

# Performance of the Structural Lightweight Concrete with Metakaolin Exposed To Elevated Temperature

Bahar Demirel<sup>a\*</sup>, Ezgi Gültekin<sup>b</sup>

<sup>a\*</sup>Technology Faculty, Civil Engineering Department, Firat University, Elazig, Turkey

<sup>b</sup>Natural and Applied Science Institution, Firat University, Elazig, Turkey

## ABSTRACT

This study examined the effect of the elevated temperature on the mechanical and physical properties of the structural lightweight concrete (SLC) specimens produced by substituting cement with metakaolin (MK) at proportions of 5%, 10%, 15% and 20% by weight. The changes of the compressive and splitting tensile strength of the series were determined at the end of the 3, 7, 28-day curing periods. Up to 15% w/w MK addition demonstrated the optimum contribution to the strength development in all three curing periods. Nondestructive tests, such as ultrasonic pulse velocity (UPV), porosity, sorptivity, results have supported this conclusion. Also, the specimens that cured 28-day were heated in an electric furnace up to 400, 600 and 800 °C and kept at these temperatures for one hour. The specimens were cooled in the furnace. Then the relative compressive strength values of these specimens were determined. While the loss of compressive strength was observed in all series depending on the elevating temperature, 15% w/w MK addition series was showed the highest compressive strength value. However, especially at a temperature of 600 °C, increasing percentage of MK, the series have less affected by the elevated temperatures. The scanning electron microscope (SEM) studies have also supported these findings.

**Keywords:** Static synchronous compensator, stability, voltage sags, control strategies.

## I. INTRODUCTION

With improving technology, many scientific research is carried out to increase the strength and durability of the concrete. Use of mineral additives obtained by various methods reduces costs by reducing the production energy of the portland cement. Consequently, it has become common to use mineral additive in the construction industry. Metakaolin, is a mineral additive that was introduced in recent years in the production of high-performance cement-based materials. Unlike other pozzolans, it is a primary product, not a secondary product or by-product, which is formed by the dehydroxylation of kaolin precursor upon heating in the temperature range of 500-800 °C [1,2]. The addition of MK, which has high pozzolanic activity, highly improves the mechanical and durability properties of cement based materials [3,4]. The reaction between the MK and calcium hydroxide (CH) creates new tobermorite gels and alumina phases [5].

Concrete is resistant to high temperatures and fires. Because it has low thermal conductivity and high specific heat. However, there is no such a rule "concrete is never affected from the high temperature". Concrete can be exposed to elevated temperatures during fire or when it is close to furnaces and reactors. High temperature may affect the concrete's mechanical properties such as strength, elastic modulus and volume deformation in a negative way. To minimize these negative effects, it is possible to correct and appropriate use of mineral additives [6].

Therefore, there have been many researches that examine the performance of concrete that exposed to high temperature containing mineral admixtures as pumice, metakaolin, fly ash and silica fume in recent years. [2, 7-12]. However, the performance of the SLC containing metakaolin that has been exposed to elevated temperature, did not found in the literature. Therefore, in this experimental study mechanical behavior and microstructure of lightweight concrete containing metakaolin was investigated.

## II. METHODS AND MATERIAL

### Experimental

As the main binding, ASTM Type I (CEM I) Portland cement was used which were obtained from Elazig cement factory. In this experimental study, as fine aggregate, pumice aggregate was collected from the Mount Meryem volcano located in the Elazig province of Turkey. Coarse aggregate was supplied in the same region as the natural river aggregate. Maximum grain size of aggregate ( $d_{max}$ ) was determined as 8 mm. The aggregates used in SLC mix proportioning were formed of 74% lightweight sand (0-4 mm) and 26% coarse aggregate (4-8 mm). Table 1 shows the grading and density values of the aggregates.

**Table 1.** Grading and density values of the aggregate

Sieve Size (mm)	8	4	2	1	0.50	0.25
Passage(%)	100	74	57	31	17	9
Specific gravity ( $g/cm^3$ )						
Fine aggregate (0-4mm)	1.85					
Coarse aggregate (4-8mm)	2.60					

Dosage of all series was determined as 400 kg / m<sup>3</sup>. Metakolin supplied from Denge Kimya Ltd. was used as mineral additive. Chemical and physical properties of cement and metakaolin are given in Table 2.

