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Study of Compressive Strength and Durability of Fly Ash and Trass Based Geopolymer Mortar

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Abstract:

In this research, the effect of natural pozzolanic trass (NT) content as a partial replacement of sand in fly ash (FA) based geopolymer mortar on setting time, workability, compressive strength, absorption, porosity, resistance in sodium sulfate (Na_2SO_4)/water solutions with wet-dry cycles and microstructure were studied. The geopolymer mortar mixtures were made with NaOH 10 M, FA/sand ratio of 0.5, Na_2SiO_3/NaOH ratio of 2.5, alkali activator/FA ratio of 0.5 and indoor curing. NT content of 0%, 10%, 20%, and 30% by weight of sand. The results showed that the setting time was shorter and the mortar flow decreased with increasing NT substitution. Absorption, porosity, and weight loss in Na_2SO_4 and water solutions showed the minimum values, while the compressive strength showed maximum results in NT substitution of 20%. The geopolymer mortar has better resistance in sodium sulfate solution than in water. The SEM results showed the formation of a denser mortar and fewer micro-cracks with NT substitution. XRD analysis shows that NT substitution increases the content of albite phase Na(Al Si_3 O_8) or N-A-S-H gel which played an important role in increasing the compressive strength and durability of geopolymer mortar.

Keywords: Compressive strength, Class C fly ash, Geopolymer, Natural pozzolan trass, Wet-dry cycles

I. INTRODUCTION

The production of Portland cement as a conventional binder emits quite a lot of CO_2 emissions in nature, which has an impact on global warming. This is because every 1 tonne of cement produces, will release about 1 ton of CO_2. Efforts to reduce CO_2 emissions through alternative binders called geopolymers were introduced by Joseph Davidovits [1].

The geopolymer binders have good strength and durability even higher than conventional binders, and can reduce about 70-80% CO_2 emissions in the atmosphere [2].

Geopolymer binders are formed by the chemical reaction of silica and alumina oxides with alkali polysilicate and form polymers with Si–O–Al bonds. The high content of silica and/or alumina oxide is found in natural and artificial pozzolans such as FA. So far, FA is the most widely used as a precursor in the manufacture of geopolymers because of its abundant availability in many countries. Along with the increasing research on geopolymers, the use of natural pozzolans has begun. Natural pozzolan in Indonesia is known as trass which is the result of weathering of volcanic deposits. Its availability in Indonesia follows the path of a series of tertiary and quaternary volcanoes [3].

The use of natural pozzolan zeolite (NZ) as a precursor in the manufacture of geopolymer mortar was studied by Degirmenci [4]. The results of the study were then compared with FA and Ground Granulated Blast Furnace Slag (GGBFS) showing that the compressive strength of mortar made from NZ was lower than FA and GGBFS due to the lower pozzolanic activity in NZ compared to FA and GGBFS. The combination of natural pozzolan and slag in the manufacture of geopolymer mortar was studied by Najimi et al. [5] with a natural pozzolan/slag composition of 70%:30%; 50%:50%; 30%:70%. The results showed that the compressive strength of mortar with 70% slag was higher than that of 70% and 50% natural pozzolan.

The results of a study from Ekaputri and Triwulan [6] on geopolymer concrete based on FA with NT as a substitute for FA stated that NT can be used for structural concrete in certain mixture compositions. The opposite result was found by Risdanareni et al. [7] showed the effect of adding NT to the geopolymer concrete mixture which caused the compressive strength of the concrete to decrease by almost 36%.

The strength and durability of geopolymer mortar made from natural pozzolan using the wet-dry cycles test were studied by Djobo et al. [8]. The results showed a decrease in strength of 24% and 14% for samples treated at 27 ^{o}C and 80 ^{o}C, respectively. Degirmenci [9] studied the effect of Na_2SiO_3/NaOH ratio on the durability of geopolymer mortar containing NZ, FA, and GGBFS in sodium sulfate, magnesium sulfate, sulfuric acid, and hydrochloric acid at concentrations of 5% and 10% for 24 weeks. The results showed that there was no significant damage to the surface of the specimen, except for those immersed in sulfuric acid solution, there was only slight damage. Geopolymer mortar from NZ suffered more damage than GGBFS and FA, also the largest weight change.

Siswati et al. [10] investigated the amorphous properties of pozzolanic materials NT, GGBFS, FA, and silica fume. The results showed that NT had a very low amorphous value/less amorphous than other pozzolans.

NT which is rich in silica but less amorphous is the focus of the study as a partial replacement of fine aggregate in the manufacture of FA-based geopolymer mortar. The compressive strength and short-term durability of Na_2SO_4 solution and water with wet-dry cycles were investigated.

