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The Effects of Natural Fiber Orientations on the Mechanical Properties of Brake Composites

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ABSTRACT

This research aims to study the usage of natural fibers as composite reinforcement and their effects on mechanical properties. Because asbestos fibers have negative impacts on the environment and public health, in the manufacture of brake composites they were replaced with natural fibers. Compared to synthetic fibers, natural fibers are cheaper, higher manufacturability, and a better contributor to mechanical properties. Pineapple leaf fibers were prepared by a water retting process, and coconut and areca fibers were prepared by a separation process after drying. The samples were prepared in the different volume fraction of fibers (2 vol% - 10 vol%) and fiber orientations adjusted as random, perpendicular, and angle of 45°. They were fabricated by hot isostatic pressing method, tested using Rockwell hardness tester, wear testing machine, and universal testing machine, and characterized using scanning electron microscopy (SEM) and energydispersive X-ray spectroscopy (EDX). The specimen in a fiber orientation of 45° performs the strongest tensile strength. The highest hardness (68 HRN) is found in the specimen with a volume fraction of 2 vol%, the lowest wear $(1,64 \times 10^{-4} \text{ mm}^2/\text{kg})$ is obtained in the sample (10 vol%), and the highest tensile strength (14,5 MPa) is resulted by a specimen in angle orientation 45°.

摘要

本研究旨在探讨天然纤维作为复合增强材料的应用及其对力学性能的影响.由于石棉纤维对环境和公众健康有负面影响,在制造刹车复合材料时, 石棉纤维被天然纤维取代.与合成纤维相比,天然纤维更便宜,可制造性更高,对机械性能的贡献更大.以菠萝叶纤维为原料,采用水脱胶法制备了椰子和槟榔纤维.在不同纤维体积分数(2 vol%-10 vol%)下制备样品,并将 纤维取向调整为随机、垂直和45°角.采用热等静压法制备,用洛氏硬度 计、磨损试验机和万能试验机进行试验,并用扫描电子显微镜(SEM)和 能谱仪(EDX)对其进行了表征.纤维取向为45°的试样具有最强的抗拉强 度.体积分数为2vol%的试样硬度最高(68hrn),磨损量最小(1,64×10-4mm2/kg),45°角试样的拉伸强度最高(14,5mpa).

Introduction

The natural fibers are originated from plant, animal, and mineral. The plants that can be taken its fibers include cotton, bamboo fiber, pineapple leaf fiber, banana frond fiber, coconut fiber, areca nut, and hemp fiber (Gokulkumar et al. 2019). The usage of organic fibers as reinforcement materials in eco-friendly composites, especially in the manufacture of brake composite, is expanding. Natural fibers are more desirable than synthetic fibers because they are low cost, nonabrasive during processing, recyclable, and biodegradable (George, Klompen, and Peijs 2001; George, Bhagawan, and Thomas 1998). The main constituent of natural fibers is cellulose (Doroudgarian, Pupure, and Joffe 2015). Each natural fiber consists of different constituents such as cellulose, lignin, pectin, and other

KEYWORDS

Betel nut fiber; brake material; coconut fiber; composite; pineapple leaf fiber 关键词: 槟榔纤维; 制动材 料; 椰子纤维; 混合成的; 萝 叶纤维

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materials so that they have different physical and chemical characteristics (Hazarika et al. 2015). The chemical composition of pineapple leaf fiber consists of alpha-cellulose (70.98 wt%), lignin (4.9 wt%), hemicellulose (15.34 wt%), fats and waxes (0.96 wt%), ash (0.95 wt%), and others (3.87 wt%) (Hazarika et al. 2017). Main constitution of betel nut (*areca catechu*) fiber includes alpha-cellulose (53.20%), hemicellulose (32.98%), lignin (7.20%), fat and wax (0.64%), ash (1.05%), and other materials (3.12%) (Hassan et al. 2010). In other studies, coconut fiber is used to strengthen composite materials. The coconut fiber contains cellulose (27.95 wt%), lignin (41.03 wt%), hemicellulose (19.78 wt%), attractive (8.60 wt%), and ash (1.30 wt%) (Leão et al. 2015). During its development, natural fibers have become an important component in the application of the textile, paper, packaging, and building materials industries (Ashori 2006). Natural fiber-reinforced polymer composites have great potential to replace materials derived from nonrenewable resources (Neto et al. 2015). Potential natural fibers to be developed include betel nut skin fibers, pineapple leaf fibers, and coconut fibers.

