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THE MECHANICAL PROPERTIES OF A WATER HYACINTH/RICE HUSK POWDERS COMPOSITE FOR TISSUE ENGINEERING APPLICATIONS

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Abstract*. In this study, composites made from water hyacinth powder (WPH) and rice husk powder (RH) were created using the hot press method, and the composites were characterized to determine their suitability for biomedical applications such as tissue engineering. The mixing ratio of WPH/RH was investigated. Fourier transmission infrared spectroscopy (FTIR) revealed the presence of chemical bonds in the composites under investigation. Tensile tests were used to investigate the mechanical properties of the composite, which revealed that adding water WPH to the rice husk composite reduced the composite's strength. A composite with a 5% WPH content had the highest tensile strength of 32.72 MPa. Meanwhile, the mechanical strength of the other composites studied ranged from 25,537 MPa to 29.43 MPa. However, the elastic modulus of the composite increased with the addition of WPH. The SEM image shows that the powder distribution is less even, the interface between WPH-RH and polyester is quite tight, and the composite contains a number of voids. Characterization of the developed composite demonstrates that the WPH/RH addition ratio can be adjusted to achieve the desired composite properties for tissue engineering and cartilage regeneration applications.*
Keywords: *FTIR; mechanical properties; rice husk; tissue engineering; water hyacinth powder.*

1. Introduction

Polymer composites have been developed for use as soft tissue to replace tissue and renew decellularized organs. Tissue engineering creates tissue-like structures by using biocompatible materials, biochemical properties, physical properties compatible with living cells, and a combination of these factors [1, 2]. Several natural and synthetic polymers have been used to create biocompatible and biodegradable polymer constructs. Collagen, chitosan, gelatin, silk fibroin, alginate, cellulose, and starch have all been widely used in skin and bone tissue engineering. Several in vivo and in vitro strategies have been used to vascularize polymer scaffolds [3, 4]. Biomaterials in tissue regeneration strategies promote the development of new tissue by providing sufficient space (porosity) and a suitable surface for cellular attachment, migration, proliferation, and differentiation of specific cell phenotypes in the formation of new tissue [5].

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Because of their unique properties, such as low density, the ability to tailor mechanical properties, degradation kinetics, bioactivity, non-toxicity, and biodegradability, natural material composites are potential candidates for various biomedical applications. As a result, the properties of biomaterial polymers can be enhanced by incorporating fillers such as rice husk powder and water hyacinth into the main polymer, which has low bioactivity in comparison to many other biopolymers. The mechanical properties of the bio composite are influenced by the addition of rice husk and water hyacinth powder to the polymer composite [6-8]. However, for general applications on hard tissue, the amount of added rice husk powder and water hyacinth must be optimized. Several researchers have previously reported the potential use of rice husks as a composite filler. As a result, rice husk waste is more commonly used in polyolefins than bioplastic polymers. Fillers ranging from 5 to 30% rice husk powder have been used in polylactic acid (PLA) and poly butylene's adipate-Co-terephthalate (PBAT) composites for bioplastic polymers. Tensile strength, Young's modulus, and elongation at break for PBAT/rice husk with a weight ratio of 70:30 are 14.5 MPa, 54 MPa, and 820%, respectively [9].

In contrast, El- vice *et al*. [10] reported the development of Eichhornia crassipes water hyacinth fiber (ECF) and *Eichhornia crassipes* maleic fiber (MoECF) as reinforcing fillers in styrene-butadiene rubber (SBR) composites in terms of mechanical, acoustic, thermal, and morphological properties. SBR composites are made with various ECF and MoECF fillers, with phr values of 1, 2.5, 5, 10, and 20. When 5 and 20 phr of MoECF ameliorate rice husk powder were added to SBR composites, the tensile strength and elongation at break increased by 8% and 310%, respectively. They were able to maintain the tensile strength of the SBR composite during the thermal aging process by using 10 phr of MoECF ameliorate. If the Eichhornia crassipes (ECs) fiber powder content is 20% or 9.29%, the strain value in the composite is known to be high, and above 20%, the strain value can decrease to around 7.52% 7.66% [11]. Meanwhile, the maximum elastic modulus of the composite is 318.3 MPa at 10% fiber content, and this value tends to decrease at ECs levels greater than 20% because composites are elastic.

As a result, the purpose of this research is to look into the effects of adding water hyacinth powder to polyester/rice husk composites. To obtain quantitative data, the concentration of water hyacinth powder was varied. Tensile properties, elastic modulus, functional groups, crystallinity index, and morphology of facture composites were studied. The findings of this study could lead to the development of a new tissue engineering material.

