

EFFECT OF HOT PRESS TEMPERATURE ON PHYSICAL AND MECHANICAL PROPERTIES OF COMPOSITES OF POLYESTER RESIN FILES WITH CHICKEN EGG (Gallus Gallus Domesticus) Shell Powder

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ABSTRACT

This study was conducted to study the effect of temperature hot press variation of mechanical chicken egg polyester chicken egg powder (Gallus Gallus Domesticus). This research was conducted in several places, namely the preparation of eggshells as fillers, composite making, and examiners. The eggshell is mashed and sifted, then the chicken eggshell powder is mixed with polyester using 80/20 and the mekpo catalyst is 1% of the mass of the matrix. After that, it is printed using compression mold with variations in temperature of 30 °C, 45 °C, 60 °C, 75 °C, 90 °C, and 105 °C. The composites formed were then analyzed for their mechanical properties and the results of the tensile strength and composite impact required for variations in temperature of the hot press 60 °C were 35.78 MPa and 8.99 J/m² were achieved.

Keywords: gallus gallus domesticus, composite, polyester, mechanical properties, temperature.

INTRODUCTION

Unsaturated polyester resin (UPR) is a thermoset material that used for matrix in composite material. UPR curing is an exothermic reaction that caused viscosity growth due to molecular weight increase from three dimensional cross-linked networks [1]. UPR is also used as a matrix [2] because UPR has a low cost and a wide variety of grades to meet specific requirements. To increase the strength of this polyester composite, it can be done by adding fillers to the polyester.

In this study, the chicken eggshell powder was chosen as filler for this polyester composite. Chicken eggshells contain calcium carbonate (CaCO₃) of 94% of the total body weight of the shell, calcium phosphate (1%), organic matter (4%) and magnesium carbonate (1%). With large calcium carbonate content, chicken eggshells have relatively very strong properties. The large number of eggshells results in more waste produced. With the many problems of eggshell waste, it is also relatively strong because it contains large enough CaCO₃, so the chicken eggshell waste is used as natural filler in the composite and is expected to improve the mechanical properties of the composite and further empower the chicken eggshell waste.

Many studies have been carried out by varying the temperature of the hot press, including Elahi, et al (2014) which made polyester composites by varying the temperature of the press (60° C, 90° C, 120° C, and 150° C) with fiber-glass fillers. From the results obtained that at a temperature of 90° C is the optimum temperature for the mechanical strength of the composite which includes tensile strength, impact strength elongation value at break and composite hardness [3]. This study aims to find out how the optimum temperature of the hot press on physical and mechanical properties of polyester composites filled with chicken eggshell powder. The tests carried out include density, tensile test, impact test, water absorption test, Fourier Transfer Infra-Red on composites and fillers, X-Ray Diffraction fillers and Scanning Electron Microscope on composites and fillers.

MATERIAL AND METHODS

Material

The main material in this study is Unsaturated Polyester obtained from PT Justus Raya, Medan, Indonesia. Whereas for fillers, namely chicken eggshells, obtained from several restaurants located in the Asia Mega Mas Complex, Medan, Indonesia. Other supporting materials were the MEKPO Catalyst obtained from PT Justus Raya. The sieve of 110 mesh is obtained from a home industry located in Semarang, Central Java.

Tests on polyester composites filled with chicken eggshell powder were carried out in several places. For tensile test, impact test, density, and water absorption test, testing was carried out at the Research Laboratory of the Faculty of Engineering, University of North Sumatra, Medan. As for the XRD test, it was conducted at the Research Laboratory of the Faculty of Mathematics and Natural Sciences, University of Medan Area, Medan. For FTIR testing, carried out in Goods Testing and Identification Center (BPIB), Jalan Sumatera, Belawan. For SEM testing carried out at the Bandung Institute of Technology, Bandung.

Preparation of Filter

The eggshell fillers obtained from various restaurants are washed thoroughly and dried in the sun for about 8-12 hours until they dry out. Then the filler was milled with a Ball Mill tool carried out at the Chemical Engineering Operations Laboratory, University of North Sumatra, Medan to form a powder which was then sieved with 110 mesh particle size.

Preparation of Polyester Matrix

Unsaturated Polyester mixed with a catalyst of Methyl Ethyl Ketone Peroxide with a ratio of 1% weight which is then stirred evenly for 10-15 minutes.

