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Mechanical and Thermal Evaluation of Diatomite Doped Fly Ash Based Geopolymers

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In this work, the properties of alkali silicate geopolymer type materials and diatomite as additive to fly coal ash was investigated using thermic coal plant's fly ash in alkaline solution. The reacted products of alumino-silicate geopolymers using fly ash plus diatomite were pseudo-amorphous aluminosilicate gel and calcite and their mechanical and thermal properties were evaluated by the addition of diatomite. The compressive strength of this geopolymer is similar to that of the Portland cement mortar of PC20 (± 20 %) when cured for 28 days while the density and thermal properties are much lower that indicates the insulator properties. Alkaline solution was produced by NaOH in different concentrations to determine the least alkaline solution molarity in the range of 1 M, 3 M and 5 M. The characterization of geopolymers were done by using XRD for phase analysis, SEM for surface and morphological evaluation, compression tests for mechanical properties and transient plane source thermal analysis for thermal insulation properties. The results showed that, 1 M of NaOH alkaline solution and 10 wt.% diatomite addition can provide enough strength of 18 MPa which is a good candidate for constructional materials. The thermal conductivity coefficient of 10 wt.% diatomite added geopolymer was evaluated as 0.0018 W/m·K which can also be a good candidate for insulator materials to be used in green lateral wall production.

Keywords: diatomite, fly ash, geopolymer, thermal conductivity, mechanical properties.

1. INTRODUCTION

Geopolymers are of great interest to researchers due to their unique properties such as low density and high thermal insulation properties and amorphous structure of high porosity. The main importance of being geopolymer is Si:Al ratio by their ionic strength [1].

The main problem of the Earth is the climate changes today and it is well known that the majority of climate changes are related to the burning of fossil fuels and production of cementitious materials. Thus, either the human dependence on these energy sources or materials which are the product of these sources must be reduced or their wastes must be reused. Some renewable sources like wind and solar are being tried to be used instead of fossil fuels and also, they gave hopeful results for producing energy with minimum natural impact. But in the cementitious materials case, there have not been many remarkable researches for the alternative materials for the production of building products. Up to now, geopolymers are seen as the best solution for the problems mentioned above. On the one hand, the low temperature of geopolymer production and on the other hand reusing of wastes like coal fly ash makes geopolymers which are giving hope of being alternatives of cementitious concretes, popular in constructional research field. And also, geopolymers, have capability of restrain CO_2 emission, low shrinkage, rapid strength development and high fire resistance [1 – 3]. In 2010, the CO_2 emission of the cement production is 9.1 % of the world's total emission. The percentage CO_2 emission of cement production is lowered while the emission value of this process is getting higher by the time [4]. For this reason, some alternatives, which are eco-friendlier, must be considered instead of cement.

Geopolymer synthesis is a very simple and inexpensive process. Geopolymerisation process is mixing of an aluminasilicate feedstock with an alkaline activator solution (usually alkali metal silicates or hydroxides) [5, 6]. The aluminasilicate source materials are in a wide range such as metakaolin (calcination product of kaolinite) [7], clay [8] and other raw materials [9] or industrial wastes like coal fly ash [10], lignite bottom ash [11], metallurgical slags [12], red mud [13] and etc.

It is well known that the coal power plants which use coal for the production of electricity, stock their wastes like fly ash in an area that belongs to that plant. And usually that waste stocks grow by the time and reach a hill height. Fly ash has very small particles and can fly with wind and affect the health of nearby livings by inhaling [14 – 18]. For this reason, with a cooperation with thermic plants, these fly ashes must be used in construction or another fields.

In this work, we have tried to utilize Kangal Coal Fly Ash into various geopolymer systems, which are synthesized in different conditions. The main aim of this project is using the coal fly ash in Turkey which is being produced about 15 million tons annually [19]. With a concrete which have average compression strength of 5 MPa, lock stones or asmolen bricks can be made. And a production for whole country can eliminate these fly ash hills and following fly ash wastes, which are produced freshly from the thermic plant, can be used instantly. Further researches can be done in this case but it seems the best way for eliminating fly ash problem, is producing fly ash based

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geopolymer products for applications that do not need high compression strengths.