In recent years, natural fibers have become attractive to researchers, engineers, and scientists as alternative reinforcing materials in the manufacture of polymer composites that are low cost and can produce good mechanical properties, resistant to wear, and recyclable (Ashok, Srinivasan, and Basavaraj 2018). Many studies have been carried out in the development of natural cellulose fiberreinforced bio-composites. A total of 8 wt% Allium sativum fiber is used by Vineeth and Senthil (2020) to reinforce composite brake linings. Physical and mechanical properties of pineapple leaf fibers include density (1.526 g/cm³), elongation at break (3 %), and tensile strength (413-1,627 MN/m²) (Devi, Bhagawan, and Thomas 2010; George, Sreekala, and Thomas 2001; Laftah and Rahman 2016). The physical properties of a single fiber of pineapple leaves include length (3-8 mm), diameter (7-18 µm), and fineness (2.5-4 tex) (Laftah and Rahman 2016). Physical properties of a pineapple leaf fiber bundle include length (10-90 mm), fineness (2.5-5.5 mm), tenacity (20-30 cN/tex), elongation (2.4-3.4%), initial modulus (570-700 cN/tex) and density (1.543 g/cm³) (Laftah and Rahman 2016). Senthilkumar et al. (2019) use pineapple leaf fibers in polymer composites and can increase the tensile strength of 23% - 25% at a fraction of 35 vol% and also increase the modulus of elasticity at additional fiber weights of 25 wt% - 45 wt%. Ashok, Srinivasan, and Basavaraj (2018) stated that the use of betel nut skin fiber along the 10 mm as much as 30 vol% fiber can increase the tensile strength of 17 MPa – 33 MPa. Areca nuts can also be used as antioxidants and anti-bacterial agents (Shen et al. 2017). The areca catechu's average moisture, diameter, density, and tensile strength were 11.70, 0.29 mm, 0.71 g/cc, and 68 MPa, respectively (Heckadka et al. 2020). Hwang et al. (2016) used coconut fiber in the composite making and can increase crack deflection, toughness index, and flexural strength. Coconut fibers have physical properties: diameter (72 µm), linear density (58 tex), and mechanical properties: tenancy (67 cN/tex), breaking elongation (33%), specific work of rupture (42 mJ/tex-m), and initial modulus (54 N/tex) (Basu, Mishra, and Samanta 2018). The use of natural fibers as polymer matrix composite reinforcement is a good momentum because it can produce composite products with good and sustainable mechanical properties (Binoj et al. 2016). Betel nut fiber, pineapple leaf fiber, and coconut fiber can be used as alternatives to reinforcing brake pads because of its abundant availability and the price is quite cheap (Silva and Walters 2006), natural fibers are also not easily abrased and are safer than synthetic fibers (Lopattananon et al. 2006).

The mechanical behaviors of composites are highly dependent on the properties of the matrix and fibers and the interactions between the matrix and fibers (Sreekala et al. 2002). There are several main problems to be solved in this study, including optimization of the content of natural fibers in composite materials, assessing the effects of fiber types, and short fiber orientations on the mechanical properties of composite brake pads. This research was conducted by referring to the research roadmap for biocomposite brake development as shown in Figure 1. The organic materials were extracted and the wastes were recycled and processed into composite brake friction materials. The effects of orientation of several natural fibers on friction and wear of composite brake linings have been reported by several previous researchers (Alshammari et al. 2018; Milosevic, Valášek, and Ruggiero 2020). This study specifically examines the effects of natural fibers orientations on the tensile strength of composite brake linings. Here, three types of natural fibers were used separately and the results are compared.



Figure 1. Research roadmap of eco-brake composite development.

