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THE EFFECT OF CHICKEN EGGSHELL (*Gallus gallus domesticus*) PARTICLE SIZE AS FILLER TO POLYESTER COMPOSITE AS MOTORCYCLE'S EXHAUST PIPE COVER MATERIAL

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ABSTRACT

This research was done to know the effect of chicken eggshell (*Gallus gallus domesticus)* powder particle size on unsaturated polyester resin mechanical properties. The eggshell was mashed and sifted to get the 50 mesh, 70 mesh, 90 mesh, 110 mesh, 130 mesh, 150 mesh, and 170 mesh particle size by using a ball mill. Then, the eggshell was mixed with an unsaturated polyester resin matrix with a ratio of 70:30 and methyl ethyl ketone peroxide (MEKP) catalyst as much as 1 % of the matrix mass. After that, it was pressed by using compression molding. The composite formed was characterized by Scanning Electron Microscopy Energy Dispersive X-ray (SEM EDX), Fourier Transform Infra-Red (FTIR), and X-Ray Diffraction (XRD). The physical properties of the composite were analyzed and the mechanical properties were tested. The SEM EDX result showed that the eggshell and composite were made up of Ca, C, and O, and the polyester was made up of C, O, and Si. The composite highest tensile strength and impact strength were at 150 mesh particle size of chicken eggshell powder which is 45.153 MPa and 9. 04 J/m^2.

Keywords: calcite, chicken eggshell, compression molding, Gallus gallus domesticus, particle size.

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1. INTRODUCTION

In recent years, polymer composite has been used in engineering applications because of its good mechanical properties compared to metals [1]. Composite is made by mixing materials with good mechanical properties as filler with matrix (polymer), resulting in better properties than the material properties [2]. The matrix that is used in this research is an unsaturated polyester resin. The benefit of using this resin is it needs less fee, thermal and dimensional stability, good insulation of electricity has good mechanical properties, resistance to chemical substances, and low density [3]. Polyester resin has low stiffness and strength. To enhance polyester resin strength, particle filler is added to the composite making. The addition of particles changes the physical and mechanical properties of polyester so it increases the stiffness, tensile strength, impact strength, and other composite mechanical properties of composite [4].

The motorcycle exhaust pipe is usually very hot after the motorcycle is used for a while. A material with heat resistance is needed to make the motorcycle exhaust pipe cover. The term "heat resistant" refers to the polymer's capability of maintaining its physical and mechanical properties at 250^{o}C for 1000 hours, at 500^{o}C for 1 hour, and up to 1000^{o}C for several seconds. Various methods have been developed to enhance the heat resistance of polymers among which the most important ones are increasing crystallinity of the polymers, cross-linking of macromolecular chains, removing weak bonds from the chemical structure, and adding heat-resistant micro- or nano-sized fillers and fibers into the polymer bulk [5]

The eggshell is chosen as a filler because eggshell is one of the materials that have high calcium carbonate content (95%) so it is expected to enhance the mechanical properties of polymer composite [6] remembering calcium carbonate is one of the mostly-used fillers in the composite making to increase composite's strength and capacity to disperse well in the polymer matrix [7]. Consequently, in recent decades, $CaCO_3$ of different particle sizes extracted from natural resources is used as reinforcement to enhance the thermal stability, degradation, strength, and physical properties of polymeric materials. $CaCO_3$ is obtained from non-renewable sources. $CaCO_3$ from the chicken eggshell decomposed after it was annealed at 900^{o}C for 4 hours [8]. Besides, the eggshell is also available in large quantities. Just from the food processing industry, reaches 250.000 tons each year [6].

Some research has used eggshell as filler in the composite making but most research focuses on the percentage of eggshell weight and usage of the metal matrix as research done by Chaithanyasai (2014) which use eggshell as aluminum 6061 filler by changing the percentage of eggshell weight and as result, the addition of chicken eggshell increase the composite strength [9]. That's why the particle size of eggshell is used to make an innovation in making polyester composite and to see the effect of eggshell size as filler to mechanical properties of polyester composite which is made with compression molding methods.

2. MATERIALS AND METHOD

2.1 Materials

The chemicals used in this research are unsaturated polyester resin as matrix and methyl ethyl ketone peroxide (mekp) as a catalyst which was obtained from PT. Justus Kimia Raya located in Jalan Putri Hijau,

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Medan. The filler used was chicken eggshell. The chicken eggshell was obtained from Alamiah Bakery, Pasar IV, Medan-Marelan. The tool used was a sieve which is made of stainless steel and the mesh is made of nylon. It was obtained from CV. Rudang.

2.2. Method

2.2.1 Preparation of filler

The chicken eggshell was washed using clean water. It was dried under the sunlight for 6 hours. It was then ground by using a ball mill for 2 hours until it became powder. The chicken eggshell powder was sifted to obtain a different particle size of the filler such as 50 mesh, 70 mesh, 90 mesh, 110 mesh, 130 mesh, 150 mesh, and 170 mesh.