There have been used different additives (aggregates or activator solutions) in their mud or pressure to make more compact structure or different curing conditions [20]. But it must be well understood that the cheapness and the maximum fly ash usage. Expensive additives, aggregates, pressure or curing conditions which have high energy consumption were refused to be used [21]. Coal fly ash components are very various due to the differences in source materials of coal-using power plants. Thus, the fly ash based geopolymer properties are dependent on region. Different geopolymer systems with different mix proportions and curing conditions are tested for understanding the best ways for producting concretes for various applications [22 – 25].

In geopolymerisation, when the alkaline solution immersed into the fly as or aluminasilicate source, water evaporates by the time and leaves AlO_4 and SiO_4 tetrahedras and an amorphous structure which shares O with tetrahedrals to bond with them, between AlO_4 and SiO_4 tetrahedras [23 – 26]. There are three common geopolymer structures: polysialate Al-O-Si chains, polysialate siloxo Al-O-Si-Si chains and polysialate disiloxo Al-O-Si-Si-Si chains [25 – 27]. C-S-H and C-S-O-H bondings are observed in XRF spectra due to CaO content of fly ash we used and these C-S-H and C-S-O-H bondings are enhancing the mechanical properties of fly ash based geopolymer concrete [26 – 28].

2. MATERIALS AND METHODS

2.1. The characteristics of fly ash and diatomite

The fly ash used in this study was kindly supplied from Kangal Thermic Plant Co. Ltd. X-Ray fluorescence (XRF) (Thermo Scientific, Niton XL3t, Hitachi, Japan) analyze results are given in Table 1 and X-Ray diffraction (XRD) (Rigaku D-Max II, Japan) patterns which are taken with a Cu Kα radiation of 1.5406 Å, are given in Fig. 1. The step size and step time was adjusted to 0.02 and $1° \cdot min^{-1}$, respectively. In XRD patterns of Kangal fly ash main peak is identified as $CaSO_4$ anhydrite. Powder morphology was irregular to round particles with an approximate particle size of 106 ± 38 microns range as sieved and analyzed by scanning electron microscopy (SEM, TESCAN® Mira3 XMU, Brno, Czech Republic).

Table 1. XRF analysis results of Kangal fly ash

Element	Concentration, wt.%
MgO	3.4480
Al_2O_3	22.382
SiO_2	24.659
SO_2	11.557
K_2O	1.3452
CaO	29.138
Fe_2O_3	7.4595
Total	100.000

As seen from XRF, the main ingredients of the mixture were $CaSO_4$ and $(Fe, Al)_2O_3 \cdot (Mg, Ca)O \cdot SiO_2$. Oxide compounds were found as CaO, $CaSO_4$, $SiAl_2O_5$, $(Ca, Na_2)O \cdot (Al, Fe)_2O_3 \cdot SiO_2$ and some other species in minor amount. Since the CaO content is high, this fly ash can be classified as Class C type of lower silicate but higher CaO due to the calcite content around coal mine. A low amount of MgO may not be seen in phase analysis due to somehow dissolution in a Ca-Na-Al-Si-OH related crystal lattice structure as well as Fe_2O_3. The reason for iron (III) oxide not to be seen in phase analysis may be attributed to substitutional solid solution with alumina in anorthite. Since alumina is the polymorph of iron oxide, Fe and Al can share the similar sites in crystals without strain in A or B sites.

The main peaks in Fig. 1 are observable to be CaO, $CaSO_4$, SiO_2, anorthite which is Na rich ordered structure. These structures are seen as water free due to the fired state of powder right after the blowing of calcite into chamber. The peaks are very sharp that indicate the highly crystalline structure and high volumetric crystals. The high-volume crystals are found where many cations were combined with various anions in different ionic sizes as S, O, C, H. Since the firing column of thermic plant was blown with $Ca(OH)_2$, it reacts with the sulphur and produces a $CaSO_4$ named as anhydrite due to the firing of gypsum that dehydrates at around 900 °C. CaO is evident to form by the dehydration of plaster. The possible presence of $CaCO_3$ in small amounts may be attributed to the low firing temperature of coal. Anorthite can easily be found in almost all kinds of fired ash due to glassy phase formation by relatively high temperature and then cooling slowly to form an ordered structure that may be capable of dissolving many other trace compounds in it. These compounds can be identified as iron oxides, magnesia, very small amount sodium to produce the glass phase to melt and then cooled down to form anorthite structures. Beyond that, SiO_2 as quartz can also be seen by the dissolution of land silicate that helps geopolymer formation to initiate tetrahedral SiO_4^-.

