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Microstructure Characterization of Fly Ash-Based Geopolymer Mortar with Substitution of Oil Palm Ash

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Abstract. Geopolymers were materials produced from the polymeric aluminosilicate geosynthesis and alkali-silicate that produce of a polymer fabric of SiO_4 and AlO_4 which were tetrahedral tethered. This paper presented the contents and microstructures of fly ash and oil palm ash geopolymers. The composition were mixture with fly ash ratio: oil palm ash = 100%: 0%, 75%: 25% and 50%: 50%, activator ratio: precursor = 0.417, NaOH ratio: Na_2SiO_3 = 2.0 with a molarity NaOH solution, of 8 M, 10 M, 12 M, 14 M and 16 M. The stages of this research include the preparation and testing of materials were used, preparation of alkali activator solution, mix design and casting process, maintenance and testing of Fresh mortar include testing of slump flow and setting time, while hard mortar includes mortar weight testing, compressive strength testing, and microstructure testing. Tests were performed at 3, 7, 14 and 28 days. The results of test was obtained a maximum compressive strength about 56.06 MPa on the composition of the FA mixture: AS = 100: 0-16 at 28 days. The microstructure of the geopolymer mortar were influenced by the concentration of NaOH solution and the application of oil palm ash.

INTRODUCTION

Geopolymer was the result of polymerization process from an organic material having silicone and aluminum elements in fly ash, metakaolin and blast furnace slag [1, 2]. Geopolymer concrete with fly ash precursor has excellent properties and was suitable for precast concrete, rehabilitation, and reinforcement of structures after natural disasters [3]. The addition of oil palm ash as a precursor of geopolymer mortar by 20% makes the compressive strength increased [4]. The microstructure test results showed mortar geopolymer with 100% fly ash mixture was denser and there was an addition of pore in the addition of oil palm ash. At a temperature of 800 ° C can be seen the geopolymer mortar morphology becomes brittle [5]. The ratio between Na2SiO3: NaOH affects the properties and microstructure of the geopolymer mortar. The test results of the compressive strength of the paste and the mortar geopolymer obtained the optimum compressive strength on the composition of the mixture with the ratio Na2SiO3: NaOH = 2 [6, 7]. This research was carried out as furthes research microstructure mortar geopolymer fly ash and oil palm ash.

MATERIAL AND METHODOLOGY

Materials was used include precursors, activators, fine aggregates, and quads. The precursor used was oil palm ash. The activator alkali solution used was Na_2SiO_3 and NaO. The composition of the mixture in this study by comparing fly ash (FA) and oil palm ash (AS) as a precursor with FA ratio: AS = 100%: 0%, 75%: 25% and 50%: 50%. While the molarity of NaOH solution = 8 M, 10 M, 12 M, 14 M and 16 M. Variable remain in this research was ratio Na_2SiO_3: NaOH = 2, activator ratio: precursor = 0,417 and ratio of fine aggregate: precursor = 2. The composition of the mixture can be seen in the Table 1.

The production of an alkaline solution of the activator begin with making the NaOH solution base to the planned molarity. Weigh the NaOH to the molarity plan then dissolve into the aquades, stir until blended and let stand