2.2.2 Composite making

In making a composite, polyester was poured into a plastic container. Then, the chicken eggshell was poured into the container with polyester with the matrix to filler ratio of 70:30 m/m [10]. The mixture was stirred and 1% methyl ethyl ketone peroxide catalyst was added to it [11]. The mixture was stirred again and then it was poured into the mold. The composite was pressed by using the compression molding method for 15 minutes. Then, the composite was taken out from the mold. The materials are used to make the motorcycle's exhaust pipe cover.

2.2.3 Density analysis (D792-08)

Density analysis was done in the Research Laboratory, University of Sumatera Utara. The tools used to find out the density of the composites were a measuring cup to find out the volume of the sample and a digital scale to find out the mass of the sample. This analysis was done to find out the mass of the composite in each volume unit. The specimen used in density analysis was round with 2 inches diameter and 0, 3 mm thickness. The equation used to find out the density of composite was [12]:

$$\rho=\frac{\text{mass}}{\text{volume}} \quad \text{........ (1)}$$

2.2.4 Water absorption analysis (ASTM D570-08)

The water absorption of polyester and polyester composite was analyzed by soaking the specimen in water at room temperature and it was weighed every 24 hours [13] until the composite did not absorb water anymore. Water absorption analysis was done in the Research Laboratory, University of Sumatera Utara. The tools used in this research were beaker glass to soak the specimen and a digital scale to weigh the specimen before and after soaking. Every 24 hours, the specimen was taken out of the water and wiped using a tissue so that there is no water on the surface. Then, the specimen was weighed and calculated by using the equation below [12].

$$Wg=\frac{We-Wo}{Wo}\times 100\% \quad \text{...... (2)}$$

Wg = composite mass increase composite
We = composite mass after soaking
Wo = composite mass before soaking

2.2.5 Tensile strength analysis (ASTM D638-10)

Tensile strength analysis is usually done to find out material deformation ability before it snaps so that the data obtained can be used to choose suitable materials for engineering applications [14]. Tensile strength analysis was done in the Research Laboratory, University of Sumatera Utara. The mechanical properties are usually learned by observing tensile strength by using a tensometer. Tensometer used in this research was Universal Testing Machine Gotech AI-7000M at 30 mm/s.

2.2.6 Elongation at break analysis

Elongation at break analysis was done to find out whether the materials can encounter elongation before it snaps when it is loaded. Elongation at break analysis was done in the Research Laboratory, University of Sumatera Utara by using Universal Testing Machine Gotech AI-7000M at 30 mm/s.

2.2.7 Impact strength analysis (ASTM D256-04)

Impact strength analysis shows the amount of energy absorbed when a specimen is fractured by high-speed impact. The ability of the specimen to resist the impact is called impact strength [15]. Impact strength analysis was done by impact tester Gotech GT-7045-MDL with 5.5 J impact energy and at the speed of 3.46 m/s. It was done in the Research Laboratory, University of Sumatera Utara. The specimen size was 65 mm x 12.7 mm x 3 mm. The method used for impact strength analysis was the unnotched Izod method.

2.2.8 Fourier Transform Infra-Red (FTIR) spectrophotometry analysis

Infra-red spectrophotometry is a method used to analyze the vibration of the functional group which shows the molecule structure of a material [16]. FTIR analysis was done at Balai Penganalisaan dan Identifikasi Barang Tipe B Medan by using Thermo Nicolet iS10. The specimen was ground before it was analyzed. The specimen was put on ZnSe Crystal as sample holding. Then the Thermo Nicolet iS10 was run to obtain the FTIR spectrum of the sample.

2.2.9 Scanning electron microscopy energy dispersive X-ray (SEM EDX)

SEM EDX analysis was done to analyze the morphology and the chemical composition of a material [17]. SEM EDX analysis was done at MIPA Laboratory, Institut Teknologi Bandung. SEM EDX analysis was done by using JEOL JSM 6510 LA Scanning Electron Microscope with 10000 and 20000 times magnification.

2.2.10 X-Ray Diffraction Analysis (XRD)

XRD analysis is a common technique to find out the crystallinity of a sample [18]. XRD analysis was done

