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STUDY OF MAKING POLYESTER RESIN MATRIX COMPOSITES USING BASALT SCORIA POWDER FILLERS TO TENSILE STRENGTH AND COMPRESSIVE STRENGTH

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Abstract
An experimental study on the manufacture of polyester resin matrix composites using basalt powder as a reinforcing filler has been carried out. Basalt is a volcanic igneous rock often found in East Lampung and has not been utilised. Basalt is chosen as a reinforcing filler because basalt has advantages such as wear resistance, corrosion resistance, resistance to chemical reactions, and high hardness. The research parameters used were variations in the size of the basalt powder, the composition of the polyester resin matrix to the basalt powder, and the percentage of the catalyst. All parameters were mixed according to the research procedure, and all samples were formed under pressure 20 kN. Tensile test results showed the highest value of 0.961 kgf/mm^2 from 60 mesh-25% polyester-1/30 catalyst variation parameter. The highest compressive strength value of 28.331 kgf/mm^2 was obtained from the various parameters of 270 mesh-20% polyester-1/20 catalyst and 270 mesh-25% polyester-1/30 catalyst. The results were not much different from those, which is 27.787 kgf/mm^2. The use of 1/30 hardener catalyst to the amount of polymer by 25 %wt gives optimal results on the characterisation material testing. However, the effect of the filler grain size gave different results for each test carried out. Therefore, to obtain the desired mechanical properties when using basalt rock powder as a filler, it is necessary to pay attention to the correct grain size.

Keywords:
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Composites;
Compressive strength;
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INTRODUCTION

Science and technology about materials in the last decade has been growing. Many research and studies have been carried out, one of which is on composite materials by combining polymeric and inorganic materials. The combination gives the advantage of increasing the value of mechanical properties and morphological properties [1]. Thermoplastic and thermoset polymers have been mass-produced industrially, and their use dominates other types of industrial material available. Due to several advantages, it has many utility and many industrial products that make use of this polymer [2][3]. One of the commonly recognised polymers of this class is polyethylene (PE) and polyester. Polyethylene is often used for its good mechanical properties, such as strength, corrosion resistance, radiation, non-toxicity, and dielectric properties [4]. However, this disadvantage of polyethylene's is its low modulus of elasticity and is easily deformed when loaded, so it is limited in its use.

For polyester, they are often used in sheet moulding compound or bulk moulding compound, so in the use of heavy work, this polymer type is more widely used. The common advantages of this polymer were the low cost production, low shrinkage and resistance to ageing. So, in Polymer Composite Matrix (PMC), this material is more selected because the advantages characteristic of this polymer is still maintained, only left to improve other mechanical properties of the PMC.

Moreover, the current world economic conditions, which require materials with good mechanical properties and have low production costs, are being sought after by world industry players. Due to these factors, many studies have combined polyester with various other filler types such as kaolin, talc, mica, chalk, silica, aerosol, titanium oxide, etc. [5, 6, 7]. These polymer composites are usually applied as deck floors, packaging materials, and household window frames to the automotive industry [8, 9, 10, 11, 12, 13]. In principle, research on the use of composite polymers with inorganic fillers has been and continues to be carried out by several researchers. Among them, HDPE and LDPE with basalt powder as a filler have been done [14]. However, there is still a gap in research that uses PET type polyester with basalt powder filler. The use of PET type polyester is based on the properties of PET which have high strength, stiffness, stable dimensions, chemical and heat resistance, and has good electrical properties. In addition, PET has low moisture absorption, as well as absorption to water.

One of the inorganic materials that are inexpensive and have good mechanical characteristics as a filler is basalt rock. The selling price of basalt rock is lower than other reinforcing fillers, requiring a long and expensive process. Each ton of basalt rock in the form of coarse is only 105,000 IDR [15].