Materials and method

Fiber extraction

Fibers were taken from local plants in Semarang, Indonesia, namely pineapple (Ananas Comosus (L) Merr) thin-leaf Queen type, areca catechu, and coconut (Cocos nucifera). In the separation of pineapple leaf fibers, the method used was water retting and scraping. The sodium hydroxide (NaOH, 50%) solution was supplied from Sigma Aldrich and then it's diluted to 5%. The water retting process was done by inserting pineapple leaves into a 5% NaOH solution for 10 h. The use of a base solution is to accelerate the process of softening cellulose. Pineapple leaves that have undergone a water retting process were then scraped using a plate or knife to remove substances that were still attached to the fiber so that the pineapple leaf fibers became strands of fibers. The fibers that have been formed were then washed and dried.

The making of coconut fibers was done by drying coconut skin for 2 days – 7 days. Dry coconut skin soaked in clean water for 5 mins, this was done to facilitate the separation of fibers. The process of making betel nut fibers was almost the same as making coconut fibers because the



Figure 2. Fiber separation results: (a) Areca nut skin fiber, (b) Pineapple leaf fiber, and (c) Coconut fiber.

texture of the two fibers has similarities, the only difference in the drying time of betel nut which was 2 days – 4 days. The produced natural fibers were shown in Figure 2.

Composite preparation

The composite brake lining was fabricated by a hot isostatic pressing method. The binding material (Araldite 506 epoxy resin, Shen Zhen Nanganghengshun Trading Co. Ltd.) as a matrix, natural fiber, and filler materials including metal powder, glass powder, and coconut shell powder were mixed until homogeneous (Sutikno, Marwoto, and Rustad 2010). Epoxy resin was chosen as a binder in brake lining composites because it has a higher strength than other polymers (Pupure, Doroudgarian, and Joffe 2014). Metal powders such as copper, brass, and bronze powders were taken from a local supplier (Pudak Scientific, Bandung, Indonesia). Glass powders (200 mesh) were made of glass wastes taken from a local supplier (Jakarta, Indonesia) and coconut shell carbon powders (Borneo activated carbon, 5% moisture) were also taken from a local supplier (Kalimantan, Indonesia). The fibers were cut to 2 mm each. The volume fraction of the fibers in the mixture of brake ingredients was made of 2 vol% up to 10 vol%. The orientation directions of the fibers were manually adjusted using the help of masks, where the masks were made based on the designs of mutually perpendicular orientation patterns and forming 45° angles to each other. Specimens with random fiber orientation were made without the aid of a mask. Each specimen was made of one type of natural fibers and does not contain synthetic fibers. The use of masks was also to facilitate the alignment between the fiber layers (Pupure, Doroudgarian, and Joffe 2014). The mixing results were then pressed using a press machine with a load of 50 kN at a temperature of 160°C for 3 h. Tensile test specimens were made by varying the orientations of the fibers namely random orientation, perpendicular orientation (George et al. 1998), and orientation of 45° according to Figure 3. The two natural fiber orientation patterns, perpendicular, and 45° angle were set manually using masks, while the random orientation was made without a mask.

Characterizations of brake composite microstructures

Observation of the microstructure of the friction surface, fiber dimension, and composition of the specimen was performed using SEM-EDX Phenom Pro-X. The specimens for the SEM study were polished and coated by Gold-Palladium alloy following standard 94 procedure. Brake composite consists of multi ingredients and in this study, the content of each ingredient was arranged based on volume fraction. Observation of the microstructures of brake lining specimens using SEM was carried out at several magnifications (400–3000x) according to the need to obtain a clear image. EDX was used to determine the elemental composition of the friction material.



Figure 3. Tensile test specimens with orientations: (a) random, (b) perpendicular, and (c) 45° angle.

Mechanical properties testing of brake composite specimens

The specimens that have been formed were tested for their mechanical properties in the form of hardness tests using Rockwell Hardness (XHR-150), wear using Ogoshi High-Speed Universal Wear Testing Machine Type OAT-U, and strength using Tensile Testing Machine Servo pulse with a load of 20 kN.