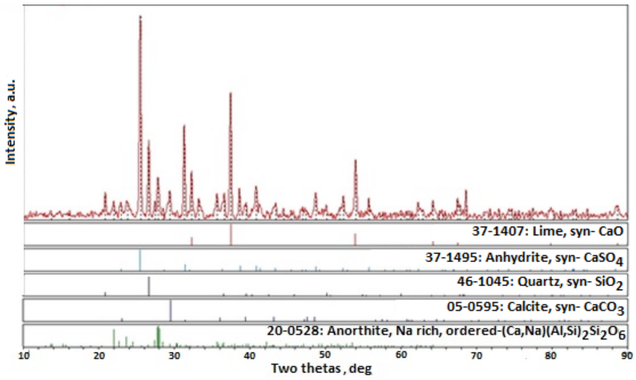

Fig. 1. XRD patterns of Kangal Thermic Coal Plant fly ash

Fig. 2 indicates the SEM-SE-BSE images of powders of interest. The spherical and sphere like particles with some irregular shaped very fine sized particles in lower magnification of fly ash image. The spherical shaped particles were related to fly ash products of $CaO\text{-}CaSO_4$ phases while fine sized particles are seen as silicates and anorthite type Ca-Al-Si-O structures. The loss of ignition of powders was negligible as about 0.59 ± 0.15 wt.%. The high magnification inset image also shows the fired products with a variety of seeds agglomerated that may be due to firing of particles to form hard and diffused agglomerates. The seeds are evident to be about lower than 1 μm with agglomerated fired powders of 10 – 15 microns. Diatomite powders were very characteristic of spheroid powders with an average particle size of 1 – 5 microns with some 10 microns round particles. The inset picture shows about 1 micron of particles with very high amount of seeds of silicates and calcite by volcanic explosions during formation. The greyish particles are known as silicates, while white ones are calcite due to density discrepancy of electron beam on BSE detector.

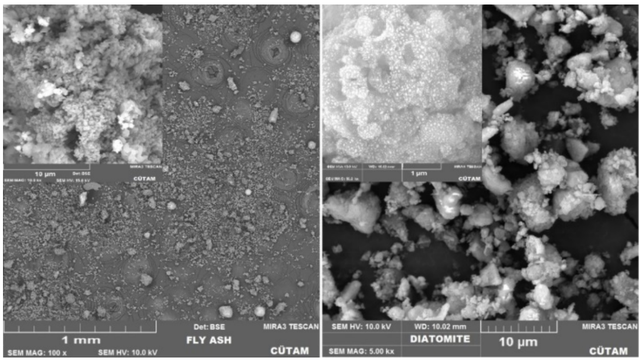

Fig. 2. SEM images of fly ash and diatomite (Inset pictures indicate the higher magnification of powders)

The diatomite was received kindly as test material from Mineral Research and Exploration General Directorate of Turkey (MTA). Powder morphology was mixed that was characterized as irregular and round-like particles with very tiny seeds. The approximate powder size was 8.3 ± 2.5 microns as seen from SEM analysis. As understood from the powder sizes, the diatomite is the filler material of fly ash based geopolymers while fly ash is expected to be puzzolanic to form the exchange of ions for concrete maturation.

Table 2 represents the elemental analysis results of diatomite.

Table 2. XRF analysis results of diatomite

Element	Concentration, wt.%
MgO	0.197
Al_2O_3	1.061
SiO_2	84.34
SO_2	0.069
K_2O	0.226
CaO	13.66
Fe_2O_3	0.324
Total	100,000

As clearly been evident, the diatomite is the source of silica to form a geopolymer structure more efficiently while producing more alkali reaction that can be resulted in more pore formation in structure. This phenomenon can increase the mass transfer and more amorphous geopolymer structure to improve thermal stability but limited strength. As seen from Fig. 3, the diatomite is a mixture of calcite ($CaCO_3$), quartz (SiO_2), and montmorillonite (an expandable and exfoliating phase with layered Ca-Al-Fe-Si-O related high volumetric crystal.

