The polymer nanocomposite characteristics on various mixtures and mixing times in simple mixing method

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ARTICLE INFO	ABSTRACT
	Science and technological developments in the material field have been currently dedicated to a super strong material potential based on nanotechnology. The super strong material can be created from the mixture of epoxy-resin polymer and SiO_2 (silicon dioxide) nanoparticles. Polymers exist as a nanoparticle adhesive due to nano-SiO_2, which possesses a high amorphic level, resulting in a stronger, more flexible, and stiffer combination than the current super strong material. The advantages of nanocomposite polymer using epoxy- resin and nano-SiO_2 produce strong and light products with an easier production process, utilizing local materials that can improve the following material quality. This study used four material variations, namely P30, P35, P40, and P45, combined with nanoparticles at 0%, 1%, 2%, 3%, and 4%. Based on the results, the highest compressive strength level was found on the PNK 40 EH2:1N1 mixture at 53.18 MPa with 1627 kg/m³ weight density. From the X-Ray Diffraction (XRD) test results, the following mixture had the lowest amorphic phase, while Fourier Transform Infra-Red (FTIR) test results showed that the following mixture absorbed more hydrogen elements, and Scanning Electron Microscope (SEM) observation on the following material mixture had more organized particle distribution.

1. Introduction

Most engineering applications in this era are currently demanding materials with higher strength ratios than their weight. Polymers are materials with suitable characteristics for engineering material requirements. Polymers are light and corrosive-resistant materials, but weaker strength can be overcome by adding the second phase material into the polymer matrix that elevates the mechanical and thermal properties (Singh, Kumar, & Jain, 2018).

Epoxy resin is one of the thermosetting polymers often used as an adhesive agent, thermal isolator, aviary material, and marine material. The epoxy resin is more advantageous than other polymers, due to its stable mass, small shrinkage value, and resistance to chemical attack. The epoxy resin has hard, insoluble, and infusible characteristics. Reactions occur between epoxy and hardener to produce polyfunctional amines, acids, phenol alcohols, and thiols. The curing agent influences the final characteristics of epoxy resin (Gu *et al.*, 2016). Epoxy-resin has high material strength and resistance levels, but the cross-link reaction can cause epoxy-resin becomes brittle. The addition of nano-SiO_2 can decrease this reaction based on the nanoparticle level used. These nanoparticles can improve the epoxy-resin strength by reducing the cracks that occurred due to highly cross-link reactions (Shameem *et al.*, 2020).

Composite material is an artificial material composed of two or more materials with different physical or chemical characteristics that provide a positive impact. The composite material can be classified based on its reinforcing materials, such as particle partition, fiber composite, and tube composite. Based on its formation materials, there are two types of main components, namely reinforcing materials as partition and acceleration agents and binding materials called matrix. Matrix is used to bind the partition materials and obtain a solid material structure that has hybrid characteristics from its formation materials. Composite material is created to obtain strength, high durability, easier production, easier improvement, and corrosion-resistant. These outcomes require an exact understanding of the formation material condition and its mixing method.

Nanotechnology is a material engineering capability in a nanoscale form. The use of nanotechnology in materials is limited to the implementation, rather than theoretical understanding of the nanoscale. The result among nano-, chemical-, and quantum mechanical technology is a chemical molecule and nanoparticle interactions, followed by their mechanical condition on a quantum scale. The impact of nanoparticles in chemical molecules results in a chemical compound originality loss in a precursor mixture, which causes a reaction based on the intuition of its composite materials. As read in the X-Ray Diffraction (XRD), Fourier Transform Infra-Red (FTIR), and Scanning Electron Microscope (SEM) test results of nanocomposite polymers, readers can no longer identify the chemical reactions due to epoxy and hardener usage, but should review the chemical reactions occurred due to the use of partition and nano-SiO_2 (Ramsden, 2017).

The nanotechnology and quantum mechanics are associative understanding, whereas quantum mechanics explain the smallest system with dynamic energy and movement. The quantum effect due to nanoparticle addition when induced perfectly on polymer molecules by shortening the electron distance and narrowing the particle room called as Bohr radius. Electrons from polymer molecules are clashed with electrons of nanoparticles, forming a quantum binding, as the nuclear atom distance is enclosed to produce more solid material, so this interaction can improve one of the object matrix characteristics (Ramsden, 2017).

Nanotechnology in construction aspect is advantageous to reduce cement production by 10% and CO_2 gas emission by 5%, these positive impacts are welcomed well by using polymer material substitution from concrete. As mentioned in this study, the binding material used resin epoxy that produced zero gas emission due to chemical reactions between pozzolan and water.

A nanocomposite is a material combination composed of nanoparticles and polymer molecules mixed with *in-situ* model (Hassan, 2019). The nanocomposite polymer is named as a nanoparticle-added composite material (Shameem *et al.*, 2020) or matrix added with the nanoscale reinforcing material (Hassan, 2019). Nanoparticles increase the material characteristics significantly compared to plain matrix (Ismail *et al.*, 2019). Therefore, this study used a similar naming as the materials were composed of polymers and nanoparticles.

Studies regarding polymers and nanotechnology combination still provide a wider discussion about the atom interaction in nanoscale condition. Discussions in nanocomposites finds several problems that should be studied further, such as partition materials as reinforcing materials from composite materials, material resistance against increased temperature, temperature and electricity propagation, material preparation. From these topics, an appropriate polymer type will be obtained to become polymer host, micro partition, and the relationship between polymer and catalyst molecules. The addition of nano-silica in elastomeric polymers is a material engineering between organic and inorganic materials that produces sol-gel reaction during polymerization process.

The nanoparticles used as nanocomposite materials include nano-clay, carbon nanotubes, carbon nanofiber, nano graphene, and nano-silica, compared to the characteristics of the polymer matrix added with the nano-clay and micro-clay, therefore the test results were discussed as the nano-effect occurred in the material (Khan, Saeed and Khan, 2019). Meanwhile, this study used nano-SiO_2 produced from the silica fusion process with silica content of 95.5%, the use of this material is expected to affect significantly.

The addition of nano-aluminum in polyethylene polymer type produced pores at 62-110 nanometer, the use of nano-SiO_2 as polystyrene polymer type reduced its polymerization level (Tatar *et al.*, 2019), while this study aimed to identify the effect of nano-SiO_2 in thermosetting polymer such as epoxy-resin, which may have a similar trend or produce a different impact.