Basalt is not flammable and can withstand high temperatures (900° C), is hard, has a toughness in accepting high loads, has insulation properties against sound and temperature, is non-reactive, and is non-radiation. Basalt is environmentally friendly and non-toxic to living things [16, 17, 18, 19, 20, 21]. The physical and chemical stability of powdered basalt (BS) against the biological environment does not endanger the environment or the life of organisms [22]. Basalt also does not contain harmful asbestos ingredients [23]. Even basalt may be used as a biomaterial for bone reconstruction in osteoplastic surgery, dentistry, and orthopaedics [24].

Commonly, the dominant element in basalt is silica, known in hardness and relatively high resistant temperature. Basalt is often porphyritic, containing larger crystals (phenocrysts) formed before the extrusion that brought the magma to the surface, embedded in a finer-grained matrix. These phenocrysts usually are of augite, olivine, or calcium-rich plagioclase. Phenocrysts have the highest melting temperatures of the typical minerals. This condition can crystallise from the melt and are therefore the first to form solid crystals.

Basalt is mainly used when it is formed in fiber shape. A lot of previous research utilising basalt is in the form of fiber as combining material in composite such as Basalt fiber reinforced polymer [25][26]. Basalt, to be formed in fiber shape, need to be melt, pulled and then spun. This technique requires advanced technology to be applied. Considering raw basalt stone still has excellent characteristics, with simple technology applied such as grinding to formed fine particles, basalt rock's opportunity to be combined with polymers, especially polyester, to get good characteristics is wide open. Based on Siswanto and Diharjo (2011) and Kartika and Pratiwi (2018) research, they utilise fine particles to form a composite with polymer matrix. Particle dimension was significant because it will influence the distribution of particles in polymer matrix solution before it is formed to PMC [27][28].

Basalt in Indonesia, especially in Lampung province still not widely used in the advanced technological process. Moreover, basalt deposits in Indonesia, especially in East Lampung, which reaches 10 million m3 and has not yet been utilised, opens opportunities for polymer composites - basalt of East Lampung to be further developed [29]. In this study, we developed an optimisation of basalt rock as a polymer composite mixture in terms of filler grain size and the effect of the concentration of polyester additives.

METHOD

The filler used in making composites is basalt obtained from Sukadana, East Lampung. Basalt is refined using a ballmill Tinius Olsen Model TO-441, then sieved using a sieve shaker MVS-1N Shaking machine to get mesh sizes of 40, 60, 150, and 270. The percentage of basalt to resin is 75%, 80%, 85%, 90%wt, while a catalyst is 1 / 30, 1/20, 1/10 percent volume of the resin used. The experimental design of the sampling follows the factors and levels as shown in Table 1.

Table 1. The experimental design of the sampling

Experimental Parameters				
Basalt particle size (mesh)	0	0	50	70
Percentage of resin (%wt)	0	5	0	5
Percentage of catalyst vs Resin (% Vol)	/10	/20	/30	

A polymer PET (Polyethylene Terephthalate) type 157® BQTN-EX polyester resin was used as a composite matrix. For the hardener catalyst of polyester, MEKPO (Methyl Ethyl Ketone Peroxide) was being used. Because the polyester resin is part of the thermoset polymer, to harden, it needs heat from the chemical reaction of polyester between the catalyst. Usually, another type of catalyst to aid in the hardening process, like Percumyl H (Cumene Hydroperoxide). Still, utilisation of this catalyst is suitable in clear polymer products and long working conditions because this catalyst has low exothermic characteristics.

The process of mixing composite materials - polymers is carried out using the Portable Mixer Stirrer Machine for 3 hours for each sample and formed with a load of 1 Ton using the TEKIRO 15 Ton hydraulic press. Furthermore, to determine the mechanical properties of polymer-basalt composites, the HT-2402 Computer Universal Testing Machines obtain the compressive strength and tensile strength which is the dimension of the specimen are 12.7 mm x 25.4 mm and 65.5 x 12 x 15 mm respectively based on ASTM D695-15, where each test is carried out 5 times for each sample being measured.