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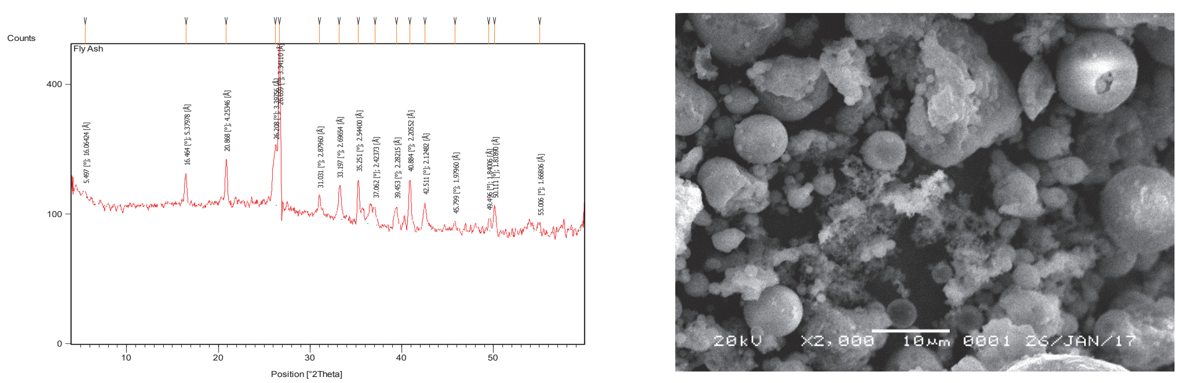
for ± 24 hours in a closed place, because when the process of mixing the temperature becomes hot. The oily NaOH was then mixed with Na_2SiO_3 in accordance with the planned ratio, stirring until smooth and then let stand for ± 24 hours until the temperature of the solution returns to normal, because when the temperature mixing becomes hot. Production of mortar geopolymer mixture begins with stirring dry precursor and fine aggregate for 3 minutes with speed 1, then input the activator solution according to the planned ratio stir back in 5 minutes with speed 2, then add speed mixer with speed 3 for 5 minutes. The geopolymer mortar mixture was ready to be insertet into the mold of the specimen. The test specimen used for the cube was 150 mm x 150 mm x 150 mm. The treatment was carried out at 90 ° C. for ± 24 hours, then the treatment was carried out by inserting the specimen into the plastic bag until the age of the test. Tests performed include slump flow, setting time, mortar weight, compressive strength, and microstructure.

TABLE 1. The composition of the mixture mortar geopolymer

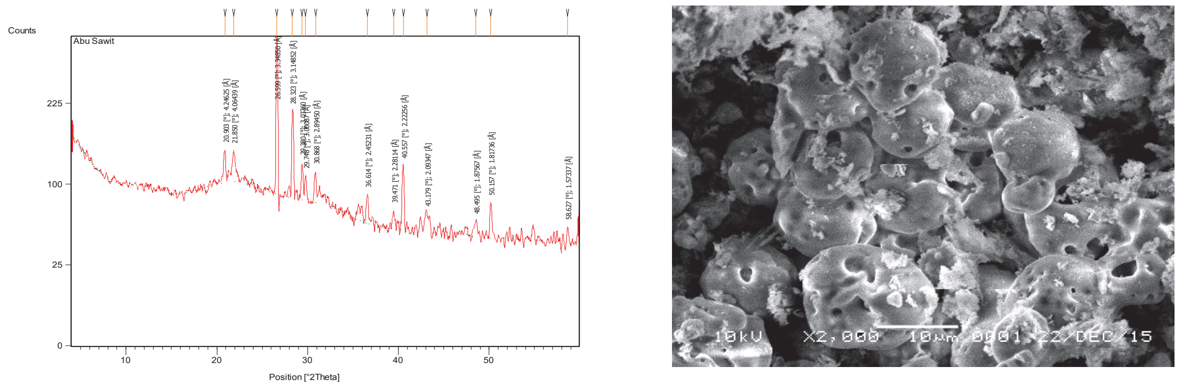
Code	*Fly ash* : Oil palm ash	Molarity NaOH	Na_2SiO_3 : NaOH	Activators : *Precursor*	Fine aggregates: *Precursor*
FA:AS-100:0-8	100%:0%	8 M	2,00	0,417	2,00
FA:AS-100:0-10	100%:0%	10 M	2,00	0,417	2,00
FA:AS-100:0-12	100%:0%	12 M	2,00	0,417	2,00
FA:AS-100:0-14	100%:0%	14 M	2,00	0,417	2,00
FA:AS-100:0-16	100%:0%	16 M	2,00	0,417	2,00
FA:AS-75:25-8	75%:25%	8 M	2,00	0,417	2,00
FA:AS-75:25-10	75%:25%	10 M	2,00	0,417	2,00
FA:AS-75:25-12	75%:25%	12 M	2,00	0,417	2,00
FA:AS-75:25-14	75%:25%	14 M	2,00	0,417	2,00
FA:AS-75:25-16	75%:25%	16 M	2,00	0,417	2,00
FA:AS-50:50-8	50%:50%	8 M	2,00	0,417	2,00
FA:AS-50:50-10	50%:50%	10 M	2,00	0,417	2,00
FA:AS-50:50-12	50%:50%	12 M	2,00	0,417	2,00
FA:AS-50:50-14	50%:50%	14 M	2,00	0,417	2,00
FA:AS-50:50-16	50%:50%	16 M	2,00	0,417	2,00