2. Methods

2.1. Materials

Fresh water hyacinth plants (*Eichhornia crassipes*) (Figure 1a) were collected from the

Central Lombok area, West Nusa Tenggara, Indonesia. Meanwhile, rice husk from *Oryza sativa* plant waste was collected from the milling factory in Jonggat District, Central Lombok. Polyester resin as matrix was obtained from PT Kimia Raya, Indonesia.

Figure 1. (a) Water hyacinth plant, (b) Water hyacinth stem, and (c) rice husks.

2.2 Water hyacinth preparation

Water hyacinth stems are cleaned and cut into 5-10 mm lengths (Figure 2a) with a knife before drying in the sun for 6 hours (Figure 2b). They are ground by machine as shown in Figure 2c. The water hyacinth powder was sieved through a 200 mesh sieve (Figure 2d) and dried in an oven for one hour at 105 ^{o}C (Figure 2e) before being stored in a plastic box. Figure 2 depicts the process of creating water hyacinth powders (WPH).

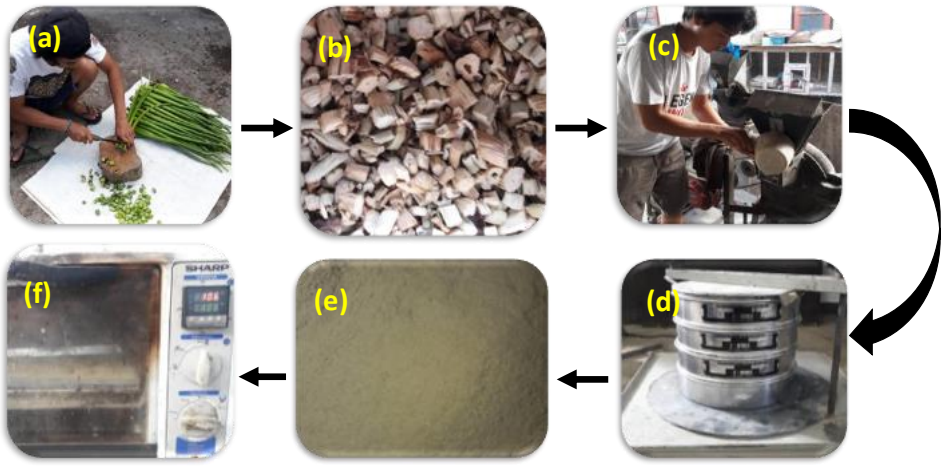

Figure 2. Water hyacinth powder preparation process.

2.3 Preparation of rice husks

A crushing machine is used to grind the chosen rice husks. They are then sifted through a sieve with a precision of 200 mesh before being baked at 105 ^{o}C for 60 minutes. They are then placed in plastic boxes for storage. Figure 3 depicts the detailed procedure for producing rice husk powder (RH).

Figure 3. Preparation of rice husk powder.

2.4 Composites preparation

A digital analytical balance is used to weigh water hyacinth powder and rice husk powder according to a predetermined composition (Table 1). The mold is made of silicon, and the specimen sizes adhere to the international standard ASTM D3039. Polyester resin is mixed with rice husk powder and water hyacinth powder first, then hardener made from methyl ethyl ketone peroxide (1% of polyester resin) is added. The dough is then poured into the prepared mold, covered, and hot pressed. The sample has been removed from the mold and is ready to be characterized.

Table 1. Composition of water hyacinth powder and rice husks in polyester composites

Sample codes	Volume fraction (%)		
	water hyacinth powder	rice husk powder	Polyester
PEA	0.5		70
PEB	1		70
PEC	2.5	7.5	70
PED	5		70
PEE	7.5		70

2.5 Characterization

2.5.1 Fourier Transform Infrared (FTIR) spectroscopy

Thermo Scientific Smart iTR instruments with horizontal attenuated total reflectance (ATR) were used to obtain FTIR spectra. The samples were set up on the ATR crystal, and the spectra were recorded in transmittance mode. Each spectrum was created using an average of 32 scans at a resolution of 4 cm^1. Using a mortar and pestle, the composite samples were ground into very fine powders. The FTIR spectra were used to determine the presence of free functional groups in the samples.

2.5.2 Tensile test

Tensile tests were performed at 25 °C on the developed rice husks biochar fiber-reinforced composites using an ASTM D3039 - Type 1 standard and a Universal Testing Machine (Tensilon) with a 20 kN load cell range and a constant crosshead speed of 5 mm/min. In each run, three samples were tested, and the average was used as the final result.