Table 2 shows the characterisation of the polyester resin 157® BQTN-EX and the MEKPO (Methyl Ethyl Ketone Peroxide) hardener catalyst [30][31], which is used for primary matrix in the composite.

Table 2. Characterization of the 157® BQTN-EX polyester resin [30][31]

Name	Value	Unit	Note
Density	1.4	gr / cm^3	at room temperature
Hardness	40	-	Barcol GYZJ 934-1
Heat distortion	70	° C	
Water absorption (room temperature)	0.188	%	24 h
	0.446	%	Seven days
Flexural strength	9.4	Kg / mm^2	
Flexural Modulus	300	Kg / mm^2	
Tensile strength	5.8	Kg / mm^2	
Modulus of elasticity	300	Kg / mm^2	
Elongation	2.4	%	

The Crystallinity and phase of basalt stone were measured by X-Ray Diffraction (XRD: Panalytical Xpert 3 Powder XRD) with a Cu-Kα as a source of X-ray operating at 40 kV and 30 mA. The sample was scanned in the range 2θ of 20-80°. The basalt chemical compositions were characterised by X-Ray Fluorescence (X-RF Epsilon 4 XRF Spectrometer from Malvern Panalytical) with operating at 50 kV and 3 mA. Meanwhile, to determine the basalt polymer composites' morphology, the observations were made using an optical microscope Nixon Eclipse MA100.

RESULTS AND DISCUSSION

As shown in Figure 1, the basalt powder from Sukadana contains pyroxene-XY(Si,Al)2O6 (COD reference number 96-900-1209), plagioclase which mainly formed from anorthite-$CaO.Al_2O_3.2SiO_2$ (COD reference number 96-100-0035) and albite-$Na2O.AL_2O_3.6SiO_2$ (COD reference number 96-900-9664), and olivine-$2(Mg.Fe)O.SiO_2$ (COD reference number 96-101-0498), which are important phases in forming natural basalt rock [32]. Usually, nearly 80% is formed by plagioclase and pyroxene as important mineral components in basalt. The plagioclase involved is an intermediate member of the An-Ab series, and Bowen showed that a very near An50 occurs most frequently in basalts. The pyroxene is chiefly calcic, which is a member of the augite series, usually more magnesian than ferrous-rich (Mg / (Mg + Fe)> 0.5), and commonly not very far from diopside, although it ranges to subcalcic augite. Calcium-poor pyroxene, such as hypersthene, may or may not be present that depend on the abundance of silica relative to other constituents. Olivine is another critical mineral in basalt classification. In alkaline basalt, it is commonly present instead of Ca-poor pyroxene. In olivine tholeiites, it is present with Ca-poor pyroxene [33][34].

