

# Mechanical And Microstructural Assessment Of Synthetic Aluminosilicate Based Geopolymer Dental Material

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## ABSTRACT

Orthodontic treatment with fixed brackets was performed to transmit the pressure from the wire to the periodontal tissues to produce tooth movement. Ceramic material can be used not only to improve the aestheticness but also maintains good mechanical properties. This study aims to determine the mechanical properties and morphological of synthetic aluminosilicate based geopolymer for aesthetic orthodontic brackets applications made by synthesizing alumina ( $Al_2O_3$ ), silica ( $SiO_2$ ), and magnesia ( $MgO$ ) from sol-gel method. Geopolymer nanocomposite was made by activating synthetic precursor with alkali solutions consists of sodium hydroxide ( $NaOH$ ) and sodium silicate ( $Na_2SiO_3$ ) solution which has molarity variations of 8M (GP-8M) and 14M (GP-14M). The test results using an X-Ray Diffractometer (XRD) showed that the crystalline phase of  $(Mg,Si)Al_2O_4$  spinel was successfully presented in the synthesized nanocomposite. The average hardness test results using the Vickers hardness tester for Geopolymer Nanocomposite with 8M and 14M alkali activator were 259.15 and 298.90 VHN, respectively. The results of the surface morphological characterization using Scanning Electron Microscope (SEM) of the samples showed the porosity of GP-14M was smaller than GP-8M, which explains the hardness test value difference. This shows Geopolymer is a potential material for orthodontic brackets manufacture.

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## ABSTRAK

Perawatan ortodontik dengan braket cekat dilakukan untuk menyalurkan tekanan dari kawat ke jaringan periodontal untuk menghasilkan pergerakan gigi. Material keramik dapat digunakan tidak hanya untuk meningkatkan estetika tetapi juga mempertahankan sifat mekanik yang baik. Penelitian ini bertujuan untuk mengetahui sifat mekanik dan morfologi geopolimer berbasis aluminosilikat sintetis untuk aplikasi braket ortodontik estetik yang dibuat dengan mensintesis alumina ( $Al_2O_3$ ), silika ( $SiO_2$ ), dan magnesium ( $MgO$ ) dari metode sol-gel. Geopolimer nanokomposit dibuat dengan mengaktifkan prekursor sintetis dengan larutan alkali yang terdiri dari larutan natrium hidroksida ( $NaOH$ ) dan natrium silikat ( $Na_2SiO_3$ ) yang memiliki variasi molaritas 8M (GP-8M) dan 14M (GP-14M). Hasil XRD menunjukkan bahwa fase kristal spinel  $(Mg,Si)Al_2O_4$  berhasil dipresentasikan dalam nanokomposit yang disintesis. Hasil uji kekerasan rata-rata menggunakan Vickers hardness tester untuk Geopolymer Nanocomposite dengan aktivator alkali 8M dan 14M berturut-turut adalah 259,15 dan 298,90 VHN. Hasil SEM sampel menunjukkan porositas GP-8M dan GP-14M yang menjelaskan perbedaan nilai uji kekerasan.

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## Introduction

Orthodontic treatment with fixed appliances can be done to correct the malocclusion so that the desired occlusion condition can be achieved. Fixed appliances are orthodontic appliances that are attached to the patient's tooth surface so that they cannot be removed and installed by the patient themselves. Fixed device components consist of brackets, bow wire or archwire, and supporting components. Based on the material, orthodontic brackets can be made from stainless steel (SS) and titanium-molybdenum alloy (TMA) [1].

Based on the type, orthodontic brackets are divided into two, namely metal and aesthetic brackets [2]. Metal brackets are made of a mixture of stainless steel, chromium and nickel. Metal brackets such as stainless steel since the beginning of their development until now are the most commonly used brackets in orthodontic treatment because they have good mechanical properties [3]. However, the nickel content in stainless steel can be released due to saliva, masticatory movements, and acidic oral conditions, causing a type IV hypersensitivity reaction [4]. Metal brackets are becoming less preferred by patients because the color and shape do not support the aesthetics.

