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Effect of Fly Ash Fraction and Mixing Process Variables On Mechanical Properties of Polymer Composites

Zainun Achmad[1], A.E Ismail[2*], Kamarul-Azhar Kamarudin[2*], Kareem AK[3]

[1]Department of Mechanical Engineering,
University of 17 Agustus 1945 Surabaya, 60118, INDONESIA

[2]Faculty of Mechanical and Manufacturing Engineering,
Universiti Tun Hussein Onn Malaysia, 86400 Johor, MALAYSIA

[3]Air Conditioning and Refrigeration Techniques Engineering Department,
Al-Mustaqbal University College, Babylon, IRAQ

*Corresponding Author

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Abstract: The development of material technology continues to be carried out to meet the needs of engineering materials which are increasing day by day and are environmentally friendly or green. In this study, the process of making epoxy resin composites with fly ash was carried out from the combustion process of a power plant. This study aims to determine the strength of the mechanical properties between the epoxy resin and fly ash weight fraction by stirring treatment in the mixing process using tensile, flexural, and impact tests, microscopic examination, FTIR, and X-RD analysis. The treatments were mixing epoxy resin and fly ash with a weight fraction of 10%, 20%, and 30%, stirring speeds of 100 rpm, 150 rpm, and 200 rpm, for 10 minutes, 20 minutes, and 30 minutes. The results showed that the F2S3T3 composite had a greater tensile strength of 31,94 MPa, while the other composites had a tensile strength above 22-30 MPa, and the lowest was 13,07 MPa in the F1S2T3 composite. The maximum modulus of elasticity is found in the F3S3T1 composite with a value of 11,12 MPa, and the lowest is found in the F1S2T3 composite at 2,06 MPa. The F3S2T2 composite has a maximum flexural strength of 33,32 MPa, and the lowest composite F2S2T1 is 17,40 MPa with a flexural modulus of 270,41 MPa. The maximum flexural modulus in the F1S1T1 composite with a value of 382,76 MPa with a flexural strength value of 28,82 MPa. The F2S1T2 composite has an impact energy of 6,94 Joules with an impact strength of 203,47 MPa, and there are 2 composites that have the same impact energy value of 5,20 Joules, namely F3S2T2, and F3S1T3. From SEM observations, the fracture surface of the composite has a concave and prominent surface, but a lot of dust is trapped so it affects the bond in the matrix and reinforcement.

Keywords: Epoxy resin, fly ash, composite, mechanical properties, process variables

1. Introduction

Particle reinforcement in composite technology continues to experience rapid development, which is being developed by many researchers. One of them is fly ash particle reinforcement. Fly ash is a material with fine particles which are dominant in solid or hollow spherical form. This material is a ferrous alumina silicate compound with the main elements Si, Al, Fe, Ca, K and Na. The purpose of providing fly ash reinforcement is to improve the mechanical properties of polymer composites [1, 2]. The use of fly ash as a composite reinforcement, namely the level of surface

*Corresponding author: emran@uthm.edu.my, kamarula@uthm.edu.my.

adhesion of the two materials is relatively low so that it does not provide effective reinforcement, so to increase the level of surface adhesion of fly ash containing ceramic particles, it is done by providing a coating using magnesium with electroless plating technique [2].

The process of mixing epoxy resin composites with fly ash reinforcement with stirring for a certain time is then carried out with a pressure molding process, where fly ash is generally used as a reinforcement to have a smooth round shape without an electroless plating process [3-5]. The ratio value of epoxy resin and resin hardener mixed at a stirring cycle for 5 minutes influences the mechanical properties of the composite [6]. Therefore, composites can increase specific strength, which is widely applied to aircraft, electronic components, and automobiles, resulting in mechanical properties that are strongly influenced by the mixing time of fly ash and epoxy resin [3]. Manoj [7] explained that the epoxy resin composite with fly ash reinforcement increased the tensile strength with increasing fly concentration, while the impact strength decreased. Ma [8] by adding fly ash reinforcement to Formant and urea plasticized thermoplastic starch and Oxysterol plasticized thermoplastic was able to increase the tensile strength and modulus of elasticity many times and was able to increase water resistance.

Shubham [9] stated that fly ash-reinforced epoxy composites treated with aminopropyl trimethoxy silane coupling agent increased strength and toughness because their bond with polymer resins resulted in low damping ability. In researching the effect of fly ash weight fraction, speed, and stirring time on epoxy resin composites, several tests will be carried out to better understand its potential. Tensile, flexural, and impact testing to get the value of mechanical properties in each variation. The scanning electron microscopy (SEM) test was to observe the surface topography and fracture of the composite, while X-ray (XRD) was to determine the composite crystal index in the amorphous and crystalline regions.