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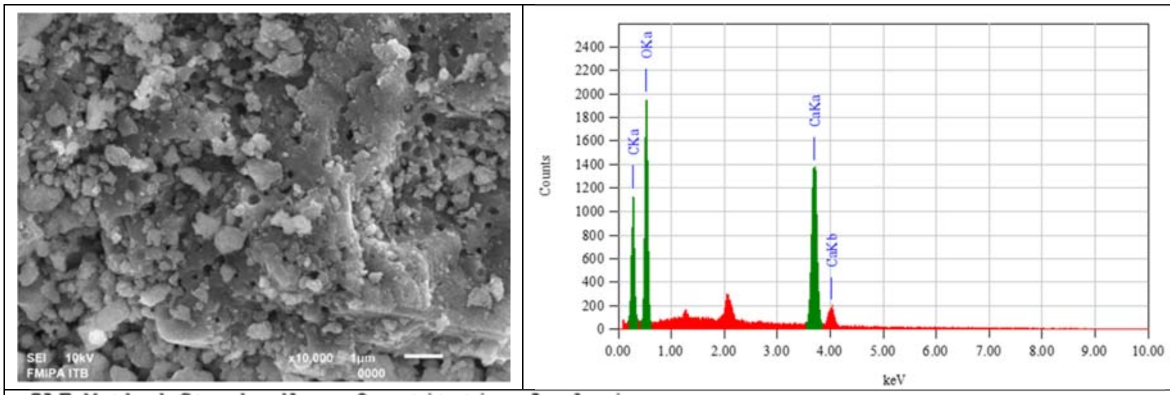
in Physic Laboratory, Medan State University by using X-Ray Diffractometer Shimadzu 6100.

3. RESULTS AND DISCUSSIONS

3.1 Filler Characterization

3.1.1 Scanning Electron Microscope Energy Dispersive X-ray (SEM EDX) Characterization of Chicken Eggshell (*Gallus gallus Domesticus*)

SEM EDX of chicken eggshell was done to find out the morphology of chicken eggshell particles and the element of chicken eggshell. The SEM EDX analysis result of 150 mesh chicken eggshells is shown in Figure-1.



ZAF Method Standardless Quantitative Analysis

Fitting Coefficient : 0.4378

Element	(keV)	Mass%	Error%	Atom%	Compound	Mass%	Cation	K
C K	0.277	23.80	0.12	36.31				21.6666
O K	0.525	41.93	0.30	48.02				30.0067
Ca K	3.690	34.27	0.53	15.67				48.3268
Total		100.00		100.00				

Figure-1. Scanning Electron Microscopy Energy Dispersive X-ray (SEM EDX) Characteristic of 150 mesh Chicken Eggshell (*Gallus gallus Domesticus*) Particle.

Figure-1 shows that the chicken eggshell particle is spherical. It consists of 23.80% of C, 41.93% of O, and 34.27% Ca (mass %). This result is supported by the research done by Ajala, *et al* (2018) who researched chicken eggshells. Ajala, et al (2018) did an SEM EDX analysis of chicken eggshells and it resulted that chicken eggshells consisted of 38.52% of C, 39.88% of O, and 21.17% Ca [19].

3.1.2 X-Ray Diffraction (XRD) Characterization of Chicken Eggshell (*Gallus gallus Domesticus*) Particle

X-ray diffraction (XRD) of the chicken eggshell particles was done to analyze crystal properties and crystallinity index of chicken eggshell by using x-ray. The crystallinity index is an index that determines the crystal structure of a material [20]. The determination of the crystallinity index of a material can be done by using the Segal method [21]. Segal method determines crystal index by comparing the intensity of crystal peak to total peak [20] which is shown in the equation below.

$$C_{IR}(\%) = (I_{200} - I_{am})/I_{200} \times 100\%$$

The result of crystallinity analysis by XRD is shown in Figure-2.

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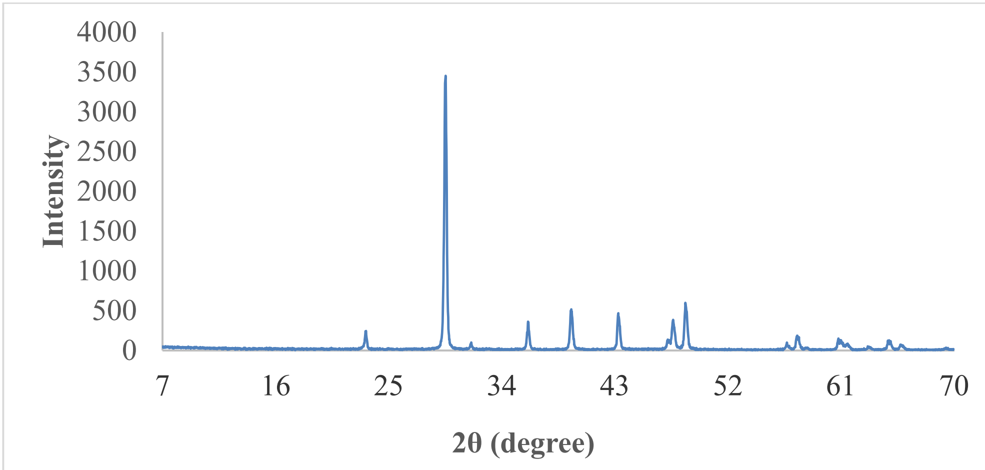