2.2. The mixture preparation process

NaOH used for geopolymerization was purchased from Sigma-Aldrich GmbH and it is specified as analytical of 99 % NaOH content. The water used for solutions were tap water. NaOH was adjusted as 1 M, 3 M and 5 M batches to be used as alkaline activator of geopolymers by weighing the appropriate amount of NaOH pellets.

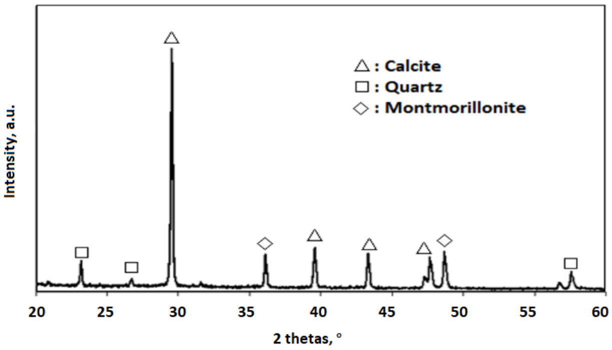

Fig. 3. XRD pattern of diatomite (a mixture of calcite, quartz and montmorillonite)

The main motivation for this paper was to reduce the alkaline solution consumption by adding a silica source to be reacted by fly ash to produce more rigid geopolymer structure with desired chain formation. The powders were weighed precisely of 0.01 g on a precision weigher (Mettler Toledo, Ohio, US). The diatomite and fly ash powders were weighed in dry basis and total powder mix was combined by activator and water as seen in Table 3. The pre-experiments were observed for geopolymer formation and after maturation; the diatomite ratio was kept between 10 wt.% – 30 wt.% of dry fly ash amount.

Table 3. Calculated and added amounts of materials and their ratios in mortar

NaOH ratio, %	NaOH wt., g	Fly ash wt., g	Diatomite wt., g	Calc. solid ratio, %
40	33.34	45	5	10
40	37.50	45	11.25	20
40	42.86	45	19.29	30

100 wt.% fly ash based geopolymerisation was done and kept as reference. The water was adjusted to 30 wt.% of dry total powder mix to evaluate the effect of water in geopolymerisation process and its structural properties. The oven (Electromag, Turkey) was adjusted to 40 °C and 80 % of relative humidity. The geopolymerisation time was 24 h for all tests. The product was put into oven by wrapping it into a seal made of silicon-based polymers. Then, the material was taken out from oven and unwrapped to stay at room temperature for another one day to finish geopolymerisation and begin maturation.

Table 3 indicates the related amounts of material added into the mortar. NaOH amount was kept constant but the molar ratio itself was changed as 1 M, 3 M and 5 M. The amount of diatomite divided by the total amount of fly ash and diatomite together gives the ratio of solid loading as from 10 % to 30 %. NaOH amount was also calculated in the same way by dividing NaOH amount in weight to the total weight amount of NaOH and powder mixture as to be constant as 40 % of mortar.

The mechanical properties of materials were measured by Shimadzu Trapezium (Japan) with a crosshead speed of 0.1 mm·min^{-1} and the thermal properties were recorded by HotDisk® TPS 3500 model (Sweden).

2.3. Measuring the thermal conductivity

The thermal conductivity of geopolymer materials were measured by HotDisk® TPS 3500 model (Sweden), a hot disk heat measurement device. Kapton® probes with a radius of 3.2 mm (5465 type) were measured in one dimensional mode of device which is suitable for materials that has height to diameter ratio bigger than 5. So, the rod like structures can be measured by putting the probe between two same type materials of interest with an insulating chamber. The room temperature of 23 ± 1 °C was adjusted for measurement compensations. The device works for meeting the ISO 22007-2 for transient plane source measurement.

The TPS theory makes an assumption as the sensor is in an infinite medium of sample, and the probing depth (penetration of heat) do not exceed the boundaries of the samples. Thermal diffusivity of a material will determine probing depth during measurements. The corresponding Eq. 1 is given below for an infinite medium according to probe radius and power divergence of hot disk [31].

$$\Delta T_s(\tau) = P_0 \left(\pi^{\frac{3}{2}}.r.k\right)^{-1}.D(\tau), \qquad (1)$$

where $\Delta T_s(\tau)$ is the temperature increase of the specimen surface; P_0 is the power output of the sensor/probe; r is the radius of the outermost ring source; $D(\tau)$ is the dimensionless time delay function variable while τ is the system delayed correction time evaluated by device. k, which is known as thermal conductivity of material of interest is calculate from the equation.

