Effect of Grafting Nano-TiO_2 on *Sansevieria cylindrica* Fiber Properties

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ABSTRACT

Natural fibers, which are abundant, environmentally friendly, and biodegradable, are used as a replacement for synthetic fibers. The composite strength can be increased by treating the surfaces of natural fibers with suitable chemicals, which can also improve the interface interaction between fiber and matrix. Application of a coupling agent in chemical treatment is utilized to reinforce the bonding between fiber and matrix. The objective of the study is to determine the influence of silane concentration on the *Sansevieria cylindrica* fiber properties. The methods included fibers treatment using ethanol and coupling agent as dissolving and TiO_2 with concentrations of 0%, 0.25%, 0.5%, 0.75%, and 1%. The mechanical strength testing was conducted through a single fiber test. Fiber morphology was observed using an electron microscope. FTIR analyzes the change in fiber chemical composition caused by TiO_2 addition. As a result, the morphology of *S. cylindrica* fibers became rougher and showed a rougher surface after a silane concentration of 1%, but with the proper concentration, some fiber surfaces provided a good interface. Ti-O bonds are formed at a wavelength of 475 cm^{-1}. The shift in a peak at 400–500 cm^{-1} indicates Ti-O-Ti group stretching vibrations believed to have originated from TiO_2 particles. The mechanical strength increases as the concentration of TiO_2 increases, with the highest fiber strength of 284.66 MPa observed at a TiO_2 concentration of 1%. This represents a 26% higher tensile strength compared to the control specimen.

Keywords: *Coupling agent, nano-TiO_2, Sansevieria cylindrica, silane, tensile strength*

I. Introduction

The past year has witnessed a significant surge in technological advancements in the field of materials, with increased demands for materials that are strong, lightweight, environmentally friendly, and cost-effective. When searching for materials with these characteristics, scientists, engineers, and researchers look for materials based on existing materials, such as composites made from natural fibers. Natural fibers are preferred because they are less expensive, healthy for the environment and people, and strong enough to compete with other synthetic materials. Because they can be compared to synthetic materials in terms of characteristics, new natural fibers are being explored more frequently in this

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industry. The automotive industry is considering switching to synthetic fiber composites from natural fibers [1]. Fiber has the potential to reduce vehicle weight by up to 80% [2].

In recent years, natural fibers have provided numerous benefits. In contrast to composites reinforced with synthetic fibers, natural fiber composites offer numerous benefits such as being low-cost, lightweight, easily separable, abundantly available, renewable, biodegradable, and posing no health risks [3]. Wheat straw is one of a number of substitutes for crop fiber sources as agricultural by-products [4], and the other is pineapple peel waste [5], switch grass [6], banana tree [7], Napier grass [8], mendong grass [9], and some of these fibers have been utilized as reinforcement in composites. Fiber from *S.cylindrica* is potential as fiber reinforcement in polymer composite due to showing a high modulus of about 7.0 GPa, a tensile strength of about 658.0 MPa, and total elongation in a range from 10% to 12%. The composition of Sansevieria fiber consists of a high water content of 84%, along with 5% fiber, 1% cuticle, and 10% dry matter, resulting in a structurally layered and robust fiber [10].

Many ways have been developed to engineer interfaces in composite materials. Natural fiber functionalization is used to increase matrix reactivity and achieve better psycho-chemical bonding. The a coupling agent is capable of enhancing the interfacial bond and improving the composite properties by converting the natural fibers from their initial hydrophilic state to a hydrophobic [11]. Coupling agents are used to change the surface to be hydrophobic so that it can bind the polymer well. It has an impact on increasing strength [12]. Mechanical interlocks can be improved by controlling the roughness and texture of the fiber surface.