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II. MATERIALS AND METHODOLOGY

II.1. Materials

The FA material was obtained from the Indonesia Amurang power station and NT from Minahasa Indonesia as the source material. The specific gravity and median particle size of FA and TR were 2.8, 40 µm, and 2.22, 65 µm, respectively. The chemical composition based on X-Ray Fluorescence (XRF) analysis is shown in Table 1. According to ASTM C618 [11], the FA could be classified as a class C because of the sum of SiO_2+Al_2O_3+Fe_2O_3 = 66.85, CaO = 26.72, and NT are class N pozzolans because of the sum of SiO_2+Al_2O_3+Fe_2O_3 = 91.85. The low CaO content (3.08%) based on the XRF test indicates that NT is not cementitious.

Fig. 1 shows its scanning electron microscope (SEM) image. The shape of fly ash was spherical while TR was angular/irregular. X-Ray Diffraction (XRD) analysis in Fig. 2 shows that NT was less amorphous than FA. Before use, NT was dried in an oven at 100 ºC for 24 hours and sieved through a 0.075 mm sieve. The passed NT is then used in the manufacture of geopolymer mortar. This condition is to activate/enhance the pozzolanic properties. Firdous et al. [12] stated that raw natural pozzolans without pre-treatment (activation) had poor reactivity due to their low solubility in alkaline media. Therefore, it needs pre-treatment before use. River sand with a saturated surface dry density (SSD) of 2.58 and a fineness modulus of 2.92 was used as fine aggregate. The alkaline activator used is a combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3).

TABLE I Chemical composition of FA and NT (% by weight)

COMPOSITION	SiO_2	Al_2O_3	Fe_2O_3	CaO	MnO	K_2O	SrO	TiO_2	BaO	SO_3
FA	32.48	9.38	24.99	26.72	0.00	0.96	0.34	1.03	0.16	3.87
NT	78.99	6.49	6.37	3.08	0.17	3.99	0.04	0.72	0.03	0.00

