

Utilization of Rubber seed shell and Palm Oil Fronds as Composite Materials for Automotive Industry

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Abstract

Rubber seed shell (RSS) and Palm Oil Fronds (POF) are types of solid waste produced from rubber and palm oil plantation that has not been fully utilized. Meanwhile, in the automotive industry, composites have been the material of choice in some of its components. For example, composite body panels have been widely used in sports cars and passenger cars. This study aimed to utilize RSS powder and POF fiber waste as reinforcing fillers for the composite matrix. The matrix used was liquid polyester resin with the addition of catalyst as a hardener. RSS, which has been carbonized, was then activated using H_2SO_4 while POF fiber was pre-treated with 5% NaOH, then characterized both fillers by FTIR and SEM. Composites filled by RSS and POF in 4 variations were tested for mechanical properties with matrix composites without fillers as controls. FTIR testing of RSS carbonized powder found that carbonyl group consisting of tar compounds and remnants of carbon dioxide compounds that lost after activation with the H_2SO_4 solution. Meanwhile in POF fibers found that carbonyl group consisted of lignin and hemicellulose disappear after pre-treatment by 5% NaOH. SEM testing of RSS and POF fillers showed changes in surface morphology. The RSS and POF surface became coarser and porous, and the fibrils of POF fiber more obvious. The mechanical properties showed that the optimum result obtained in the composition of Matrix/POF/RSS (92.5/2.5/5).

1. Introduction

The growing area of rubber plantations in Indonesia means the higher production of rubber. Consequently, more waste is produced. One of the waste is a rubber seed shell, which is an outer rubber seed wrap after rubber skin with brown features and a hard texture [1]. Rubber seed shells are plantation waste that has not been fully utilized. Several studies that have been done before by utilizing rubber seed shell waste is to find out whether rubber seed shells could be used as an alternative source of activated carbon in the pack carburizing process [2]. Rubber seed shell can be utilized and potentially add economic value

by being processed into briquettes as an alternative fuel with the carbonization process. The shell of rubber seeds is carbonized at a temperature of 600°C for 2 hours to produce rubber seed shell (RSS) charcoal [3].

In 2014, the area of oil palm reached 10.9 million hectares, with the production of 29.3 million tons of CPO [4]. Side products of oil palm plants that are available in large quantities and have not been utilized optimally are leaf fronds, palm sludge, and oil palm cake [5]. Palm oil fronds are a type of solid waste produced throughout the year by oil palm plantations. The amount is enormous,

almost equal to the number of fresh fruit bunches (FFB) production.

Palm oil fronds (POF) and leaves are the by-products obtained when harvesting fresh fruit bunches. The number of fronds and fresh leaves that can be obtained for each hectare of oil palm reaches more than 2.3 tons of dry matter. Assuming 1 hectare consists of 30 trees, each tree can produce 22–26 fronds/year with frond weight and palm leaves 4–6 kg/frond. Even, the frond production can reach 40–50 fronds/trees/year with a weight of 4.5 kg/frond [4]. The waste has not been fully utilized as a product that has high economic value and is thrown away into mulch/fertilizer in the garden, which then become a nest for pests and insects. Therefore, more attention is needed hence the waste does not have a negative influence on the environment.

Previous research that utilized oil palm fronds as a raw material for zephyr boards, which were resistant to termite attacks was carried out by Wardani *et al.* [6]. Palm oil fronds are very well utilized as raw material for zephyr boards [7]. The physical-mechanical test results of the zephyr board of oil palm fronds are quite good because they meet the JIS A 5908–2003 Type 18 particleboard quality standards [8]. Furthermore, the quality of zephyr boards from palm frond waste can be compared with other composite boards, even with the quality of plywood made from wood with better material quality [9].

The utilization and use of composites have developed rapidly and are widespread in this country. In the automotive industry, composites have been the material of choice in several components. Currently, composite body panels have been widely used in sports cars to passenger cars. In 2000, the American automotive industry used 318 million pounds of composites [10]. There are many reasons for the growth of composite applications, however the main factor is that products fabricated from composites are more durable, lighter, corrosion-resistant, more economical, and so forth. Composite is a material formed from a combination of two or more forming materials through non-homogeneous mixing, where the mechanical properties of each of the forming materials are different. Composites consist of matrices that function as adhesives or binders and fillers to function as reinforcement for composites [11].