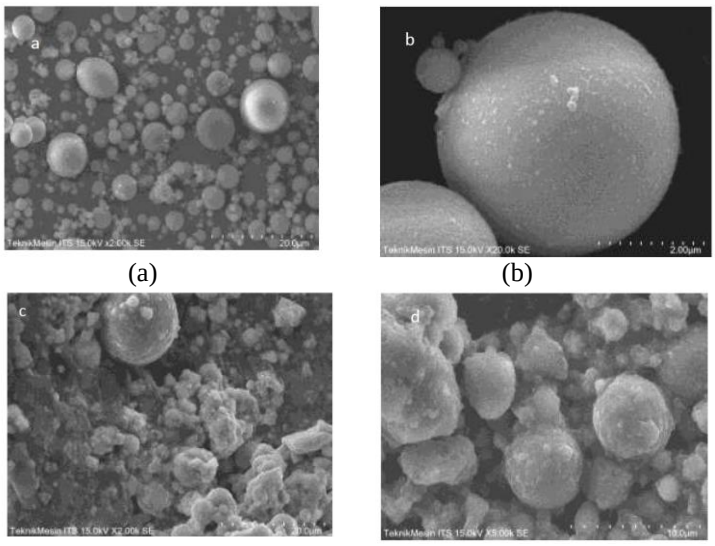
2. Methodology

The resin material used is Epoxy Resin with the Bakelite trademark EPR 174 from Korea and Hardener Resin with the brand Versamid 140 marketed in Indonesia by PT. Justus Kimia Surabaya Branch. The mixing process of epoxy resin – resin hardener is 10:4. The fly ash material was obtained from the city of Malang, East Java, with a particle size of 250-300 mesh, and the electroless platting process was carried out as follows: (1) Sifting coal bottom ash to a size of 250-300 mesh, (2) Washing coal bottom ash with 96% alcohol, as in Figure 1.1, (3) After washing with alcohol 3 times, clean coal bottom ash was put into a bowl and then in the oven to dry the coal bottom ash at 100 °C, (4) The process of stirring the HNO3 liquid in the Erlenmeyer tube and stirring with a magnetic stirrer for 5 minutes, then 20 grams of fly ash is added and stirred for 30 minutes, then 0.5 grams of epoxy is added then stirred using a magnetic stirrer and heated at 100 °C for 10 minutes, (5) Add 0.1 g of fine Mg powder, then stir using a magnetic stirrer and heat at 100 C for 60 minutes and (6) Dry the electroless coating powder in an oven at 200 C for 240 minutes to allow the oxidation process to occur. SEM micrograph test results fly ash after washing with alcohol and the electroless plating process, as shown in Figure 1.1.

The first stage is the mixing process of epoxy resin and fly ash with a weight fraction of 10%, 20%, and 30%, then stirred at speeds of 100 rpm, 150 rpm, and 200 rpm, for 10 minutes, 20 minutes, and 30 minutes, and poured hardener according to the ratio of epoxy and hardener 10:4 before 5 minutes of stirring ends. (experimental run as shown in Table 1.1). Then it is poured into a 29 cm diameter mold with normal curing for 10 minutes, then the mold is closed with a pressure of 40 kg for 24 hours. After drying, the composite mold can be removed and made into a slab. Then the composite plate was made of five test objects for tensile, bending, and impact tests according to ASTM standards then cut using a CNC machine and ready to be tested. Testing mechanical properties to determine tensile, flexural, and impact strength. Tensile testing using ASTM D638 standard, flexural testing using ASTM D790-02 standard, and impact testing according to ASTM D256. Each test object is 5 samples in each treatment.