Initial tests are XRF, XRD and SEM precursor. Based on XRF test results, fly ash contains elements of $SiO_2 + Al_2O_2 + Fe_2O_2$ = 88.82%> 70%, SO_3 = 0,51 and CaO = 3.33 <10%. Fly ash used in this research was fly ash type F in accordance with ASTM C-618. Element of $SiO_2 + Al_2O_2 + Fe_2O_2$ = 88,82%. XRF test results, oil palm ash containing $SiO_2 + Al_2O_3 + Fe_2O_3$ = 59.32% <75% ASTM C-618 requirement. The results of XRD fly ash and oil palm ash testing can be seen in Figures 1 and 2.



(a) XRD (b) SEM

FIGURE 1. XRD and SEM fly ash test results



(a) XRD (b) SEM

FIGURE 2. Results of XRD and SEM test of oil palm ash

Based on Figure 1 it can be seen that the surface of fly ash grains was denser than oil palm ash. The shape of fly ash was perfect but the rounded oil palm ash but not irregular. This make the mixture of use fly ash workability higher than the mixture using of oil palm ash as a precursor.The results of XRD fly ash and oil palm ash testing showed more dominant amorphous structures than crystal structures. This makes fly ash and oil palm ash easier to react than other elements. With a high molarity of NaOH solution, fly ash and oil palm ash become more reactive to form a solid bond between fly ash and oil palm ash with an activator alkali solution.

RESULTS AND DISCUSSION

Fresh Mortar Test Results

Slump flow testing was done to determine the thickness of mortar geopolymer mixture. The results of slump flow testing for all compositions of the mixture can be seen in Figure 3.

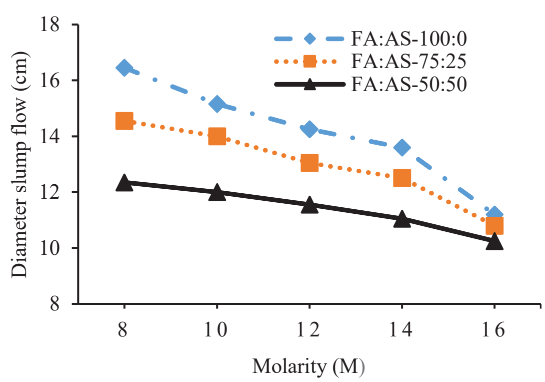
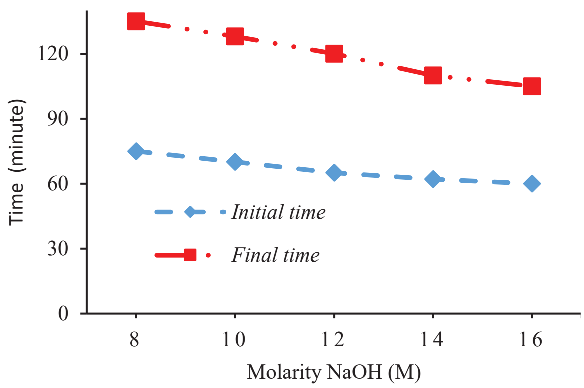