The device is presented in Fig. 4 a and b is the Kapton® sensor of device coded as 5465 having a radius of 3.2 mm, Fig. 4 c is the specimen geometry to be put onto each other where Kapton sensor was placed between the specimens of same type, Fig. 4 d shows the device measurement set up prior to covering with steel environmental shield.

Fig. 4. a – the real time picture of device; b – Kapton sensor; c – one dimensional mode specimen geometry; d – measurement set up

3. RESULTS AND DISCUSSION

3.1. The morphological analysis of geopolymer batches

SEM was employed for morphological analysis of geopolymer batches of maturation for 28 days. Fig. 4 represents the surface features of geopolymers, pure and diatomite added materials. Fig. 5 a shows the pure geopolymer mortar after 28 days with fly ash where 1 M NaOH used as activator. The fully reacted fly ash particles with Na related reaction areas are clearly seen. The ellipses indicate the reacted regions with Na-Ca hydroxides with spherical fly ash particles accompanied to reaction by maturation.

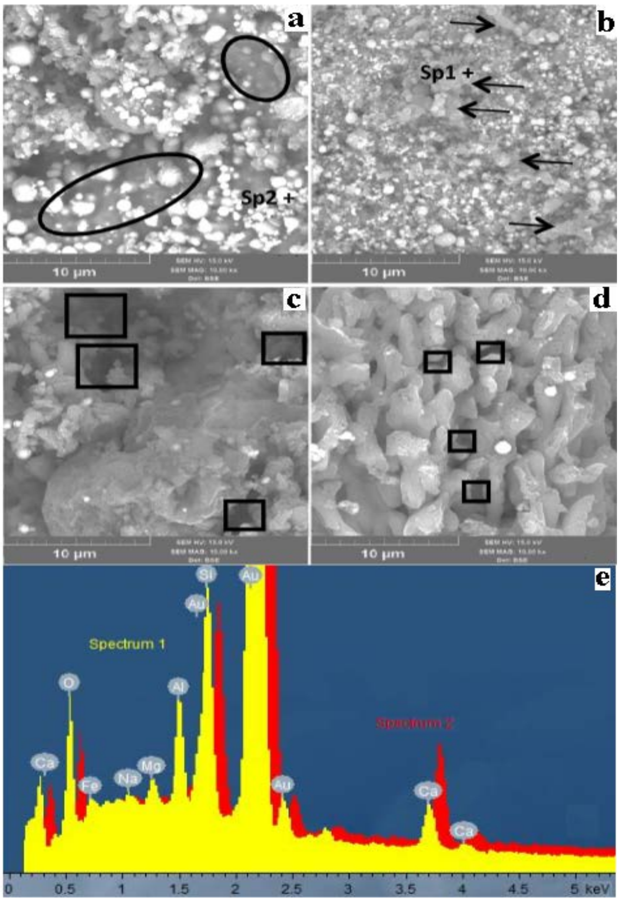

Fig. 5. SEM-SE images of: a – pure geopolymer (GP); b – 10 wt.% diatomite added GP (D10GP); c – 20 wt.% diatomite added GP (D20GP); d – 30 wt.% diatomite added GP (D30GP); e – corresponding EDX spectrum of Sp1 and Sp2 as seen in (a) and (b). Black arrows stand for added diatomite, ellipses stand for fully reacted GP areas around and under fly ash particles, big-small squares stand for formed coarse and fine pores

When 10 wt.% diatomite is added to geopolymer (D10GP) mortar for Fig. 5 b, the reaction slows down or some unreacted regions were seen. The black arrows indicate the diatomite particles above the fly ash particles without any Na-Ca reaction. A small amount of pore formation has begun to take place due to lack of reaction. But, the main structure without large pores remains with fine particle size of fly ash. These fly ash particles seem to be fully reacted and very fine Na-Ca rich regions were seen. The diatomite is evident to partially react with fly ash particles and seeded to precipitate on probably Ca rich round particles. The diatomite may react with Na activator but not enough for full reaction even in 28 days. The activator contact areas are evident with calcite and quartz regions of diatomite which is not sufficient for montmorillonite mixtures. Fig. 5 c represents 20 wt.% diatomite added geopolymer mortar, there has been an increasing size of pores with some reacted and agglomerated fly ash particles. The squares show the pores coarsened and by the addition of diatomite, the reaction regions were decreased and pores got bigger. As known from the feature of backscattered image analysis of SEM (SEM-BSE), the whiter the electron beam seen, the higher the compound crystal densities, which indicate the presence of fly ash particles were found.