The bracket industry tries to redesign the brackets periodically and develop aesthetic brackets made from ceramic material [5]. Fulfilling this request requires a material that is aesthetically pleasing to the patient and adequate for the clinician to carry out treatment such as *In vitro* evaluation [6]. Aesthetic brackets are brackets with colors that match the teeth. The materials used as aesthetic brackets are plastic and ceramic [7]. Plastic brackets made of polycarbonate and plexiglas are less desirable in orthodontic practice because they discolor with time of use. Plastic brackets have poor integrity in the wire slots so that the energy generated by the wire causes distortion in the bracket and pressure is not transmitted to the teeth [8]. Commonly used ceramic brackets are made of zirconia and alumina. Pure alumina brackets are available in monocrystalline and polycrystalline forms.

Ceramic brackets have good hardness and color stability so they don't get discolored even in long-term use. Its use in orthodontic practice is less desirable because of the brittle nature of ceramics so that fractures often occur when torsional movements or tipping are carried out [9]. Fractures generally occur at the base or the base of the winglet because that location is the location that experiences the highest stress and becomes the fulcrum when installing and removing the archwire [10].

One of the factors that affect the hardness and color of ceramics is the size of the constituent particles. The alumina particles used are on a micrometer scale and heated at a temperature of 1800°-2100°C. The burning or sintering process will cause the alumina particles to solidify and crystallize to produce a transparent color. The particle size of the alumina used is about 0.3-30µm. The particle size that is getting closer to 30µm will reduce the strength of the ceramic. The development of research in the field of nanotechnology materials is able to produce materials with good strength. The end result of this research is to change technology that is generally based on materials with a micrometer scale to nanometers to obtain a number of superior physical properties. Nanoparticles have good properties and various applications[11].

Composite consists of matrix and filler. The main function of the matrix is to protect the composite from environmental influences such as pressure and temperature and to bind and transfer the load received by the composite to the filler so that the material becomes stronger. Composite that has large surface area cause many interactions between particles. The more interactions that occur, the greater the mobility of the polymer chains is reduced. This makes the bonds between particles stronger so that the mechanical properties of the material increase, but the addition of nanoparticles will not always strengthen the mechanical properties of the material. Addition to a certain point will make the material experience saturation so that the strength of the material will decrease [12]. One of the material that can be used as composite matrix is Metal Oxide/Ceramic such as Aluminum oxide ( $Al_2O_3$ ) or alumina.

This compound is a material derived from bauxite that has a high melting point and is chemically very stable and non-reactive in the body, so it is widely used as a biomaterial. Alumina has high hardness, is resistant to abrasion and corrosion [13]. The addition of magnesium oxide (MgO) or magnesia can reduce the porosity of the material after sintering. To achieve high transparency, a material must have a full density or low porosity because residual pores result in a strong optical scattering effect [14]. The addition of silicon dioxide (SiO<sub>2</sub>) or silica is used to produce alumina and silica polymer chains as a stronger framework.

Based on the description above, this study was conducted to synthesize aluminosilicate-based Geopolymer as the base material for aesthetic orthodontic brackets. Resulting powder was carried out by X-Ray Diffraction (XRD) testing. Mechanical properties of Geopolymer was carried out through Vickers microhardness test. The microstructure of Geopolymer was observed through Scanning Electron Microscope (SEM).

## Materials dan Methods

### Materials

The precursors for synthetic aluminosilicates are alumina nitrate nanohydrate (Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O), magnesium chloride hexahydrate (MgCl<sub>2</sub>·6H<sub>2</sub>O), and tetraethyl orthosilicate TEOS (Si(OC<sub>2</sub>H<sub>5</sub>)<sub>4</sub>), and Ammonia (NH<sub>3</sub>). Alkali solution to activate aluminosilicate was made by mixing NaOH and Na<sub>2</sub>SiO<sub>3</sub> solution in a 1:2 ratio. The molarity variations of NaOH were 8M and 14M. The sodium hydroxide (NaOH) which functions to react the elements to produce strong polymer bonds and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) which will accelerate the polymerization.

### Aluminosilicate synthesis

The precursors were mixed with Al:Mg:Si ratio of 70:25:5, homogenized using magnetic stirrer for 30 minutes. Then it was evaporized using 100°C oven for 24 hours followed by refining with mortar.

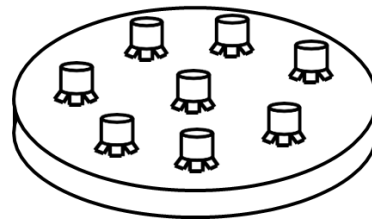
Resulting powder then transferred into 1000°C furnace for two hours and then kept in sealed container. Comparison of compositions between alumina, magnesia, and silica can produce materials in various phases. Manipulation of composition with a certain mole fraction can produce material with spinel phase that has good physical and mechanical characteristics, making it suitable for orthodontic bracket applications [15]. It has wavelength transmittance values of 0.25-5.0 m and hardness of 16 GPa.