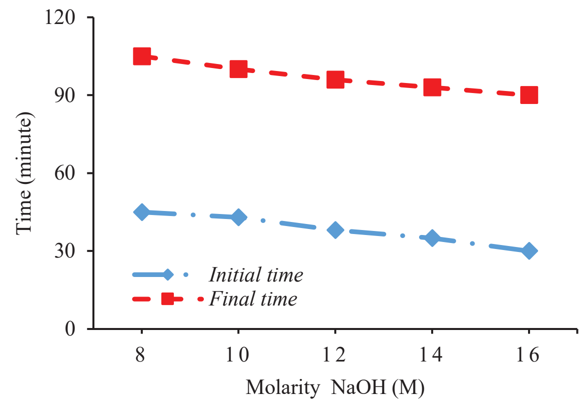
FIGURE 3. The result of the geopolymer mortar slump flow test

The results of the test in Figure 3 show that the higher molarity of oil palm ash also affects the slump flow value of more oil palm ash which was used slump flow value becomes smaller. This result refers to the results of SEM ash and fly ash testing of Figures 2 and 3 showing the more rounded shape of fly ash particles compared to oil palm ash, in addition, the molarity of NaOH solution also affects the value of slump flow. High molarity of NaOH solution makes the mortar mixture thicker so that the slump flow value becomes smaller. The time setting test was performed to determine the time of mortar geopolymer bond. The result of testing time mortar geopolymer setting can be seen in Figure 4.

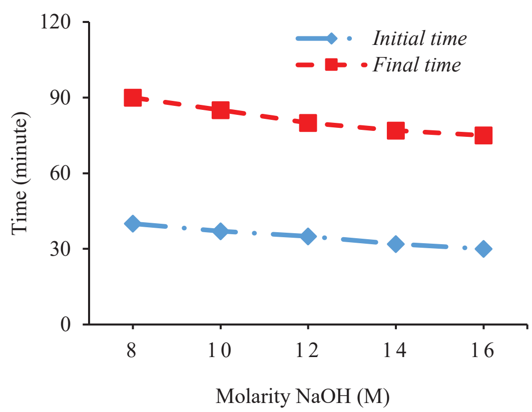
Figure 4 shows the fastest starting and ending time on the composition of the FA mixture: AS = 50%: 50%, while the longest binding time occurs in the composition of the FA mixture: AS = 100%: 0%. The geopolymer mortar bonding time for all mixed compositions was influenced by the molarity of NaOH solution, the higher the molarity of the solution the faster the binding time was obtained. This was because the higher molarity of NaOH solution makes the OH-ions increase so that the chemical bonding reactions are contained in the precursor more quickly. This speeds up the early and late binding of the geopolymer mortar. The addition of oil palm ash precursors also affects the timing of the geopolymer mortar, the more oil palm ash used in fastening time becomes faster. The higher CaO content of oil palm ash than fly ash causes the timing of geopolymer mortar ties that use oil palm ash to be faster. The CaO element in oil palm ash accelerates the hydration reaction of the geopolymer mortar mixture, resulting in faster binding times. The timing of mortar on compositions using oil palm ash was faster not because oil palm ash was more reactive than fly ash, but because the composition lacks an alkaline solution of the activator since it was absorbed by the CaO element. This makes the polymerization reaction less than optimal and forms a less dense bond



(a) Mixed composition FA: AS = 100%: 0%



(a) Mixed composition FA:AS=75%:25%



(b) Mixed composition FA:AS=50%:50%

FIGURE 4. The result of the geopolymer mortar time setting test

Test Result Mechanical Properties of Mortar Geopolymer

Geopolymer mortar weight testing was done at 28 days. The resulted of mortality weight testing could be seen in Figure 5.