Carbon-fiber composites made from waste have not been widely developed in Indonesia, while available organic waste is very abundant. Therefore, this research is focused on increasing the added value of organic waste from non-valuable materials into materials that are technologically useful and has a high value. Carbonized rubber seed shell, and oil palm fibers will be used as reinforcement fillers for composite matrices for the application of raw materials in the automotive industry. The composite matrix is in the form of resin and assisted by the catalyst using a simple mixing method. The resin

used in this study is a polyester resin, with the consideration that the polyester resin is liquid, so it is relatively easy to seep and wet the surface of the reinforcement, is cheap, and has an excellent adhesive function.

2. Methodology

2.1. Equipment/Material

The equipment used in this study were beaker glass, measuring cup, blender, hot plate, crucible cup, furnace, mortar, analytic balance, sterile fainting, oven, hydraulic press machine, FTIR spectrophotometer, SEM (Scanning Electron Microscope) and composite mechanical properties testing tool. Whilst, the materials used were rubber seed shells, oil palm fronds, polyester resin, MEKPO catalyst, NaOH, and distilled water. The materials used were obtained from the Regencies in Riau Province, the Chemical Laboratory of the Muhammadiyah University of Riau and the Physical Chemistry Laboratory of the University of Riau.

2.2. Oil Palm Fiber Processing

Preparation of frond fibers raw material was intended to obtain oil palm fronds with uniform moisture content, type, and size. First, the palm fronds were soaked with distilled water at room temperature for 3 x 24 hours. The soaking function was to remove other substances found in palm wood particles. Then the fronds were flattened to become a fiber. After that, the fronds were dried out in the sunlight for 2 x 24 hours, and in the oven, until the air content reached about 5–10%. Then the fronds were copped by the desired size. After the fiber was successfully obtained, the fiber dried in the sun for about 3 hours and then soaked with NaOH solution with a concentration of 5% for 2 hours. After being soaked, the fiber was rewashed with clean water to remove the NaOH solution residues attached to the fiber. Then the fibers were dried again for 3 hours in the sunlight and able to be used as reinforcement fillers for making composite materials.

2.3. Carbonization of Rubber Seed Shells

Firstly, the rubber seed shell was soaked with distilled water at room temperature for ±24 hours. The function of immersion was to remove other substances found in eggshell particles. After that, the shell was dried out in the sunlight for 2 x 24 hours. Then, the shell was mashed into a powder then being furnace at 750°C for 3 hours to obtain carbon black. Carbon black powder from rubber seed shell (RSS) was then activated by soaking the powder into 1 M H₂SO₄ solution for 2 hours. After being soaked, the powder was rewashed with clean water to remove the H₂SO₄ solution residues attached. The powder was dried again for 3 hours in the sunlight and can be used for composite materials used as reinforcement fillers [12].

2.4. Mixing Process

Mixing was carried out in an aluminum pan while heated on an electric stove $\pm 100^\circ\text{C}$ to homogenize the resin and filler. Rubber seed shell carbon fiber, and oil palm fronds fiber and a mixture of both materials (7.5% w/w) were sprinkled with four variations of composite composition mixture of carbon fiber shell powder and oil palm fiber while stirring for 7 to 10 minutes.