Table 1 - Trial code

Code	% FA	Speed (rpm)	Mixing Time (minute)
F1S1T1	10	100	10
F1S2T3	10	200	30
F1S3T2	10	300	20
F2S1T2	20	100	20
F2S2T1	20	150	10
F2S3T3	20	200	30
F3S1T3	30	100	30
F3S2T2	30	150	20
F3S3T1	30	200	10



(a) (b)

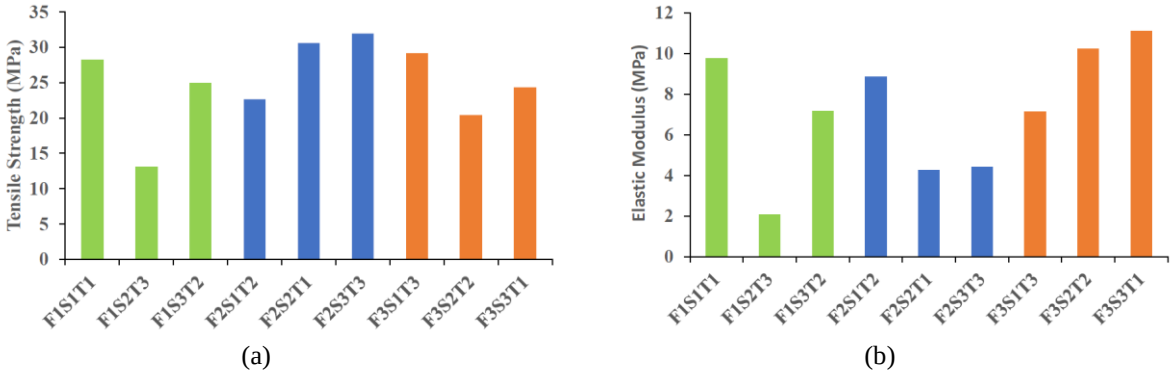
(c) (d)

Fig. 1 - SEM micrograph test results (a) and; (b) fly ash after washing with alcohol, and; (c) and; (d) after the electro less plating process

3. Results and Discussion

3.1 Tensile Strength of Epoxy Resin Composite with Fly Ash Reinforcement

Figures 2 (a), and 2 (b) show the tensile strength, and modulus of elasticity of the epoxy/fly ash composite treated with the addition of fly ash (F), stirring rotation (S), and stirring time (T). The increase in fly ash varied from 10 to 30%, rotation from 100 to 200 rpm, and stirring time from 10 to 30 minutes under normal curing conditions. The addition of fly ash reinforcement, rotation, and stirring time encourage changes in the tensile strength of the composite. The highest tensile strength of 31,94 MPa was obtained from the F2S3T3 composite, and 15,89% higher than the epoxy-hardener matrix polymer composite with a ratio of 10:4. The increase in the tensile strength of the composite is closely related to the increase in the bond formed between the reinforcement and the matrix. This is indicated by the increase in the percentage of fly ash strengthening, mixing process, and crystallinity. While the decrease in tensile strength of the F1S2T3 composite resulted from 10% fly ash reinforcement mixed with 150 rpm for 30 minutes, the tensile strength value is lower than the strength of the epoxy-hardener polymer composite of 16,11% or 13,07 MPa. The F3S3T1 composite has the highest elastic modulus of 11,12 MPa, and the lowest is 2,06 MPa of the F1S2T3 composite.



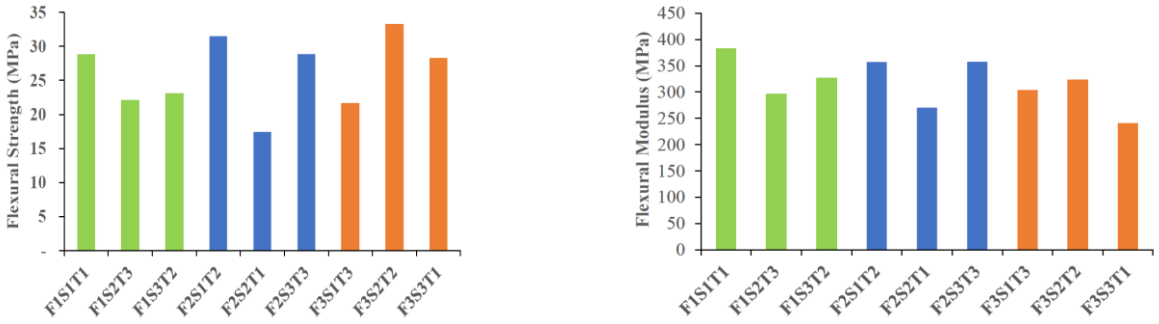
(a) (b)

Fig. 2 - Tensile strength of epoxy resin composite with 10%, 20%, and 20% fly ash reinforcements (a) tensile strength and; (b) modulus of elasticity