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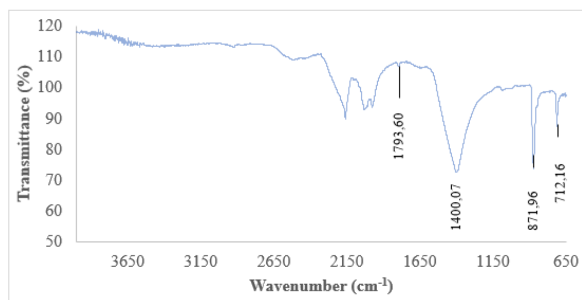
<Card List>
No.  Card      Chemical Formula                          S       L          d      I      R
               Chemical Name (Mineral Name)                      Dx     WT%    S.G.
  1 5-0586 CaCO3                                     0.890 0.579(11/42) 0.764 0.863 0.382
              Calcium Carbonate ( Calcite, syn )                 -----  -----  -------
  2 5-0586 CaCO3                                     0.890 0.579(11/42) 0.764 0.863 0.382
              Calcium Carbonate ( Calcite, syn )                 -----  -----  -------
```

Figure-2. X-Ray Diffraction (XRD) Characteristic of 150 mesh Chicken Eggshell (*Gallus gallus Domesticus*).

From Figure-2, the crystallinity index of the chicken eggshell particle can be determined by the Segal method according to maximum intensity data at a sharp peak. The maximum intensity of chicken eggshell particle is 3450 at 2θ =29, 54° and the minimum intensity is 2 at 2θ =54, 18°. The crystallinity of the chicken eggshell is 99.94%. Figure-2 also shows that the constituent component of chicken eggshell is calcite ($CaCO_3$). This result is supported by the SEM EDX result which shows that there is Ca, O and C in chicken eggshell. The XRD analysis result is also supported by Naemchan, *et al* (2008) who researched to analyze the crystallinity of chicken eggshells and compare it to commercial calcite. Their result showed that there are similar peaks of chicken eggshell XRD result and commercial calcite XRD result [22].

3.1.3 Fourier Transform Infra-Red (FTIR) of Chicken Eggshell Particle (*Gallus gallus Domesticus*)

FTIR characterization of the chicken eggshell particle was done to find out the functional group of chicken eggshells. FTIR characterization result of chicken eggshell is shown in Figure-3.



Functional group analysis [23]:
- 1850-1500 cm^{-1} : C=O
- 1400-1150 cm^{-1} : C-O
- 800-600 cm^{-1}: C-Cl

Figure-3. Fourier Transform Infra-Red (FTIR) Characteristic of 150 mesh Chicken Eggshell (*Gallus gallus Domesticus*) Particle.

Figure-3 shows that there are several peaks to indicate the functional group of chicken eggshells. The wavenumber 1793.60 cm^{-1} indicates C=O, the wavenumber 1400.07 cm^{-1}indicates C-O and the wavenumber 712.16 cm^{-1} indicates C-Cl.