The benefit of epoxy nanocomposite is rarely studied (Gu *et al.*, 2016), so this study used the following material as part of synthetic material discussions in construction field. The nano-boron conditioned with a couple agent to increase the nanoparticle adhesion in polymer molecules could improve the strength by 21 – 54%, compared to the purified epoxy-resin. The addition of nanoparticles in epoxy reduces the electrical conductivity and material resistance against high temperature (Gu *et al.*, 2016), so the test materials are expected to be used for partition or construction materials in the buildings. The technology application in the construction sector can exceed the concrete technology (Metaxa *et al.*, 2021), based on the compressive strength and specific gravity obtained beyond expectations, resulting in a light density and high strength.

In this study, nanocomposite polymer material fabrication was applied based on the previous literature approach, then

adopted following the easiest method. The approach was formed as a nanocomposite material fabrication with simple mixing method to obtain high suppressive strength and light weight density material.

2. Materials and Methods

The material types and methods used in this study were based on the trial and error experimental results, following the combination and comparison of several previous literatures, which reported that use of nanoparticles and polymer materials.

2.1. Materials

In this study, the material composition contained epoxy-resin, hardener, sieve no 16-passed sand, and nano-SiO$_2$ particles. This composition was combined from the previous literatures about the impact of nano-silica sand on epoxy-resin and the use of nano-SiO$_2$ in epoxy-resin (Zheng *et al.*, 2019).

The use of nano-SiO$_2$ on the test materials were at 0%, 1%, 2%, 3%, 4%, whereas these numbers were odd numbers which were occurred its effect on the test material, the ratio of epoxy resin and hardener was 2:1 as the standard of epoxy fabrication resin used in this study. As for previous studies regarding the use of nano-SiO$_2$ on epoxy resins, the 2%, 4%, 6%, 8% dose with the ratio of epoxy and hardener at 100:30 were applied (Zheng *et al.*, 2019), while another literature used nano-SiO$_2$ used at 1%, 1.5%, 2%, 2.5% with a ratio of epoxy and hardener at 100:80 (Singh, Kumar and Jain, 2018).

The fine aggregate percentages were used at 30%, 35%, 40%, and 45%. This consideration was applied based on the SEM results on a micro-usage, producing a sharp angle and an irregular surface that caused a great crack occurrence. In contrast, nanoparticles produced finer surface and higher strength level due to relatively stable energy dissipation (Tatar *et al.*, 2019). In this study, the partition used was the nano-microparticles and microparticles mixture, whereas micro partition was used to find the optimal and economical mass material, while nanoparticles were used as accelerator and reinforcing agent in the polymer host, producing new characteristics on the nanocomposite polymers.

The materials used were named based on their compositions, which produced the first variation of PNK P.30-E.H.2:1-N.0. The following material code was based on several literatures by taking the abbreviation of the materials such as polymer matrix composite (PMC). For simplifying the material name, therefore:

PNK   = Nanocomposite polymer
P   = Sand
E.H   = Epoxy and Hardener
N   = Nano-SiO$_2$

2.2. Methods

The methods used in this study was a simple mixing method. There were no special treatments during the material preparation, mixing, and composite material pouring. In this method, the material was produced through direct mixing, solution mixing, and in-situ polymerization process (Shameem *et al.*, 2020). Increased temperature was absent in the mixture variation and material formwork, for ensuring the optimal particle distribution, the first stage, the epoxy resin was mixed with the SiO$_2$-nanoparticles for 10, 15, 25, and 30 minutes at room temperature used for mixing variations had an optimal composition. After the nanoparticles were evenly mixed, hardener and fine aggregate filler were added and mixed together for 5 minutes, then poured in a steel formwork. The simple mixing method used in this study was different from the previous study conducted by (Singh, Kumar and Jain, 2018), whereas the method used the addition of methyl ethyl ketone solution to reduce the viscosity level and could evaporate at 70°C with 10 minutes of mixing time, while (Zheng *et al.*, 2019) performed a curing process in a vacuum oven, then stood in a water bath for 16 hours at 40°C. A special treatment was absent as the epoxy-resin used had a factory specification with extremely low viscosity level, so the simple mixing method is advantageous to simplify the material production and suitable for commercial scale. The material preparation method carried out by (Kanniyappan *et al.*, 2017) applied nano-silica particles dried in an oven at 120°C for 5 hours to ensure that no moisture content, in contrast to the simple mixing method used to prepare the nano-SiO2 without using an oven, as the nano-SiO$_2$ could melt when exposed to heat. The experiment flow chart can be seen further in Fig. 1.

Fig. 1. Flow chart of nanocomposite synthesis with simple mixing method

3. Results

The test results on the materials produced included setting time, weight density, suppressive strength, X-Ray Diffraction (XRD), Scanning Electron Microscope (SEM), and Fourier-Transform Infrared Spectroscopy (FTIR). These test results are commonly used to identify the effect of ratio aspect, material function, and total fractions (Linec and Muši˘, 2019).

3.1. Setting Time

The setting time test was performed to determine the initial and final hardening process period. The hardening period of PNK P.40-E.H.2:1-N.0 material was obtained at 65 minutes with the final hardening period was obtained at 140 minutes. This hardening period was longer than the PNK P.40-E.H.2:1-N.1, PNK P.40-E.H.2:1-N.2, PNK P.40-E.H.2:1-N.3, and PNK P.40-E.H.2:1-N.4 materials, that were respectively shorter. Shorter binding time was caused by the addition of nano-SiO_2 in the material composition. The addition of nano-silica could reduce the hardening period (Kanniyappan *et al.*, 2017). The combination of inorganic partition and nanoparticles in polymer matrix results in a hardening initiation on the polymer composite (Khan, Saeed and Khan, 2019), which reduces the formwork removal time on the test material. The addition of nanoparticles in polymers also affects the polymer solution viscosity level, whereas the higher nanoparticles addition level, the higher viscosity gained. Greater nanoparticles addition causes the slower hardening process in the polymers, as optimal nanoparticle numbers can induce the initial hardening period called polymer nucleation.