**Table 2.** Chemical composition of the cement and metakaolin

Oxide compounds(mass%)	CEM I 42.5 N	Metakaolin (MK)
Silica (SiO <sub>2</sub> )	21.12	52-54
Alumina (Al <sub>2</sub> O <sub>3</sub> )	5.62	41-44
Iron oxide(Fe <sub>2</sub> O <sub>3</sub> )	3.24	<1.5
Calcium oxide(CaO)	62.94	<0.5
Magnesia (MgO)	2.73	<0.4
Sulfur trioxide(SO <sub>3</sub> )	2.30	-
Sodium oxide(Na <sub>2</sub> O)	-	<0.1
Potassium oxide(K <sub>2</sub> O)	-	<2
Titanium dioxide (TiO <sub>2</sub> )	-	<1
Density Density ( $g/cm^3$ )	3.15	2.60
Blaine (cm <sup>2</sup> /g)	3379	22000
Loss on ignition	1.78	-

As seen Table 3, five different series were prepared for investigating the effects of the metakaolin on the SLC spicemens. M0 represents control SLC without metakaolin. In M5, M10, M15 and M20 series, cement was replaced with MK at four proportions (5%, 10%, 15% and 20%) by weight.

**Table 3.** The mixture details of the prepared series ( $kg/m^3$ )

Series	Water	Cement	MK	Fine Aggregate (0-4mm)	Coarse Aggregate (4-8mm)
M0	190	400	-	493	285
M5	190	380	20	493	285
M10	190	360	40	493	285
M15	190	340	60	493	285
M20	190	320	80	493	285

Prepared mixtures pour into cubic and cylindrical molds, in order to determine compressive and splitting tensile strength, respectively. The specimens, which were demolded from the molds after 24 hours, were cured in lime saturated water for 3, 7 and 28 days.

For each series, total of fifteen pieces of SLC specimens were prepared, with five specimens being taken from each curing age (3, 7, and 28 days). Because five different series are used in the experiments, a total of 75 cubic specimens (100 × 100 × 100 mm) were prepared in order to determine the properties of the concretes such as compressive strength, apparent porosity, sorptivity and UPV.

To determine splitting tensile strength of SLC at the end of 28 days, total of 15 cylinder specimens (100x200 mm) were prepared, including 3 for each series. The UPV test that is an in-situ, nondestructive test was conducted by passing a pulse of ultrasonic wave through concrete to be tested and measuring the time taken by pulse to get through the structure. Wave speed was calculated by using Eq. (1) [13],

$$V = \left(\frac{h}{t}\right) * 10^6 \quad (1)$$

Where V = ultrasonic wave speed (m/sec), h = distance between the surface of concrete specimen from which the ultrasonic wave is sent and the surface the wave is received (m), t = time passed from concrete surface

from which the ultrasonic wave is sent and the surface the wave is received ( $\mu\text{s}$ ).

The porosity was calculated through Eq. (2). (% P) value was determined on cubic specimens according to Archimedes principle related to the weights of saturated specimens in air ( $W_{\text{sat}}$ ), in water ( $W_{\text{wat}}$ ) and the oven-dried specimen weight ( $W_{\text{dry}}$ ).

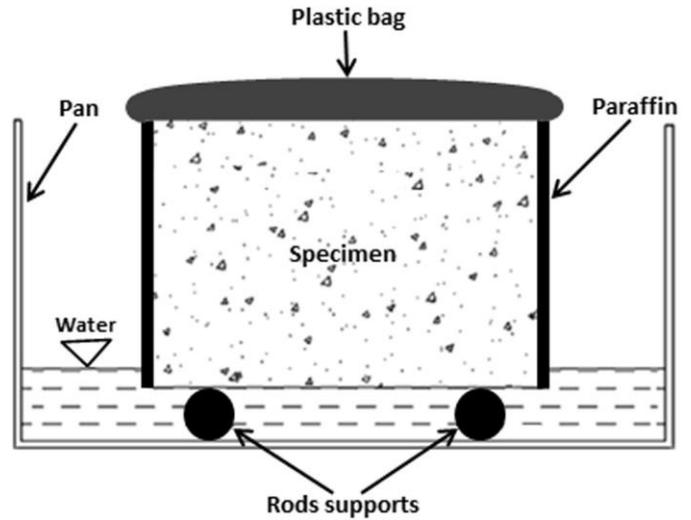
$$P = \frac{W_{\text{sat}} - W_{\text{dry}}}{W_{\text{sat}} - W_{\text{wat}}} * 100 \quad (2)$$