### Geopolymer synthesis

Geopolymer was made by mixing synthetic aluminosilicates and alkali activator to form slurry paste with variations conformed to Table 1. It was then poured into Ø5mm x 5mm height cylindrical mould (Figure 1) and heated in a 60°C ovens for 1 hours. After setting, it was removed from mould then calcined at 1400°C for 2 hours.

**Table 1.** Mix Design of Geopolymer

No	Name	Aluminosilicate: Activator ratio	NaOH molarity
1	GP-8M	3:2	8M
2	GP-14M		14M



**Figure 1.** Cylindrical mould for Geopolymer

### Characterization

The hardness of resulting Geopolymer was measured using LECO-Japan M-400-H1/H2/H3 instrument with 100g indentation for 15 seconds in Solid Oxide System Laboratory, Department of Metallurgical Engineering Institut Teknologi Bandung. The Vickers Hardness (HV) value was calculated using equation (1).

$$HV = 1,8544 \times \frac{P}{d^2} \quad (1)$$

The Geopolymer was crushed into powder form after hardness testing and was collected for characterization purposes. The X-Ray Diffraction (XRD) measurement was performed on Philips Diffractometer PW1710 with Cu as anode. Resulting diffraction pattern was compared to the Joint Committee on Powder Diffraction Standards (JCPDS). Scanning Electron Microscope (SEM) measurement was performed with HITACHI SU3500. Since the specimens are not conductive, it was coated with gold or carbon with Ion Sputtering Method. Both of these characterizations were conducted at the Center of Advanced Sciences (CAS) Institut Teknologi Bandung, Indonesia.

**Result and Discussions**

*1. Vickers Microhardness Analysis*

Table 2 shows the resulting Vickers microhardness test of Geopolymer.

**Table 2.** Vickers Microhardness result

No	Name	Vickers Microhardness (HV)
1	GP-8M	259.15
2	GP-14M	289.90

The hardness values of the two sample groups were analyzed using the Statistical Product and Service Solutions (SPSS) Software version 17.0. The hardness value tests was analyzed with normality and homogeneity tests. The normality test used the saphiro-wilk test with p-values in GP-8M and GP-14M are 0.398 and 0.119, respectively. The ( $p>0.05$ ) indicating that the data were normally distributed. The homogeneity test of the data was carried out using the Levene test with a p-value in GP-8M and GP-14M of 0.631 ( $p>0.05$ ) indicating that the data were homogeneous. The data were then statistically tested by independent t-test to determine the difference in hardness of the two sample groups. The results of the independent t-test showed a p-value of 0.271 ( $p>0.005$ ).

This indicates that there is no significant difference between the two sample groups. The mean, standard deviation, and independent t-test results of synthetic aluminosilicate based Geopolymer with 8M and 14M activator solution variations was shown in Table 3.

**Table 3.** Mean, Standard Deviation, and Results of Independent T-test of Geopolymer

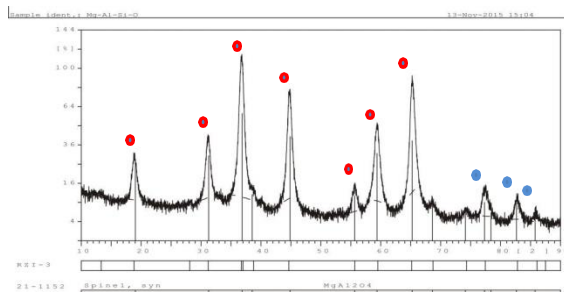
No	Name	VHN Average $\pm$ SD	p-value
1	GP-8M	259.15 $\pm$ 9.45	0.271
2	GP-14M	289.90 $\pm$ 7.75	