The effect nanoparticle addition can further affect the hardening transition period that occurs whenever the material is in a soft state, before heading to the initial hardening period. The increased and decreased transition period are influenced by the type of particles and their interactions with polymer molecules. The hardening transition is also influenced by the working method and material condition, before and after pouring the material. Nanoparticles also reduce the cross-linking reaction level in thermosetting polymers, as nanoparticles can reduce the catalytic effect (Khan, Saeed and Khan, 2019). The material hardening period can be seen in Fig. 2.
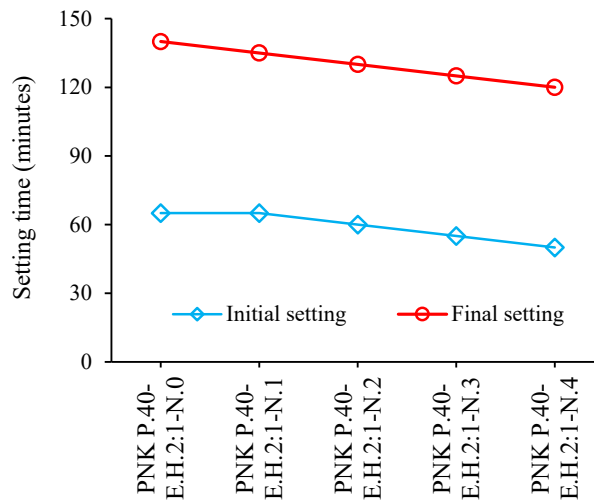

Fig. 2. Setting time

3.2. Density

Density of four mixture variations increased respectively, following the volume of sand usage. The first mixture of PNK.P30EH2:1N0 obtained 1,621 kg/m³ density, and the maximum density was obtained from the fourth mixture of PNK.P45EH2:1N0 at 1,807 kg/m³. This condition occurred due to gravity specificity of the sands that extremely influenced the epoxy matrix density, whereas the higher sand volume applied, the smaller volume ratio with epoxy-resin obtained. Decreased weight density was occurred after the addition of nano-SiO_2, whereas the use of nano-SiO_2 at 1, 2, 3, and 4% showed a lowering graph. There was a weight density comparison obtained from several nanocomposite materials, such as MMT polymer composite at 2,830 kg/m³ and epoxy-nano silica composite at 1,920.5 kg/m³ (Linec and Muši˘, 2019), while weight density in this study obtained the lowest value at 1577 kg/m³ and the highest value at 1683 kg/m³. The density obtained had a lighter value than two previous studies, as this study used fine aggregate partition to gain an optimal mixture. Density obtained could be classified as light concrete type among 1,500-2,200 kg/m³ (Murali, 2019), therefore the materials produced can be used a partition or a construction material due to having a light weight value.
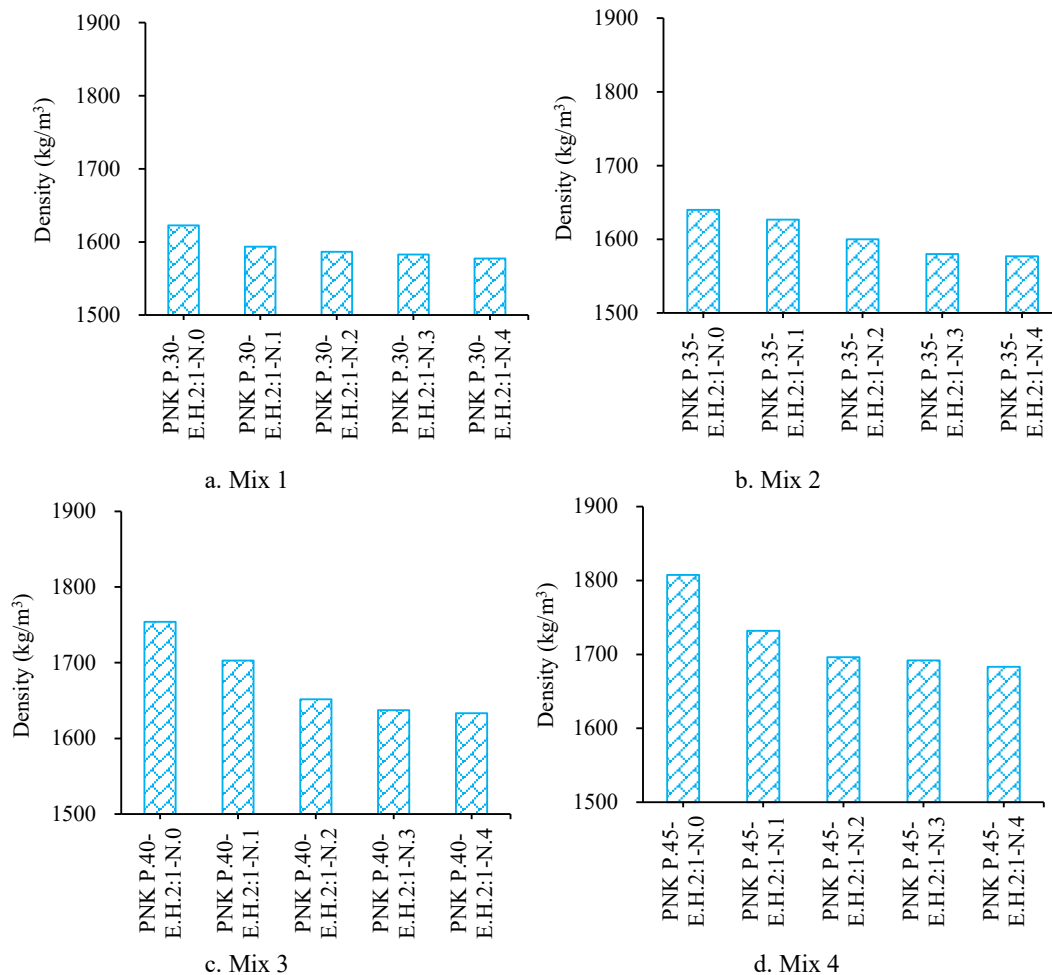
Fig. 3. Density

The addition of nano-silica sand in epoxy-resin should have increased the density, proportional to the level of nano-SiO_2 used, however this study obtained different results, whereas the higher nano-SiO_2 level used, the lower material density gained. This phenomenon was caused by differences in the types of nanoparticles. In this study, nanoparticles from the fusion of SiO_2 compounds were very susceptible to the increased temperature, as the nano size used reached 100 nm. The addition of nano-silica sand to the epoxy-resin should have increased the density, proportional to the levels of nano SiO_2 used, however different results were obtained in this study. This phenomenon was caused by the different types of nanoparticles used. The density of materials obtained can be seen in Fig. 3.