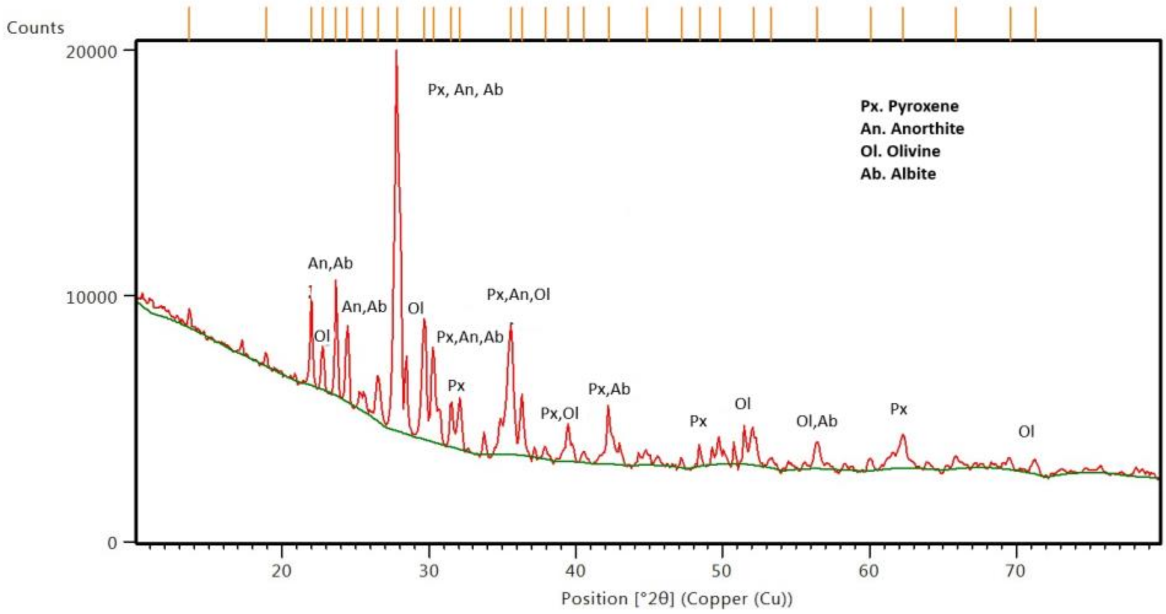

Figure 1. The result of XRD analysis of basalt rock in Sukadana, East Lampung

As seen in Table 3, Sukadana Lampung Timur's basalt rock is dominated by silica (SiO2) of 44,651%. Like most other silica-based minerals, this mineral's main characteristics are its high toughness and hardness. When used as a component of polymer composites, the characteristic properties of polymer composites obtained will increase, especially for its tough hardness. As seen in Table 3, besides silica oxide as the dominant element in basalt rock, other metal minerals are quite dominant from other elements in the rock, such as alumina and iron oxide, which are quite large, respectively, namely 15,544% and 19,537%. The presence of these metal elements can increase the mechanical properties of the resulting composite material [35][36].

Polymer composites - basalt is made by mixing polyester resin with 10% -25%wt against powdered basalt raw materials. The powdered basalt itself is varied into four particle sizes, namely 40 mesh and 60 mesh, representing the grain size of coarse particles and 150 mesh and 270 mesh, representing the fine grain size [37]. In the use of polyester resin, a hardener catalyst is required where its use differs depending on the material mixed into the polymer [38, 39, 40, 41, 42]. So, to determine the optimal level of catalyst used on polyester mixed with basalt powder, this study varied to 1/10, 1/20 and 1/30 of the concentration of resin used.

From the tensile and compressive strength test data in Figure 2 and Figure 3, the minimum concentration was obtained for polyester resin as a matrix in the manufacture of polymer composites - basalt. It can be seen that for adding 10% and 15% polyester to the composite mixture at all variations in grain size and catalyst content. The results could not be measured in the test equipment due to the lack of polyester quantity to spread evenly to bind basalt powder particles in the composite. The composite results obtained in this variation are easily crushed and broken.

The particle size influences the composite's mechanical properties, especially its toughness because the smaller particle size has a larger surface area than the coarse or large particle size. This surface area will affect each component of the particles attractive forces in the stress distribution system in the composite. The stress will be evenly distributed throughout the composite with a large contact area so that the load received can be greater. Based on Figure 2, with fine particles, the gap created will be smaller than coarse particles, so the particle is easier to fill the gap and distributed.

Table 3. Results of XRF analysis of basalt rock in Sukadana, East Lampung

No.	Element	%WT	Oxides	%WT
1	Mg	1.998	MgO	2.28
2	Al	12.275	Al_2O_3	15.544
3	Si	33.073	SiO_2	44.651
4	P	0.575	P_2O_5	0.766
5	K	1.010	K_2O	0.677
6	Ca	17.587	CaO	13.301
7	Ti	2.219	TiO_2	1.863
8	V	0.06694	V_2O_5	0.05841
9	Cr	0.122	Cr_2O_3	0.08994
10	M N	1.265	MnO	0.783
11	Fe	28.974	Fe_2O_3	19.537
12	Ni	0.05845	NiO	0.03104
13	Cu	0.04639	CuO	0.02497
14	Zn	0.05591	ZnO	0.02986
15	Ga	0.00693	Ga_2O_3	0.00399
16	Rb	0.00697	Rb_2O	0.00319
17	Sr.	0.195	SrO	0.09851
18	Y	0.00605	Y_2O_3	0.00328
19	Zr	0.02689	ZrO_2	0.01551
20	Mo	0.00136	MoO_3	0.00088
21	Sn	0.05857	SnO_2	0.03244
22	Te	0.02382	TeO_2	0.01308
23	Eu	0.331	Eu_2O_3	0.186
24	Hg	0.00135	HgO	0.00055
25	Pb	0.01263	PbO	0.00583
26	Re	0.00442	Re	0.00203