Composites

The prepared polyester is then mixed with fillers with a comparison of polyester and eggshell powder is 80:20 and stirred evenly. After being mixed evenly, pour the mixture into a 200 x 200 x 3 mm mold that has been smeared with glycerin as a lubricant. Then the mixture is pressed by a compression molding device with a temperature variation of 30 °C, 45°C, 60 °C, 75 °C, 90 °C and 105 °C for 15 minutes. After the printing time has ended, let the composite dry and remove it from the print. Composites that are still in the form of thin plates are then drawn according to the ASTM D-638 14 standard and cut.

Composite Analysis and Characterization

The composite specimens that have been cut will be tested. Tests carried out include density test, tensile test, impact test, water absorption test, EDX SEM, FTIR, and XRD.

Analysis of Density of ASTM D792-08

The density analysis was carried out at the Research Laboratory, University of North Sumatra. This analysis aims to determine the composite mass per unit volume. Density analysis is done by using a digital balance to determine the mass of specimens and measuring cups to determine the volume of the specimen.

Analysis of Water Absorption ASTM D570-98

Analysis Water absorption is carried out at the Research Laboratory, the University of North Sumatra with the help of beaker glass and digital balance. The composites that want to be analyzed are immersed in water in a beaker glass and weighed every 24 hours until the composite can no longer absorb water.

Analysis of Tensile Strength of ASTM D638

This test was carried out with the help of the Gotech Al 7000-M tool located at the Polymer Laboratory, University of North Sumatra. Tensile strength testing was carried out with a tensometer for each specimen with a thickness of 4 mm. Tensometer is first conditioned on a load of 100 kgf with a speed of 20 mm/minute, then clamped firmly with a dial that is dialled. The engine is

turned on and the specimen will be pulled up to the specimen observed until it breaks, the maximum stress and strain are recorded [4].

Analysis of ASTM D4812 Impact Strength

This test is also carried out with the help of the Gotech Impact Tester tool which is in the Polymer Laboratory, University of North Sumatra. The specimens are clamped vertically which will be bumped with a hammer. The impact strength of the composite will appear on the screen of the device.

Scanning Analysis Electron Microscope Energy Dispersive X-Ray (SEM EDX)

This testing was carried out at the Bandung Institute of Technology with the help of the JSM-6510 LA JEOL tool. The composite sample is inserted into the chamber with the initial parameters Rotation stage and 0° slope angle. This SEM tool has to be made to adjust the apartment, alignment and filament centering. After the sample is ready for characterization, the image will be taken according to the desired magnification.

Fourier Transform Infra-Red (FTIR) Analysis

This test was carried out at the Goods Testing and Identification Center (BPIB), Jalan Sumatera, Belawan. To do this test, a tool called Thermo nicolet is10 is used. The analyzed specimens will be mashed and placed on top of the ZnSe Crystal as a sample place which will later be obtained FTIR spectrum from the sample analyzed

X-Ray Diffraction (XRD) Test

The XRD test is currently the most common technique for knowing the crystallinity of a sample. The X-Ray Diffraction (XRD) test was carried out at the Physics Laboratory, Medan State University using the X-Ray Diffractometer Shimadzu 6100.

RESULTS AND DISCUSSIONS

Characterization of Scanning Electron Microscope Energy Dispersive X-Ray (SEM EDX) Chicken Egg Shell (*Gallus Gallus Domesticus*)

The purpose of the characterization of Scanning Electron Microscope (SEM) is to find out how the morphology of chicken eggshell powder fillers. The characteristics of SEM EDX from chicken eggshell powder can be seen in Figure-1.

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Figure-1. Scanning Electron Microscopy Test Results on 110 mesh Chicken Egg Shell Powder.

Fillers 10,000x Magnification From the results of SEM EDX, it was found that the constituent elements of chicken eggshell were elements C, O and Ca, where element C composed 46.01% of the mass of chicken eggshells, element O compiled 35.66% of the mass of chicken eggshell and Ca compose 18.33% of chicken eggshell powder. In the study conducted by Ajala et al [6] which conducted research on chicken eggshells, it was found that in the SEM EDX analysis, the chicken eggshell contained C, O, Cl and Ca which had a successive value of 38.52%, 39.88 %, 0.43% and Ca 21.17 %. This result almost resembles the results of the SEM EDX analysis

carried out in this study where there are levels of C, O and Ca.