Previous research on *S. cylindrica* fiber only used alkaline treatment[10]. In this study, additional treatment on *S. cylindrica* using a coupling agent varied by TiO_2. The coupling agents improve the adhesion between the matrix and reinforcement. It also influences surface roughness to reduce the hydrophilicity of natural fibers. Adding a coupling agent in fiber composites will increase the mechanical properties of the fiber up to 61% when compared to without a coupling agent because the coupling agent can increase the bond between organic and inorganic materials [13]. The fiber is processed with addition material using a TiO_2 to increase the mechanical strength. The influence of TiO_2 treatment can increase the tensile strength of natural fibers up to 61% [12] when compared with natural fibers without treatment. The TiO_2 addition can improve fiber's inter-organic and inorganic bonding [4]. The effect of the concentration TiO_2 will determine the mechanical properties of the fibers. Objective of this study is to determine the influence of variations in the concentration of TiO_2 on the properties of *S. cylindrica*.

II. Material and Methods

1. Materials

The study used material Chemical reagents, including silane coupling agent (Merck, Germany), ethanol (Merck, Germany), NaOH (Technical grade, Indonesia), and TiO_2 (Sigma Aldrich). The *S. cylindrica* fiber used in the study was sourced from a homestead in Malang, East Java, Indonesia (Figure 1). The leaf length was about 80 to 100 cm, and samples were chosen when the plants were 6 to 8 months old. The *S. cylindrica* fiber consists of 72.14% cellulose, 20.22% hemicellulose, 3.44% lignin, 4.22% t extractive compounds, and 4.22 to 5.22% moisture [9].

Fig. 1. *Sansevieria cylindrica* plant

2. Fiber Extraction

The fiber was extracted mechanically by beating the stems of *S. cylindrica* while still wet, then cleaning them in an aqueous medium until the fibers were separated from the connective tissue. The fiber is then soaked in water for a week. *S. cylindrica* fiber was collected, cleaned, and allowed to air dry.

3. Alkali Pretreatment

Alkali pretreatment was carried out by immersing the fiber in alkali (NaOH 5%) for two hours and then rinsing it with distilled water up to five times. After being dried at room temperature until they were completely dry, the fibers were wrapped in plastic wrap and stored in a dry box with a humidity level of 40% [15].

4. Nanoparticles Grafting

Mixing the silane coupling agent in alcohol with concentration of 10% and nanoparticles TiO_2 with a stirring process for 30 minutes. Nano TiO_2 with a concentration of 0.1% shows good hydrophilic characteristics when compared to a higher concentration of nano TiO_2 1%[16]. The TiO_2 concentrations used were 0%, 0.25%, 0.5%, 0.75%, 1% since a higher concentration can degrade the fibers' mechanical qualities. The fibers were soaked in each solution using an ultrasonic homogenizer for 15 minutes, washed with distilled water for 1 hour, then dried using an oven at 75°C for two hours.

5. Morphology Analysis

Surface morphology observations were conducted 1000 times magnification with Scanning Electron Microscope (SEM), FEI type Inspect S50, with a voltage range of 10.00 to 15.00 kV. The surface of the nanocomposite was coated with a sputter coater to enhance the surface conductivity and investigate the morphology of the nanocomposite surface.

6. Functional Group Analysis

The FTIR test in this study was to see the characterization of the chemical properties of the functional groups of *S. cylindrica* fiber with TiO_2 grafting process compared to Sansevieria fiber without TiO_2 grafting using FTIR merk Shimadzu, type: IR-Prestige21. The spectrum was scanned at a range of 400-4000 cm^{-1}.

7. Tensile Strength Analysis

The tensile strength of *S.cylindrica* fibers was measured at fiber length of 30 mm, using five specimens at ambient temperature. The fiber tensile tester had a maximum load of 50N, and a loading rate of 3.5 mm/min. The fiber cross-section area was determined by horizontal photographic observation using an optical microscope. It was assumed that the fiber had a circular cross-section, and the ImageJ software was used to measure its diameter at five different points along the photograph. After the area measurements were recorded, the average cross-sectional area of fiber was calculated. Figure 2 depicts the single fiber tensile test specimen.