3.3. Compressive strength

The addition of 4% nano-SiO_2 in epoxy nanocomposite increased the tensile strength level at 30.57%, flexural strength at 17%, and flexural modulus at 76% (Singh, Kumar and Jain, 2018). The addition of 1.5% nano-SiO2 in epoxy-resin obtained 36.38 MPa pressure level (Zheng et al., 2019). These results were still below the material strength obtained from different compositions and without special treatment at 54.18 MPa. Furthermore, the use of 10% nano-clay with the mixing homogeneity level reached exfoliated phase could increase the epoxy-resin at 7% higher than the plain state, while this study used 1% nano-SiO_2 in matrix composite with the optimum partition level of 40%, besides increasing the strength at 17.14% higher than the material with similar partition and without using nano-SiO_2. The use of nano-silica sand in epoxy-resin at 15-20% could reduce the strength level, while the nano-SiO_2 level among 2-4% in nanocomposite polymer obtained a relatively decreased strength level material. The compressive length of the materials can be seen in Fig. 4.
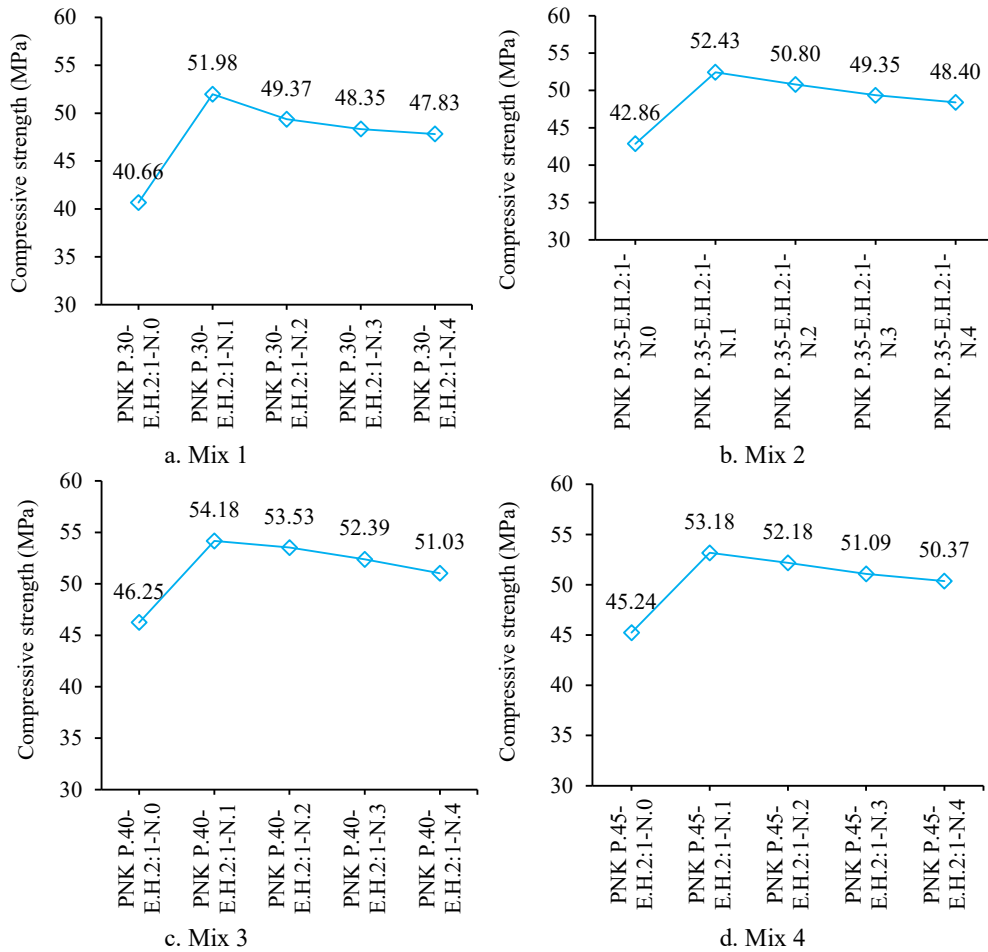
Fig. 4. Compressive strength

In Fig. 5, the mixing time affected the strength level of the nanocomposite polymers. This condition occurred due to nanoparticles in the matrix polymer were evenly distributed. The distribution of nanoparticles was proportionally linear to the mixing time. Materials with more homogenous composition had more stable intermolecular attractions and received a better load. The PNK 40 EH 2:1 was the most optimal material from the previous experiment with a relatively light weight and high strength. The mixing time effect on the PNK 40 EH 2:1 material can be shown below with the addition of 1% nano.
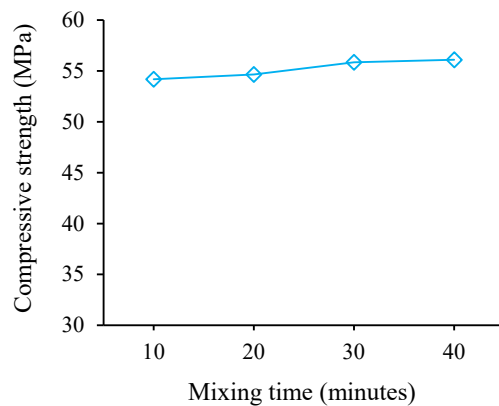

Fig. 5. Compressive strength of PNK40EH2:1N1

3.4. X-Ray Diffraction

The XRD test was performed to measure the atomic distance among the crystal layers formed (Kanniyappan et al., 2017). The nano-silica content was 0% in the P.40-E.H.2:1-N.0 material. The XRD graph of P.40-E.H.2:1-N.0 material was identified quantitatively to have dominant amorphic phase, whereas the more amorphic, the better mixture formed in the material. At

0% nano-silica used, the material obtained the amorphous region at 10526 with the crystalline area of 583.45 or 5.5%. The addition of 1% nano-SiO$_2$ in P.40-E.H.2:1-N.1 material had more stable XRD pattern than the addition of 0% nano-SiO$_2$ with the amorphous region at 13296.8 and crystalline area of 5.45%. The addition of 4% nano-silica in the P.40-E.H.2:1-N.4 obtained the increased peak phase. Quantitatively, the crystalline area formed in the P.40-E.H.2:1-N.4 mixed material was 989.84 with the amorphous region of 9064. The crystalline percentage in the P.40-E.H.2:1-N.4 material was obtained at 14.9% greater than the previous test materials. This condition was caused by the excessive use of nano-silica that could influence the polymer bond, reducing its adhesive power to become brittle. In P.40-E.H.2:1-N.4 material, the O$_2$Si compound was found as the chemical formula of nano-silica and fine aggregate. There were also some additional materials such as F$_7$H$_{12}$N$_3$Zr as ammonia material, produced from the decomposition of organic material that was possibly carried from fine aggregate.