Hardness testing aims to determine the measured value of hardness in the brake pad composite specimens. The hardness testing method used was the Rockwell B hardness test ASTM E10-01. In the Rockwell B (HRB) hardness test, a load of 98.07 N was initially applied to the indentor. After the initial load was applied, it was followed by giving an additional burden to the identifier until it reached a total load of 980.7 N (Ma, Low, and Song 2002). Within a certain period, after the maximum load was applied, the load is released until it returns to the initial load of 98.07 N.

Wear testing was carried out to determine the level of wear of a specimen due to continuous mechanical movement using SNI 09–0143-1987. This test referred to the Reiken Ogoshi method (revolving disc), with the width of the disc plate used, which was 3 mm, the radius of the plate 14 mm wear, the compressive force of wear 6.36, the wear distance of 200 m, and the time of testing 60 s. The width of the wear on the test specimen was measured using a microscope at magnification 100X.

A tensile strength test aimed to determine the mechanical properties of the brake pad specimens against tensile loads including tensile strength, flexibility, and length increase in response to external forces based on ASTM E8. Tensile testing was done by loading the specimen in the direction of the test machine or longitudinally. In this study, the variation in the orientation of the fiber toward the direction of the load force was used as an independent variable and, the tensile strength and elasticity as the dependent variable.

Results and discussion

Hardness numbers of brake composites

The results of Rockwell hardness testing of composite brake lining reinforced betel nut skin fiber, pineapple leaf fiber, and coconut fibers at different volume fractions are shown in Figure 4. The test results show that the increasing volume of natural fiber is inversely proportional to its hardness value. The higher the fiber volume fraction causes the level of hardness to decrease, conversely the number of fiber volume fractions decreases the greater the hardness of the specimen. Brake lining with coconut fiber ingredient contributes to greater hardness than the use of other fibers. The use of pineapple leaf



Figure 4. Rockwell hardness numbers for natural fibers-reinforced brake composite at different volume fractions of fibers.

fibers in the specimen gives the smallest hardness value, while the use of betel nut skin fibers produces a hardness value between the two. The test results showed that the specimens containing 2 vol% coconut fibers had the highest hardness value (68 HRB), while the lowest hardness value, 42 HRB, was found at the specimens using pineapple leaf fiber ingredient. The difference in the hardness number is caused by plastic and elastic deformation during compaction. Specimens with smaller natural fiber content tend to produce greater densities so that the transfer of forces between the particles in them becomes large (Maleque et al. 2012). When the fiber volume fraction increased, the glass powder volume fraction lowered.

Wear of brake composites

The results of wear testing of natural fiber-reinforced composite brake pads at different volume fractions are shown in Figure 5. The results of wear tests in Figure 5 show that an increase in wear value in the specimen is in linear relation with the increase in natural fiber volume fraction. Specimens with higher fiber fraction have larger wear values while specimens with lower fiber fraction wear values tend to be smaller. The highest level of wear observed in the specimens of brake lining with a fiber volume fraction of 10 vol% and the lowest wear is owned by brake lining with a fiber fraction of 2 vol%. This level of wear is inversely proportional to the level of hardness. The wear test curve shows the tendency of the wear curve more toward the positive while the hardness value forms the curve in the negative direction. The curves of both the hardness and wear test show that the smaller the level of hardness of the specimen, the greater the specimen wear. This increased wear value is due to a large amount of material lost due to a lack of bonding between the constituent particles during the friction process. The use of pure fibers without treatment allows adhesion forces between the constituent particles to be blocked because there are still many layers of lignin that cover the surface of the fiber (Kumar 2010). When the volume of fiber continues to increase and friction occurs in the specimen, the material that previously functioned as a reinforcement material is not optimal so that the specimen will be easily abrased.

Tensile strength of brake composites

The results of the tensile test of each specimen of brake pads reinforced separately by areca nut fiber, pineapple leaf fibers, and coconut fibers are shown in Figure 6. Among the three natural fibers,



Figure 5. Wear rates of specimens at different volume fractions of fibers for each pineapple leaf, areca nut, and coconut fiber.