The characteristic peak of calcite is in the range of 1530-1320 cm^{-1} with a broad peak which indicates C-O [24]. FTIR result shows that there is a broad peak at that range so it can be stated that the chicken eggshell is composed of calcite. This result is supported by XRD and SEM EDX analysis result which showed that there is C, Ca, and O in chicken eggshells. This result is also supported by the research done by Alvin, *et al* (2021) who did an FTIR analysis of calcite extracted from the chicken eggshell. The result showed a significant peak at 1400cm^{-1} which indicates calcite contained in chicken eggshells. This peak also appeared in this research. Besides, peaks that indicate carbonate in chicken eggshells were 1792 cm^{-1} and 712 cm^{-1} and those peaks also appeared in the FTIR analysis of this research [25].

3.2 Composite Characterization

3.2.1 Scanning Electron Microscopy Energy Dispersive X-ray (SEM EDX) Characterization of Polyester and Polyester Composite Filled by Chicken Eggshell (*Gallus gallus Domesticus*) at 70:30 Polyester to Filler Ratio

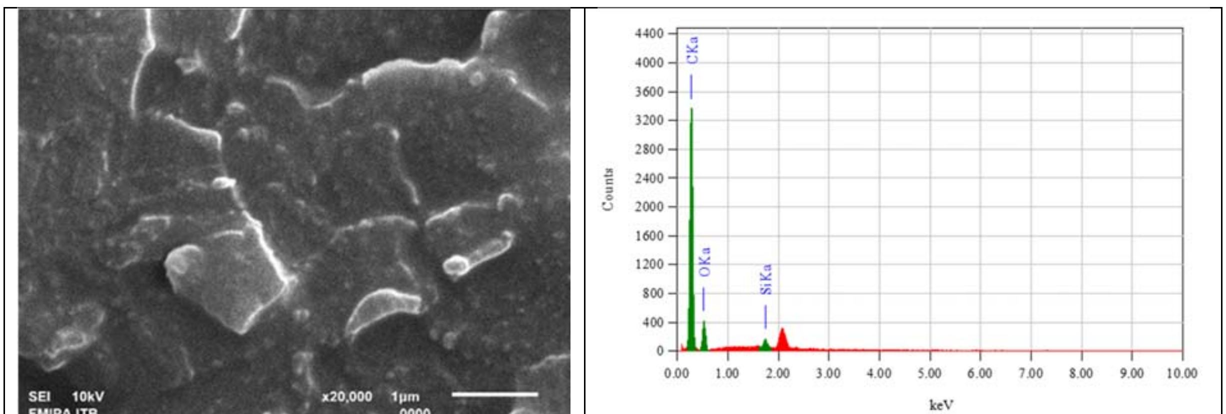
SEM EDX characterization of polyester and polyester composite filled by 150 mesh chicken eggshell was done to find out the morphology and the constituent element of fracture composite. SEM EDX characteristic of polyester and polyester composite filled with chicken eggshell is shown in Figure-4.

Figure-4 (a) shows that the fracture surface is smooth. It shows that the polyester is fragile and damages easily if it is loaded. SEM EDX analysis resulted that polyester consisted of 87.76% of C, 11.19% of O, and 1.05% of Si (mass %). This result is supported by Hassan, *et al* (2012) research which showed that there was C, O, and H in polyester but there is no Si [26].

Figure-4 (b) shows the surface morphology of the 150 mesh chicken eggshell-filled polyester composite fracture. The surface looks rougher which indicates that the chicken eggshell particle is dispersed uniformly in the polyester matrix and has a good interface bond. SEM EDX showed that chicken eggshell-filled polyester composite is made of 80.08% of C, 12.01% of O, and 7.91% Ca (mass %). This result is supported by Hassan, *et al* (2012) research. They analyzed SEM EDX of composite filled with chicken eggshell and observed that there were C, Ca, and O in the composite. However, there is not any Si [27].

3.2.2 Fourier Transform Infra-Red (FTIR) Analysis of Polyester and Polyester Composite Filled with Chicken Eggshell (*Gallus gallus Domesticus*)

FTIR of polyester and chicken eggshell-filled polyester composite was done to identify a functional group of polyester and chicken eggshell-filled polyester composite. FTIR characteristic of polyester and chicken eggshell-filled polyester composite is shown in Figure-5.