Based on the XRD graph, the P40.EH 2:1 N 1-4 material had a higher amorphic phase than its crystalline level. The graph peaks formed showed that the nanoparticles and polymer molecules were unbonded. The epoxy-resin is a polymer with high amorphic phase, and nanoparticles were added to this polymer due to its size closed to the polymer molecules. From the XRD results, the PNK P40 EH 2:1 N1 material was the optimal material by showing the irregular graph pattern and amorphic characteristics.

The nano effect provides increased characteristics beyond the predicted hypothesis, whereas the nano effect was not found in partitions with sizes exceeding the nanoscale. This condition was caused by the degree of crystallization and the degree of crystallinity, which affected the composite material hardening process. The use of nanoparticles with spherulitic shape was evenly distributed to each other, resulting in the interlocking surface bonds between nanoparticles and polymer molecules. The XRD results of PNK P 40. EH 2:1 N0 – 4 material can be seen in Fig. 6.
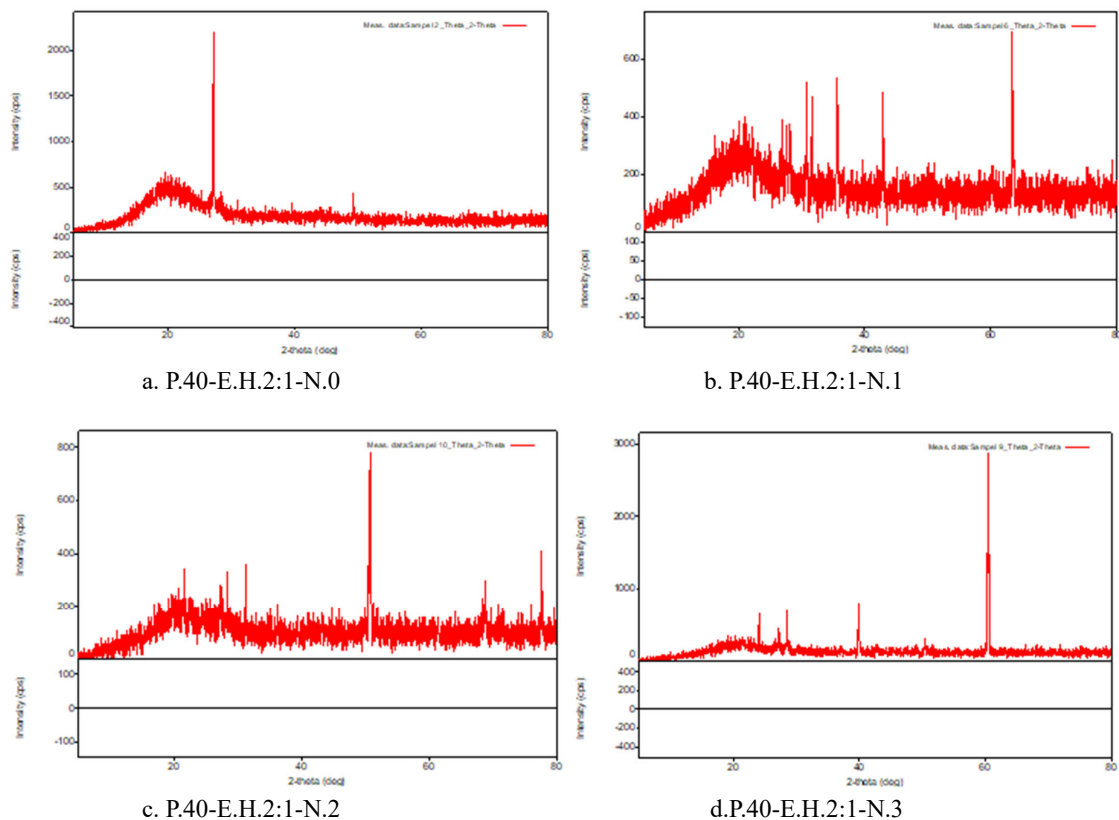
a. P.40-E.H.2:1-N.0

b. P.40-E.H.2:1-N.1

c. P.40-E.H.2:1-N.2

d.P.40-E.H.2:1-N.3

Fig. 6. The x-ray diffraction

3.5. Scanning Electron Microscope

The use of nano- and microparticles in matrix polymers has different impact. From the SEM results, the use of microparticles obtained a sharper angle and an irregular surface, which caused a great crack occurrence. In contrast, nanoparticles produced plainer surface and higher hardness level due to relatively stable energy dissipation (Kanniyappan *et al.*, 2017). Nanocomposite is a material combination of nanoparticles and polymer molecules within the *in-situ* method. Based on the particle distribution and homogeneity level, nanocomposites are divided into three types, namely immiscible, intercalated, and exfoliated. In the SEM observation results, the P40.EH 2:1 N 1-2 materials had an intermediate mixing level or intercalated type, as the homogenization process still used the simple mixing method. The SEM test results from P40.EH

2:1 N 0-2 materials showed a relatively homogenous nano-SiO_2 particle distribution that could be seen vaguely, so the nano-SiO_2 distribution in epoxy-resin has entered the intercalated level, as a good beginning to produce nanocomposite polymers with exfoliated level that had no different between the polymer molecules and nanoparticles using an ordinary magnification.

Therefore, the mixing and stirring method for material production has been sufficient to fulfill the quality standard of nanocomposite polymers. The SEM observation results in epoxy-composite showed many pores and cracking patterns on the material surface, due to the increasing levels of nano-silica sand, which were unmixed maximally, causing a decreased material strength level (Kanniyappan *et al.*, 2017). From the SEM results, material with 1% nano-SiO_2 had plainer morphology and more even particle distribution, in contrast to the material with 2% nano-SiO_2, which had more messy morphology and heterogenous particles, followed by many cracks, causing the decreased strength level, proportional to the increased nano-SiO_2 percentage. Based on the SEM observational results on epoxy-resin, the nano-SiO_2 concentration at 1-1.5% showed plainer surface due to the formation of new surface in greater numbers that could improve the relationship of nano-SiO_2 particle and epoxy to receive greater energy dissipation distributed in the new surface that produced a significant material characteristic improvement. In nano-SiO_2 concentration at 2-2.5%, heterogenous particle distribution was occurred, resulting in an agglomeration in several area due to particle bulking (Zheng *et al.*, 2019). In addition, the SEM results of this study showed a plain surface and a relatively homogenous distribution in the material with 1% nano-SiO_2, while messier surface was found in the material with 2% nano-SiO_2. These phenomena become the main reason of strength level in the material.