After measuring the UPV and porosity values, these specimens were also used for sorptivity measurement. Measurements of capillary sorption were carried out using specimens pre-conditioned in the oven at about  $50^\circ\text{C}$  until constant mass was achieved. Specimens were cooled down to room temperature.

Test specimens were prepared as shown in Fig. 1 [12,13]. Test specimens were exposed to the water on one face by placing them on a pan. The lower areas on the sides of the specimens were coated with paraffin to obtain unidirectional flow. At various times (such as such as 0, 5, 10, 20, 30, 60, 180, 360 and 1440 min.), the weight of the specimens was measured using a scales. Then the amount of water that absorbed was calculated and normalized with respect to the cross-section area of the specimens exposed to the water. The sorptivity coefficient ( $k$ ) was obtained by using the following equation (3):

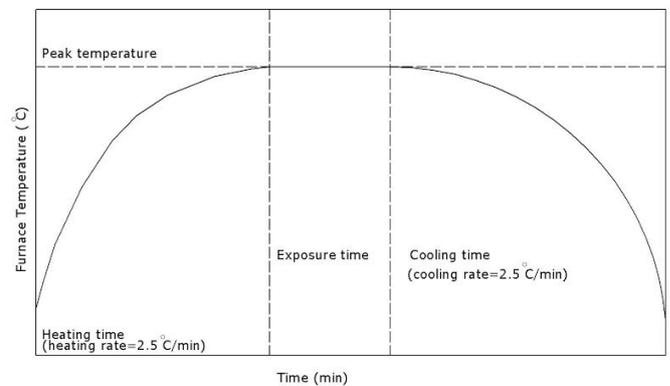
$$\frac{Q}{A} = k \cdot \sqrt{t} \quad (3)$$

Where  $Q$  = the amount of water absorbed in ( $\text{cm}^3$ );  $A$  = the cross-section of specimen that was in contact with water ( $\text{cm}^2$ );  $t$  = time (s) and  $k$  = the sorptivity coefficient of the specimen ( $\text{cm}/\text{s}^{1/2}$ ). To determine the sorptivity coefficient,  $Q/A$  was plotted against the square root of time ( $t$ ), and then,  $k$  was calculated from the slope of the linear relation between  $Q/A$  and  $t$ . This technique has been used in many previous studies [12-16].



**Figure 1.** Experimental setup for sorptivity test [12]

All non-destructive tests such as UPV, porosity and capilarity were made for specimens completed the 28-day curing period. To determine microstructure and strength changes of SLC with MK after high temperature, 28 days cured samples were used. These specimens were dried in an oven at about  $50^\circ\text{C}$  until a constant mass was achieved at the end of 28days. Then, five specimens for each temperature were heated to 400, 600 and  $800^\circ\text{C}$  using a Protherm HLF 150 electrical furnace. The average heating rate of the electric furnace used in the experiments was  $2.5^\circ\text{C}/\text{min}$ .



**Figure 2.** Temperature-time curve heating, exposure, cooling

The specimens were keep at these temperatures for one hour to achieve a thermal steady state. Then, the samples were cooled in the oven by about  $2^\circ\text{C}/\text{min}$ . Fig. 2 represents the temperature–time curve showing heating time, exposure time and cooling time of the peak temperature.

### III. RESULTS AND DISCUSSION

Table 4 presents the physical and mechanical properties of the hardened concrete samples after 28 days without exposure to high temperatures.