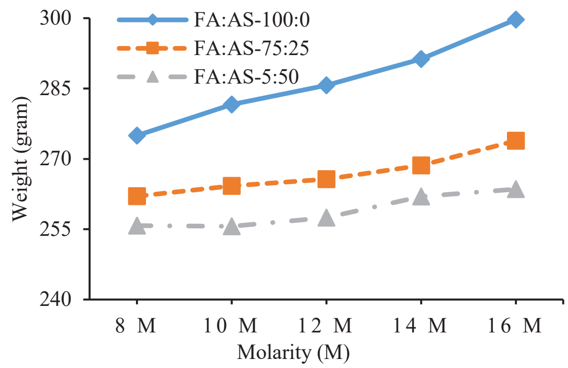
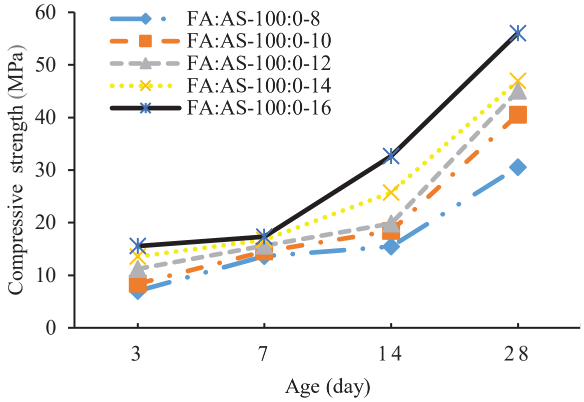


FIGURE 5. Result of weight test of geopolymer mortar age 28 days

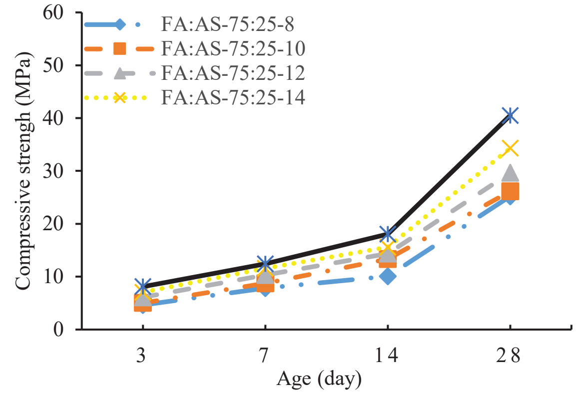
Based on the test result can be seen more and more oil palm ash used heavy mortar weight. This refers to the results of XRF testing of oil palm ash which shows the element of Al_2O_3 of oil palm ash which was smaller than the fly ash, so the polymerization process was less than the maximum and the bonds are formed less dense. The molarity of NaOH solution also influences the weight of the geopolymer mortar the higher concentration of NaOH solution makes mortar heavier. At high molarity of NaOH, polymerization reactions are faster, making the mixture form a solid bond between the precursor and the activator alkali solution. It could be concluded that the addition of oil palm ash as a precursor and the molarity of NaOH solution affect the weight of mortar geopolymer. Almost all the weight of geopolymer mortar was lighter than the weight of cement mortar. The mortar weight exceeding the weight of cement mortar occurs only at the age of 14 and 28 days in the composition of the FA mixture: AS = 100%: 0% with a molarity of 14 M and 16 M NaOH solution. It could be seen that the mortar geopolymer takes the longest time to process the polymerization so that at the age of 14 and 28 days there was the addition of mortar geopolymer weight. The maximum weight of geopolymer mortar occurred at age 28 for all compositions of the mixture.