Characterization of X-Ray Diffraction (XRD) Chicken Egg Shell Particles (Gallus Gallus Domesticus)

The purpose of X-Ray Diffraction (XRD) characterization of fillers of chicken eggshell powder was to analyze the properties of crystals and the crystallinity index of chicken eggshell powder fillers obtained using X-rays. The results of crystallinity testing using XRD can be shown in Figure-2.



Figure-2. Characteristics of X-Ray Diffraction (XRD) Chicken Egg Shell Powder Filler.

Determination of the crystallinity index of a material can be done using the Segal method. The Segal method determines the crystal index by comparing the intensity of the crystal peak with the total intensity indicated by the following equation [6]:

$$C_{\rm IR}(\%) = (I_{200} - I_{\rm am})/I_{200} \ge 100\%$$
(1)

Based on the data obtained, the maximum intensity at sharp absorption peaks (sharp peak) of the spectra produced by samples of chicken eggshell powder is at $2\theta = 29.52$ ° with an intensity of 3210 and the minimum intensity value is at $2\theta = 59.42$ ° with an intensity of 2. The crystallinity index of chicken eggshell powder is 99.93%. Figure-2 also shows that the results of the XRD qualitative analysis show that the components of the chicken eggshell are calcium carbonate. Calcium carbonate contained in chicken eggshells is supported by

SEM EDX analysis which shows the presence of elements Ca, O and C. Naemchan, *et al* (2008) conducted a study comparing the results of commercial calcium carbonate analysis with chicken eggshells. From the results of research conducted by researchers, it was found that the similarity of the peak of the calcium carbonate between commercial calcium carbonate and chicken eggshell [7]. This can indicate that chicken eggshells contain calcium carbonate.

Characterization of Fourier Transform Infra-Red (FTIR) Chicken Egg Shell Particles (Gallus Gallus Domesticus)

Characterization of the FTIR (Fourier Transform Infra-Red) filler of eggshell powder is to identify the functional groups of the eggshell powder itself. The FTIR characteristics of the eggshell powder can be seen in Figure-3.

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www.arpnjournals.com 120 110 **Fransmittance** (%) 100 90 80 712,01 70 [792,41 870,99 400,98 60 50 2150 1650 3650 3150 2650 1150 650 Wavenumber (cm⁻¹) Description [8]: $1850-1500 \text{ cm}^{-1}$: C=O

Figure-3. Characteristics of Fourier Transform Infra-Red (FTIR) Chicken Egg Shell Powder.

Based on Figure-3, it can be seen that there are several key peak peaks which can indicate a group as a special feature of a compound. The FTIR test results showed that there was a C = O bond which was shown at peak with a source of 1792.41 cm⁻¹. In addition, there is also a C-O group, which is at a peak with a source of 1400.98 cm⁻¹ and a C-Cl group at 712.01 cm⁻¹.Typical CaCO₃ peak peaks are in the range 1530-1320 cm⁻¹ with broad peaks indicating the C-O function group [6].

Based on this theory, the results of FTIR analysis on chicken eggshells showed that chicken eggshells were composed of calcium carbonate. This is supported by the results of SEM and XRD analysis which shows the presence of elements of Ca, C and O in chicken eggshells. In the study conducted by Ongo, et al (2016) who performed FTIR analysis on calcium carbonate, it was found that calcium carbonate from chicken eggshell extract showed a significant peak at 1404 cm⁻¹ which showed the presence of carbonates in eggshells. This peak also appears in the FTIR analysis carried out on eggshells in this study. In addition, the peak that shows the presence of carbonate in the eggshell is at 709 cm⁻¹ and 871.8 cm⁻¹ and the peak also appears in the FTIR analysis of chicken eggshells in this study [9].

Characterization of Energy Dispersive X-Ray (SEM EDX) Scanning Electron Microscopy Polyester and **Chicken Egg Shell (Gallus Gallus Domesticus) Composite Composites with Composition** Composition 80:20 with 60°C Temperature

The aim of the characterization of Scanning Electron Microscopy Energy Dispersive X-ray (SEM EDX) is to determine the fracture morphology of a composite. Fault morphology will provide additional information about how damage occurs to the fault section. The characteristics of SEM EDX from polyester composites filled with chicken eggshell powder can be seen in Figure-4.