3.2 Flexural Strength Epoxy Resin Composite with Fly Ash Reinforcement

The flexural strength and modulus of elasticity of the epoxy/fly ash composite with the percentage of weight, rotation, and stirring time varied under normal curing conditions are shown in Figures 3 (a), and 3 (b). The flexural strength of the epoxy/fly ash composite has the same tendency as the epoxy-hardener matrix polymer composite. The flexural strength of the F1S1T1 composite was 28.83 MPa, then there was an increase in the rotation time, and the

mixing time in the F1S2T3 and F1S3T2 composites decreased by 19,9%. Composite F2S1T2- F2S3T3 has a flexural strength with the same tendency. Meanwhile, composites F3S1T3- F3S2T2- F3S3T1 have the opposite trend, namely 59-33,20, and 28,20 MPa, increasing by 53,77% and decreasing by 23,16% from 21,59 MPa. Composites with 30% fly ash reinforcement at a stirring speed of 150 rpm for 20 minutes had greater flexural strength than other composites. This indicates that the decrease in strength may be due to the presence of a fly ash layer. The results of the epoxy-fly ash mixing process occur homogeneously, and the crystallinity strength of the polymer matrix and fly ash reinforcement is generally below 35%.

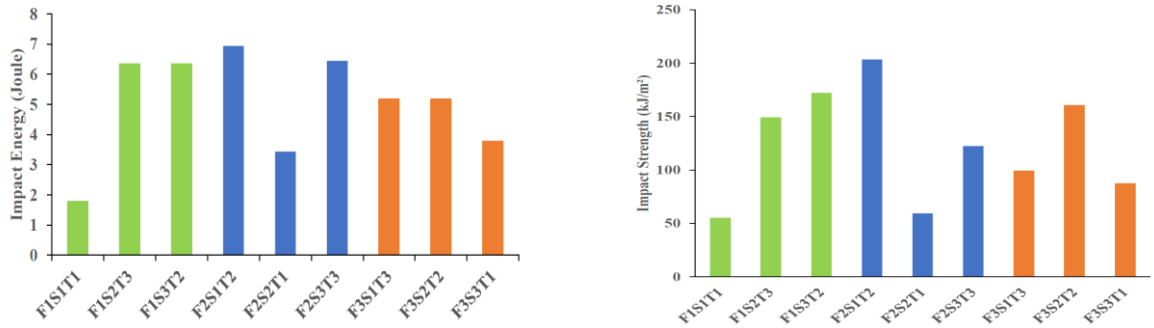


(a) (b)

Fig. 3 - Flexural strength of composites with 10%, 20%, and 30% fly ash reinforcements (a) flexural strength, and; (b) flexural modulus

3.3 Impact Strength of Epoxy Resin Composite with Fly Ash Reinforcement

Figures 4 (a), and 4 (b) show the impact energy, and impact strength of the epoxy/fly ash composite with various weight percentages of fly ash reinforcement (F), stirring rotation (S), and stirring time (T). The maximum impact energy is found in the F2S1T2 composite of 6.94 Joules with an impact strength of 203.47 kJ/m², while the lowest impact energy is found in the F1S1T1 composite. The impact energy of the F1S1T1-F1S3T2 composite has an increasing trend, F2S1T1-F2S3T3 has a decreasing trend, as well as the F3S1T3-F3S3T1 composite. This happens because the addition of fly ash, rotation, and stirring time has an effect on the amount of impact energy, so that the bond between the matrix and reinforcement occurs strongly, when viewed from the impact energy, almost all composites have almost the same value above 5 Joules. When the impact load is applied, the composite fracture path area passes through the matrix interface bond with the reinforcement, so that the strength of the interfacial bond increases the impact energy.



(a) (b)

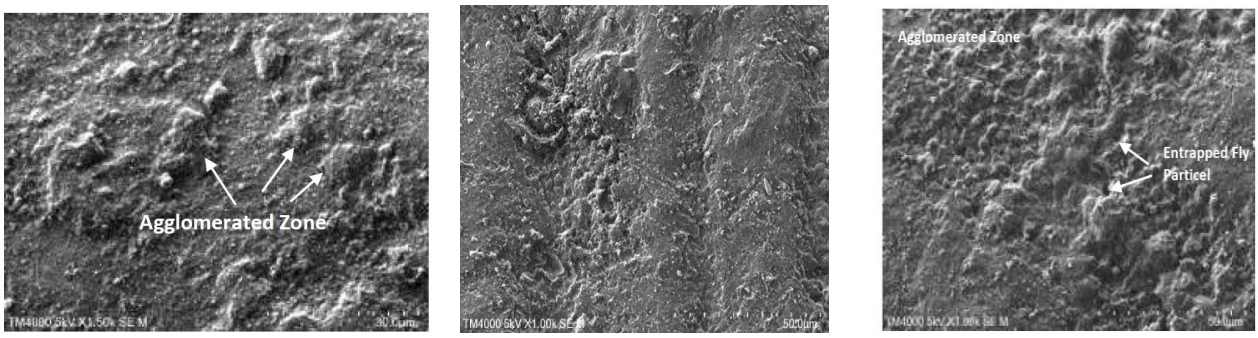
Fig. 4 - Epoxy resin composite with 10%, 20%, and 20% fly ash reinforcements (a) energy impact, and; (b) impact strength