Figure 6. Tensile strengths (a) and elasticity modulus (b) of brake composites against fiber orientations for each coconut fiber, pineapple fiber, and areca nut fiber.

coconut fibers contribute the most to the strength of the friction material compared to pineapple leaf fibers and areca nut fibers, this is presumably due to the different crystallinities of celluloses for each natural fiber (Leão et al. 2015).

The tensile strengths and elasticity modulus of specimens were affected strongly by the fiber orientations. The highest tensile strength of specimen, 14.5 MPa, was owned by specimens with fiber orientation 45°, while specimens with random orientation occupy the smallest tensile strength of 8.06 MPa. The use of coconut fibers also produces greater tensile strength, followed by betel nut skin fiber and pineapple leaf fiber. The tensile strength curves in Figure 6 show that the magnitude of the elasticity value is directly proportional to the tensile strength. In general, the increase in tensile strength and modulus of elasticity is due to the presence of natural fibers which plays a role in resisting deformation (Takamizawa et al. 2018). This change occurs when the orientation direction is varied, resulting in different tensile strength. The fibers serve to strengthen the composite structure in maintaining its shape by resisting deformation. In composites where the fibers are arranged perpendicular to each other, which plays a role in restraining the rate of deformation and maintaining the shape of the composite specimen, that is, only the fibers are parallel to the tensile direction. Composites containing longitudinally oriented fibers have a higher relaxation rate than composites containing transversally oriented fibers (George et al. 1998; George, Bhagawan, and Thomas 1998). If the mixing of materials is carried out randomly, the longer fibers in the composite structure tend to form local clusters with the same fiber orientation, while the shorter fibers behave more individually, making it more difficult to create transverse cracks. Shorter and thinner fibers have the advantages of being more flexible and easier to orientate (Pupure et al. 2018). Cracks are difficult to find in composites reinforced with short fibers (less than 5 mm). The degree of the load carried from the matrix to the fiber is a function of (i) the length of the fiber, which is called the length or critical aspect of the fiber ratio, (ii) fiber orientation and direction relative to each other. If fiber orientation and direction are not in line with the applied stress, bonded failure happens easily. Unidirectional fiber composites tend to transmit better external stresses, that is why hand-laid fiber composites perform better mechanically (Shesan et al. 2019).

In this study the matrix used is a type of epoxy resin, it is known that thermoplastic resins or polymers have a semicrystalline crystal structure consisting of neatly arranged polymer chains and amorphous polymer chains. When the specimen receives traction force (stress), the polymer chain will be in line with the direction of the traction load. The presence of reinforcing fibers causes the resin (matrix) to be filled with reinforcing particles so that the deformation that occurs in amorphous polymers can be minimized by reinforcing fibers. Tensile strength will increase when the stress that occurs can be transferred to the fiber, otherwise, the tensile strength will decrease if the energy transfer cannot be continued to the fiber to the maximum so that the tensile load tends to be retained by the resin. Besides it, the increased tensile strength and modulus of elasticity are also due to the better bond between the reinforcement and the matrix. Bonding between particles will cause strong adhesion forces and improve mechanical properties (Hristov et al. 2004). In general, fibers that increase composite hardness will increase

the modulus of elasticity. This is because hardness is a function of fiber volume and modulus (Srinivasan et al. 2011).

Microstructures of brake composites

Observation of brake composite microstructures aims to determine the surface, cross-sectional structure, the particle size of the specimen constituent, and dimensions of natural fibers. The SEM images of natural fiber-reinforced composite brake linings are observed in magnifications (400–3000x) and the clear images of magnifications 3000x and 410x are shown in Figure 7.

SEM images show the existence of a micro-particle-size constituent of brake lining material and the dimensions of natural fibers used. Microstructures that have bright colors are metals that have high thermal conductivity and high hardness. In the microstructures of the brake lining specimens, the carbon element looks black while the metal elements such as Cu, Zn, Al, Mo, and Fe look brighter. The light-dark pattern formed from each phase indicates the difference in the conductivity of the material (Sutikno, Marwoto, and Rustad 2010).