As shown in Table 4, the compressive strength of all mixtures improved by the age of concrete irrespective of the MK amount. This is an expected result. But, the rate of the compressive strength development was considered more significant at M15 series compared to the control series (M0). It was observed to be in the

ranges of 108.99%–83.28% for M15 and M0, respectively. This could be due to the pozzolanic reaction of MK which is higher at the 28 days curing ages compared to earlier ages. This results demonstrate compliance with the findings of past studies [17,18]. In addition to this, for all curing ages, the results of compressive strength indicated that these strengths increased with the increasing MK replacements up-to 15% weight per weight. This increase can be attributed to MK particles pores filling effects, the acceleration of cement hydration and the pozzolanic reaction of MK with the calcium hydroxide [19,20].

**Table 4.** Test Results

	Compressive Strength (MPa)			Splitting tensile Strength (MPa)	Nondestructive Testing		
	3 days	7 days	28 days		Ultrasonic pulse velocity (Km/s)	Porosity (%)	Sorptivity coefficient x 10 <sup>-3</sup> (cm/s <sup>1/2</sup> )
	3 days	7 days	28 days	28 days	28 days	28 days	28 days
M0	15.91	17.52	29.16	2.327	3.424	11.01	1.315
M5	16.85	18.25	31.86	2.436	3.484	9.35	0.994
M10	16.97	19.72	31.92	2.637	3.572	8.75	0.990
M15	17.23	21.30	36.01	2.631	3.787	3.16	0.256
M20	18.74	20.95	34.34	2.582	3.610	2.91	0.201

The reduction of the strengths for series of M20 compare to M15 is elucidated as the result of dilution effect which reduces the  $\alpha\text{C}_3\text{S}$  and  $\beta\text{C}_2\text{S}$  main phases in blended cement. The dilution effect is a result of substituting a part of cement with the equal amount of MK. At higher ratio replacements, the MK particles are pelleted around the cement particles and prevent the hydration process. Less hydration products occurs; causing to the improvement of fewer points of contact which act as binding centers between cement particles. Therefore, there is an optimum MK replacement for mechanical strength of concrete containing MK. In this study, since the efficiency appears to reduce over a replacement ratio of 15% w/w can also be accepted as an optimum ratio for MK considering the economic efficiency. This assertion was collaborated in literatures [1, 5, 12, 21]. Also in this case, Morsy and others [6] explains as follows, "when metakaolin is used in higher proportions, and prevents the hydration process.". When the ultrasound results are analyzed in Table 4 it is seen that the series of M15 has an optimum value. This situation is probably due to the following.

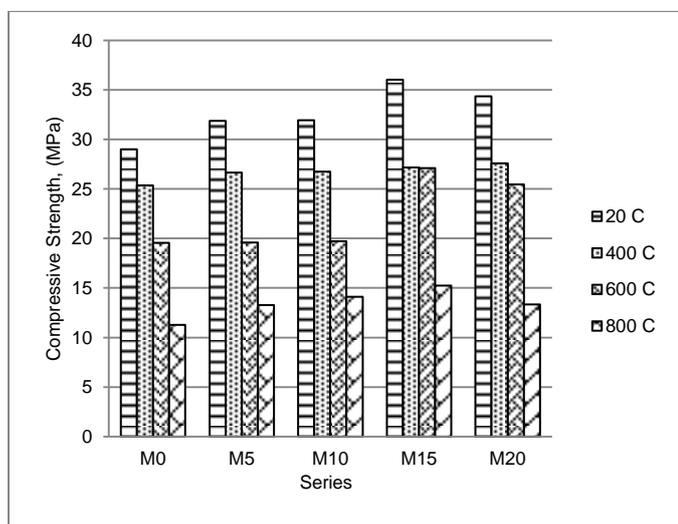
By filling the voids inside the concrete, kaolin having a very thin structure creates a more compact concrete. Thus, ultrasound waves pass more quickly in concrete. An information that metakaolin fills the gaps due to its fine structure is available in the literature. Khatip [22] mentions as the results of his work, MK physical particles provides tightening, that act as a filler which fills the interstices within the structure of the hardened cement mortar. Also, metakaolin produces extra CSH (Calcium Silica Hidrate) and create contact points acting as binding centers between the cement grains.