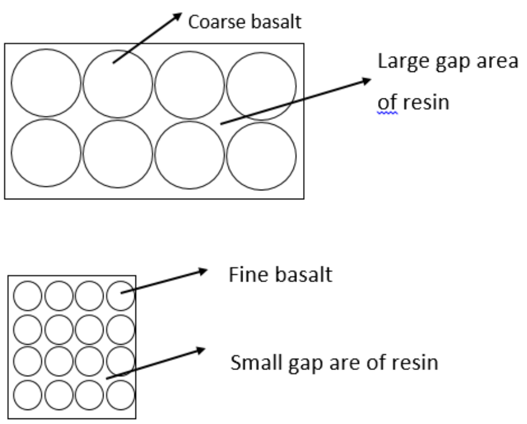

Figure 2. Illustration image of basalt particle in matrix composite based on the size of a grain

This result contradicts the test results of tensile stress, where larger particles tend to have better tensile strength. The factor that influences the tensile test is the elasticity of the polymer itself. With the small contact area of the fill particles (coarse or large particle size), the polymer resin will fill in the gaps between the particle gaps due to pressure in the molding process. Then the stress will be evenly distributed throughout the composite so that the load received can be greater.

In the tensile strength test data, as shown in Figure 3, the largest value is dominated by the particle size of coarse powder (40 and 60 mesh), using basalt powder reaching 75%. But suppose it is viewed from how optimal the addition of hardener catalyst content is. In that case, the value obtained is when using 1/30 catalyst volume on 25%wt polyester with a basalt powder size of 60 mesh. The use of a hardener catalyst affects the strength of the polymer formed. In polyester resin, adding a catalyst will accelerate the polymer's hardening process, which is characterised by the heat reaction in the polymer. If the addition of a catalyst is too little, forming a solid and hard polymer takes a long time. With a large addition, it will accelerate the hardening of the polymer, in its use as a composite matrix. The use of too much catalyst will give too short a time to mix the composite material so that there will be an inhomogeneous distribution of particles or voids or air bubbles in the composite mold due to trapped air. If too little catalyst is used, the resulting composite will not harden and tend to soften [42][43].

In Figure 4, the compression test shows the dominance of fine particle size variations (150 and 270 mesh) having a higher compression value. Especially for the 270 mesh particle size, the application of polyester–hardener catalyst 20% - 1/20 and 25% - 1/30 has a compression value that is not much different. According to Jaya et al. (2016), under pressure conditions, composites with finer particles are better able to withstand pressure than composites with larger particle sizes [44].

