 mixtures were 6.9, 9.3, 4.4, 4.9 and 2.9%, respectively. Although UPV values increase with the cement content and aging there are cases where the UPV values remain constant in two consecutive cement contents or two different curing periods. Then it can be said that UPV values increases with aging and OPC content but this increases were not as evident as the increase in compressive strength.

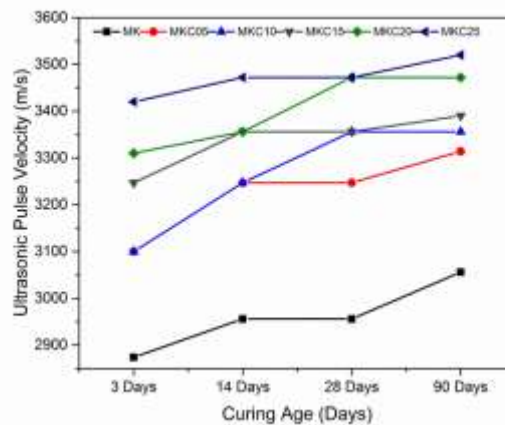


Figure 3: UPV results of AACM specimens at different ages

The ultrasonic device is used to measure the transit time of ultrasonic waves sent to specimen from one surface to another and so the wave velocity is calculated. The relationship between the calculated super-sonic wave velocity and the compressive strength of the AACM specimen is approximately obtained[4]. Fig. 4 shows the relationship between compressive strength and UPV results at the end of 28 days of curing. As mentioned earlier, there was a high relationship between pressure resistance and UPV results. Coefficient of determination (R^2) was 0.9721. This means that we can estimate the compressive strength over the UPV results by 97.21%. Coefficient of determination (R^2) proved strong relationship between these two variables.

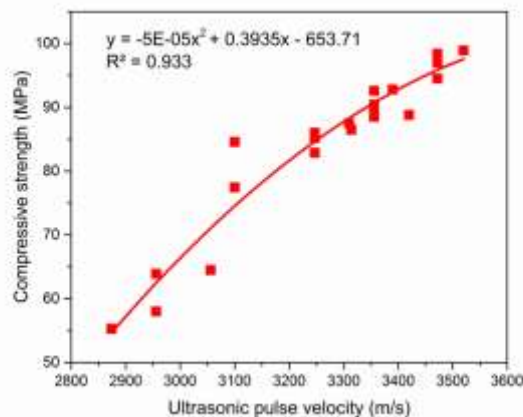


Figure 4: Relationship between the compressive strength and UPV at the age of 28

3.3 Water absorption

Figure 5 shows the water absorption and compressive strength of the specimens taken from all mixtures at the end of the 28 days of curing period. The water absorption percentages of the samples obtained from the mixtures of MK, MKC 05, MKC 10, MKC 15, MKC 20 and MKC 25 were 21.51, 17.52, 17.73, 16.82, 13.17, and 12.53%, respectively. It is evident that the water absorption reduces with increasing in OPC content in all specimens. Hydration and C-A-S-H increases with increasing OPC content. The new products fill the pores and thus reduce water absorptions with corresponding increase in compressive strengths[7]. Although the rate of water absorption decreased as the amount of OPC added increased, the water absorption values were high due to the fact that the metakaolin used was very fine grained. Similar values for water absorption in alkali active cements were obtained in other studies [6,7].