As seen from Fig. 4 e, in EDX spectrum 1 and 2, higher Ca content was evaluated as fly ash while low Ca but high Si containing oxide mixture was evaluated as diatomite regions with an approximate mixture of CaO, SiO_2 and montmorillonite as described by XRD. As illustrated by SEM and found from EDX, whiter regions are fly ash while greyish ones are seen as diatomite. These regions are getting less by increased diatomite amount. Within the increase of diatomite, the pores also get coarsened to result in lack of reaction.

3.2. The mechanical properties of geopolymer materials

Fig. 6 shows the mechanical property evaluation of geopolymers as mentioned in graphs.

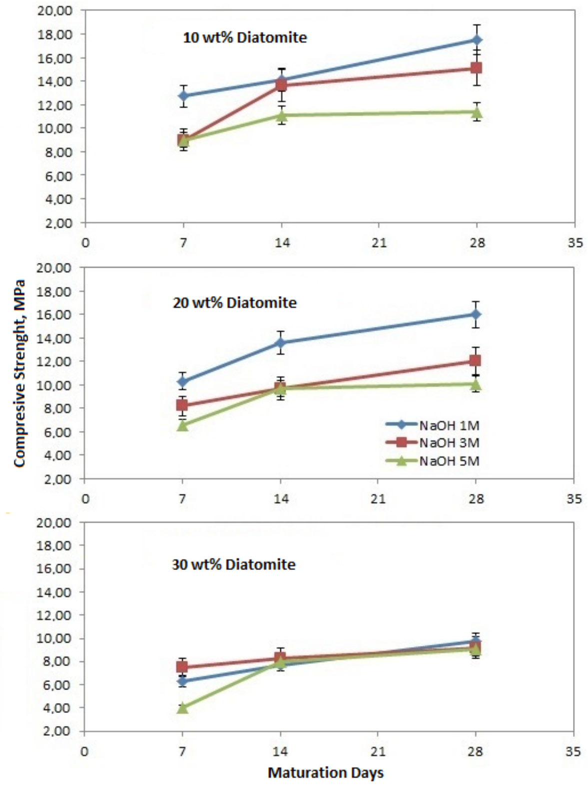

Fig. 6. The compressive strength change by increasing maturation time of geopolymers

The reference geopolymer had a compressive strength of 22 MPa, which was not seen in the graphs. Since the minimum limit of PC20 is around ± 20 % of 20 MPa. The lowest limit is 16 MPa, 10 wt.% of diatomite with 1M NaOH seems to have a compressive strength of 18 MPa and the increasing molarity of NaOH decreases the compressive strength of mortars after 28 days of maturation. The diatomite addition of 20 wt.% has also given compressive strength of 16 MPa that goes down to 12 MPa and 9 MPa with increasing molarity of NaOH in mortar with forwarding days of maturation. By increased diatomite addition to 30 wt.%, decreases the overall compressive strength of mortar even for 28 days of maturation. If the maturation times were thought, all samples improved the mechanical properties but when the molarity was thought, increasing molarity decreases the mechanical properties for all samples.

The diatomite addition decreased the strength due to highly porous structure of diatomite which in turn increases the total open porosity of geopolymer material while favoring the rupture. The limit addition of 20 wt.% was attributed to the mixture of quartz, calcite and montmorillonite as well. The quartz and calcite intend to increase the mechanical property while montmorillonite interfere with the property stability due to high water adsorption and swelling of montmorillonite which makes the material more water containing and collapse the water particles inside the body. This water elongates the period of maturation as well as restraining the pore closure and reactions. The lowest diatomite addition is seemed to be more stable and low energy consuming within low amount of NaOH addition (1 M).