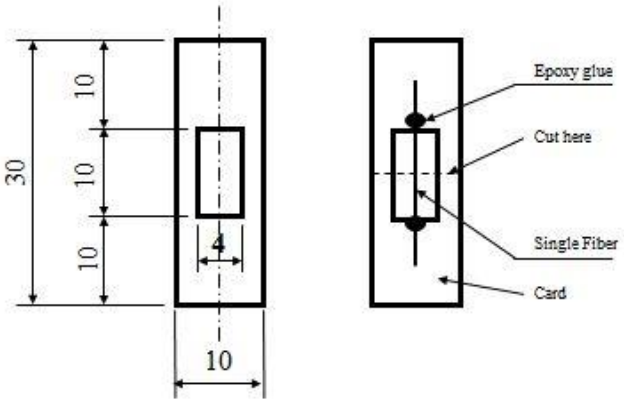

Fig. 2. Single fiber tensile test specimen

III. Results and Discussions

1. Morphology Analysis

The morphology was analyzed using SEM with 1000 times magnification to observe fiber morphology. Morphological results of *S. cylindrica* fibers without TiO_2 from the SEM observation are shown in Figure 3a, addition 0.25% TiO_2 in Figure 3b, addition 0.25% TiO_2 in Figure 3b, addition 0.50% TiO_2 in Figure 3c, addition 0.75% TiO_2 in Figure 3d, and addition 1% TiO_2 in Figure 3e. As shown in Figure 3a, 3b, and 3c, the surface of the fiber becomes more evenly tight and smoother. The higher concentration of TiO_2, the rougher surface of *S. cylindrica* fibers. The addition of 1% TiO_2 resulted in a rougher surface morphology. By incorporating additional materials and subjecting fibers to chemical treatments, the surface of the fibers can become rougher. This is because the silane coupling agent causes the hydrolysis of the lignin content in the fiber, resulting in a rougher interface on the fiber [17]. The nano-TiO_2 particles agglomerated on the fiber surfaces, with the quantity increasing as the nano-TiO_2 content increased [18].