Table 1. Variation of Composite Composition

No	Matrix (Resin + Catalyst) %	POF Fibers %	RSS Powder %	Composite's Name
1	100	0	0	Matrix (100)
2	92.5	0	7.5	Matrix/RSS (92.5/7.5)
3	92.5	2.5	5	Matrix/POF/RSS (92.5/2.5/5)
4	92.5	5	2.5	Matrix/POF/RSS (92.5/5/2.5)
5	92.5	7.5	0	Matrix/POF (92.5/7.5)

2.5. Hot Compaction Process (Hot Press)

The powder mixture was compacted in a mold and pressed with a manual hydraulic press while heated. The sample mold design was formed rectangular with a length of 25 cm with a width of 25 cm. The print wall thickness was 1 cm. The distance of the mold wall to the heating wall was 1 cm. The lower part of the print lid was made of an invisibility hole to place the tip of the thermocouple detection to detect the temperature that occurs in the mold. The heater design was made a maximum of 600°C . Then, the press was printed with a manual hydraulic machine with 11 US ton hydraulic press maximum pressures. The height of the base of the hydraulic press machine can be adjusted according to the needs of the stroke length [12]. RSS and POF powders were characterized using FTIR and SEM instruments, and composites were tested for mechanical properties, including tensile strength, pounding strength, and hardness test.

3. Results and Discussions

3.1. FTIR Functional Groups

Figures 1 (a) and (b) show the changes in the position of the peak frequency of RSS after activation compared to the position of the peak frequency of RSS before activation. After activation with H_2SO_4 , the OH peak in the range of $3700\text{--}4000\text{ cm}^{-1}$ increases. This is because the amount of crystalline cellulose has been washed with H_2SO_4 solution, and amorphous cellulose (active) increases. The OH peak becomes stronger, showing the increasing number of free OH groups not involved in hydrogen bonds [13]. Aromatic bracelet peaks (C=C) from tar and carbonaceous remnants at 1654.03 cm^{-1} and 1412.92 cm^{-1} are lost after activation [14].

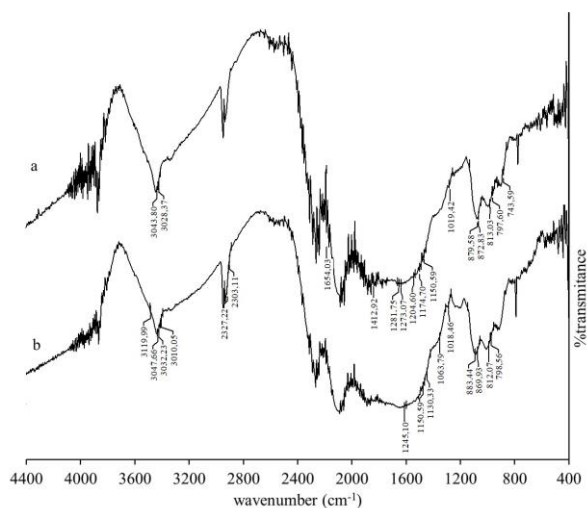


Figure 1. FTIR of Rubber seed shell Char (RSS) before (a) and after H_2SO_4 activation (b)

Activation is a physical change where the surface area of carbon rises sharply due to the removal of tar compounds and carbonaceous remnants. The absorption of activated carbon gets stronger along with the increasing concentration of the activator added. This gives a strong influence to bind tar compounds out through the micropores of activated carbon so that the surface of the activated carbon is wider, which results in more excellent absorption of activated carbon [15]. This acid activation causes the RSS surface to become rougher. This will increase the maximum interaction and adhesion with the matrix. Acid leaching opens more active OH sites on the surface of the RSS, causing the powder to become hydrophobic and reduce water absorption.

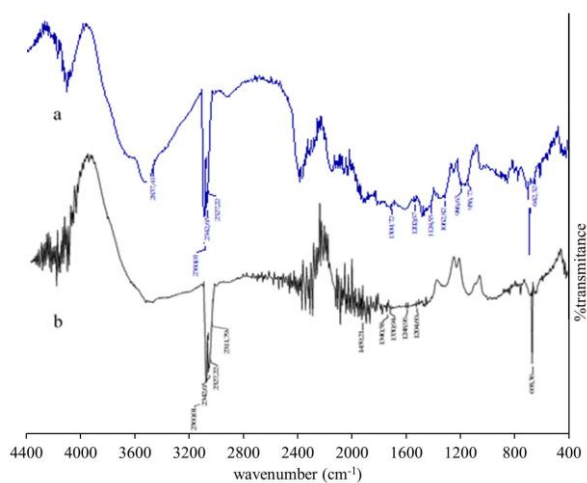


Figure 2. FTIR of Palm Oil Frond Fibers before (a) and after washing NaOH (b)