ZAF Method Standardless Quantitative Analysis

Fitting Coefficient : 0.6039

Element	(keV)	Mass%	Error%	Atom%	Compound	Mass%	Cation	K
C K	0.277	87.76	0.23	90.84				92.0296
O K	0.525	11.19	0.80	8.70				6.7304
Si K	1.739	1.05	0.44	0.47				1.2400
Total		100.00		100.00				

(a)

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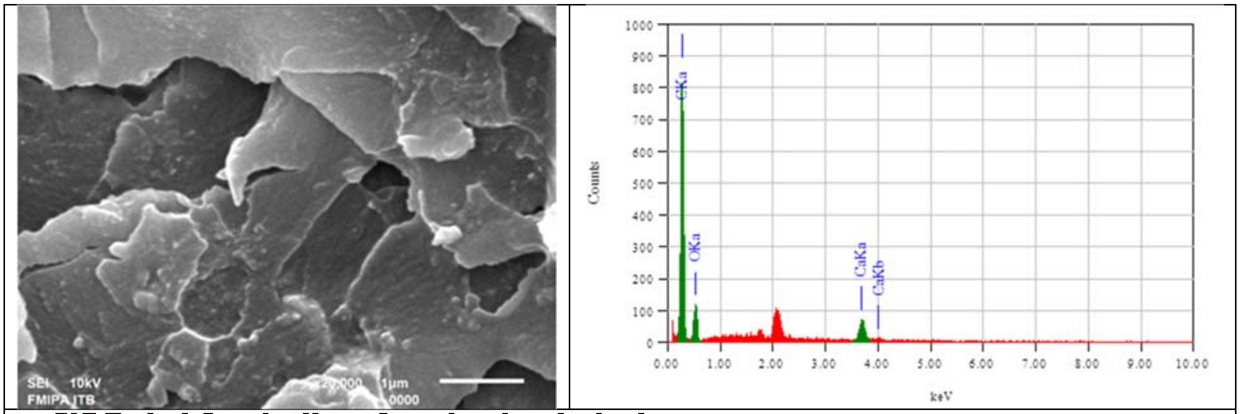


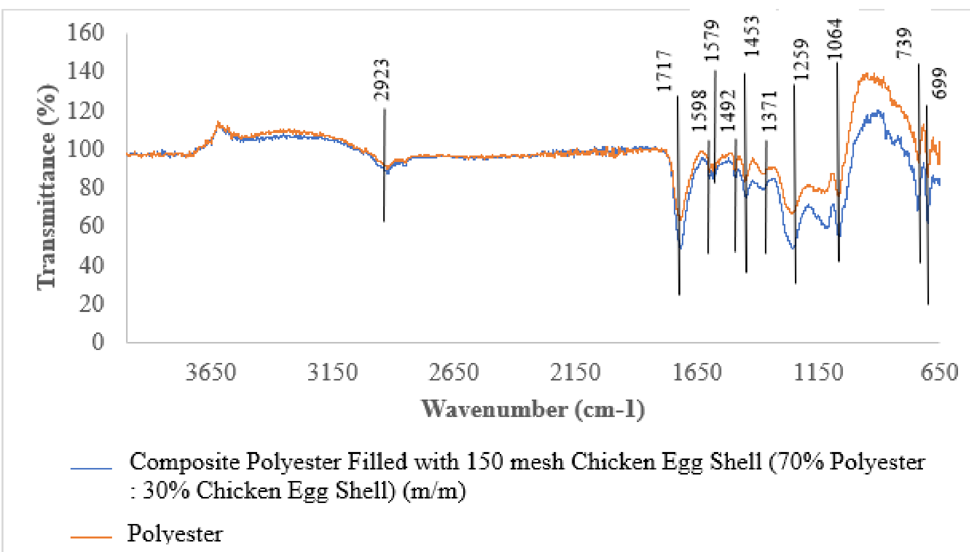
ZAF Method Standardless Quantitative Analysis

Fitting Coefficient : 0.6945

Element	(keV)	Mass%	Error%	Atom%	Compound	Mass%	Cation	K
C K	0.277	80.08	0.02	87.55				83.5004
O K	0.525	12.01	0.02	9.86				6.7614
Ca K	3.690	7.91	1.32	2.59				9.6201
Total		100.00		100.00				

(b)

Figure-4. SEM EDX Characteristic of (a) Polyester Fracture (b) Polyester Composite Filled With 150 mesh Chicken Eggshell Fracture at a 20000-time magnification



Wavenumber[28]:
- 2923 cm^{-1} : C-H stretch
- 1717 cm^{-1} : C=O stretch
- 1598-1371 cm^{-1} : C=C stretch
- 1259 cm^{-1} : C-O-C stretch
- 1064 cm^{-1} : C=C-H
- 739-699 cm^{-1} : C-H bending

Figure-5. Fourier Transform Infra-Red (FTIR) Analysis of Polyester and Polyester Composite Filled with 150 mesh Chicken Eggshell (*Gallus gallus Domesticus*).

Figure-5 shows that both polyester and chicken eggshell-filled polyester composite have C-H stretch at 2923 cm^{-1}, C=O stretch at 1717 cm^{-1}, and C=C stretch at 1598-1371 cm^{-1}, C-O-C stretch at 1259 cm^{-1}, C=C-H bending at 739-699 cm^{-1}. This FTIR result is supported by the research on the polyester ortho-phthalic resin of Dholakiya (2012). His research showed C-H stretch at 2985cm^{-1}, C=O stretch at 1736 cm^{-1}, C=C stretch at 1306 cm^{-1},C-O-C stretch at 1145 cm^{-1}, C=C-H at 1004 cm^{-1}, and C-H bending at 755 cm^{-1}. Dholakiya (2012) also stated that the CH=CH bond almost disappeared and caused the sharper peak as shown in the 2923 cm^{-1} wave number in this research. Besides, crosslinking caused the C=C peak at 1453 cm^{-1} to become clearer [28].

3.2.3 Density analysis

The density of polyester composite filled with chicken eggshell is shown in Figure-6.

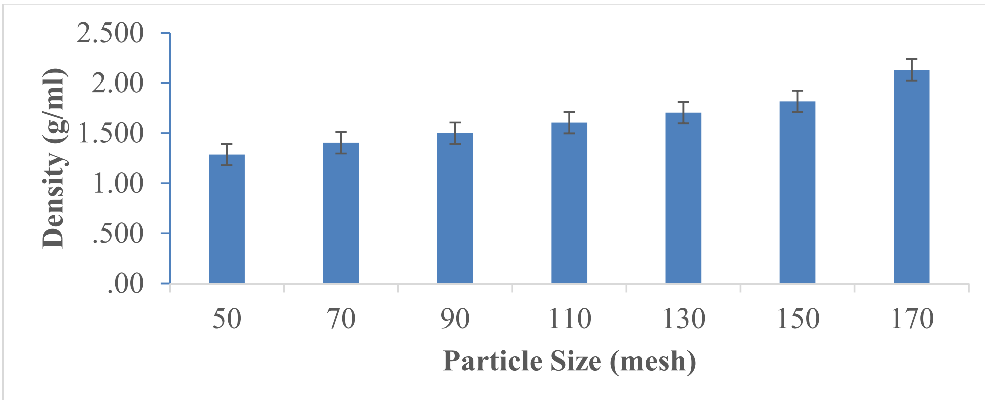


Figure-6. Effect of particle size on the density.