As illustrated from Fig. 5, in SEM photos, the increasing pores by increasing diatomite decreases the mechanical properties due to low amount of connection between particles with highly unreacted regions from about 18 MPa down to 8 MPa. The occurrence of unreacted regions may be attributed to the increased amount of montmorillonite in diatomite by high amount of water dissolved in the body that restrained the reacted area increase. The lower the activator addition, the shorter the maturation with high compressive strength occurs. These advantages give possibility of high usability in application areas such as road construction, lock stone production, panel wall manufacturing for lateral forces.

3.3. Thermal conductivity of geopolymer materials

As in Fig. 7, the increasing molarity increases the mechanical strength of materials but also increases the thermal conductivity of geopolymers for 1 M NaOH added 10 wt.% diatomite doped fly ash based geopolymers [30].

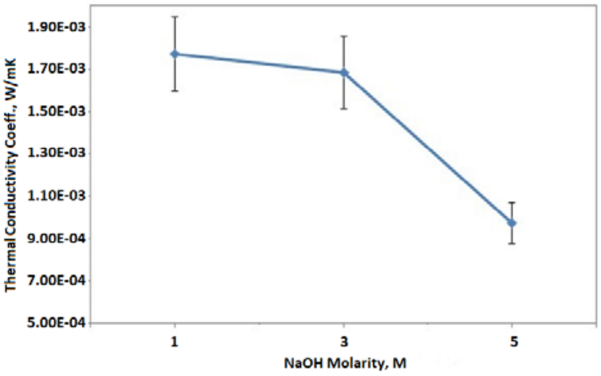

Fig. 7. The effect of NaOH molarity on the thermal conductivity coefficients of different diatomite added geopolymer materials after 28 days of maturation

Since the porosity of materials for lower molarity decreases, the mechanical properties increase while the air bubble probability of material decreases which in turn makes the thermal conductivity coefficient higher. The highest thermal conductivity coefficient was evaluated as 0.0018 W/m·K for 1 M NaOH added 10 wt.% diatomite doped geopolymer structure. In contrary, higher molarity of

NaOH up to 5 M, decreases the thermal conductivity coefficient down to 0.0009 W/m.K which makes a promising candidate material with an acceptable mechanical strength about 18 ± 2 MPa for 10 wt.% diatomite added fly ash geopolymers in the mentioned conditions. Similar trends were found for 20 wt.% and 30 wt.% diatomite added geopolymers as seen in graph. The decreased compression strength by increased pores resulted in decreased thermal conductivity coefficient for both 20 wt.% and 30 wt.% diatomite added geopolymer structures. This phenomenon is very well known by increased closed pores improves the thermal insulation properties while decreasing the strength by production of stress intensity and crack propagation areas. An average of 0.001 W/m·K of thermal conductivity coefficient values for almost all molarity value can be a good candidate but deterioration in compression strength due to excess amount of diatomite in structure compasses the reaction areas. Since the highest strength geopolymer for doped materials is 10 wt.% diatomite added ones, the thermal conductivity coefficient was as low as the known good insulators to be a good candidate for construction wall panel materials [31, 32].

4. CONCLUSIONS

The waste material management has attracted an interest for researcher. The waste residue fly ash of a thermic plant was attempted for utilization as non-load bearing construction materials. To produce geopolymers by doping a silicate source as diatomite was evaluated by means of mechanical and thermal properties. The following derivations can be concluded from this study:

1. The geopolymer mortars were produced and materials were manufactured in humid environment. By increasing maturation time, an acceptable and measurable strength was obtained to a certain extent. The maximum strength was found as much as 18 ± 2 MPa which is very close to PC20 as candidate materials for construction wall panel product;
2. The increasing NaOH molarity decreased the mechanical strength while decreasing the insulation properties. Improved pore amount enhances the thermal properties as being more insulator. The pore size can be controlled by alkaline molarity that can be adjusted from a few microns to ten microns by ion exchange capability and reaction possibility;
3. High volumetric pore-containing and average compressive strength geopolymer materials were obtained successfully. The better the compressive strength, the higher the thermal conductivity coefficients can be obtained;
4. XRF, XRD and SEM analysis and elemental distribution, phase formation and pore structure as well as grain morphology were evaluated, respectively. Class C type high Ca and sulphate rich powder doped with diatomite was produced;
5. A good candidate material for construction wall panel for lateral forces is well established for low energy consuming future generation buildings without any cement used.

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