As seen in Table 4. the porosity and sorptivity values of the series support UPV results. The amount of used metakaolin increases up to 15%, series demonstrated lower sorptivity and porosity. In some sources says that, usage of metakaolin increases the need for water used in the concrete and due to the concrete mortar receives inside enough water during the mixing process, the water absorption ratio of the concrete decreases [23].

Splitting tensile results are parallel to the compressive strength results. Optimum percentage of splitting tensile strength is also 15%. The literature especially

emphasized that tensile strength also increases as if compressive strength with increasing replacement up to 15% by weight [6].

As shown in Fig. 3, the compressive strength values of all series are decreased with increasing temperature. In fact, whatever the content of the concrete, a slight increase in compressive strength was observed up to 400 ° C. This peak value of the compressive strength varies in each study. For example, in a study, It was observed that the compressive strength peaked at the 250° C [6], in another study, it's seen that this value is 200 ° C [24]. In a study of Demirel and Kelestemur's, which is prepared by nine different series of concrete, some of them increase up to 400 ° C and then it shows a sharp decline [13].



**Figure 3.** Relationship of Temperatures-Compressive Strength Series

This situation in the literature explains that, especially before 350 ° C, the degradation of Ca(OH)<sub>2</sub> (CH) doesn't occur, thus there isn't any loss of strength [13,26]. Unlike literature, any strength increase was not observed in this study. In this study, the compressive strength of concrete was investigated for 0 and 400 C. However we estimate that, if the compressive strength was considered more another temperature values (for example 300 ° C or 350 ° C), a peak value certainly could observe. Although the strength loss was observed depending on the temperature increase, especially at the 600 ° C, while the MK percentage was increasing, response rates from high temperatures of the series has decreased. This situation can be seen more clearly in Table 5. For example, at 600 ° C while resistance loss of M5 series 38.49% and resistance loss of M10

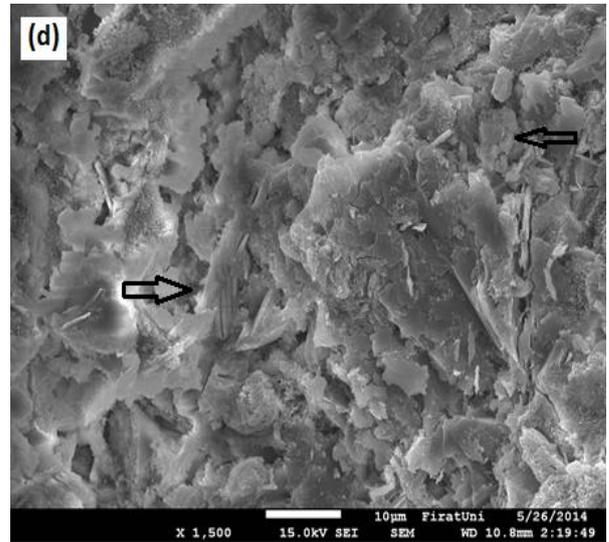
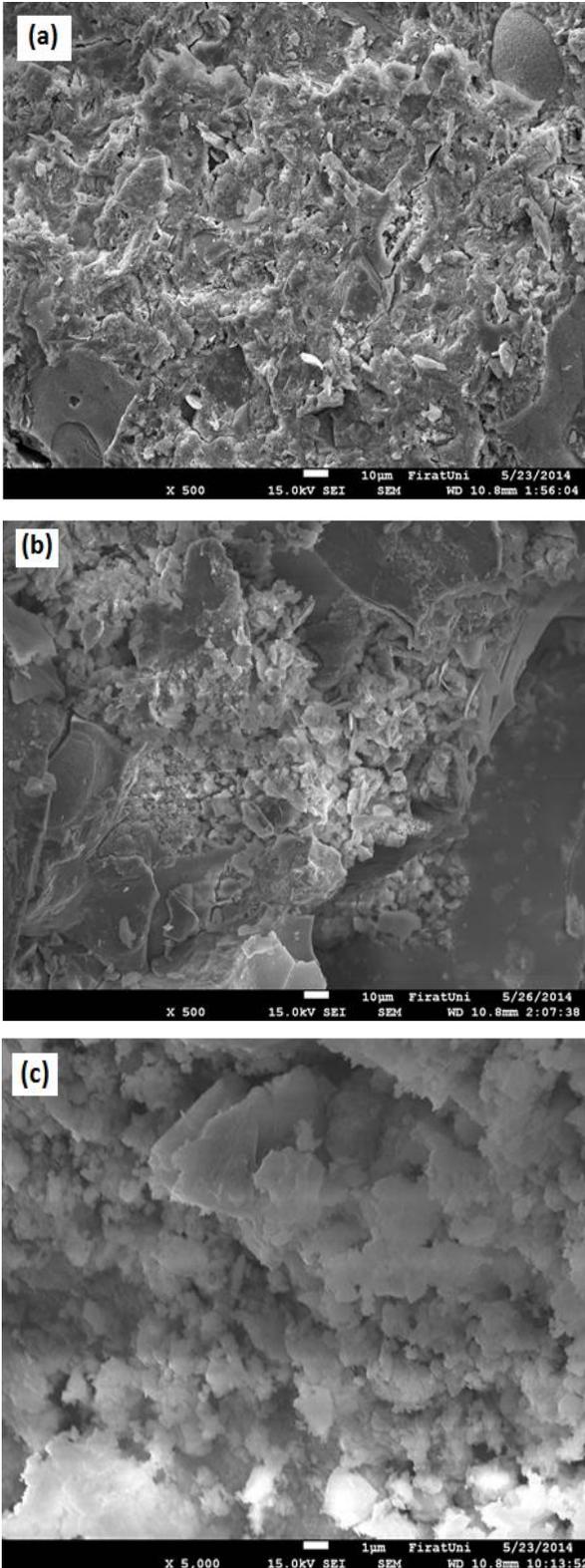
series %37,84, resistance loss of M15 series has dropped to 24.75% level.