Figure-6 shows that the smaller the particle size resulted in the bigger the composite density. The biggest density is 2.240 g/cm^3. It is shown in the composite with 170 mesh chicken eggshells. This is caused by the density of chicken eggshell (2.147 g/cm^3) [29] which is higher than the density of polyester (1.10 g/cm^3) [30]. Smaller particle size also causes less void formed so that it enhances the density of the composite[8]. This result is supported by Ameh, *et al* (2015) who varied the particle \ size in making polyester composite. The particle size used in their research was 0.5 mm, 2.0 mm, and 2.8 mm. Their research reported that the biggest composite density was obtained at 2.8 mm, the smallest particle size used [31].

3.2.4 Water absorption analysis

The water absorption analysis result is shown in Figure-7.

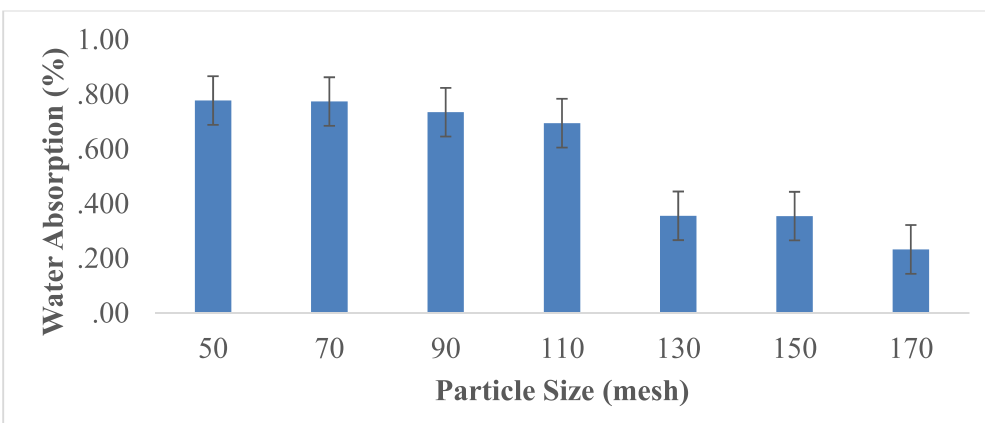


Figure-7. The effect of particle size on water absorption.

Figure-7 shows that the smaller the particle size causes the less water absorbed by the composite. The most water absorbed was 0.904% by polyester composite filled with 50 mesh chicken eggshell and the least water absorbed was 0.114% by polyester composite filled with 170 mesh chicken eggshell. Composite with bigger filler particle size has the most water absorbed because water diffusion happened easier in bigger particle size [30]. This research is supported by the research of Norhidayah, *et al* (2014). They varied Bertam particle size as filler in polyester. The result showed that the least water

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absorption happened on the smallest filler particle size used, which was 284 µm [32].

2.2.5 Tensile strength analysis

The result of tensile strength analysis of polyester composite filled with chicken eggshell is shown in Figure-8.

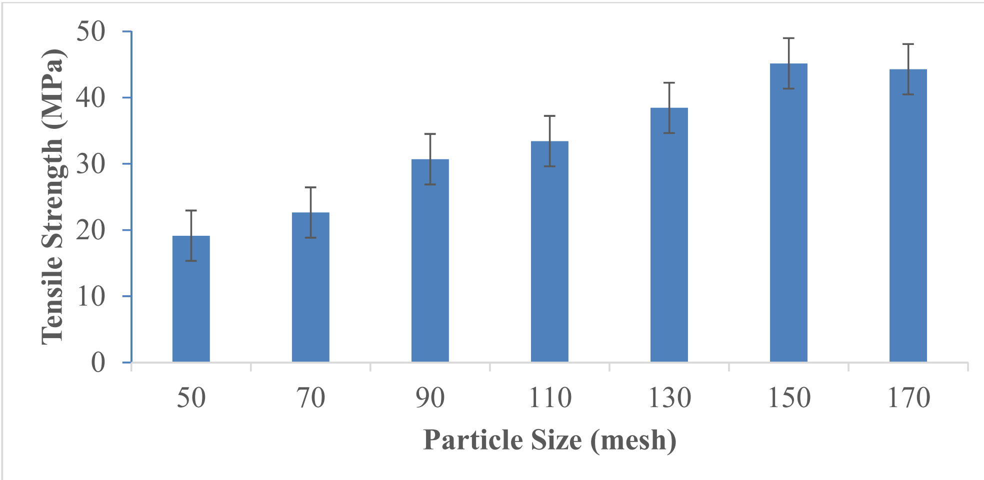

Figure-8. The Effect of Particle Size on Composite Tensile Strength.

Figure-8 shows that the tensile strength of the composites increases as the particle size gets smaller and reaches the highest tensile strength of 45.154 MPa by 150 mesh particle size. This is caused by the smaller the particle size used; the surface will get bigger so that the interface between filler and matrix becomes stronger [33] which causes the enhancement of the mechanical properties of the composite. This result is supported by SEM EDX result which showed that polyester composite filled with 150 mesh chicken eggshell showed the chicken eggshell was dispersed uniformly in the matrix so that the good interface happened. This is also supported by Shehu, *et al* (2014) research which varied the particle size of palm seed shells as filler in the polyester matrix and resulted that the smaller the particle size used, the bigger the tensile strength obtained [34].

3.2.6 Elongation at break