3.4 Composite Surface Morphology

The topography of the microstructure of an epoxy composite with fly ash reinforcement with a normal curing process is shown in Figure 5 a. The micro-photo shows the irregular shape of the powder, which is characterized by gray powder with a white border and is a composite reinforcement powder. The morphology of the F1S1T1composite surface, the fly ash reinforcing powder was evenly distributed, and several agglomeration zones were seen, but there was still a lot of dust attached to the particle surface so that it became the boundary between the matrix and the fly ash particles so that the interface bonds be weak.

In F3S3T1composites, the surface morphology of the matrix with the reinforcement was well mixed, but there

were particles decomposed without bonds between the particles, so these particles become foreign objects in the composite section, which makes it unable to accept external loads, causing the composite strength to decrease as shown in Figure 5(b). On the surface morphology of the F2S2T1composite, it shows the presence of fly ash particles trapped in the matrix which forms cavitation, so that it will inhibit the flow of stress which causes an area of stress concentration. That's because the proper interfacial bonding and uniform distribution of fly ash particles in the matrix material, so that fly ash particle are trapped in the matrix, can hinder the flow as shown in Figure 5(c).

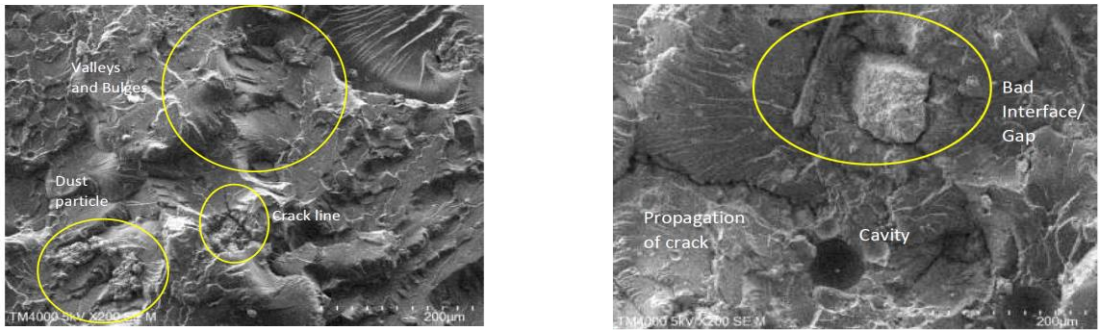


(a) (b) (c)

Fig. 5 - Surface topography of composite with fly ash reinforcements (a) the F1S1T1 composite with 10% fly ash, 100 rpm rotation, stirring time 10 minutes; (b) the F3S3T1 composite with 30% fly ash, 200 rpm rotation, stirring time 10 minutes, and; (c) the F2S2T1 composite with 20% fly ash, stirring at 150 rpm for 10 minutes

3.5 Fracture Surface of Tensile Test Specimen

Figure 6(a) Surface morphology of the F1S3T2 composite under normal curing conditions of the fracture surface of the tensile test specimen shows that the fracture surface has valleys and peaks. The structure that forms peaks or protrudes is seen because of tension so that the matrix material dislocations due to external loads. The dust particles are trapped and do not adhere to the matrix so there is this poor interface that forms cavitation holes, when there is tension there is no bond with the matrix due to the accumulated stress concentration which causes crack initiation and propagation. The fracture surface of the F3S2T2 composite under the same curing conditions has a valley and prominent surface. but a lot of dust is trapped so that it is not bound to the matrix which causes cavitation as the center of stress concentration, this is an early defect that can cause early cracks and crack propagation when receiving external loads. This dust is not bound to the matrix as shown in Figure 6(b). This happens because of the effect of the large content of fly ash, stirring cycle, and time during the mixing process.