**Table 5.** Relative strengths of the specimens

Specimen Code	T (°C)	Compressive Strength (MPa)	Relative Strength (%)
M0	20	29,16	100
	400	25,36	86,96
	600	19,55	67,04
	800	11,29	32,92
M5	20	31,86	100
	400	26,66	83,67
	600	19,60	61,51
	800	13,28	41,68
M10	20	31,92	100
	400	26,74	82,59
	600	19,72	62,16
	800	14,12	44,23
M15	20	36,01	100
	400	27,18	75,47
	600	27,1	75,25
	800	15,23	42,29
M20	20	34,34	100
	400	27,59	80,34
	600	25,47	74,17
	800	13,33	38,81

According to Morsy and others [6], the decrease in compressive strength with increase in temperature above 200 ° C may be due to the dehydration of CH at about 500 ° C producing CaO and H<sub>2</sub>O. Strength loses over 600 ° C, caused by basically decomposition of CaCO<sub>3</sub> and subsequent CO<sub>2</sub> escape from CaCO<sub>3</sub> especially at 600°C, while the amount of MK increases, the response rates of the series from high temperatures decrease. It can be explained as follows: increasing MK reacts with CH which caused by the hydration of cement and it leads to decrease of CH. In other words, CH which will be dehydrated decreases.

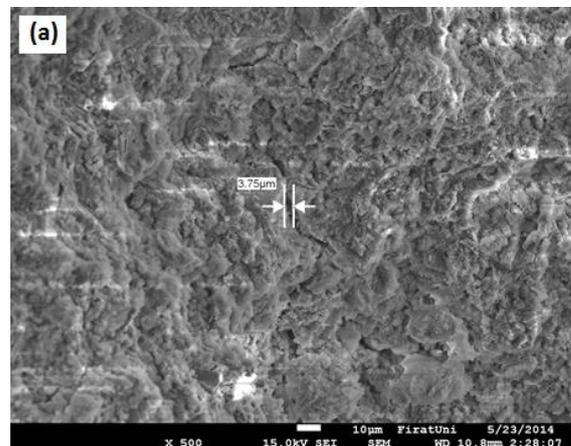
SEM analyses of the SLC specimens before and after elevated temperature were carried out at the Central Research Laboratory of Firat University using a Jeol JSM7001F scanning electron microscope. The microstructure of the series that hasn't been exposed to temperature are shown in Fig.4.