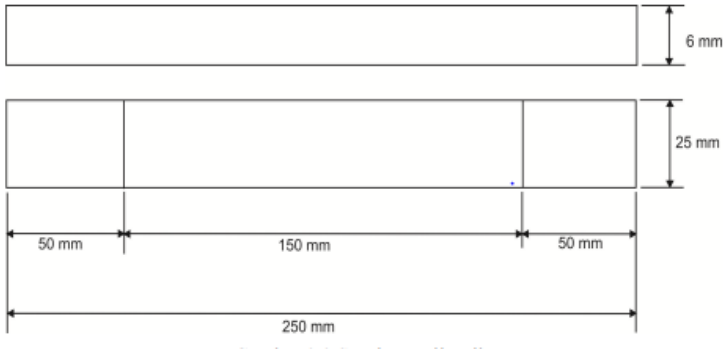

Figure 4. Schematic of ASTM D3039 tensile strength test samples.

2.5.3 XRD

On the X'pert Analytical (Model: PW3040/60), the composite crystallity index using Cu Kα radiation (λ = 0.154) resulted in an operating voltage of 40 kV and 30 mA. The XRD pattern of the sample is scanned and recorded between 5° and 80°. The crystallity index (CrI) value is then calculated using equation 1 [12].

$$CrI\,(\%) = \left[\frac{I_{002} - I_{am}}{I_{002}}\right] \times 100 \qquad (1)$$

Where, I_{002} ($2\theta = 22.6^{\circ}$) represents a crystalline region and I_{am} ($2\theta = 18^{\circ}$) represents the amorphous region of each composite sample.

2.5.4 Scanning electronic microscopy (SEM)

The optimized composite samples were examined with a Tescan Vega 3 scanning electron microscope. Samples were mounted on double-sided adhesive carbon tape, vacuum dried, and scanned at a 10 kV acceleration voltage.

3. Results and Discussion

3.1 FTIR analysis

Figure 5 depicts the FTIR spectrum of WPH composite reinforced RH/polyester composites. Figure 5 shows that the curves of all samples show the same trend. The large band in the 3000-3500 cm^{-1} range is attributed to the O-H stretching of intramolecular and intermolecular H-bonds [13]. C=O stretch is assigned to the band around 1713 cm^{-1}. O-H stretching and deformation vibrations are assigned to the band between 1645 and 1652 cm^{-1} [14]. The bands around 2911,

2917, 2849, 1417, 1423, 1266, and 917 cm^{-1} are attributed to the stretching and bending vibrations of the C-H bonds of the methyl or methylene groups, respectively [13]. C-O deformation vibrations are assigned to the bands around 1329,1087,1374, and 1088 cm^{-1}, while C-C stretching vibrations are assigned to the bands around 834 and 837 cm^{-1} [14]. The results show that as the volume fraction of WPH increases, the band intensity assigned to C=O stretching decreases [13, 15-16]. It is worth noting that the band associated with the O-H stretching vibration, which was previously located at 3266 cm^{-1} composites, has shifted to a higher wavenumber (3278 cm^{-1}).