(a) (b)

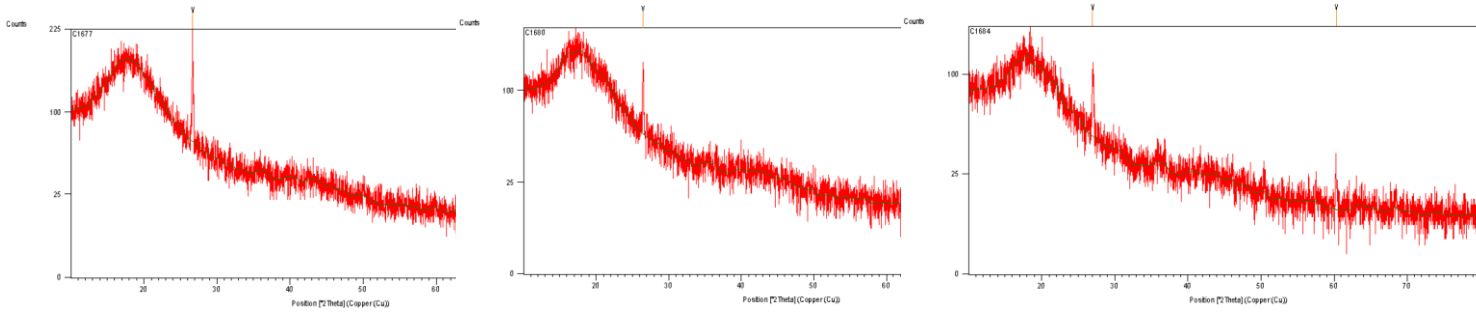
Fig. 6 - Surface fracture of composite with fly ash reinforcements (a) the F1S3T2 composite with 10% fly ash stirring at 200 rpm for 20 minutes, and; (b) the F3S3T2 composites with 30% fly ash stirring at 150 rpm for 20 minutes

3.6 XRD Analysis

The F1T1S1 composites had a higher degree of crystallinity, namely 44,45%, while those with fly ash content and agitation cycles were greater at the same time and experienced a decrease in the degree of crystallinity as shown in Table 2. The strongest peak appears in the crystal plane (110) at around 2Θ= 16,29°- 18,89° which is called the amorphous region, which is a large and wider peak. While the next strong peak appears in the plane (200) which occurs in area 2Θ= 26,52°- 27,02°, this shows the crystal area in the form of a rigid peak. Figure 9 shows an X-ray diffractogram on a composite under normal curing conditions, where the wider peak is the amorphous phase while the sharp peak is the crystal phase.

Table 2 - Degree of crystallinity of composites with fly ash reinforcement under normal curing conditions

Treatment	Fly Ash (%)	Speed (rpm)	Time (minutes)	Crystallinity (%)
F1S1T1	10	100	10	44,45
F2S2T1	20	150	10	27,60
F3S3T1	30	200	10	31,00



(a) (b) (c)

Fig. 9 - XRD analysis of composites with fly ash reinforcement under normal curing conditions (a) F1S1T1 composite; (b) F2S1T1 composite, and; (c) F3S1T1 composite

4. Conclusion

From the results of the above discussion, it can be concluded that the F2S3T3 composite has a greater tensile strength of 31,94 MPa, while the other composites have a tensile strength above 22-30 MPa and the lowest in the F1S2T3 composite of 13,07 MPa. The maximum elastic modulus is found in the F3S3T1 composite with a value of 11,12 MPa, followed by the F3S2T2 composite with a value of 10,22 MPa, and the lowest is found in the F1S2T3 composite of 2,06 MPa. The composite with maximum strength has an elastic modulus of 4.42 MPa. The F3S2T2 composite has a maximum flexural strength of 33,32 MPa, and the lowest composite F2S2T1 is 17,40 MPa with a flexural modulus of 270,41 MPa. The maximum flexural modulus in the F1S1T1 composite with a value of 382,76 MPa with a flexural strength value of 28,82 MPa. The F2S1T2 composite has an impact energy of 6,94 Joules with an impact strength of 203,47 MPa, and some composites have the same impact energy value of 5,20 Joules, namely F3S2T2, and F3S1T3. From SEM observations, the fracture surface of the composite has a concave and prominent surface, but there is a lot of dust trapped so that there is no strong bond in the matrix and reinforcement, this causes the stress concentration center which is an early defect that can cause crack initiation and crack propagation when accepting external loads.

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