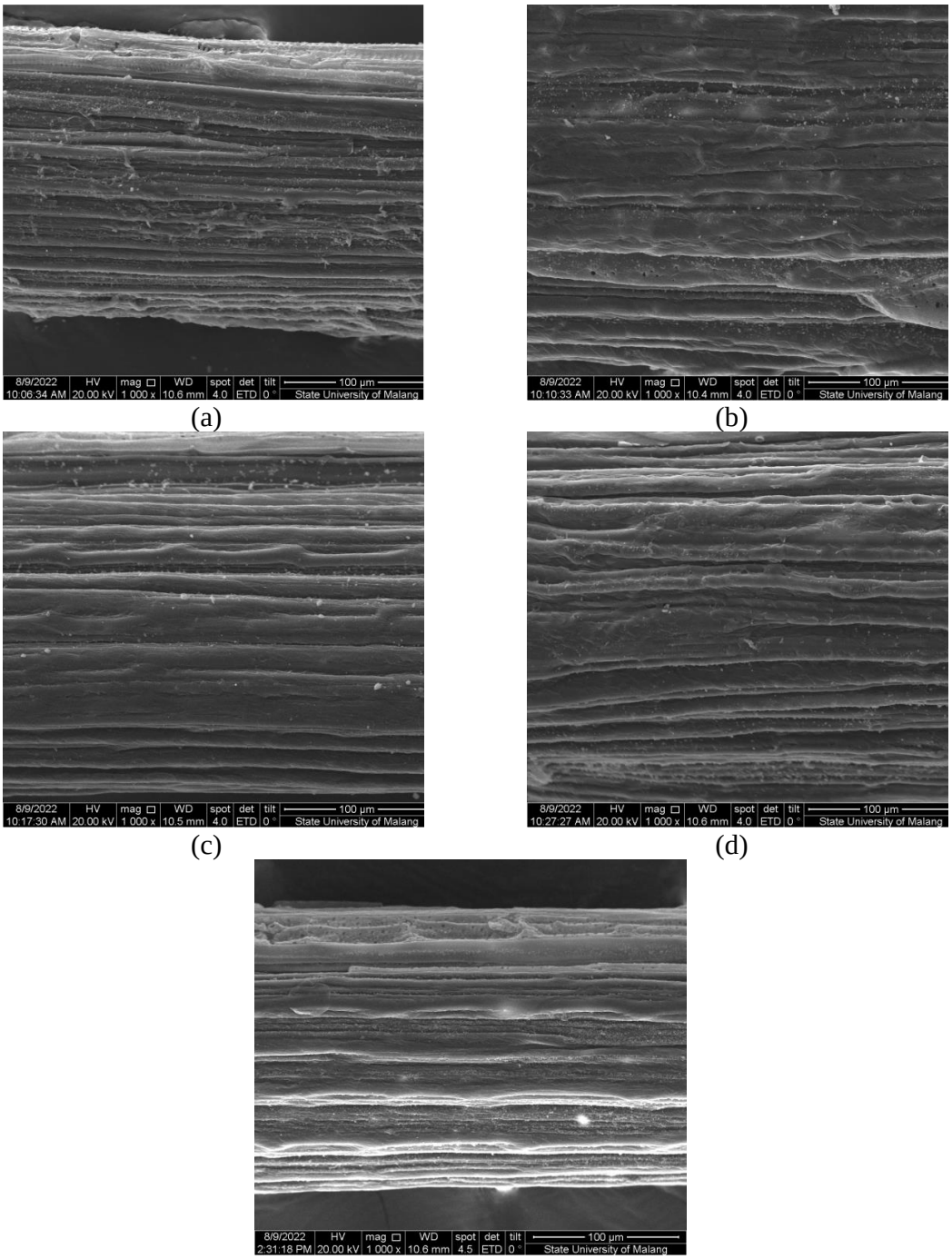

Fig. 3. Morphology of *S. cylindrica* fibers with TiO_2 concentration of (a) control; (b) 0.25%, (c) 0.50%; (d) 0.75%, and (e) 1%.

2. Functional Group Analysis

The fibers were a type of heterogeneous lignocellulosic substrate, displaying a spectral pattern containing absorption bands that were relatively distinct in nature. The addition of TiO_2 to the cellulosic fiber caused it to swell, removing lignin, hemicellulose, and other impurities from the fiber surface. FTIR was used to analyze the change in the fiber functional group caused by TiO_2 addition. Chemical treatments involving coupling agents can

strengthen the interactions at the interface by reinforcing chemical bonds through the presence of chemical functional groups and an acid-base interaction mechanism. This, in turn, can improve the overall effectiveness of the treatment [19].

As shown in Figure 4, the broadband ranging from 3000 to 4000 cm^{-1} indicates the OH stretching present in hemicellulose, cellulose, and lignin. Changes at the peak around the value of O-H undergo strain, and peak shift at the stretch number 3100-3600 is seen in fibers. This is due to the surface hydrolysis reaction of the silane coupling agent in *S.cylindrica* fibers [20]. The C-H bond, frequently seen in alkane groups, was thought to be responsible for the wide band of 2850-3000 cm^{-1}. At 1765-1715 cm^{-1}, the hemicellulose compound exhibits sharp peaks for carbonyl and ketone C=C groups. At 1730 cm^{-1}, peak was disappeared after fibers were treated with 5% NaOH. It is evidence that hemicellulose decreased following alkalization process. The alcohol compounds and aromatic lignin structure (C=O bond) present in the fiber were referenced by the peaks at 1450 and 1650 cm^{-1}. The observed peak at 890-900 cm^{-1} indicated the -glycosidic linkage of fiber, which is contributed by both hemicellulose and cellulose [21].