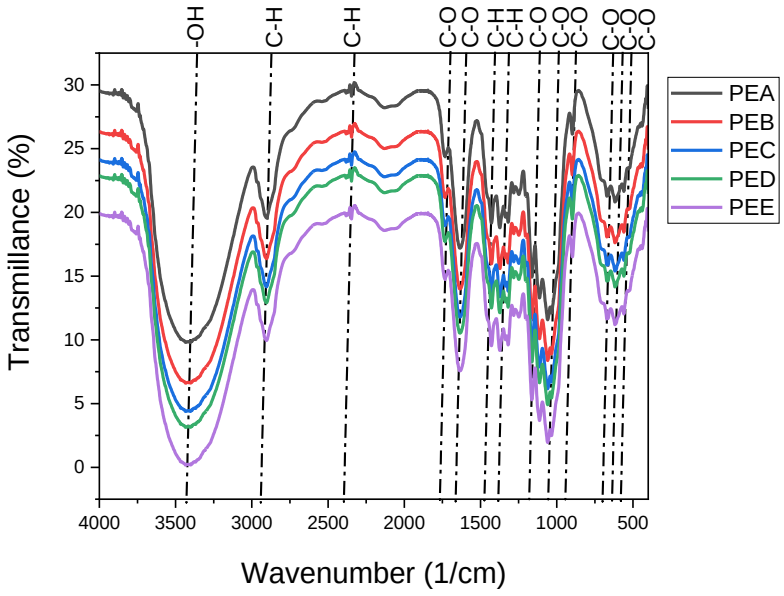

Figure 5. FTIR of WPH/RH reinforced composite polyester

3.2 Tensile analysis

Figure 6a shows that, when compared to the other samples tested, the PEA composite sample had the highest tensile strength value of 32.72 MPa. The presence of 7.5% rice husk and 0.5% WPH is thought to tighten and strengthen the interface bond between the powder and resin, resulting in a higher tensile strength. Meanwhile, for the WPH-RH volume fraction of 1%:7.5% (PEB sample), the tensile strength decreased by 23.33 MPa, which was associated with the increase in the WPH volume fraction. However, when the WPH volume fraction in the composite was 1.5%-5%, the tensile strength of the composite increased slightly, reaching 25.54 MPa-29.43 MPa. This is thought to be because the WPH in the RH/polyester composite also serves as reinforcement to withstand the load transmitted by the resin and RH. However, when the WPH content was 7%, the tensile strength decreased because a number of voids were discovered in the composite, which was caused by the manufacturing process. These results have been confirmed from SEM test images.

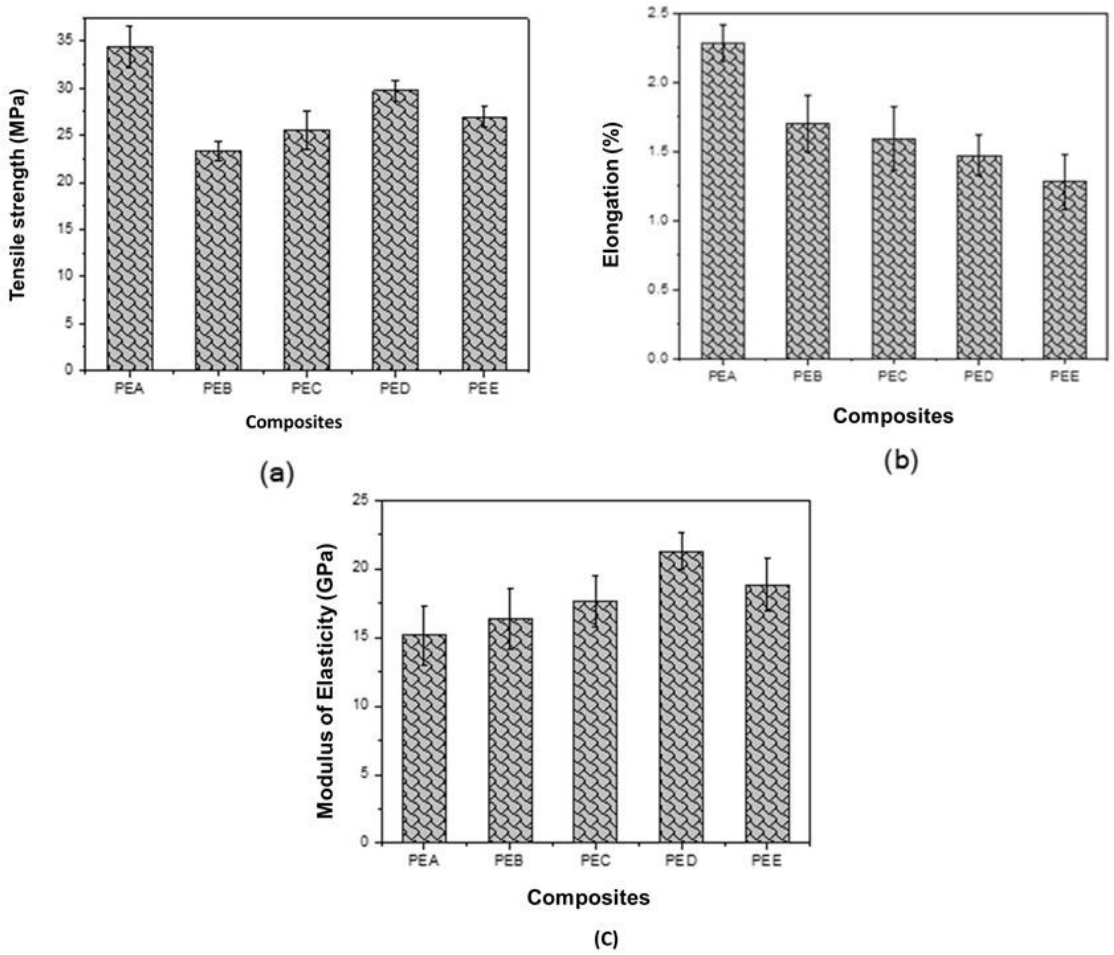

Figure 6. (a) Tensile strength, (b) Elongation, and (c) Modulus of elasticity of water hyacinth powder/rice husk reinforced composite polyester