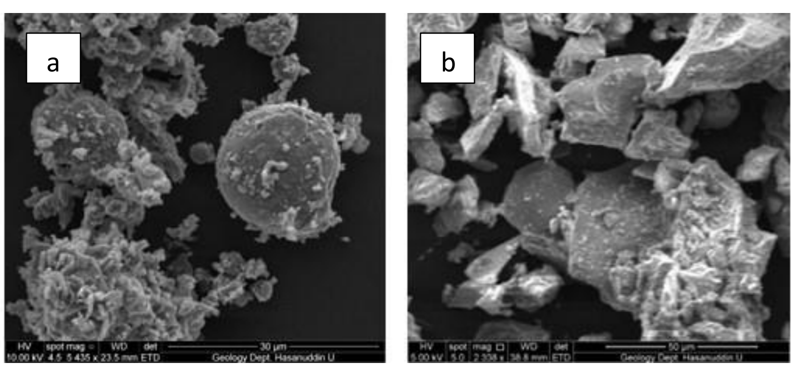

Fig 1: SEM images (a) FA, (b) NT

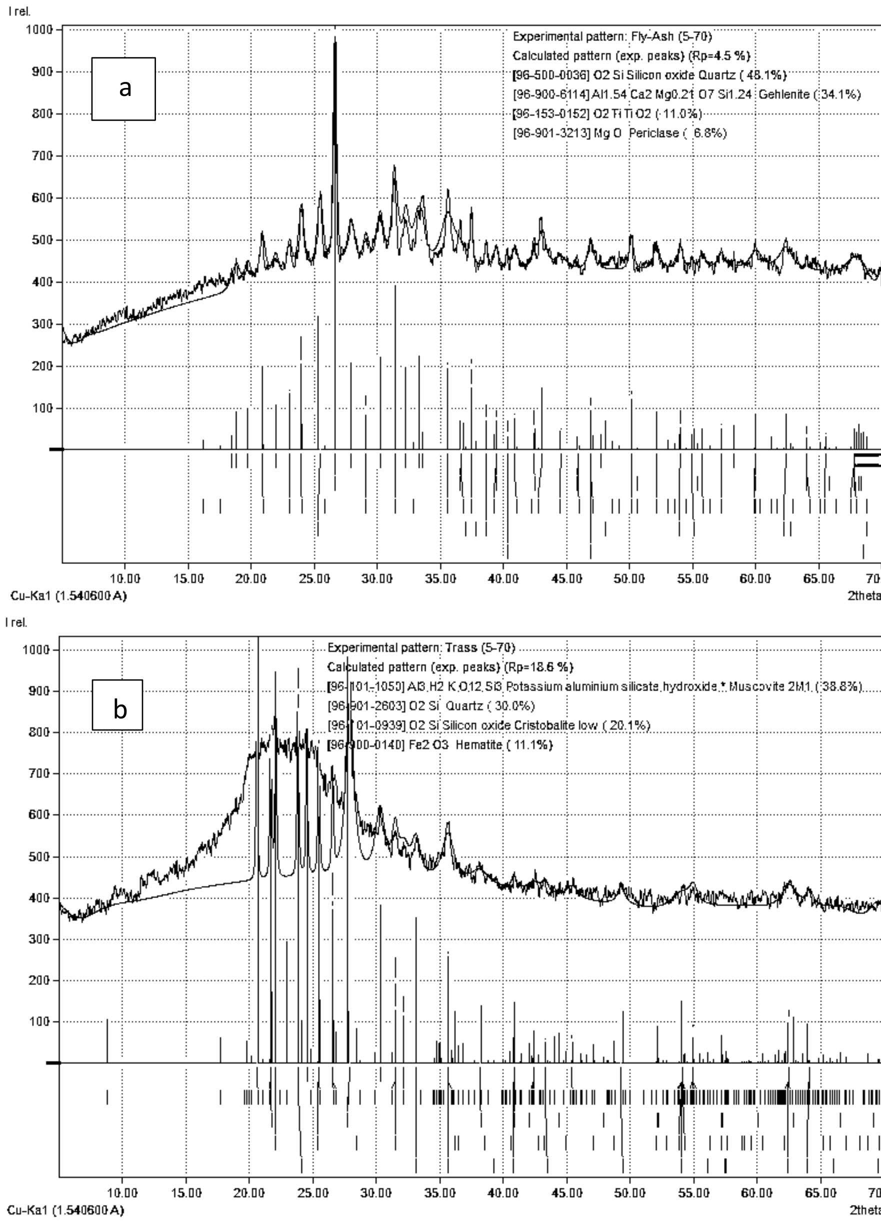

Fig 2: XRD Results (a) FA, (b) NT

II.2. Mix Proportion

The geopolymer mortar mixtures were prepared with NaOH 10 M, FA/sand ratio of 0.5, Na_2SiO_3/NaOH ratio of 2.5, alkali activator/FA ratio of 0.5, and indoor curing at 28 ^oC. Determination of composition is based on previous research [13]. Variations in NT content of 0%, 10%, 20%, and 30% by weight of sand. The mixture proportions for 3 cubes with a side size of 50 mm are shown in Table 2. The mortar mixing process used a separate mixing method [14, 15].

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TABLE II Proportion of geopolymer mortar mix

ID MIXTURE	% NT	FA (gr)	NT (gr)	SAND (gr)	NaOH (gr)	Na_2SiO_3 (gr)
M0-NT (CONTROL)	0%	300	0	600	43	107
M10-NT	10%	300	60	540	43	107
M20-NT	20%	300	120	480	43	107
M30-NT	30%	300	180	420	43	107

II.3. Sample Testing

The setting time of the geopolymer paste was tested according to the ASTM C191 standard [16], workability according to the ASTM C1437 [17], and compressive strength according to ASTM C109 [18]. The compressive strength value was the average value of 3 specimens in each mixture at the age of 7, 28, and 60 days. Absorption, porosity, and resistance tests on the samples aged 28 days. Each value was the average value of 3 specimens in each mixture. Absorption and porosity tests refer to the ASTM C642 [19], the test of resistance to 5% Na_2SO_4 and water refers to SNI-03-0691-1996 with the wet-dry cycle method for 5 cycles [20]. One cycle consists of soaking for 16-18 hours, then heating at 105±5 ^oC for 2 hours. The durability of the geopolymer mortar was evaluated by the weight loss of the specimen after cycling. The smaller the weight loss, the better the level of resistance.

III. RESULTS AND DISCUSSION

III.1. Setting Time, Workability, and Compressive Strength of Geopolymer Mortar

The results of the setting time test for FA geopolymer paste with the addition of NT are shown in Fig 3. The setting time is faster with increasing the amount of NT by 115, 90, 62, 39 minutes for the initial setting time and 195, 150, 120, 90 minutes for the final setting time at 0%, 10%, 20%, and 30% NT, respectively. This condition is influenced by the surface area of the material [12] in this case NT. The more NT in the mixture, the greater the total surface area of NT to react with the alkali activator so that the setting time is faster.

The workability test results in Table 3 show that the mortar flow decreases by 210, 180, 152, and 102 mm for NT substitution of 0%, 10%, 20%, and 30% of the weight of sand. These results when compared with the workability criteria of geopolymer mortar from Ghosh & Ghosh [21] in Table 4, the workability of M0-NT is high, M10-NT and M20-NT is moderate, M30-NT is very stiff. These results are related to the FA and NT shapes (Fig.1). The spherical of FA make the mixture easy to flow while the NT with the angular shape can resist the movement of the mortar so it can't flow easily. The surface area of the material can also affect workability [22]. The smaller the surface area of the material, it will increase the more workability and vice versa. In this study, NT as a substitute for sand has a finer grain size than sand, so that the surface area of NT per unit weight is larger, which causes the flow value to be smaller.