Compressive Strength Test

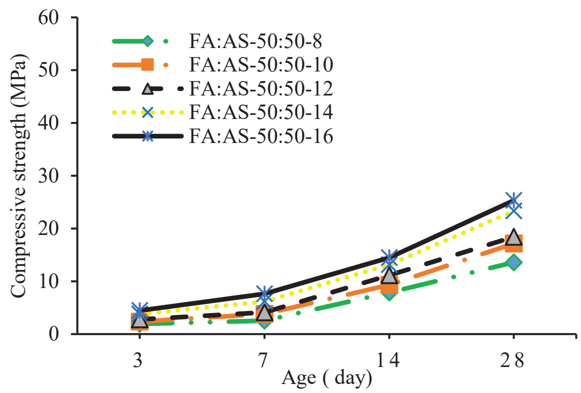
The test results of compressive strength of mortar geopolymer on the age of composition testing of FA: AS = 100%: 0% was presented at 13, FA composition: AS = 75%: 25% was presented in Figure and for FA composition: AS = 50%: 50% presented in Figure 6. Based on Figure 6 it can be seen that the maximum compressive strength of geopolymer mortar occurred at 28 days with the molarity of 16 M. NaOH solution. The maximum mixture composition was FA: AS = 100%: 0%. The compressive strength increases with the life of the test. Can be concluded maximum composition with FA ratio: AS = 100%: 0%. This was because the mineral content of SiO_2 and Al_2O_3 fly ash was higher than that of oil palm ash. In a composition which utilizes 100% of the binding reaction between the precursor and the activator alkali solution was maximal so as to make a solid bond and produce a higher compressive strength. Compressive strength with the ratio of FA: AS = 50%: 50% low refers to the results of XRF testing of oil palm ash SiO_2 and Al_2O_3 elements are lower than the fly ash, so the polymerization reaction was less than the maximum and the bonds are formed less solid. This causes the compressive strength of the geopolymer mortar to decrease as the addition of oil palm ash, the more oil palm ash that was used the compressive strength gets decreased. NaOH serves to activate the chemical elements contained in the precursor thus making a strong bond of geopolymer matrix. The OH ion in NaOH was an essential element in the polymerization process. When high levels of OH-ions make the polymerization process faster, the bonds are more solid. The decrease in OH-ion levels makes the polymerization reaction slower, resulting in less than optimal yield. It can be concluded that the molarity of NaOH solution influences the compressive strength of the geopolymer mortar, the higher the molarity makes the compressive strength increases.



(a) Mixed composition FA: AS = 100%: 0%



(b) Mixed composition FA:AS=75%:25%



(c) Mixed composition FA:AS=50%:50%

FIGURE 6. Compressive strength mortar geopolymer

SEM Analysis

Microstructural testing was done by SEM method. The result of SEM mortar geopolymer testing can be seen in Figures 7,8 and 9. All SEM images were taken with 10 kV electron and magnification of 2,000. Based on Figure 7-9, the microstructure of fly ash and oil palm ash was influenced by the addition of oil palm ash and the molarity of NaOH solution. FA Composition: AS = 100%: 0% indicates microstructure that was more dense compared to FA composition: AS = 75%: 25% and FA: AS = 50%: 50%. At 28 days of age, nearly all compositions of the mixture show the formation of pores, cracks and unresolved precursor granules. It can be seen that the microstructure of mortar geopolymer was getting better as the molarity of NaOH solution increases. The round granules of unreacted precursors are present in almost all mixed compositions. This result refers to the SEM fly ash and oil palm ash test in Figures 1 and 2 showing the dominant round grain shape.

At 28 days there are still unresolved fly ash and oil palm ash. This suggests that there are some grains of precursors that take longer to react with the activator alkali solution. Cracks can be seen in all mixed compositions. The cracking occurs during the compressive strength test, the overly high treatment temperature and the Na_2SiO_3: NaOH = 2 ratios. The high use of Na_2SiO_3 makes the polymerization process faster but causes the number of cracks in the geopolymer mortar. This makes the microstructure mortar of the fly ash geopolymer and oil palm ash indicate the presence of cracks. FA Composition: AS = 100%: 0% shows the strong bond between the precursor and activator alkali solution, but there was pore and crack in all molarity of NaOH solution.