The results of the Vickers hardness test in shows different mean hardness values in the two groups. GP-8M and GP-14M have a mean hardness of 259.15 and 289.90 VHN, respectively. Sampling of the two groups was carried out with a similar procedure and at the same time. The difference between the two groups is in the concentration of NaOH in the activator solution, so it can be said that the difference in hardness values between GP-8M and GP-14M is influenced by the concentration of the activator solution. GP-14M with a concentration of 14 M has a higher hardness than 8 M. This is because the OH- ions in sodium hydroxide has a role in producing bonds between Al and Si to form the main framework. The main skeleton will form a chain of siloxo, siloxo, and disiloxo chains. Na+ ions will strengthen the bonds between the main framework, so that the activator with a higher amount of NaOH will have a higher hardness. Higher water contents lead to a decrease in compressive strength [16]. H+ ions in water will interfere with the activity of OH- ions in producing bonds between Al and Si. The lack of OH- ions in the polymerization process will cause the crystallization to take place imperfectly. The bond between Al and Si will form the main framework of sialate (Si-O-Al-O), siloxo sialat (Si-O-Al-O-Si-O), or disiloxo sialate (Si-O-Al-O-Si-O-Si-O).

Na<sup>+</sup> ions will strengthen the bonds between the main framework to form (Na<sup>+</sup>)-(Si-O-Al-O) and also balance the negative charge [17]. The Vickers hardness test produces data that varies, but the results of the normality and homogeneity test with p-value > 0.05 indicate that the distribution of data between the two groups is normal and homogeneous. The data were then tested differently using independent t-test with p-value > 0.005 indicating that there was no significant difference in the two sample groups. The test results, which showed that there was no difference between the two sample groups, were due to the fact that the hardness values at several points in the two sample groups had very small differences. Difference in hardness values was due to the uneven distribution of particles in the sample since the hardness test is local at random points, so that the indentation in the hardness test affects different particle components. The hardness value of Titanium-based orthodontic brackets is 165-371 VHN. The resulting hardness test shown that the average hardness value of both GP-8M and GP-14M were in the metal-based bracket hardness value range. This shows that Synthetic aluminosilicate based Geopolymer are potential alternative material for metal-based brackets.

## 2. XRD Analysis

XRD characterization results in the form of a diffractogram then processed using X Powder software. Figure 2 shows the diffractogram of XRD results of synthetic aluminosilicate powder.