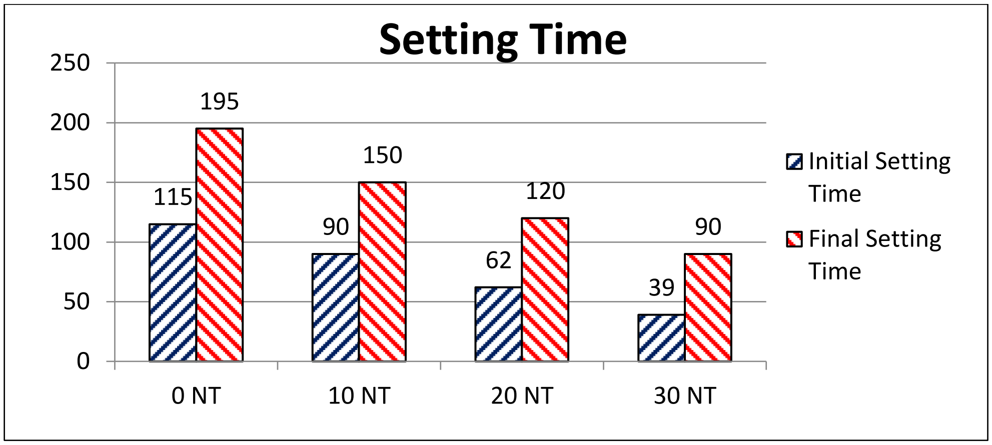

Fig 3: The setting time of FA geopolymer paste with % NT

TABLE III Values of mortar flow

ID MIXTURE	M0-NT	M10-NT	M20-NT	M30-NT
FLOW DIAMETER (MM)	210	180	152	102

TABLE IV Criteria for geopolymer mortar workability [21]

NO.	FLOW DIAMETER (MM)	WORKABILITY
1	ABOVE 250	VERY HIGH
2	180 – 250	HIGH
3	150 – 180	MODERATE
4	120 – 150	STIFF
5	BELOW 120	VERY STIFF

The results showed that the mortar mix with 10-20% NT had good workability. This result is following the research of Ghosh and Ghosh [21] which stated that the minimum flow diameter should be 150 ±10mm so that the geopolymer mortar is easy to work. The test results also show that NT substitution of 10-20% can improve the flow criteria from high to medium workability.

Fig. 4 shows the increase in compressive strength of all mixtures using NT at the age of 28 and 60 days. This increase indicates the effect of NT substitution on the FA-based geopolymer mortar mixture. The silica content of 78.99% (Table 1), becomes an additional precursor, which then reacts with the alkaline activator solution in the polymerization process and affects the increase in compressive strength. Similar results were presented by Najimi et al. [5] that the compressive strength increased with increasing natural pozzolan content compared to the low natural pozzolan content. Based on Fig. 4, the use of NT as a sand substitute in a mixture of FA-based geopolymer mortar with 10 M NaOH and a Na_2SiO_3/NaOH ratio of 2.5 can increase the compressive strength. These results are similar to those of Ekaputri et al. [6] who recommend that NT can be used for structural concrete provided that the ratio of Na_2SiO_3/NaOH is between 2–2.5 and the concentration of NaOH solution is 10 M – 14 M.

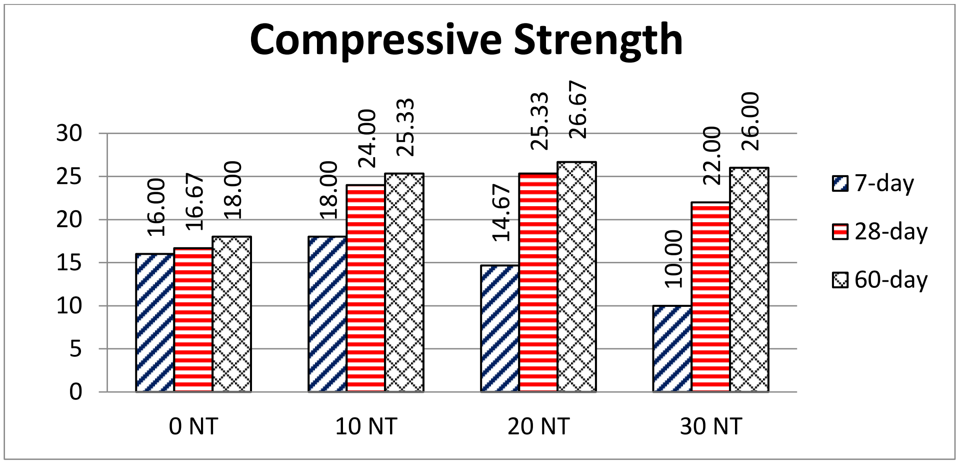

Fig 4**:** Compressive strength of geopolymer mortar

The effect of NT on the compressive strength at the age of 7 days has not been seen and is only seen after the age of 28 days. This indicates that the NT reaction in the geopolymer mortar mixture is slow. The results of Najimi's research [5] also stated that the reaction rate of natural pozzolans increased with increasing curing age. The same behavior was also found by Djobo et al. [8] on a geopolymer mortar made from volcanic ash which was cured at room temperature of 27 ^{o}C. The 7-day compressive strength was 8 MPa and 28 days increased to 20.5 MPa. The effect of curing at room temperature is also one of the reasons for the slow increase in the compressive strength of FA-based geopolymers [23, 24].