Figure 2 (a) and (b) indicate the change in the position of the POF peak frequency after washing NaOH compared to the peak frequency POF before washing. The aromatic bracelet peaks (C=C) of lignin and hemicellulose in 1459.21 cm^{-1} are lost after alkali washing [14]. The alkyl nitrile strain in $2220\text{--}2500\text{ cm}^{-1}$ is increasing after washing NaOH, and the amine strain at $1080\text{--}1360\text{ cm}^{-1}$

also increases in intensity. This increase is predicted to form nitrocellulose compounds, which are shown by the replacement of the hydroxyl (-OH) group in cellulose by groups (-NO₂) to form nitrocellulose. The best nitration process occurs when the entire -OH group on cellulose has been replaced by the -NO₂ group characterized by the absence of the -OH group in nitrocellulose [16].

Alkaline washing as a treatment is carried out on Palm Oil Frond (POF) fibers intended to remove part of the hemicellulose, wax, and lignin that lies on the surface of the frond while changing its morphology and chemical composition. Palm oil fronds that alkali-treated have a more open structure because hemicellulose has dissolved, and there is an intra and inter fibril enlargement, which increase the active cellulose number on the surface of the fiber. This treatment causes the surface of the fiber to be rougher. This increases the maximum interaction and adhesion with the matrix.

3.2. SEM Morphology

Figures 3 (a) and (b) showed the surface morphology of RSS carbon before and after activation observed with an induced electron microscope (SEM) at 500x magnification. After the activation process with the H₂SO₄ solution, the surface of the rubber shell carbon becomes coarser and more porous, caused by some layers of tar, lignin, hemicellulose, and other carbon compounds that have been dissolved in H₂SO₄ solution. Activated carbon has a porous structure so that molecules can pass it.

Activation with H₂SO₄ solution causes physical changes where the surface area of carbon increases sharply due to the removal of tar compounds and carbonaceous remnants. This activation has a strong influence to bind tar compounds out through the micropores of activated carbon so that the surface of the activated carbon becomes wider, which results in more excellent absorption of activated carbon [15].

Figures 4 (a) and (b) shows the surface morphology of POF fibers before and after washing NaOH observed with an induced electron microscope (SEM) at 5,000x magnification. Palm frond fibers before alkali washing are seen to have a smooth and flat surface with the presence of compounds such as lignin, hemicellulose, and wax (wax) on its surface [17].

After washing alkali (5% NaOH), the surface of the palm fronds shows a rough and uneven surface caused by a portion of the lignin and hemicellulose layers that have been dissolved in the alkaline solution. Moreover, palm frond fractionation occurs as a result of removing lignin and hemicellulose layers that bind to microfibril structures. Fibrillation increases the aspect ratio and specific surface area of palm fronds for interaction with the polymer matrix. Thus, alkaline washing is expected to increase the composite mechanical interaction between the POF-Matrix as well as the POF-RSS Matrix [18].