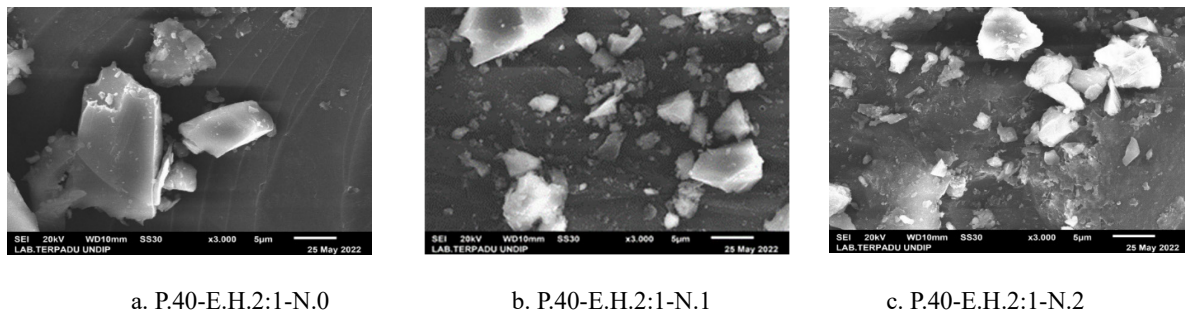| a. P.40-E.H.2:1-N.0 | b. P.40-E.H.2:1-N.1 | c. P.40-E.H.2:1-N.2 |

Fig. 7. Scanning Electron Microscope

The P.40-E.H.2:1-N.0 material can be qualitatively observed in Fig. 7a. A shiny area was found, as a polymer piece that resembled acrylic properties, which could reflect the light. The darker area was identified as a fine aggregate used in the material composition, which could absorb light. In addition, the silica content was obtained from the fine aggregate used in the material composition of the test object. In the P.40-E.H.2:1-N.0 material, cracks were found due to the crystalline phase occurrence based on the XRD test, which resulted in high epoxy and hardener reaction that caused cracks in the material. The P.40-E.H.2:1-N.1 material was a material with 1% nano-silica. The P.40-E.H.2:1-N.1 material had the highest compressive strength supported by the qualitative SEM results, based on its more homogeneous particle. The homogeneous particle dispersion could increase the material strength to accept the load. The shiny area was an epoxy-resin that resembled acrylic properties, which could reflect x-ray rays. The epoxy-resin serves as a material composition binder. The darker area was the fine aggregate used in the material composition. In the P.40-E.H.2:1-N.1 material, there were fine particles scattered in each material area as nano silica particles used in the material. The distribution of nano-silica was more homogenous at material with 2% nano-SiO_2 concentration, which means that the material has more compressive strength level. The SEM results in the P.40-E.H.2:1-N.1 material can be shown in Fig. 7c. In this material, the compressive strength of material with 2-4% nano-silica decreased. The materials found problems in sustaining their shape, when accepting the axial load, causing a great lateral deformation, so the material is very suitable for receiving the tensile loads at high concentration levels. By adding nano-silica in epoxy-resin, the material can increase its adhesion level, so it will be more flexible (Jahangiri, 2019). In the PNK P.40-E.H.2:1-N.2 material, strength reduction was found at 1.88% smaller than the PNK P.40-E.H.2:1-N.2 material. The strength reduction was occurred due to agglomeration or uneven distribution of nano-silica particles in the material. Agglomeration could decrease the mechanic characteristics of PNK P.40-E.H.2:1-N.2 material, when accepting the loads and sustaining its shape (Kumar *et al.*, 2018).

3.6. FTIR

The FTIR was performed in the nanocomposite polymer material sample without nano-silica particles and with nano-silica particles at 1, 2, 3, and 4%. The results obtained from the FTIR test of the material is shown in Fig. 8. The data obtained from the FTIR results were interpreted using a correlation table for data comparation. Fig. 8a is the reading results of Table 1. All materials had a SiO_2 bond. The SiO_2 bond was identified from the vibration range of 1000-1250 cm^{-1}, whereas this vibration range indicates a vibration absorption of distributed Si-O-Si (Molina *et al.*, 2020). The absorption of Si-O-Si in each material was caused by the material composition with and without the nano-SiO_2 which similarly used a fine aggregate as material partition. The fine aggregate had a silica mineral as a main content of natural sand material. The chemical group absorption of P.40-E.H.2:1-N.1, P.40-E.H.2:1-N.2, P.40-E.H.2:1-N.3, and P.40-E.H.2:1-N.4 materials had seven bond groups.

The FTIR test in the P.40-E.H.2:1-N.0 material quantitatively can be seen its correlation data in Table 1. In the Table 1, the P.40-E.H.2:1-N.0 had seven bond groups with C-H bond as the reaction product of benzene and amine molecules in epoxy-resin and hardener. The carbon bond formed caused the material to become harder and more brittle. There was a vibration absorption of NO_2 and C-O as the chemical reaction product of polymer molecules and nano-SiO_2 particles. The absorption of C-O alcohol could reduce the material weight due to evaporation regarding the increasing temperature. The NO_2 compound could evaporate due to increasing temperature during the hardening process of the material. Qualitatively, the FTIR results of P.40-E.H.2:1-N.0 material is shown in Fig. 8.

Table 1. Material data interpretation

Group	Standard Frequency	P.40-E.H.2:1-N.0 (cm^{-1})	T	P.40-E.H.2:1-N.1 (cm^{-1})	T	P.40-E.H.2:1-N.2 (cm^{-1})	T	P.40-E.H.2:1-N.3 (cm^{-1})	T
C-H (alkane)	2850-2970	0	0	2884	108	0	0	0	0
C-H (aromatic)	690-900	695	80	694	96	695	90	695	85
O-H (hydrogen)	3200-3600	0	0	3245	106	0	0	0	0
O-H (acid)	2500-2700	0	0	0	0	2515	101	2515	100
C=C (alkene)	1610-1680	0	0	0	0	1641	103	1606	99
C=C (aromatic ring)	1500-1600	1504	90	1508	101	1547	103	1508	95
C=C (alkyne)	2100-2260	2159	91	2160	100	2159	90	2159	92
C=O(aldehyde)	1690-1760	0	0	0	0	1691	103	0	0
C-N (amine)	1180-1360	1180	85	0	0	0	0	0	0
C-O (alcohol)	1050-1300	1010	64	0	0	1691	103	0	0
NO_2 (nitro)	1500-1570	1504	90	1508	101	1503	100	1508	95
SiO_2 (silica)	1130-1160	1010	85	1007	91	1010	83	1008	68