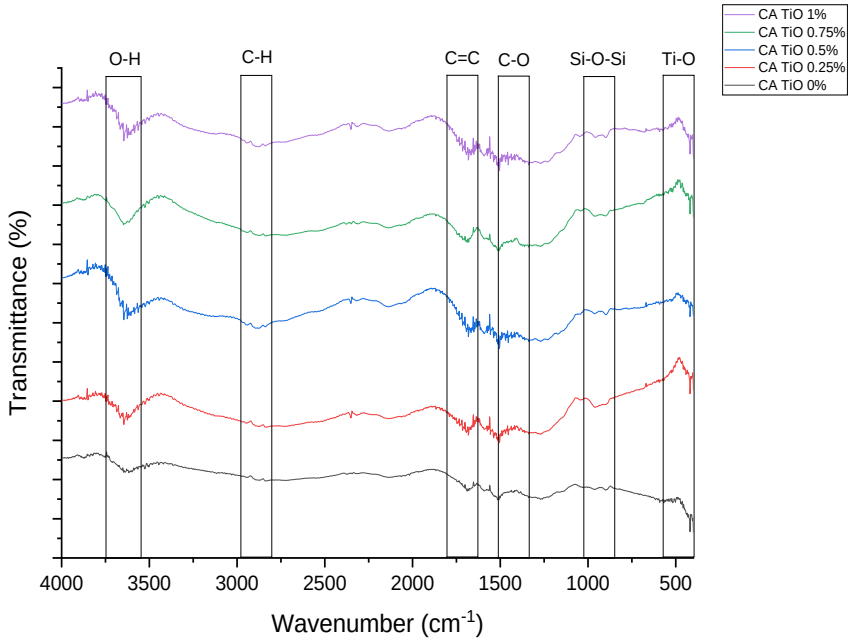

Fig. 4. FTIR test results of *S. cylindrica* fibers with variations in TiO_2 concentrations of 0%, 0.25%, 0.5%, 0.75% and 1%.

The reduction of hydroxyl groups in these fibers is due to the hydrolysis of the silane coupling agent, resulting in a new peak formed in fiber around the peak value of 1157cm^{-1} (Si–O–Si), where the fiber without silane coupling agent treatment does not have such a peak. Si–O–Si indicates the condensation reaction of a silane coupling agent that reacts between fiber and silane [11]. The addition of TiO_2 to the other silanes constructs a valley that represents each molecular bond between the CA-TiO_2 fibers. The valleys that are visible in detail indicate the presence of O-H bonds at 3600 cm^{-1}. C-O bonds exist at wavelengths ranging from 1500 to 1600 cm^{-1}. Ti-O bonds are formed at a wavelength of 475 cm^{-1}. The shift in the peak at 400–500 cm^{-1} indicates Ti-O-Ti group stretching vibrations believed to have originated from TiO_2 particles [23]. Stretching of C-O and carbonyl (C=O) is the result of the decomposition of CH_2OH groups in cellulose with the help of TiO_2 as a catalyst [24].

3. Tensile Strength Analysis

Figure 5 represents the tensile strength of *S. cylindrica* fiber with variation addition TiO_2. The strength of control fiber and treated fiber using silane are differrent because control fibers have hydrophilic properties, they cannot bond well with hydrophobic polymers like epoxy. Fibers treated with silane coupling agents can acquire hydrophobic surfaces, allowing them to adhere to active groups in hydrophobic polymers. Silane converts hydroxyl groups (-OH) in the fiber into silanol groups (Si - O - Si), increasing the bonding between the fiber and matrix and thus increasing the interfacial shear strength of the composite with the silane bridging material [12].