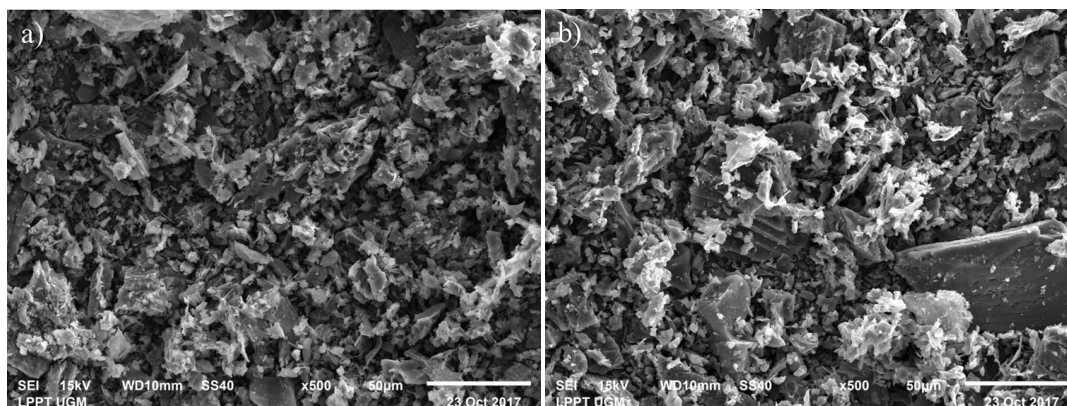


Figure 3. SEM of carbonized RSS powder, before (a) and after activation with H₂SO₄ (b)

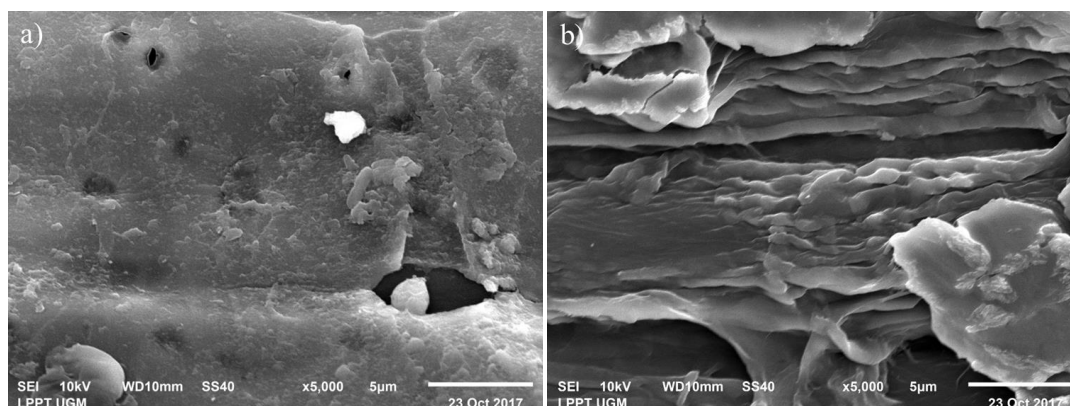


Figure 4. SEM fiber POF before (a) and after washing NaOH (b)

3.3. Composite Mechanical Testing

3.3.1. Tensile Strength

The tensile strength of the four composite and matrix variations as a control can be seen in Table 2. It is found that the tensile strength of the resin/catalyst matrix with a composition of 100/1 is 297 MPa. After the matrix is added with rubber shell filler (RSS) and palm fronds (POF) with Matrix/POF/RSS composition (92.5/5/2.5), the tensile strength value decreases to 186.9 MPa. This is caused by a lack of harmony between the hydrophobic matrix system and the RSS and POF fillers, which are initially hydrophilic, which in this variation, the composition of RSS is less than the POF composition in the composite.



Figure 5. Composite sample of each variation

In the variation of matrix/POF/RSS composites with a composition of 92.5/2.5/5, where the composition of RSS is more than the composition of POF, the tensile strength value has increased even though not significantly, that is from 186.9 MPa to 193.2 MPa. This is because the RSS composition of carbon powder more fully transfers compatibility to the composite and increases the ratio between fillers and matrices.

Table 2. Composite Mechanical Properties Test Results

Test Parameter	Composite Composition				
	Matrix (100)	Matrix/POF/RSS (92.5/5/2.5)	Matrix/POF/RSS (92.5/2.5/5)	Matrix/RSS (92.5/7.5)	Matrix/POF (92.5/7.5)
Tensile Strength (MPa)	297	186.9	193.2	164.2	136.2
Impact Strength (kJ/m ²)	139.6	139.5	139.45	138.6	139.45
Hardness (Hv)	424.67	658.67	626	530.3	693.67