Based on the FTIR results in the P.40-E.H.2:1-N.1, this material was produced by adding the 1% nano-SiO_2. The relationship of increased material strength at 1% concentration can be observed from the quantitative data of test sample. In Table 1, the P.40-E.H.2:1-N.1 material had seven groups, i.e. 5 groups of hydrogen and carbon bond. In optimal level, the use of nano-SiO_2 could produce more carbon bonds, producing a harder material level. In the P.40-E.H.2:1-N.1 material, there was a O-H hydrogen bond as the chemical reaction product of epoxy and hardener, this hydrogen bond was a binder agent of the material composition. Moreover, there were also NO_2 and C-O compounds that could evaporate due to the increasing temperature during material hardening process, causing the material weight density reduction. The FTIR results of P.40-E.H.2:1-N.1 material can be seen in Figure 8b. In the P.40-E.H.2:1-N.2, P.40-E.H.2:1-N.3, and P.40-E.H.2:1-N.4 materials, strength reduction was occurred. The strength reduction was caused by the formation of carboxylic acid vibration absorption in the materials. The carboxylic acid was a reaction product between polymer molecules that received nano-SiO_2 above the reaction capacity of epoxy and hardener. This condition produced the carboxylic acid with the O-H group. The more nano-SiO_2 used, the more carboxylic acid produced in the materials. The acid content caused the materials more susceptible, which reduced the adhesive capability of acrylic epoxy-resin. This susceptibility can be shown in the SEM results in Fig. 8c. The P.40-E.H.2:1-N.2, P.40-E.H.2:1-N.3, and P.40-E.H.2:1-N.4 materials also had alcohol and nitro groups. Both groups caused the material weight reduction, therefore the use of nanoparticles in excessive level can reduce the material weight level.
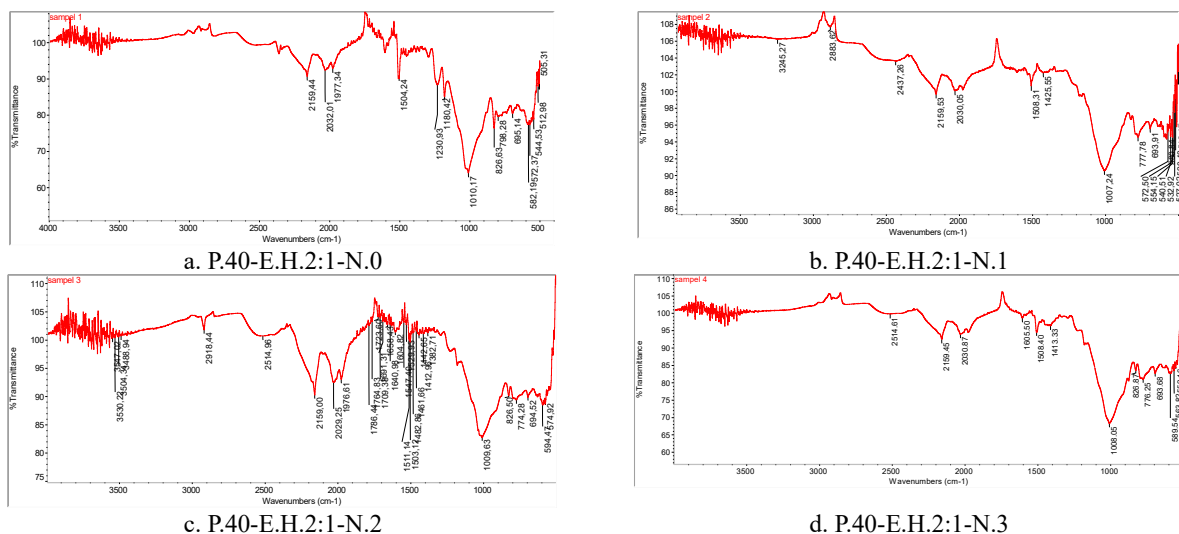

a. P.40-E.H.2:1-N.0

b. P.40-E.H.2:1-N.1

c. P.40-E.H.2:1-N.2

d. P.40-E.H.2:1-N.3

Fig. 8. FTIR

4. Discussions

4.1. Correlation of compressive strength and weight density

The weight density and compressive strength test results obtained the minimum compressive strength in PNK P.30-E.H.2:1-N.0 mixture at 40.66 MPa and weight density of 1,623 kg/m³. The optimum compressive strength was obtained from the PNK P.40-E.H.2:1-N.3 mixture for 30 minutes of mixing time at 70.76 MPa and weight density of 1,637 kg/m³. In Figure 9, the correlation of compressive strength and weight density indicates to be high, whereas the material weight was similar to the material density. High material density could increase the compressive strength obtained. Figure 9 describes that the PNK 40 EH2:1 material has a correlation with the strength and weight density values. The highest material strength was found when the material had a maximum weight density. This condition occurred as high weight density resulted in high density that could receive and distribute the load well. The average correlation results of weight density and compressive strength on nanocomposite polymer was 0.8784. The determinant coefficient (R^2) had a value approaching to number one value. This means that the weight density affects the concrete compressive strength, therefore producing the proportional weight density and compressive strength obtained from the linear regression equation based on the optimal material condition, namely: $f_c' = 0.0244 \, \rho + 11.647$.
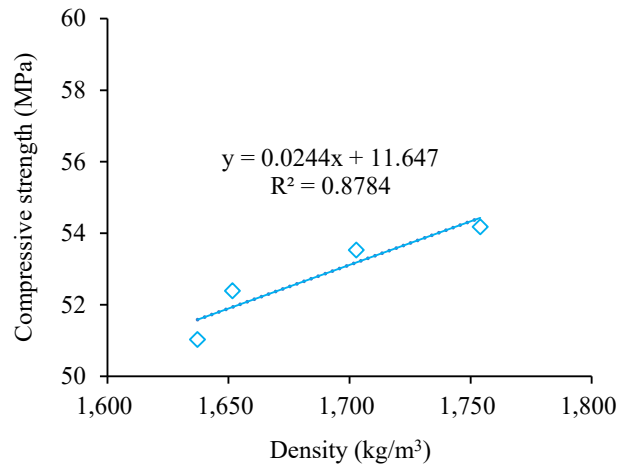