Coconut fibers have the largest average width, 402.55 um, as seen at the SEM image in magnification of 3000x. The fibers of pineapple leaves and areca nuts have average widths of 4.12 um and 3.52 um, respectively. In Figure 6, the porosities of the composites are found and it's suspected that there is an interaction between the fibers and the matrix. The microstructures of the coconut fiber-reinforced brake composites showed fewer porosities, compared to the pineapple leaf and areca catechu fiberreinforced brake composites. The greatest porosity was found on the surface of the pineapple leaf fiber-reinforced brake composite. As the density of composite increases, the hardness of composite increases as well. Fiber porosities and micro porosities around the fibers occurred because it was triggered by composite failures. The formation of the pores can be due to the stress transfer which removes the amorphous constituents of the fibers. The adhesion between the fibers and the composite matrix is marked with a lighter color on the outer sides of the fibers which indicate the growth of new layers and small granules appear attached to the fibers. The coated surfaces are the result of the aminosilane groups coupling with the surface hydroxyl groups of the fibers (Dharmalingam et al. 2020). This can improve the mechanical properties of brake composites (Azeez et al. 2018).



Figure 7. Microstructures of specimen surfaces of brake composites reinforced by (a) Areca nut skin fibers 850X, (b) pineapple leaf fibers 850X, and (c) coconut fibers 410X.



Figure 8. EDX curves of brake linings reinforced by (a) areca nut fibers, (b) pineapple leaf fibers, and (c) coconut fibers.

	Fibers in brake composites		
Elements (%)	Areca nut fiber	Pineapple leaf fiber	Coconut fiber
С	38.7	39.8	60.0
0	34.9	36.9	33.0
К	-	0.2	2.0
Ν	23.8	21.5	-
Na	0.9	0.9	2.1
Si	0.5	0.3	-
S	1.0	-	-
Cl	0.3	0.2	2.2
Sr	-	-	0.7

Table 1. The percentages of chemical elements for each brake composites using different fibers.

Elemental chemical composition of brake composites

The EDX characterization aims to determine the chemical composition contained in the samples of natural fiber-reinforced composites. Figure 8 and Table 1 show the results of EDX curves of natural fiber-reinforced composites and the percentages of chemical elements contained.

The difference in the composition of the chemical elements in the EDX data can occur because the brake composite is composed of multiple ingredients, and different natural fibers are used. The nitrogen element (N) found in the composite reinforced with areca nut and pineapple leaf fibers, but not found in fiber-reinforced composites, is thought to have come from the metal powders used. This nitrogen is in the form of amino silane which comes from other ingredients of brake composites. This was confirmed by the discovery of other elements such as silicon (Si), potassium (K), sulfur (S), chlorine (Cl), and strontium (Sr). This EDX data shows that the presence of natural fibers in the specimen is still relatively dominant. The carbon is the largest element formed by natural fibers in plant tissue so that almost all samples are dominated by carbon elements, while the oxygen content in EDX analysis is mainly because of the presence of cellulose, and lignin. This inhomogeneous mixing causes porosities in the specimens. Table 1 proves that specimens with better mechanical properties have less carbon content than specimens with lower mechanical properties. Pineapple leaf fibers in this study contained a carbon element (39.8%) smaller than the results of the study of Najeeb et al. (2020) (53.56%).

Conclusion

Based on the results and discussion it can be concluded that the less use of the natural fiber fraction results in high hardness and decreases the level of specimen wear. The best result in this testing is found at coconut fiber-reinforced brake pad specimen with 2 vol% fraction because it

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conforms to SNI 09–0143-1987 specifications with a wear value of $5x10^{-4}$ mm²/kg – $5x10^{-3}$ mm²/kg and hardness of 68mm²/kg – 105mm²/kg. The tensile test shows the specimen with 45° orientation produces the highest tensile strength followed by the specimen with perpendicular and random orientation. Future work should focus on utilizing hybrid fibers in the development of brake pads and examining their effects on their mechanical properties and investigating interfacial adhesion phenomena.

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Disclosure statement

The authors declare no competing financial interest.

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