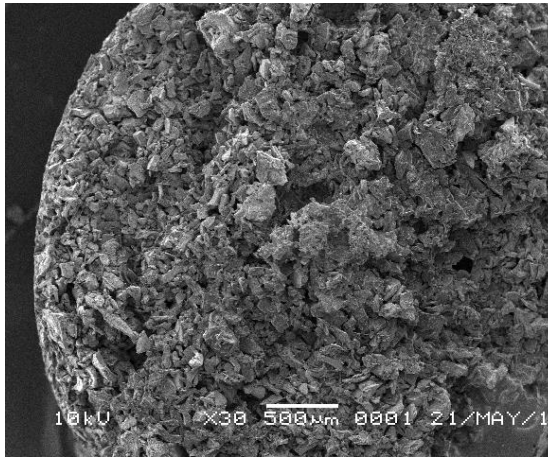


**Figure 2.** XRD diffractogram of synthetic aluminosilicate (Note: spinel (red) and cristobalite (blue))

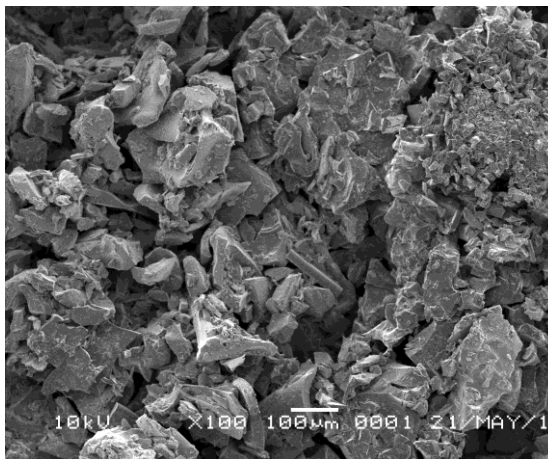
XRD characterization analysis showed that the nanocomposite powders were spinel and cristobalite. The peaks on the diffractogram are narrow and sharp indicating that the material is crystalline. The first seven peaks indicate spinel with the formula  $MgAl_2O_4$  with a monoclinic crystal form. The next peaks indicate the crystalline phase of cristobalite with the formula  $SiO_2$  with a tetragonal crystal form. Cristobalite formed from residual silica compound that does not react completely. The presence of cristobalite did not affect the physical and mechanical properties of spinel because the silica used was only 5% of the total volume of the initial solution. This XRD analysis shows that the composition of the nanocomposite powder with a mole fraction of alumina magnesia silica of 70:25:5 will produce spinel in accordance with the predictions of the ternary diagram of  $MgO:Al_2O_3:SiO_2$ . The ternary diagram shows the materials that can be formed from a mixture of alumina, magnesia, and silica. A number of comparisons between the three compounds will produce different materials with different characteristics. Compounds that can be formed from these three oxide are sapphirine, spinel, periclase, corindon, fouesterite, mullite, cordierite, protoenstatite, trydimite, cristobalite, and enstatite. XRD results show a picture of narrow and sharp peaks. This indicates that spinel has a crystalline phase. The crystalline phase is expected because in this phase spinel has a stable and unreactive crystal so that its physical and mechanical properties are better. The crystalline phase is achieved through a calcination process or a heating process at a temperature below the melting point to produce a stable and unreactive oxide compound. This indicates that the calcination temperature of 1000°C used in this study is optimal to produce spinel with a crystalline phase. A stable spinel has been formed at 700°C, but pure crystalline has only been formed at a temperature of at least 800°C. The peak intensity will gradually increase until the calcination temperature is 1000°C-1200°C. Through this analysis, it can be concluded that the synthesis of spinel nanocomposites using a bottom-up approach with the sol-gel method was successful.

### 3. SEM Analysis

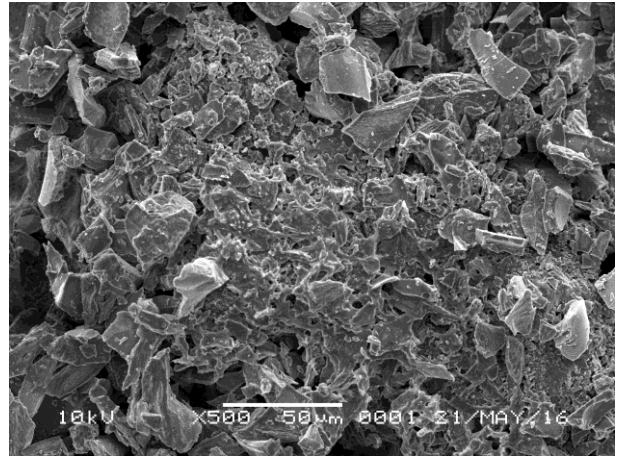
SEM characterization was carried out on hardened Synthetic Aluminosilicate based Geopolymer samples that had been sintered at a temperature of 1400°C with a handling time of 2 hours and was carried out by observing the nanocomposite cross-section. Resulting images of GP-8M and GP-14M with different magnification were presented in Figure 3-8.



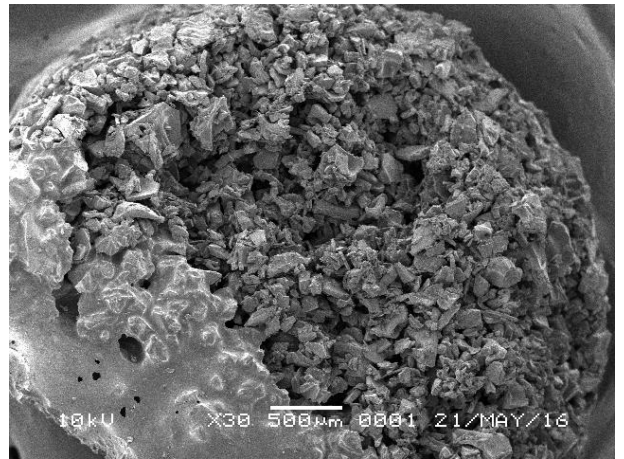
**Figure 3.** SEM Images of GP-8M (Magnification 30X)



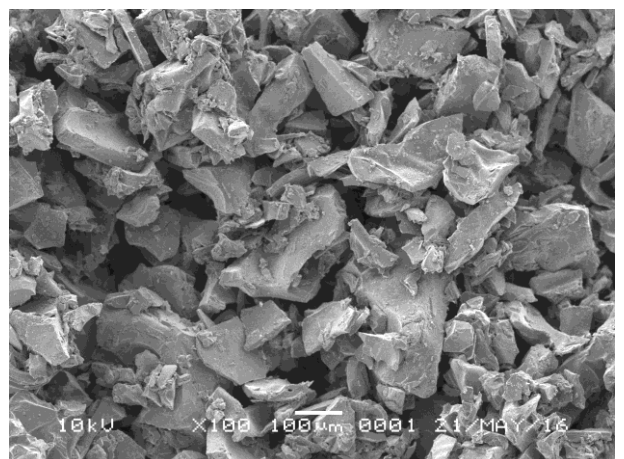
**Figure 4.** SEM Images of GP-8M (Magnification 100X)