III.2. Absorption, Porosity, and Durability of Geopolymer Mortar

Mortar durability is influenced by its absorption and porosity properties. In Fig. 5, the relationship between %NT on absorption and mortar porosity shows the same trend, which is decreasing at 0-20%NT, then increasing at 30%NT. Absorption values were 18.92%, 18.66%, 18.43%, 19.19% and porosity were 21.35%, 20.89%, 20.39%, 22.48% on NT substitution of 0%, 10%, 20%, and 30%, respectively.

The decrease in absorption and porosity values indicated the effect of NT substitution in the geopolymer mortar mixture. The fineness of the NT grains allows it to fill the voids so that the mortar becomes denser. The distance between the grains is getting smaller to reduce the pore size of the geopolymer mortar. In the use of NT by 30%, there is an increase in the value of absorption and porosity. These two properties are related to the presence of voids in the mortar that occur due to a less workable mixture so that the mortar is difficult to make and compact.

The resistance test using the wet-dry method is shown in Table 5. Before and after the cycle, the samples were dried in an oven at a temperature of 105±5 ^{o}C to a constant weight. A significant increase in weight occurred in the first cycle and then decreased in the second cycle and was seen to be stable in the fifth cycle. The empty mortar pores increased the ability to absorb the solution in the first cycle.

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TABLE V Weight change of geopolymer mortar with variation of NT

SOLUTION	% NT	% WEIGHT CHANGE						
		BEFORE	1	2	3	4	5	AFTER
Water	0 NT	0.00	5.16	0.56	1.39	5.68	6.00	(3.72)
	10 NT	0.00	6.00	1.58	3.24	6.71	6.37	(2.65)
	20 NT	0.00	7.93	5.78	5.28	4.72	7.18	(1.84)
	30 NT	0.00	8.99	6.57	6.51	5.79	8.81	(2.52)
Na_2SO_4	0 NT	0.00	6.64	1.37	5.41	7.19	8.17	(2.86)
	10 NT	0.00	7.44	4.47	6.86	8.42	8.72	(1.46)
	20 NT	0.00	9.94	7.64	6.97	7.23	9.03	(1.03)
	30 NT	0.00	11.38	7.83	7.03	7.31	10.25	(1.52)

The results in Table 5 show that there was an increase in weight in all samples for 5 cycles, and the increase in weight in Na_2SO_4 solution was greater than in water. These shows that the Na element in the sulfate solution can react with the elements in the geopolymer mortar. The test results showed the greater the substitution of NT, the greater the increase in weight, indicating the influence of silica content in NT.

The change in weight after the cycle is complete is shown in Fig. 6. The weight loss of mortar in Na_2SO_4 is 2.86%, 1.46%, 1.03%, 1.52% and in water is 3.72%, 2.65%, 1.84%, 2.52% for NT substitution of 0%, 10%, 20%, and 30%, respectively. Fig. 5 and 6 show the same trend, indicating that the durability of mortar is affected by absorption and porosity. The smaller the absorption and porosity values, the smaller the mortar weight loss and vice versa. The test results in Fig. 6 show that the mortar weight loss is smaller with the substitution of 10-20% NT in both water and Na_2SO_4. At 30% NT substitution, weight loss increased. This condition is due to the large voids indicated by the absorption value and porosity of M30-NT, making it easier for the solution to enter and attack the mortar structure. The amount of mortar weight loss can be compared with the study of Djobo et al. [8] on a sample of geopolymer mortar made from volcanic ash which was immersed continuously for 120 days in 5% sulfuric acid solution, the weight loss was 3.51%. This shows that FA-based geopolymer mortar by replacing some of the sand with NT has good resistance.

In Fig. 6, the mortar is immersed in water, the weight loss is greater than in the Na_2SO_4 solution. This condition is caused by the release of some Si-O-Na bonds and hydrolysis (chemical breakdown of a compound due to reaction with water) some silica –Si-O-Si- bonds which are generally soluble in water [12, 25]. The results of the study of Djobo et al. [26, 27] also found a decrease in compressive strength in geopolymer materials based on volcanic ash mixed with oyster shells soaked in water for 24 hours. This is mainly due to the synthesis that causes the formation of various types of mineral zeolite or sodium silica with -Si-O-Si- bonds which are partially released/broken in the water.