The result of elongation at the break of polyester composite filled with chicken eggshell can be seen in Figure-9.

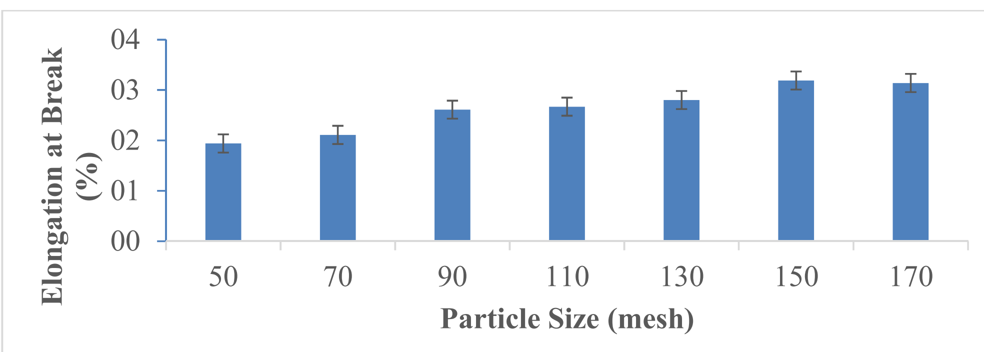

Figure-9. The effect of particle size of the elongation at break of composite.

Figure-9 shows that the elongation at the break of composite filled with chicken eggshell keeps increasing until it reaches the highest value, 3.19% by using 150 mesh particle size. This is caused by the bigger particle size used causes the filler is not uniformly distributed so that the interface of filler and matrix is not strong enough and it causes the composite to be broken when it is only pulled for a while so that the elongation at break value is low. This result is supported by Onuoha, *et al* (2017) who varied the particle size in making polypropylene composite reinforced by snail shell powder. The result showed that the elongation at break increased by using a smaller particle size of filler [35].

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3.2.7 Impact strength analysis

The effect of particle size on the impact strength of polyester composite filled with chicken eggshell can be seen in Figure-10.

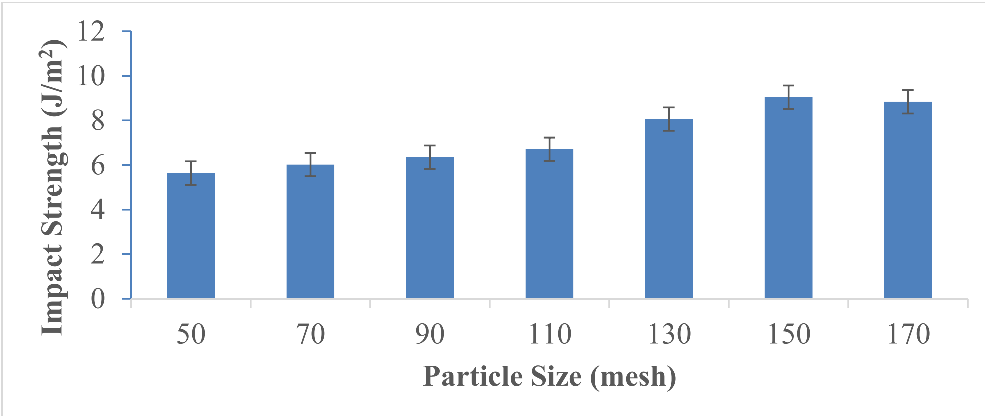

Figure-10. The effect of particle size on the impact strength of polyester composite filled with chicken eggshell.

Figure-10 shows that the impact strength of polyester composite filled with chicken eggshells keeps increasing as the particle size gets smaller. The highest impact strength was 9.04J/m^2 by using 150 mesh chicken eggshells. The enhancement of the impact strength was caused by the smaller the particle size, the interface area gets bigger so that the bond between matrix and filler becomes stronger [36]. This research is supported by Daniel, *et al* (2017) who varied the particle size of rice husk and coconut shell as filler. The size used was 75 µm, 50 µm, and 300 µm. The result showed that the impact strength increased at 150 µm and decrease at 300µm [37].

4. CONCLUSIONS

The chicken eggshell is made up of calcite. It consists of C, O, and Ca which was shown in the SEM EDX result. The smaller the particle size is used to enhance the physical and mechanical properties of the composite. The highest density was at 2.240 g/cm^3 and the lowest water absorbed was 0.114% by using 170 mesh chicken eggshell particle size. The highest tensile strength, elongation at break, and impact strength were 45.153 MPa, 3.19%, and 9.04 J/m^2 respectively by using 150 mesh chicken eggshell particle size.

ACKNOWLEDGMENT

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