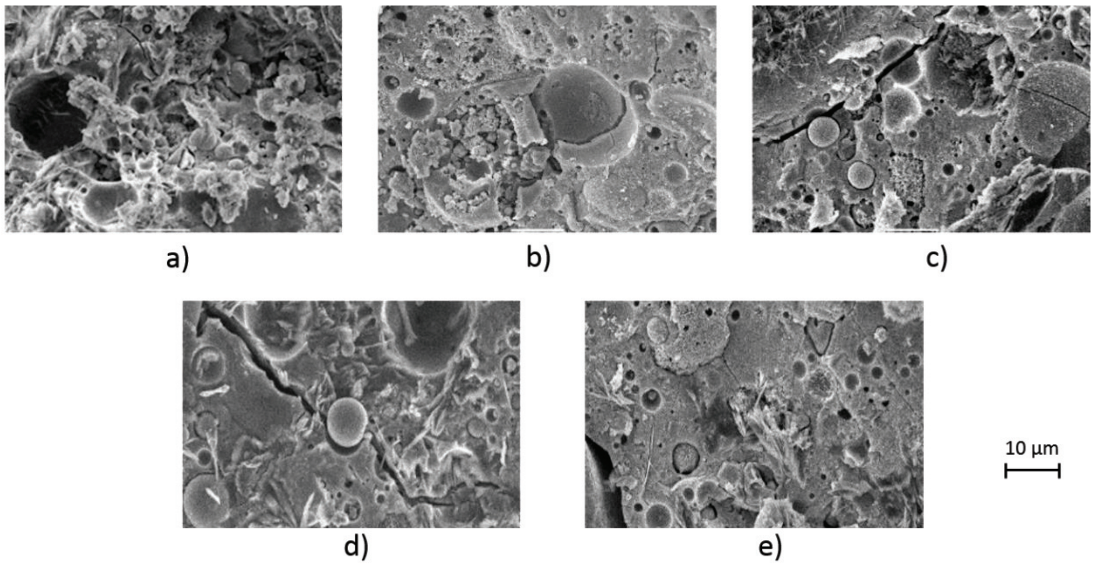


FIGURE 7. SEM test results composition 100% fly ash: 0% oil palm ash based on different molarity, M - a) 8, b) 10, c) 12, d) 14 and e) 16

The reaction product was indicated as a polymer zeolite. Zeolite was a crystalline element that has many pores, besides high curing temperature causes the formation of air bubbles in a mixture of mortar geopolymers. The formed air bubbles are trapped in a mixture of faster geopolymer mortar reacting due to high curing temperatures. In FA composition: AS = 75%: 25% and FA: AS = 50%: 50% there was unconfined ettringite formation. Ettringite was calcium hydroxide ($Ca(OH)_2$). The formation of ettringite in large quantities leads to the development of volume, thus making the mortar shatter and the compressive strength obtained was not maximal. This was due to the higher CaO in oil palm ash compared with fly ash.

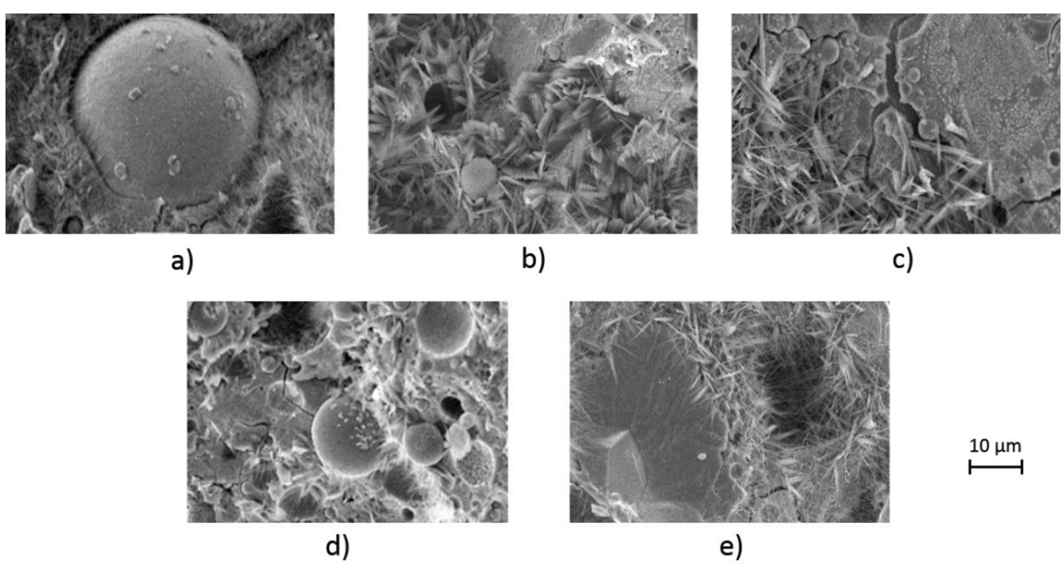


FIGURE 8. SEM test results composition 75% fly ash: 25% oil palm ash based on different molarity, M - a) 8, b) 10, c) 12, d) 14 and e) 16