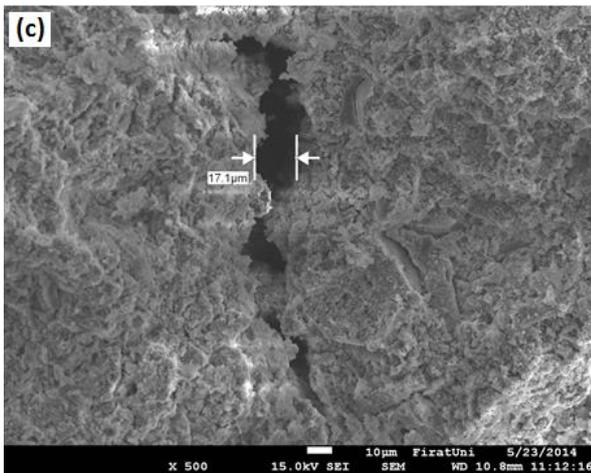
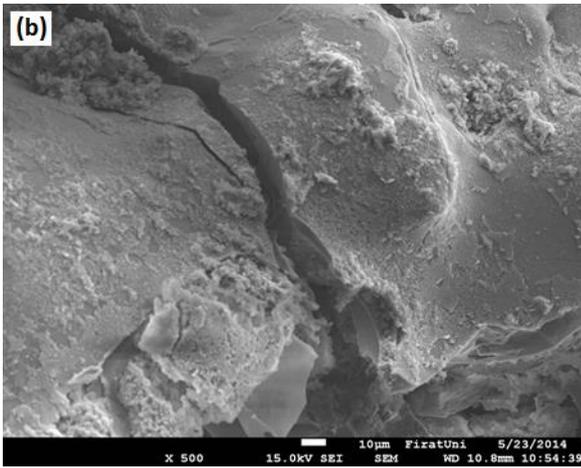


**Figure 4.** SEM at 20 ° C series of images a) M0, b)M5, c)M15, d)M20

When the four pictures in the Fig. 4. is examined; while MK percentage of the series increases, a layered look is emerging. This is consistent with the literature. Because; Kong and others, emphasize that metakaolin particles in the sample is in the form of a sheet-like structure, appears in the form of plaques [25]. In Fig. 4(d), these layers are indicated by arrows.

In Fig. 5, the microscope images of the series exposed to 800 °C are given together. While MK percentage of the series increases, it is obvious that the internal structure of the SLC specimens takes a denser look [6, 26]. At this temperature (800 °C), as a result of CH and CSH deformation the internal structure has been distortions and cracks occurred. While the kaolin percentage increases, it is noteworthy that the the size of the crack grow up.





**Figure 5.** The series exposed to 800 °C a) M5, b) M15, c) M20

#### IV. CONCLUSION

The results obtained from this experimental study that performed are given below.

MK developed the performance of the SLC specimens with MK. The optimum value of 15% was observed clearly in both splitting tensile and compressive strength. The reduction of the strengths for series of M20 compare to M15 is elucidated as the result of dilution effect which reduces the  $C_3S$  and  $bC_2S$  main phases in blended cement. At higher ratio replacements, the MK particles are pelleted around the cement particles and prevent the hydration process.

The increase in the ratio of MK resulted in a rise in ultrasonic pulse velocity value of the SLC specimens, but at the same time it lead to reduce in porosity and sorptivity values. By filling the voids inside the concrete, metakaolin having a very thin structure creates a more

compact concrete. Thus, ultrasound waves pass more quickly in concrete.

The compressive strength values of all series are decreased with increasing temperature. The results were showed that, no matter what the temperature value (400, 600 or 800 °C), M15 series exhibited the highest compressive strength.

When SEM images examined, while MK percentage increases, internal structure of SLC specimens exhibited layered appearance.

SEM images displayed that the microstructure of the SLC specimens containing MK up to 15% w/w was more uniform and denser. SEM images showed that, at 800 °C, as a result of CH and CSH deformation the internal structure has been distortions and cracks occurred.

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