The PEE sample has the lowest tensile strain value of 1.28%, as shown in Figure 6b. The magnitude of the composite tensile strength does not correspond to the magnitude of the tensile strain. According to Sharm *et al*. [17], the greater the value of the increase in length (L), the greater the tensile strain. The decrease in composite elongation is thought to be caused by the powder's and matrix's low interfacial strength. If the interface is weak, tensile forces applied to the composite can cause powder release from the matrix, reducing the amount of elongation that can occur. Furthermore, the presence of air bubbles, cracks, or inclusions in the composite results in a weak structure, reducing the elongation that can occur and limiting the composite's ability to provide elongation.

Figure 6c depicts the tensile elastic modulus values of all composites investigated. The lowest elastic modulus value obtained from the PED sample was 15.18 GPa, which tended to increase to 16.35 GPa to 21.28 GPa as the WPH content in the composite increased. This increase in modulus of elasticity value indicates that the presence of WPH gives the composite the ability to resist deformation and provide optimal load transfer, increasing the overall stiffness of the composite; thus, the more WPH, the stiffer the polyester/RH composite is. Furthermore, this

increase is thought to be due to the uniform distribution of powder in the resin. Load transfer between the powder and the matrix can occur efficiently when the powders are evenly distributed in the matrix, increasing the overall elastic modulus of the composite.

In contrast, the elastic modulus of the PEE samples decreased due to a weak bond between the powder and the matrix: If the bond between the fibers and the matrix is weak, the elastic modulus of the composite can decrease. When these bonds are weak, applying force to the composite can cause fiber detachment from the matrix, resulting in reduced load transfer and a low elastic modulus [18]. This WPH/RH composite material can be used for tissue engineering and cartilage regeneration applications after considering all of the results and properties such as tensile strength and elastic modulus.

3.3 X-Ray Diffraction (XRD)

Figure 7 illustrates the crystallinity indices of the various composites investigated. The crystallites index value was discovered to be obtained at an angle of 2θ = 19.2° - 21.5°. The PEA sample had the highest crystallinity index at 59.7%, followed by the PEB, PEC, and PED samples, which had crystallinity indexes of 42.2%, 39.5%, and 34.3%, respectively. The composite crystallinity index value is high because RH has more regular and crystalline structural groups than synthetic fibers, which is also supported by WPH structural groups. RH is primarily composed of natural polymers with a regular and crystalline molecular structure, such as cellulose or keratin. This crystal structure is thought to give natural fibers strength, stiffness, and strong mechanical properties. Furthermore, the presence of the composite matrix influences high composite crystallinity index values because the matrix can affect the structure of RH dan WPH and reduce crystallinity. However, in the PEE sample, the crystallinity value increased by 38.7%, indicating that the PEE composite's crystal structure was more crystalline than the other samples. Sari *et al*. [19] and Zarrintaj *et al*. [20] also stated that the higher the crystallinity index peak, the higher the crystalline fraction, and the lower the intensity peak, the amorphous fraction. The developed composite has a higher crystallinity value than low-density polyethylene composites (33-34%) [21].