Fig. 9. Correlation of compressive strength and density

4.2. Correlation of Compressive Strength and XRD

The optimal compressive strength was obtained from the P.40-E.H.2:1-N.0, P.40-E.H.2:1-N.1, P.40-E.H.2:1-N.2, P.40-E.H.2:1-N.3, and P.40-E.H.2:1-N.4 materials. The highest compressive strength value occurred in the P.40-E.H.2:1-N.1 material was 54.14 MPa, as shown in Fig. 4c. The XRD results of P.40-E.H.2:1-N.1 material can be seen in Figure 6a. The P.40-E.H.2:1-N.1 material obtained a compressive strength value at 54.14 MPa as 17.09% higher than the material without nano-SiO₂ particles. From the XRD results, the nano-SiO₂ had silica purity level by 95.05%. The XRD results showed that the nano-SiO₂ was in an amorphic phase with irregular peak phase, as shown in Figure 6. The addition of nano-SiO₂ in polymer molecules increased the O-H hydrogen bond due to C-H epoxy and nano-SiO₂ Si-O-Si reaction that increased the matrix polymer, and the bonding power was increased in proportion to the increased material strength. The P.40-E.H.2:1-N.4 was a material with the highest nano-SiO₂ concentration at 4%. The compressive strength reduced at 51.03 smaller than the P.40-E.H.2:1-N.3 material. The P.40-E.H.2:1-N.2, P.40-E.H.2:1-N.3, and P.40-E.H.2:1-N.4 material reductions can be seen in Figure 4. The XRD results of P.40-E.H.2:1-N.3 material showed the cause of material strength reduction. The strength reduction was occurred due to the crystalline formation in the mixture product. The XRD results of P.40-E.H.2:1-N.4 material can be seen in Fig. 6.

4.3. Correlation of Compressive Strength and SEM

The scanning electrone microscope (SEM) test was performed on the optimal composition found in the P.40-E.H.2:1-N.0, P.40-E.H.2:1-N.1, and P.40-E.H.2:1-N.2 materials. The compressive strength value of P.40-E.H.2:1-N.0 material was 46.25 MPa, as shown in Figure 4. The SEM results of P.40-E.H.2:1-N.0 material showed a plain surface material, caused by the main material composition, namely epoxy-resin. When the epoxy-resin has become stiff with acrylic properties, this condition produced a plain surface like glass that could refract the x-ray light. In Fig. 5a, the nano-SiO₂ particles were absent due to the composition of P.40-E.H.2:1-N.0 material as a standard material without the addition of nano-silica, resulting in brittle characteristics.

The P.40-E.H.2:1-N.1 material obtained a compressive strength improvement of 54.18 MPa. This condition was occurred

due to the 1% nano-SiO$_2$ addition. The SEM results of P.40-E.H.2:1-N.1 can be shown in Fig. 7. In this figure, several brighter parts indicated as the results of acrylic refraction of epoxy-resin, while the darker parts were fine aggregate used. In Figure 7b, the nanoparticle distribution showed a fine spot in all sample areas. In 1% nano-SiO$_2$ concentration, the distribution of nano-SiO$_2$ was relatively even without agglomeration that could increase the material strength (Patel *et al.*, 2018). The observation results of P.40-E.H.2:1-N.2 material with 2% nano-SiO$_2$ addition obtained uneven particle distribution shown in Fig. 5c. The unhomogenized particle distribution or agglomeration could reduce the material strength and affect the mechanic relationship of microstructures in the material (Patel *et al.*, 2018).

4.4. Correlation of XRD and SEM

The correlation of XRD and SEM could be compared between the P.40-E.H.2:1-N.0 material and the SEM image of P.40-E.H.2:1-N.0. Based on the XRD results of P.40-E.H.2:1-N.0 material, a crystalline phase was obtained, when compared with Fig. 5a, the P.40-E.H.2:1-N.0 material had a plain surface and several cracks. The cracks formed indicate that the material is brittle. In the P.40-E.H.2:1-N.1 material, the correlation of XRD and SEM can be seen in Fig. 6 and Fig. 7. The P.40-E.H.2:1-N.1 material was in a great amorphic phase with low crystalline level. The crystalline phase formed showed a maximum hydrogen bond. The maximum hydrogen bond was observed on the SEM image of P.40-E.H.2:1-N.1 material, whereas the following material had a good density with less cracks. The P.40-E.H.2:1-N.2 material had a higher crystalline phase due to unmaximized hydrogen bond, resulting in non-reactive material. The reduced hydrogen reaction in the P.40-E.H.2:1-N.2 material observed from SEM results can be seen in Fig. 7c. In this figure, the material surface had more porosity level and irregular particle organization.

4.5. Correlation of XRD and FTIR

The correlation of XDR and FTIR tests could be observed from the P.40-E.H.2:1-N.0 material, whereas this material had no nano-SiO$_2$ in the material composition. The XRD results of P.40-E.H.2:1-N.0 showed that the Si-O-Si content based on the FTIR test obtained the Si-O-Si absorption in a wide shape with the SiO$_2$ vibration length at P.40-E.H.2:1-N.0 material at 1010 cm^{-1} and intensity of 85 T. The P.40-E.H.2:1-N.0 material obtained crystalline phase with the absorption of C-O group (alcohol, ester) and NO$_2$ (nitro). These compounds were unable to react with epoxy-resin, forming a crystalline phase. In the P.40-E.H.2:1-N.1 material with 1% nano-SiO$_2$ addition obtained the XRD results with amorphous region at 10526 larger than its crystalline level. From the FTIR test results, the P.40-E.H.2:1-N.1 material had more hydrogen group absorption level. The optimum use of nano-SiO$_2$ could increase the hydrogen bond formed. The quantitative data of FTIR test in the P.40-E.H.2:1-N.1 material can be seen in Table 1.

The XRD results in the P.40-E.H.2:1-N.2 and P.40-E.H.2:1-N.3 materials an increased crystalline level. The increased crystalline could be correlated with the FTIR results due to the existence of carboxylic acid absorption. The carboxylic acid in the material was corrosive that reduced the chemical reaction of epoxy-resin polymer molecules.

5.   Conclusions

The mixture composition of nano-silica, epoxy-resin, hardener, and fine aggregate produced new materials with light and strong characteristics. These characteristics were extremely suitable for building as partition or structural materials. The addition of sand in the material was aimed to determine the optimal mixture weight that could decrease the material strength. The maximum strength of the produced materials was averagely found in the use of 1% nano-SiO$_2$, which reached an optimum content. Decreased strength was occurred due to several causes such as agglomeration as shown in the SEM observation results, several acid and alcohol reactions from the FTIR results, and several compounds carried by the fine aggregate. For further studies, partition material against the increasing temperature should be studied, therefore the material can finally be implemented in the construction sector.

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