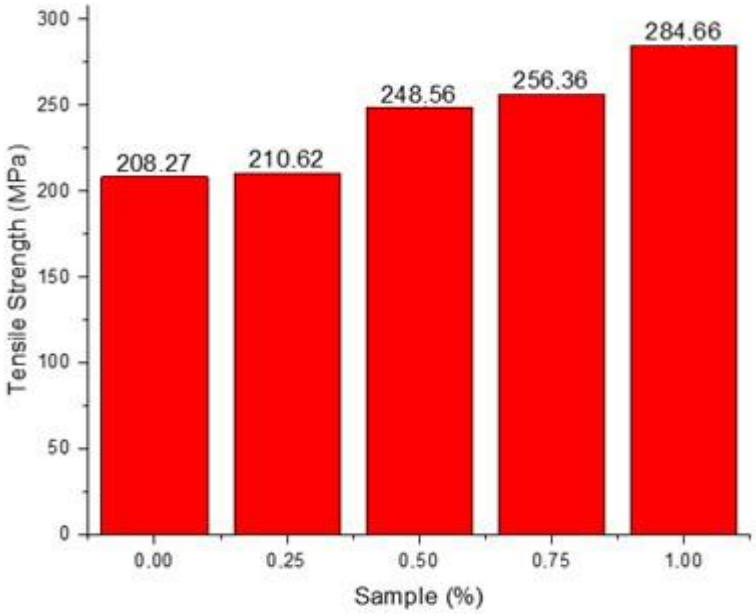

Fig. 5. Tensile strength *S. cylindrica* fiber with variation concentration of TiO_2.

The fiber surface has an intrinsically high surface atomic energy so that adhesion to rough surfaces can be effective. When expressed per unit nominal area, the increase in the area due to various factors also increases the surface energy. When stress is applied to a roughened surface, the stress is redistributed, and the resulting interface is strengthened because roughness can change the fracture mechanism from a small one to a more energetic mode. When two incompatible polymers come into contact, the mechanism shifts from chain to other types of plastic deformation. An extended single fiber will undergo configuration deformation of the macromolecular chain as an elastic response because the macromolecular chains in the cell wall of the fiber are not oriented to the length of the fiber axis [18].

The addition of TiO_2 to a single fiber of *S. cylindrica* fiber can influence the interface shear stress, increasing the fiber strength. The tensile test results showed that the highest value was 284.66 MPa at 1% TiO_2 concentration. At 0% TiO_2 concentration, the lowest tensile strength value is 208.27 MPa. Overall, the addition of TiO_2 increases the *S.cylindrica* strength. Tensile strength increased to 210.62 MPa, 248.56 Mpa, and 256.36 MPa with a TiO_2 addition of 0.25%, 0.5%, and 0.75%, respectively.

The influence of nano-TiO_2 on the fiber surface after silane treatment can be attributed to the increase in fiber strength and breaking strain. The silane coupling agent layer filled with nano-TiO_2 can improve the fiber tensile strength and reduce the stress concentration on

the fiber surface. The effect of grafting nano-TiO_2 on the strength of *S. cylindrica* fiber was restricted when compared to the treatment that involved solely the addition of a coupling agent. Furthermore, the concentration of nano-TiO_2 grafted on sansevieria fiber can increase its tensile strength.

IV. Conclusion

The treatment of *S. cylindrica* fiber was carried out to obtain the characteristics of the fiber. Fiber specimens with a 5% concentration of silane coupling agent with the addition of TiO_2 with a concentration of 1% have a coarser structure so as to improve mechanical properties. Surface variation in fiber on 1% TiO_2 surface additive has the highest tensile strength value of 284.66 MPa. FTIR results indicate that adding TiO_2 to the other silanes constructs a molecular bond between the fiber-silane-TiO_2. Incorporating TiO_2 nanoparticles can enhance the bonding properties and tensile strength of the material. It is suggested to modify the surface of natural fibers through nanoparticle grafting to attain optimal mechanical properties for natural fibers.

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