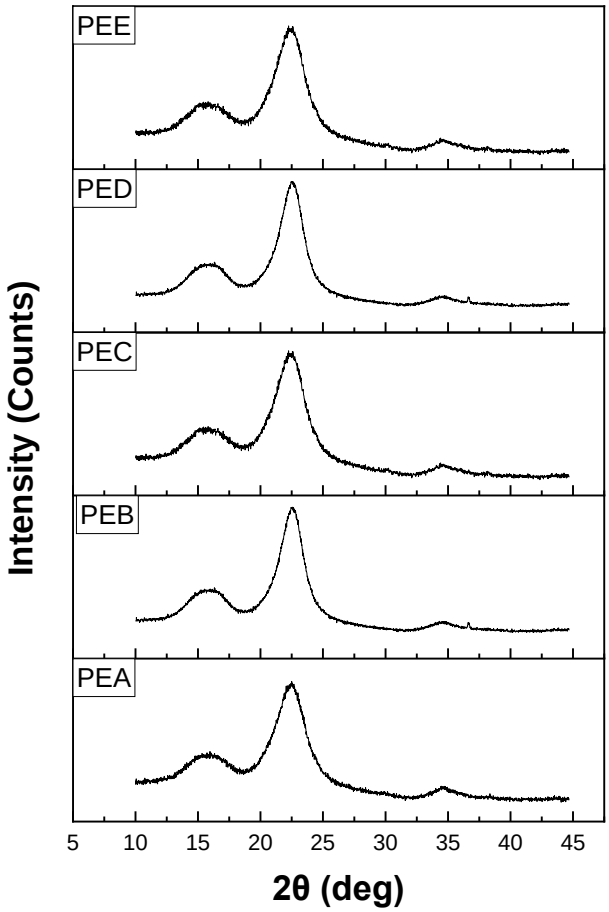

Figure 7. XRD of WPH/RH reinforced composite polyester

3.4 SEM

SEM analysis was performed on the fracture part of the composite sample to evaluate the effect of adding WPH on the morphology of the composites. Figure 8 depicts the micro-sectional morphology of PEA, PEB, PEC, PED, and PEE samples. Figure 8a shows that when compared to other samples (Figs. 8b, 8c, 8d, and 8e), a stronger density and interfacial bonding between RH-WPH-polyester and a more even distribution of RH are observed on the surface of the PEA composite, which strengthens the reason why the tensile strength and elongation of PEA composite is greater than that of other samples.

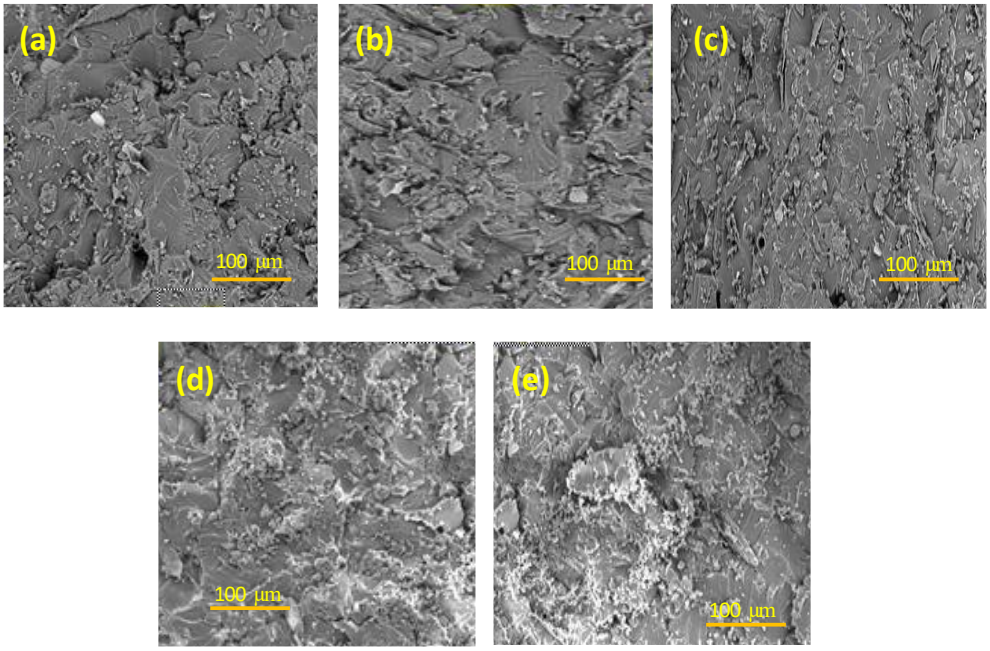

Figure 8. SEM photos of composites sample, (a) PEA, (b) PEB, (c) PEC, (d) PED dan PEE.

4. Conclusions

The hot pressing method was used to create composite samples, and their characterization was investigated. The effect of adding WPH ratio was studied using FTIR, XRD, tensile strength tests, and SEM. The addition of WPH to the polyester/RH composite reduces the tensile strength while increasing the elastic modulus. The optimal mechanical strength of a composite with a WPH content of 5% and RH of 7.5% (PEA sample) was 32.27 MPa. Meanwhile, the mechanical strength of the other composites studied ranged from 25.54 MPa to 29.43 MPa. Taking into account all of the findings and the ability to control properties such as tensile strength and elastic modulus, this WPH/RH composite material can be used for tissue engineering and cartilage regeneration applications.

Acknowledgment

The authors declare that they have no known competing interests that could have influenced the literature reported in this paper.

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