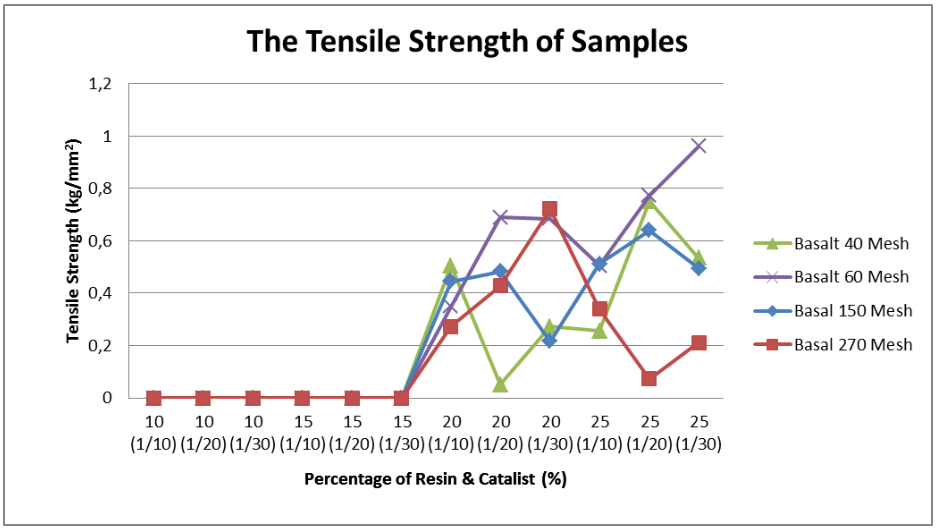

Figure 3. The results of the polymer-basalt composite tensile test

Based on Figure 3, we can see the tensile strength result and the Indonesian Standard SNI 03-2015-2006, as shown in Table 4. Compared with standard, particle board with basalt powder has a tensile strength 30 percent higher than required. The sample with the smallest tensile strength is the particles size 40 mesh - 20 wt% polyester resin - 1/20 vol% catalyst with a value of 0.05 kgf/mm^2. Nevertheless, the tensile strength value still meets the standard as a particle board.

Table 4. Tensile strength comparison with SNI 03-2105-2006 [45]

No.	Comparison		
			Tensile Strength (kgf/mm2)
1	Experiments		
	Area: 12 x 15 mm 2	180 mm2	0,961
	Force (kgf)	173 kgf	
2	SNI 03-2105-2006		
	Particle board (Ordinary & decorative)		0,031
	Particle board (Structural)		0,031

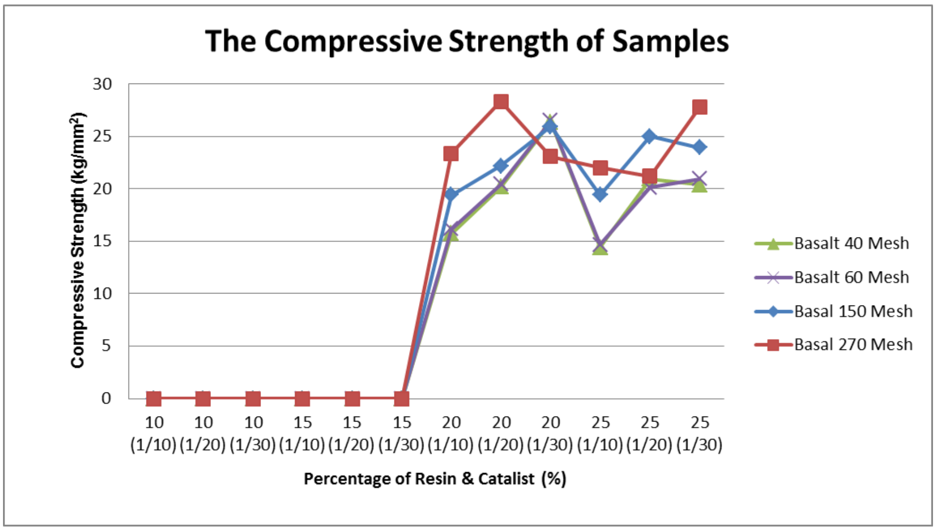

Figure 4. The results of the basalt-polymer composite compressive strength test

The volume of a polymer that fills between the filler particles will be more than the composite with finer particle size. As shown in Figure 5, the highest value in the Tensile test (60 mesh-25% polyester -1/30) shows that the particles are rarely scattered where the voids are filled by a polyester matrix even though voids still occur caused by trapped air during the composite molding process. So that during the test process, the tendency for the polyester's elastic properties to be more dominant.