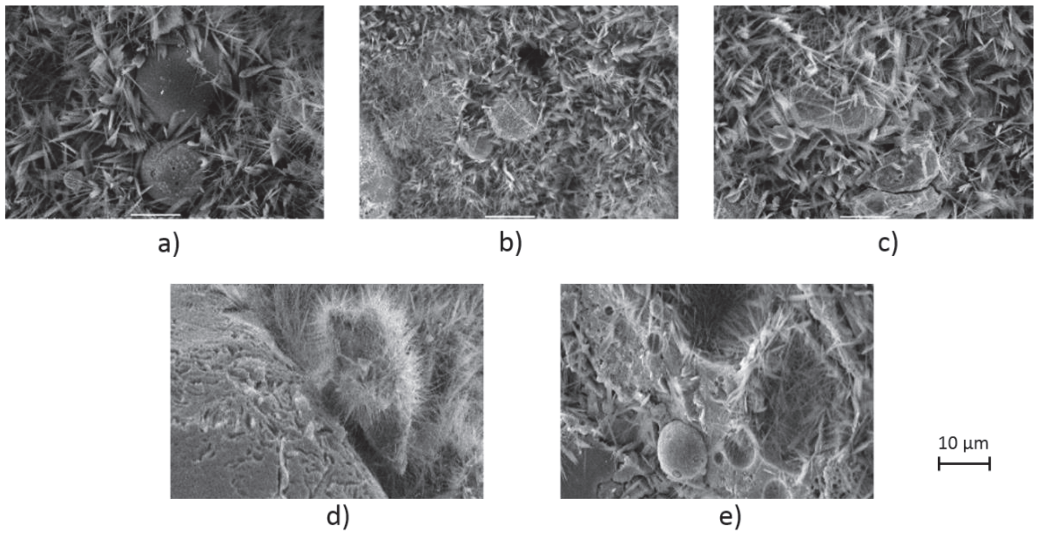


FIGURE 9. SEM test results composition 50% fly ash: 50% oil palm ash based on different molarity, M - a) 8, b) 10, c) 12, d) 14 and e) 16

CONCLUSIONS

Based on the results and discussion got some conclusions, namely:

The use of oil palm ash as a precursor of geopolymer mortar makes the compressive strength of the geopolymer mortar down to every addition. This result was directly proportional to the mortar weight, the more oil palm ash used to make the mortar weight drop. Oil palm ash also affects the microstructure of mortar geopolymer. The use of fly ash as a precursor makes the compressive strength of mortar higher and makes the weight of mortar increased. Based on SEM test results, more oil palm ash leads to ettringite formation and an increase in each addition of oil palm ash was used, otherwise, the mixture using fly ash was almost invisible ettringite. More and more ettringite was formed resulting in a decrease in the compressive strength of mortar geopolymers. The addition of oil palm ash made mortar weight lighter, mortality decreased by 5-9% and decreased compressive strength by 15-45% to cement mortar on 25% of oil palm ash. In the addition of 50% of oil palm ash , there was a decrease in mortar weight of 8-11% and a decrease in compressive strength of 47-71% to cement mortar. The decrease in the weight of the geopolymer mortar was indicated because the surface of the oil palm ash was less dense, thus the weight becomes lighter. At 28 days there was an increase of compressive strength of mortar geopolymer by using fly ash with 16 M31 NaOH solution molarity of cement mortar.

The maximum composition of the geopolymer mortar is FA: AS-100: 0-16, the maximum compressive strength of 56.06 MPa at 28 days. As for the composition of the mixture FA: AS-75: 25-16 of 40.51 MPa and for mixed composition FA: AS-50: 50-16 of 25.33 MPa. It can be concluded that the maximum composition of all mixtures was at 100% fly ash with a molarity of 16 M NaOH solution

The maximum molarity of NaOH solution for all compositions is 16 M, the higher the compressive strength NaOH concentration is increased.

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