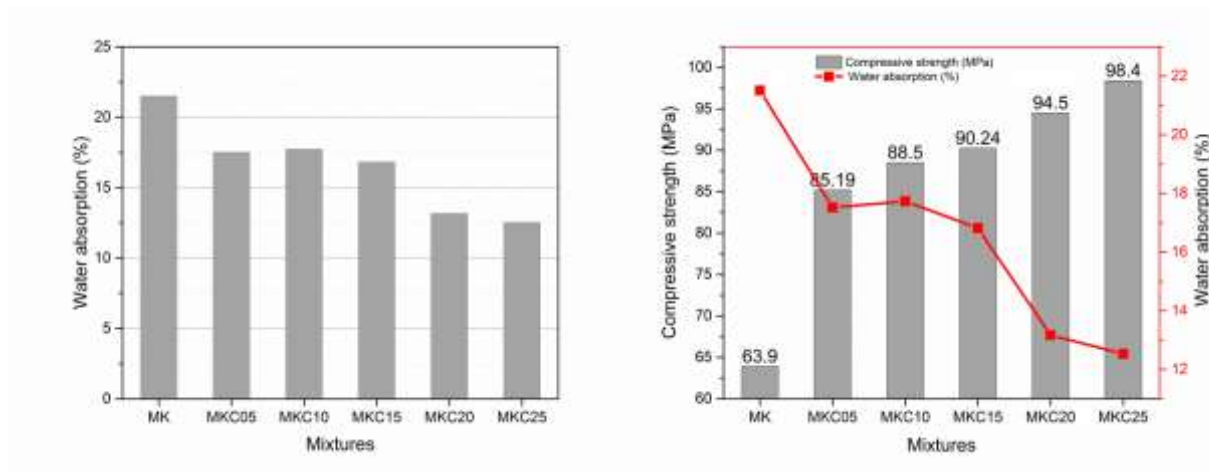


Figure 5: Water absorption and compressive strength of AAMC specimens at the age of 28

As shown in Fig. 6, there is a close relationship between water absorption and compressive strength of AAMC specimens. High water absorption capacity has lower strength. The strong correlation between these two variables was supported by the coefficient determination (R squared). $Ax^2 + bx + c$ is the polynomial equation that best matches the R² values for these variables. This R² value is 0.9774. That means the compressive strengths can be predicted by water absorption percentage by 97, 74% accuracy.

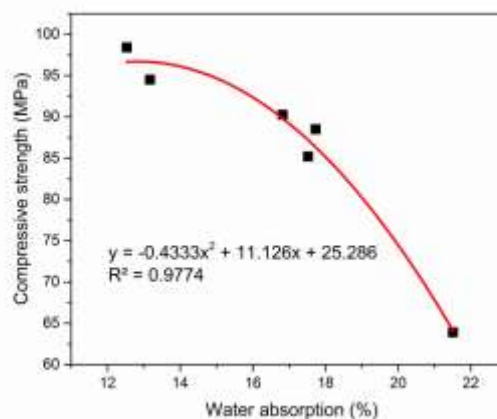
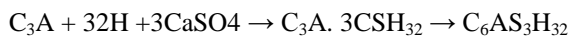


Figure 6: Relationship between the compressive strength and water absorption at the age of 28

3.4 SEM analyses

SEM image and EDX results of AAC specimens are shown in Figures 7-10. The main reaction product formed from activation of metakaolin is $\text{Na}_2\text{O}-\text{Al}_2\text{O}_3-\text{SiO}_2-\text{H}_2\text{O}$ (N-A-S-H) geopolymer gel which can be regarded as a zeolite precursor [8]. In Fig. 7, specimen having 100% metakaolin, it can be seen that N-A-S-H gel have formed. Elemental compositions obtained from EDX results confirm that these have occurred. The reaction products forming during of alkaline activation of OPC and low CaO contents materials (such as metakaolin or type F fly ash) are N-A-S-H and C-S-H [8]. A research has evinced that these two products don't evolve as two separate gels. There are composition and structural change through interaction of between these gels [9]. As can be seen from EDX results in Figures 7-10, Ca increased as OPC replacement increased. The increase in calcium demonstrates the co-existence of N-A-S-H and C-S-H gels. We can see from paste SEM images with OPC increase in our mixes they seem more condensed. In cement hydration process at aqueous state mainly involve of C_3S , C_2S , C_3A , and C_4AF also clinker sulfate and gypsum. In the hydration process, C_3A , C_4AF , C_3S , and C_2S will carry out a complex hydration reaction to form ettringite, calcium hydroxide, and C-S-H gel. The Hydration of tricalcium aluminate will form ettringite. The reactions has been shown in below:



$\text{C}_6\text{AS}_3\text{H}_{32}$ = (Calcium Aluminate Tri Sulphate Hydrate or Ettringite)

The reaction of pure C_3A with water is very fast and this may lead to prohibition from flash setting gypsum is added for cement. Needle shape gel prepared a new type amorf materials that have been obviously seen in figures C,D,E also Unreacted or partially reacted OPC particles have not seen in the matrixes. The geopolymer gels produced by the dissolution of metakaolin generally have insignificant amount of calcium, which is primarily sodium alumino-silicate hydrate (N-A-S-H). In shape (a) we can see only N-A-S-H gels with partially cement hydration product. In the mix samples of this research, presence of calcium in the geopolymeric gel is confirmed as shown in typical EDX diagrams shown in Figures 7-10. Inclusion of ordinary Portland cement supplies additional calcium and thus contributes to formation of the binding product containing calcium ion. these new combined matrixes are more strength i.e. the more Ca ions the more strength .in other words our hypothesis these two materials don't evolve as two separate gels and Composition and structural change have been occurred from interaction of gels has been proved.