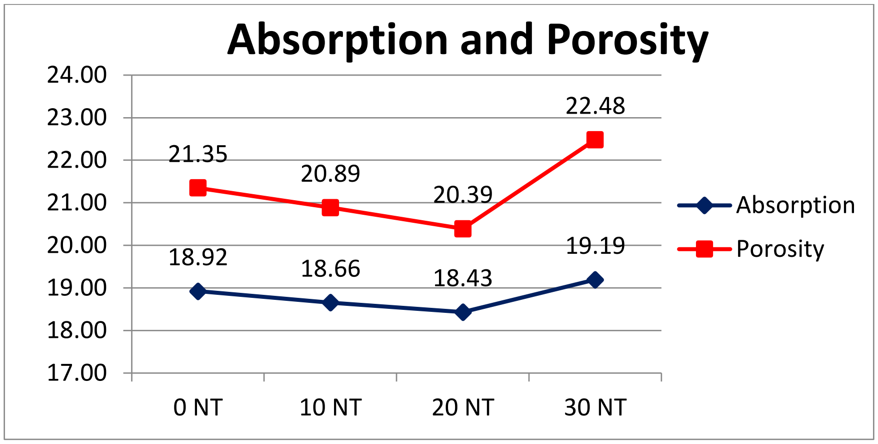

Fig 5: Water absorption and porosity of mortar geopolymer

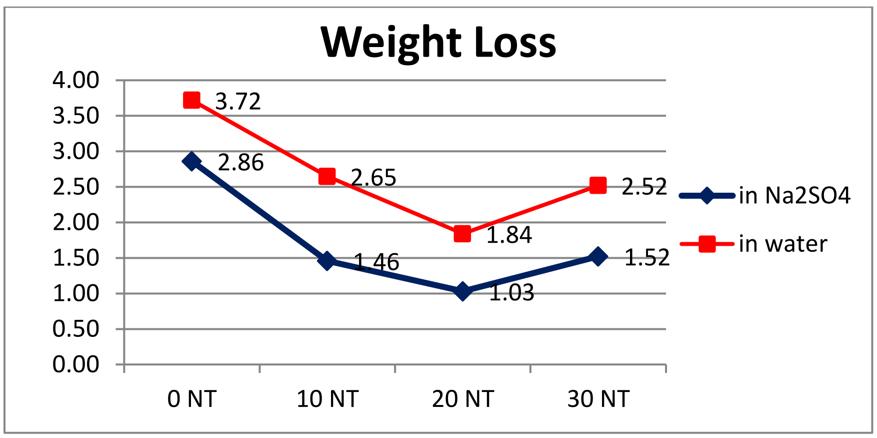

Fig 6: The effect of %NT on a weight loss of geopolymer mortar

On the other hand, the mortar immersed in the Na_2SO_4 solution lost less weight, probably due to the silica (Si) of NT reacts with sodium (Na) and exerts a positive impact on the geopolymer mortar in the form of Si-O-Na bonds. Djobo et al. [8] also stated the presence of sodium (Na) in the specimen, increasing resistance to sulfate attack. From the visual results, there were no visible defects on the surface of all test specimens, indicating that the geopolymer mortar was stable in a wet-dry environment, both in water and in sulfate.

III.3. Geopolymer Mortar Microstructure

III.3.1 Scanning Electron Microscope (SEM)

The results of SEM analysis on mortar aged 28 days with a magnification of 2500x in Fig. 7 show that a homogeneous and dense matrix has been formed, but in Fig. 7a (M0-NT) it can be seen that there are micro-cracks on the surface of the mortar. These microcracks are caused by matrix shrinkage. With a high flow mortar (Table 3), it shows the mixture has high water content. The high water content in the activator can be one of the causes of shrinkage [23, 28].