Based on the tensile and compressive test graph in Figure 3 and Figure 4, there is an optimal point for adding polyester resin and the catalyst's use at the end of 80% basalt powder - 1/20 catalyst. The tensile and compressive test data's tendency for each variation shows an increasing value all except for the particle size variation of 40 mesh-20% polyester-1/20 catalyst. In this variation, the tendency to get the same results increases. Therefore, it is possible to obtain the addition of powdered basalt rock as a filler. When viewed from Table 2 about the characteristics of polyester, the addition of crushed basalt rock in various sizes can increase the value of mechanical strength that polyester itself can provide. Especially its tensile strength is many times greater than polyester itself.

In contrast to the morphology for fine-particle composites, as shown in Figure 6, composite samples with variations in particle size of 270 mesh-75% basalt-1/20 catalyst powder show that basalt particles spread closely together compared to Figure 6 with the scale ratio at 100x magnification—the tendency of the composite character to be denser. However, voids still occur due to trapped air.

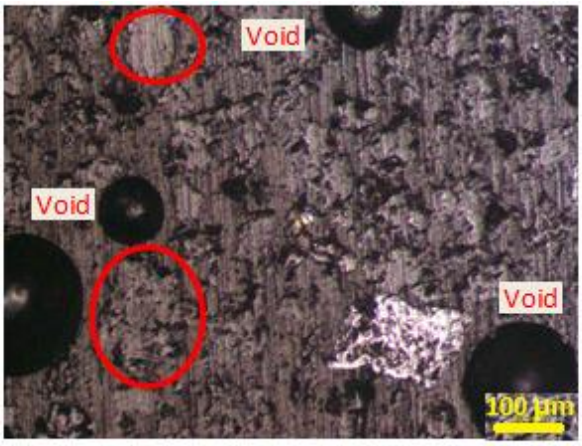

Figure 5. Cross-sectional morphology of polymer-basalt composite with a variation of 60 mesh-75% basalt-1/30 catalyst powder

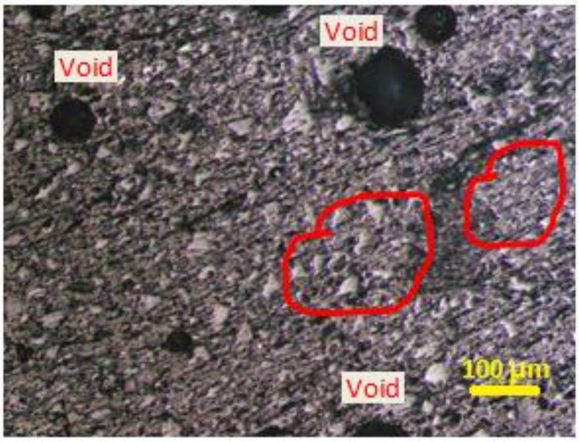

Figure 6. Cross-sectional morphology of polymer-basalt composite with a variation of 270 mesh-75% basalt powder-1/20 catalyst

CONCLUSION

Scoria-type basalt Sukadana Lampung Timur, rich in silica, alumina, and iron oxide as a filler component, can improve polymer composites' mechanical properties in a polyester resin matrix. The highest tensile strength value is obtained from a mixture of 75% basalt powder variations - 1/30 hardener catalyst with 60 mesh basalt grain size of 173 kgf. While the best results for the compressive strength value were obtained with basalt powder with a particle size of 270 mesh, both for variations of polyester - catalyst 20% - 1/20 and 25% - 1/30, respectively 3477 kgf and 3410 kgf. The optimal results without decreasing the value of tensile strength and compressive strength were obtained from the use of basalt powder and the hardener content in the addition of 80% basalt powder - 1/20 catalyst for all sizes of basalt grain variations. Using 1/20 and 1/30 hardener catalyst to the amount of polymer by 25%wt mostly gives optimal results on the characterization material testing. However, the effect of the filler grain size gave different results for each test carried out. Therefore, to obtain the desired mechanical properties when using basalt rock powder as a filler, it is necessary to pay attention to the correct grain size.

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