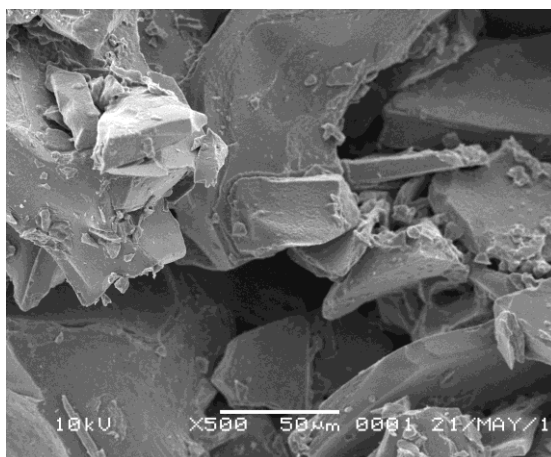
**Figure 5.** SEM Images of GP-8M (Magnification 500X)



**Figure 6.** SEM Images of GP-14M (Magnification 30X)



**Figure 7.** SEM Images of GP-14M (Magnification 100X)



**Figure 8.** SEM Images of GP-14M (Magnification 500X)

Figures 3-5 shown the results of surface morphological characterization of GP-8 magnified 30, 50, and 100 times, while Figures 6-8 shown the results of surface morphological characterization of GP-14 magnified 30, 50, and 100 times, respectively. The image shows that bonding has occurred between nanocomposite particles, but the structure is not yet solid which is indicated by the presence of porosity. SEM images of GP-8M and GP-14M with magnification of 500X shows that the two groups have different particle sizes. The average particle size of GP-8M and GP-14M were 1.25-25 $\mu$ m and 6.25 – 87 $\mu$ m, respectively. Sampling of the two groups of nanocomposites in this study was carried out with a similar procedure and at the same time. The difference between GP-8M and GP-14M are the concentration of NaOH which will increase the pH of the activator solution. The concentration of the activator solution affects the particle size and pores of the nanocomposite. Particle size of GP-14M was higher because of 14M activator solution having a higher pH which means there are a greater alkali activation strenght due to the higher sodium (Na) means higher alkali content in NaOH. This compound act a metal center will attract other compounds to bind, resulting polymerization in GP-14M will occur in a shorter time. This causes compaction to occur rapidly and causes the formation of larger particles.

Larger particle size is produced by a higher pH [18]. The ratio of water and NaOH will affect water absorption which has an impact on particle size [19]. The SEM test results in all figures show that both GP-8M and GP-14M have porosities which indicated that the material has a non-solid structure. This is caused by the compaction process or an imperfect sintering process. The sintering process is closely related to the heating temperature. The temperature used is not optimal because it is still far from the spinel melting point of 2135°C. This causes the particles to only diffuse and leave gaps or pores between the particles. The maximum density can be achieved by heating close to the melting point of the material. Diffusion accelerates at higher temperatures [20]. Sintering at temperatures close to the melting point will cause solidification of the material.

#### Acknowledgements

We thank Advanced Materials Processing Laboratory, Institut Teknologi Bandung as research venue for this study.

#### Conclusions

This study concludes that Synthetic Aluminosilicate powder made from alumina, magnesia, and silica precursors with a composition ratio of 70:25:5 was successfully synthesized by the sol-gel method. XRD test showed that the spinel crystalline phase was formed. Independent t-test statistical test showed that there was difference in hardness between synthetic aluminosilicate Geopolymer nanocomposite with different activator solution concentrations. Geopolymer with 14M activator solution concentration had a higher mean hardness value (259.15 VHN) than 8M activator solution (289.90 VHN). The resulting SEM images showed the morphological characterization of Geopolymer, where GP-14M had a larger particle size than GP-8M. The hardness of synthetic aluminosilicate based Geopolymer can be be used as an alternative material for metal-based brackets according to hardness value.

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