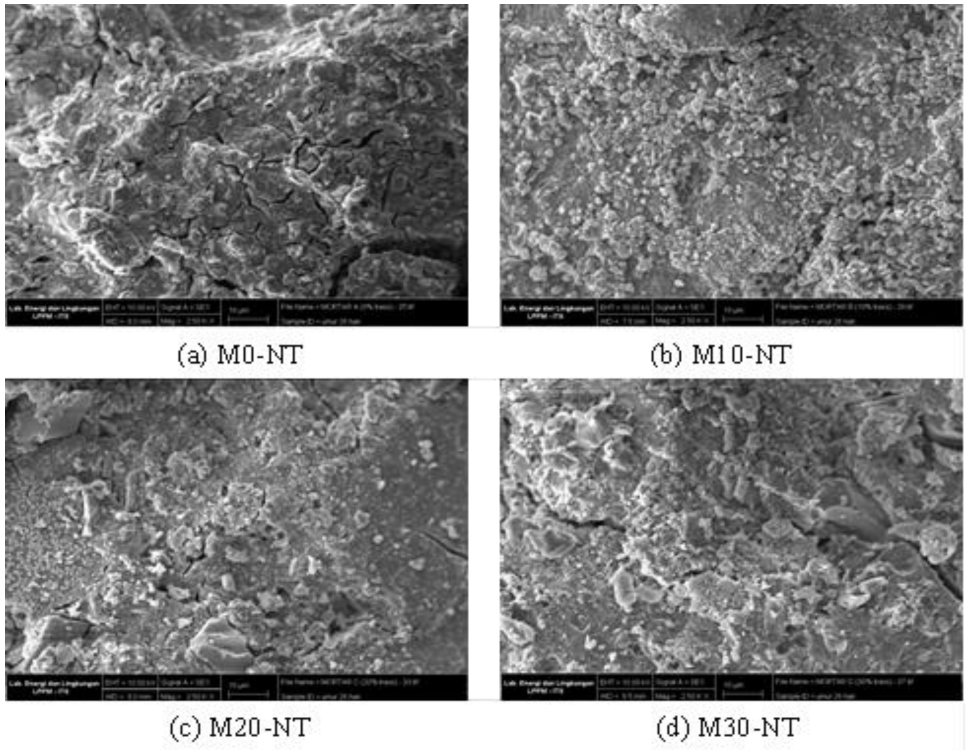

Fig 7**:** SEM image on 28 days

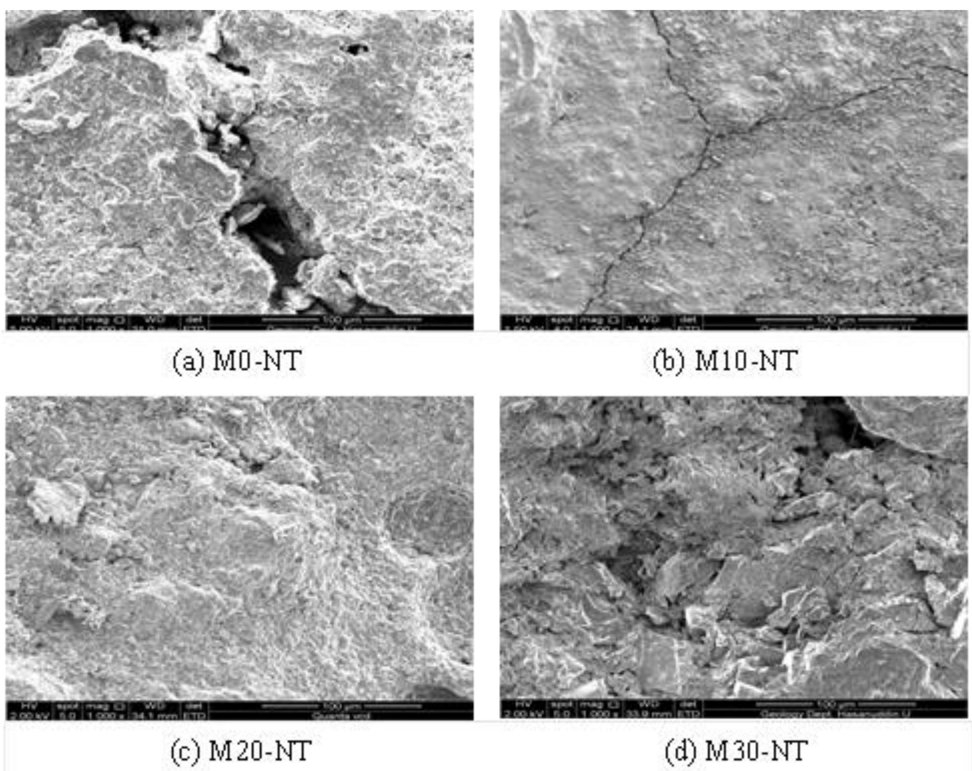

Fig 8**:** SEM image in Na_2SO_4

Fig. 7b, c, d show fewer micro-cracks, possibly because the water in the activator is absorbed by the NT so that the shrinkage is very small. The fineness of the NT also contributes to the density of the matrix, helping to reduce micro-cracks by filling voids thereby increasing the compressive strength [29]. These SEM results explain the compressive strength results (Figure 4).

Fig. 8 shows the SEM results with 1000x magnification of the mortar samples tested for resistance in Na_2SO_4 solution. Fig. 8a shows fairly wide micro-cracks compared to Fig. 8b and Fig. 8c does not show micro-cracks. Fig. 8d shows the cavities which indicate that the mortar is less dense. This is related to the very small flow value so that the mortar is less workable. This SEM result shows that NT substitution up to 20% can increase the geopolymer mortar resistance to Na_2SO_4 and confirm the mortar resistance test results in Fig. 6.

III.3.2 Energy Dispersive X-Ray (EDX) Analysis

EDX analysis of mortar samples at the age of 28 days is presented in Table 6. The EDX results showed an increase in silica in the mortar using NT. This follows the content of NT which is rich in silica (Table 1). There is also an increase in the element sodium (Na). Elemental Na is mainly required in the polymerization reaction and plays an important role in increasing the compressive strength [30, 31]. The presence of Na in the specimen also increases the resistance to sulfate attack [8]. The EDX results confirmed the effect of NT on increasing the compressive strength and durability of geopolymer mortar.