In the variation of the matrix/RSS composition (92.5/7.5), the tensile strength value is found to be 164.2 MPa. The much lower value of this filler mixture is due to the agglomeration of the rubber shell carbon powders, which weakened the interaction of the filler-matrix surface. Variations in the composition of the Matrix/POF of (92.5/7.5) are found to have a tensile strength of 136.2 MPa. The value of the tensile strength of the composite with palm frond fillers washed with NaOH is not enough to increase the tensile strength of the resulting composite. Surface modification of fibers with NaOH solution is not influential enough to increase the strength of the material [19]. This decrease is also caused by

uneven fiber size, which causes the distribution of fillers in the composite to be uneven. Moreover, the filler composition in the composite, which is not maximal enough, also reduces the value of the tensile strength. Alkali washing parameters include concentration and immersion time also affect the filler in the composite, which weakened the interaction between fillers and so forth on will reduce the value of composite tensile strength.

3.3.2. Impact Strength

Table 2 shows the comparison of the value of the strength of the matrix and the four variations of composite composition with the addition of fillers of RSS and POF. The value of the impact strength of the blank matrix without the addition of fillers is 139.6 kJ/m². The addition of fillers to the matrix system is found to reduce the value of the material impact strength because of noise disturbance on the homogeneity of interactions between the phases and the arrangement of the matrix chain, although not significantly. The impact strength value of the matrix/POF/RSS composite at composition 92.5/5/2.5 is 139.5 kJ/m². Composite Matrix/POF/RSS in composition 92.5/2.5/5, the value of impact strength is 139.45 kJ/m². The impact strength value of the matrix/RSS composite with a composition of 92.5/7.5 is found to decrease to 138.6 kJ/m² due to the weak interaction between fillers and matrices, which caused the uneven displacement of the load which caused a decrease in composite impact strength. The impact strength of the matrix/POF composite with a composition of 92.5/7.5 is 139.45 kJ/m². POF washing with NaOH solution removes surface impurities and provides better interaction between POF face and matrix, which in turn increases the impact strength of composite results, although not too significant.

3.3.3. Hardness

The hardness value of the four composite and matrix variations as a control can be seen in Table 2. The value of the blank matrix hardness without the addition of fillers is 424.67 Hv. The hardness value of the matrix/POF/RSS composite at composition 92.5/5/2.5 is 658.67 Hv. The addition of fillers to the matrix system is found to increase the value of material hardness caused by increase matrix-fiber adhesion and better bond formation between filler-matrix. With the formation of better bonds, the composite becomes harder and stiffer.

Composite Matrix/POF/RSS in composition 92.5/2.5/5, the hardness value obtained is 626 Hv. The hardness value of the matrix/RSS composite with a composition of 92.5/7.5 is found to decrease into 530.3 Hv. This decrease is due to phase separation and agglomeration of RSS particles, which may occur, resulting in uneven distribution of filler particles in composite systems. The filler agglomeration gives the results of a non-dense and soft sample, which further decreases composite hardness. Composite hardness is

influenced by filler composition, filler sprinkling, and interaction between fillers. The value of composite hardness of the Matrix/POF with a composition of 92.5/7.5 is 693.67 Hv, which is much higher compared to other composites [20].

The addition of bamboo fiber in natural rubber increases the hardness of the material. The addition of the filler composition causes the composite to become stiffer and harder. The scattering of uneven fillers in the matrix causes agglomeration of fillers, which in turn produces a non-dense and soft sample. Activation of rubber shell carbon and alkali washing on the palm fronds causes better bond formation between filler-matrix. With the formation of better bonds, the composite will become harder and stiffer [21].

4. Conclusion

The overall results of this study can be summarized as follows: Testing of FTIR functional groups in RSS found carbonyl groups consisting of tar compounds and compounds of carbon dioxide remnants lost after activation with H_2SO_4 solution. In POF, it is found that carbonyl groups consisting of lignin and hemicellulose also disappear after alkali washing with NaOH. SEM testing of RSS fillers and palm fronds (POF) has changed in the surface morphology. The RSS surface becomes coarser and porous as well as the POF surface, moreover the fibrils of POF fiber are more noticeable. Composite mechanical properties of the four composition variations showed the composition of composites with a combination of RSS and POF fillers, giving the optimum mechanical properties.

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