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Figure-4. Scanning Electron Microscopy EDX Test Results (a) After Samples Testing Tensile Strength (b) Samples Before Testing the Tensile Strength of Composite Polyester Chicken Egg Shell (Gallus Gallus Domesticus) with Comparison of 80/20 Matrix/Fillers and Hotpress Temperature 60 °C.

In Figure-4 (a), it can be seen that the morphology of the sample surface after testing the tensile strength of a chicken egg-filled polyester composite (Gallus Gallus Domesticus) with an 80/20 matrix / fill ratio and a temperature of 60° C has a rough but evenly distributed surface. This shows that fillers can be well dispersed, and this is reinforced by the absence of void seen in the sample. In Figure-4 (b), it can be seen that the morphology of the sample surface before the tensile strength testing of chicken egg-filled polyester composite (Gallus Gallus Domesticus) with 80/20 matrix / fill ratio

and 60° C hot press temperature looks finer compared to sample after tensile testing in Figure-4(a).

From the results of the SEM EDX that has been done on a broken composite sample (a), it was found that the constituent elements of chicken eggshell were elements C, O and Ca, where unus C composed 84.28% of the mass of the chicken eggshell, element O compiled 11.08% of masses of chicken eggshells and Ca elements comprise 4.64% of chicken eggshell powder. Whereas in intact composite samples (b), it was found that the constituent elements of chicken eggshells were elements



C, O and Ca, wherein C composes 81.04% of the mass of chicken eggshells, element O composes 17.01% of the mass of chicken eggshells and elements Ca composes 1.95% of chicken eggshell powder. In a study conducted by Hassan et al (2012) who conducted SEM testing of EDX on chicken egg shells, it was found that chicken shell powder contained elements of Ca, Si, O, C and Mg [9]. This shows that the results of the research conducted have similarities because, in the tests carried out, the composite of chicken eggshell powder also contains C, O and Ca.

Characterization of Pure Polyester Fourier Transform Infra-Red (FTIR) and Composite Polyester Filled Chicken Egg Shells (Gallus Gallus Domesticus)

The purpose of the characterization of Fourier Transform Infra-Red (FTIR) of polyester and polyester composites filled with chicken eggshell powder is to identify functional groups of pure polyester compounds and polyester composites filled with chicken eggshell powder. FTIR characteristics of pure polyester and polyester composites filled with chicken eggshell powder can be seen in Figure-5.



Figure-5. Characteristics Fourier Transform Infrared (FTIR) of Polyester and Composite Polyester Filled Chicken Egg Shells (Gallus Gallus Domesticus).

From Figure-5, it can be seen for pure polyester and chicken shell-filled polyester composites having CH stretch group at 2926 cm⁻¹, C = O stretch at 1716 cm⁻¹, C =C stretch at 1598-1366 cm⁻¹, COC stretch at 1258 cm⁻¹, C = CH at 1060 cm-1 and CH bending at 737-698 cm⁻¹. The results of the FTIR analysis were supported by previous research conducted by Dholakiya (2012) who carried out FTIR analysis on ortho-phthalic resin pure polyester. The results obtained showed that the peak obtained in his study was CH stretch at 2985 cm⁻¹, C = O stretch at 1736 cm⁻¹, C= C stretch at 1306 cm⁻¹, COC stretch at 1145 cm⁻¹, C = CH at 1004 cm⁻¹ and CH bending at 755 cm⁻¹. Dholakiya (2012) also stated that the CH = CH bond which is almost lost in the composite causes a sharper peak as in this study, which is at source 2923 cm⁻¹. In addition, the presence of crosslinking causes a peak in the C = C group in the 1453 cm⁻¹ source to be clearer [11].

Density

Density testing aims to determine the physical properties of a composite which will later affect the mechanical properties of Composites. Besides density testing can also see whether the product weight is directly proportional to the volume of material used. The results of density testing on composites can be seen in Figure-6.



Figure-6. Density test.

Figure-6, shows that the higher the temperature of the press, the greater the value of the obtained density. The highest composite density value is at the quenching temperature of 105° C which is equal to 1.487 gram/ml,

while the lowest density value is at the temperature of Hotpress of 30°C which is equal to 1.308 gram/ml. From Figure-6, it can be seen that the composite density is

directly proportional to the increase in the temperature of the press, where the composite density is always increasing.