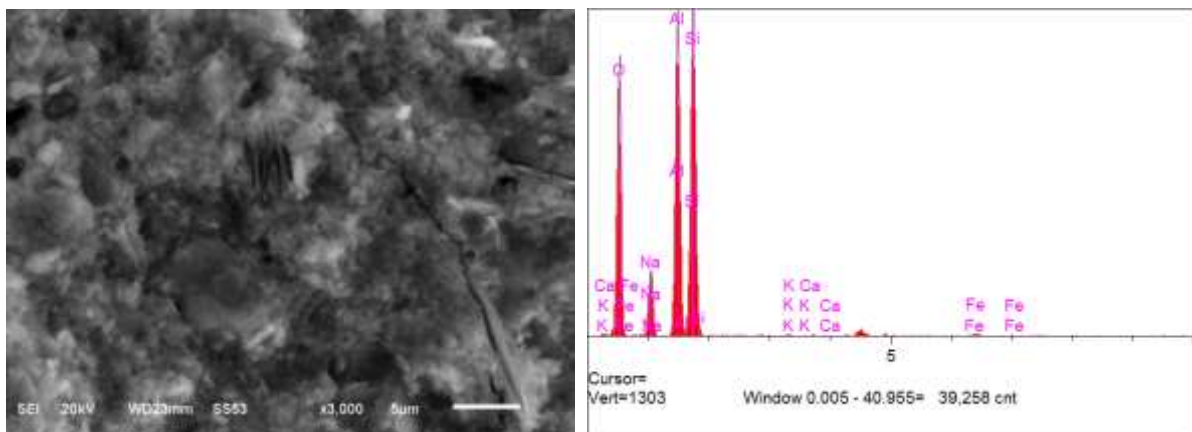


Figure 7: SEM micrographs for MK at the age of 28

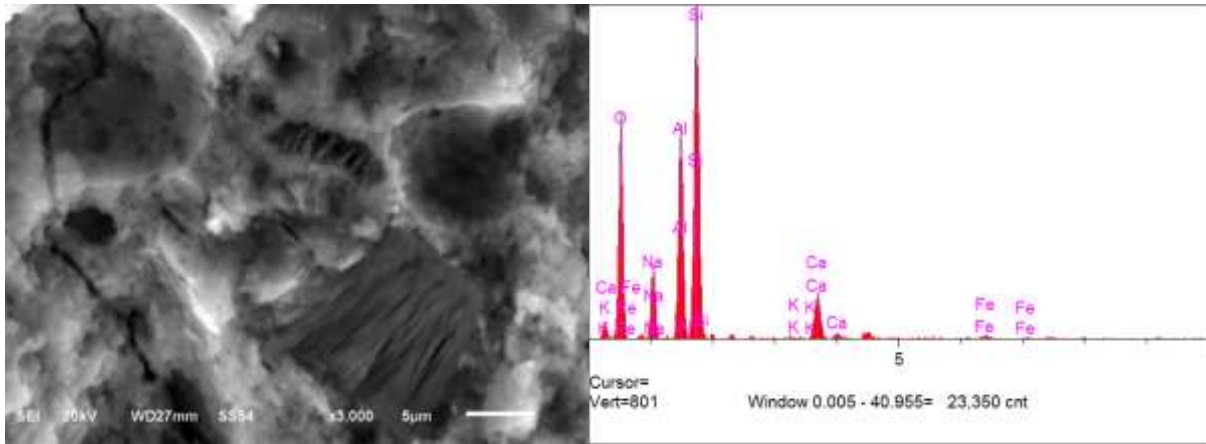


Figure 8: SEM micrographs for MKC05 at the age of 28

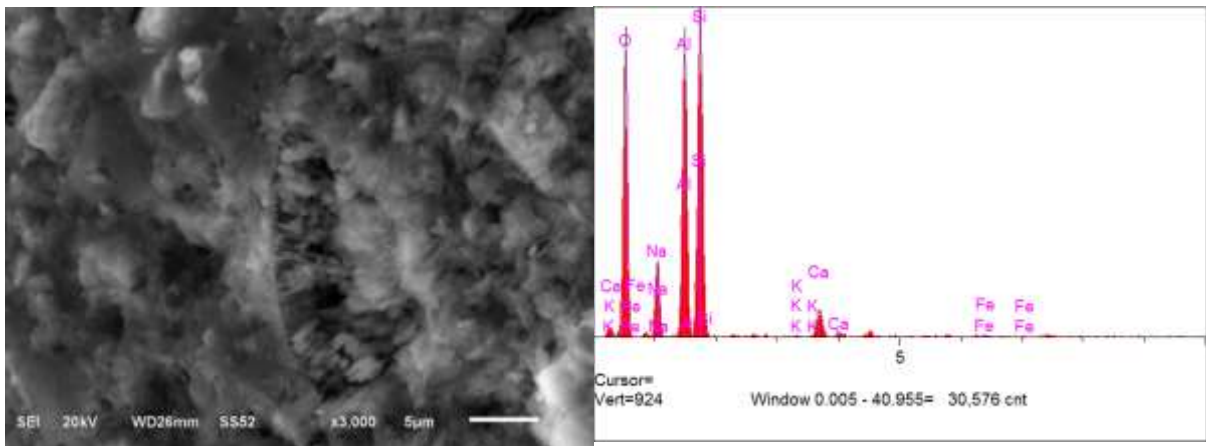


Figure 9: SEM micrographs for MKC10 at the age of 28

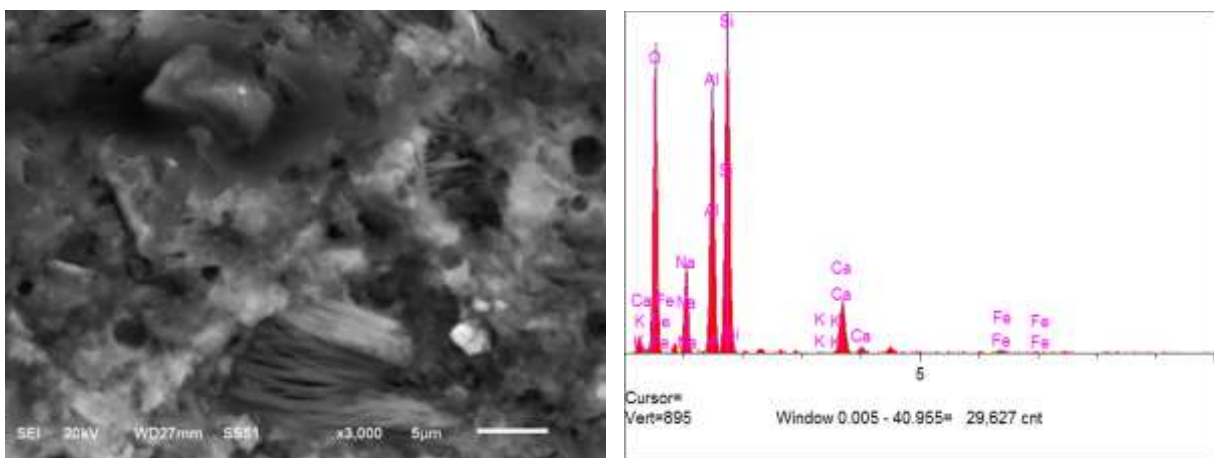


Figure 10: SEM micrographs for MKC20 at the age of 28

4. CONCLUSIONS

The main purpose of this study was to investigate the compressive strength of alkali activated metakaolin based cements activated with mixture of NaOH and Na₂SiO₃ solutions at the ratio of 1:3. Significant increases in compressive strength were observed with the replacement of OPC. At the end of the 28 days in 5% OPC substitution which is the lowest percentage of replacement, compressive strength was 1.33 times higher than mixtures produced with fully metakaolin. This value was 47.9 for 25% OPC substitution.

When investigating effect of strength gaining it is seen that a significant part of the final strength is gained at the end of 3 days of curing period. these final strength gaining rates are 16.6, 11.6, 6.9, 8.9, 11.7 and 11.4% for MK, MKC05, MKC10, MKC15, MKC20 and MKC25, respectively. During the 90 day of curing period none of the compressive strength of the specimens decreased with aging. This type of cements can be used as building material because of reaching desired strength and not decreased the strength during the curing period. The water absorption of the MK samples were too high but with increasing percentage of OPC, water absorption decreased considerably. Increase in compressive strength of OPC containing mixtures compared to blends produced 100% kaolin is a result of formation of new gel structures.

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