III.3.3. X-Ray Diffraction (XRD)

Table 7 shows the results of XRD Mortar geopolymers aged 28 days which were analyzed using Rigaku Miniflex II XRD and PDXL2 Software.

TABLE VI Elements in geopolymer mortar based on EDX analysis

ELEMEN	M-0NT 28 hr (Wt.%)	M-10NT 28 hr (Wt.%)	M-20NT 28 hr (Wt.%)	M-30NT 28 hr (Wt.%)
O	78.33	62.38	74.87	61.60
C	5.24	1.01	6.49	7.16
Ca	4.10	3.82	3.57	11.01
Si	4.24	11.47	6.26	8.66
Na	2.46	5.45	3.21	4.03
Al	0.88	4.51	1.38	1.67
Cl	0.24	0.34	0.39	0.29
Mg	0.21	0.30	0.12	0.21
Fe	4.31	10.73	3.69	5.36

TABLE VII XRD analysis results on geopolymer mortar aged 28 days

ID MORTAR	PHASE NAME	FORMULA	CONTENT (%)
M0-NT	Quartz Low, Syn	$Si\ O_2$	41
	Albite	$Na\ (Al\ Si_3\ O_8)$	43
	Magnesium Ferrous Oxide	(MgO)0.239 (FeO)0.761	15.5
M10-NT	Quartz	$Si\ O_2$	21
	Albite, Syn	$Na\ Al1.08\ Si2.92\ O_8$	67
	Magnesioferrite	$(\ Mg\ Fe_2\ )\ O_4$	12
M20-NT	Quartz Low, Syn	$Si\ O_2$	22
	Albite	$Na\ (Al\ Si_3\ O_8)$	61
	Magnesioferrite, Aluminian, Syn	$Mg1.01\ Fe1.77\ Al.22\ O_4$	17
M30-NT	Quartz Alpha, Alpha-Si O_2	$Si\ O_2$	30
	Albite	$Na\ (Al\ Si_3\ O_8)$	58
	Ferrosilite, Magnesian	$(\ Mg.465\ Fe1.535\ )\ Si_2\ O_6$	12.1

In the M0-NT mortar, phase quartz, albite, and magnesium ferrous oxide were formed, then in the M10-NT, M20-NT, and M30-NT mortar, the magnesium ferrous oxide phase changed to magnesioferrite, magnesioferrite aluminian, and ferrosilite magnesian. This change is due to the addition of NT which contains a lot of Si, Al, and Fe elements so that they react during the polymerization process and form new compounds. From the phases formed, the most mineral compound content is phase albite. Phase albite in the M0-NT mortar which was initially 43%, increased in the M10-NT, M20-NT, and M30-NT mortar, respectively, by 67%, 61%, 58%. On the other hand, the quartz phase in the M0-NT mortar, which was initially 41%, decreased in the M10-NT, M20-NT, and M30-NT mortars by 21%, 22%, and 30%, respectively. This indicates that some of the reactive silica elements in NT react with alkaline activator solutions (NaOH and Na_2SiO_3) and form an albite phase so that the quartz phase content decreases but the albite phase content increases. Phase albite ($NaAlSi_3O_8$) known as N-A-S-H [29, 32] plays an important role in increasing the compressive strength of geopolymer mortar [33]. This analysis shows conformity with the results of compressive strength as shown in Table 4.

Phase N-A-S-H also provides resistance to chemical attack [5]. The results of XRD analysis showed conformity with the results of mortar resistance to Na_2SO_4 solution where the smallest weight loss occurred in M10-NT and M20-NT mortars according to the highest number of phase albite, namely 67 and 61%.

IV. CONCLUSION

The effect of NT as a partial replacement of sand in a mixture of FA geopolymer mortar with 10 M NaOH, the ratio of $Na_2SiO_3/NaOH = 2.5$, the ratio of activator/FA = 0.5, and the ratio of sand/FA = 2 obtained the results:

1. Setting time was shorter and mortar flow decreased with increasing NT substitution.

2. The high silica content of NT increases the compressive strength with optimum results at 20% NT substitution.
3. The fineness of NT affects the decrease in absorption value and porosity of geopolymer mortar for NT substitution by up to 20%
4. Durability properties of geopolymer mortar based on absorption, porosity, and weight loss showed that the substitution of NT 20% gave the best results. The geopolymer mortar has better resistance in sodium sulfate solution than in water.
5. SEM analysis showed the formation of a denser mortar and fewer micro-cracks while EDX analysis showed an increase in silica in the presence of NT substitution. XRD analysis showed an increase in the content of phase albite $Na(Al\ Si_3\ O_8)$ or N-A-S-H gel in the presence of NT substitution which played an important role in increasing the compressive strength and resistance of geopolymer mortar against Na_2SO_4 attack.

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