Analysis of Water Absorption

The purpose of the water absorption test is to find out how much water is absorbed by the composite. Water absorption for polyester composite filled with chicken eggshells with the highest percentage of water absorption and the least percentage of water absorption seen in Figure-7.



Figure-7. Analysis of water absorption.

From Figure-7, above, it can be seen that with the increase in temperature of the hot press, the absorption of water in the composite will decrease further. From the picture, it can be seen that the highest water absorption at the quake temperature is 30°C, which is 0.50%, while the absorption of the lowest water is at the quenching temperature of 105°C, which is 0.286%. According to Deng, *et al.* (2010) who made polypropylene composites with fillers by varying the temperature of the hot press (20°C, 45°C, and 65°C); it was found that the increase in temperature of the hot press caused a decrease in water absorption in the composite [12].

Analysis of Tensile Strength

The purpose of tensile strength testing is to find out how much force is needed to pull the material apart. The greater the tensile strength of material means the greater the force needed to attract the material. The results of testing the tensile strength of composite can be seen in Figure-8:



Figure-8. Analysis of tensile strength.

Figure-8 shows the relationship of the temperature of the compression to the composite tensile strength where the highest composite tensile strength is at the temperature of the hot press 60°C of 35.78 MPa, while the value of the lowest tensile strength is at the temperature of hot press 30°C of 29.13 MPa. This explains that the high temperature of the hot press can increase the tensile strength of the composite until it reaches its optimum point, before experiencing plunging. This is caused by the increase in temperature at the time of composite printing can increase the interface bond between the matrix and filler, so that the composite density increases and reduces the voids in the composite, this will increase the mechanical strength of the composite. But when the pressing temperature is too high, the tensile strength will decrease. That is because the interfacial bond between the matrix and filler decreases. If the interface bond decreases, there will be a reduction in voltage which can be transferred from the matrix to the filler. This is supported by Elahi, et al. (2014) who tested the mechanical strength of glass fibers with polyester matrices and found that by varying the temperature of the hot press (600°C, 900°C, 1200°C, 1500°C), it was found that at 900°C the tensile strength reached a point optimum before going down at 1200°C and 1500°C [3].

Analysis of Elongation at Break

Analysis for elongation at break aims to determine whether the material can undergo deformation or elongation when given a load. The results of testing elongation at break can be seen in Figure-9:



Figure-9. Analysis of elongation at break.



The picture above shows the relationship of temperature of the press to elongation at break composite where the highest composite elongation at break value is at temperature of hot press 60 °C is 0.0355, while the lowest elongation at break is at temperature of hot press 30 °C is 0.0301. This explains that the temperature of the hot press can increase the elongation value when breaking up to the optimum point, before decreasing. In a study conducted by Nasution, *et al* (2018) which varied the temperature of the forklift (120°C, 130°C, 150°C and 170°C) on Styrofoam composites with wood powder fillers, it was found that the elongation value at the optimum reached 130°C and then decreased at temperatures of 150°C and 170°C [13].

Analysis of Impact Strength

Impact Strength is an indicator of whether a material is strong or brittle. Impact strength shows how much energy is produced to destroy material through impact on a surface. The results of the impact strength testing on composites can be seen in the following Figure-10.



Figure-10. Analysis of Impact Strength.

From the picture above it can be seen the highest composite impact strength at the temperature of hot press 60°C which is equal to 8.99 J / m^2 , while the value of impact strength (Impact Strength) is the lowest temperature of hot press 30°C at 7.54 J / m². This explains that the temperature of the hot press can increase the value of composite impact strength until it reaches its optimum point, before experiencing a decline. The pressing temperature is also needed to combine the bond between the matrix and the filler to make it more dense and fused. The pressing temperature significantly affects the impact strength to withstand sudden loads. In a study conducted by Nasution et al. (2018) which varied the temperature of the hot press (120°C, 130°C, 150°C and 170°C) on Styrofoam composites with wood powder fillers, it was found that the impact strength increased to the optimum point (130°C), then decreased the impact strength value [13].

CONCLUSIONS

The results obtained egg shells containing elements C, O, and Ca, where element C composed 46.01% of the mass of chicken eggshells, element O

compiled 35.66% of the mass of chicken eggshell and Ca compose 18.33% of chicken eggshell powder. The best composite at hot press temperature of 60°C, Tensile strength properties of 35.78 MPa, xandImpac strength of 8.99 J/m^2 .

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