

## Evaluation of Silane Coupling Agent Treatment on *Sansevieria cylindrica* Fiber as Reinforcement in Epoxy Composite

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### Abstract

The load transfer effectiveness from matrix to reinforcement is depended on the interface condition of the fiber surface and matrix. Chemical treatment can change fiber properties. The current study aims to evaluate the influence of chemical treatment using silane solution on interface shear strength (ISS) of *Sansevieria cylindrica* fiber (SCF) reinforced epoxy composite. The research procedure includes silane treatment on SCF with percentage silane concentrations of 0.0, 2.5, 5.0, 7.5, and 10.0%. ISS was evaluated by a single fiber pullout procedure. SCF crystallinity, functional group, and morphology were evaluated using X-ray diffraction, Infrared spectrometry, and electron microscope. The result shows that the surface morphology of SCF became rough and the surface rougher after silane treatment of 7.5%. SCF with no treatment has a degree of crystallinity of 66.0%, but after being treated with silane treatment, the degree of crystallinity increased, and the highest value of 75.0% was obtained from the silane treatment of 10.0%. The ISS increases following the increasing silane concentration. The highest ISS of 91.3 MPa was obtained at a silane treatment of 10.0%, with an increasing ISS of 129.0% compared to the control specimen.

**Keywords:** Epoxy composite, silane, *Sansevieria cylindrica*, interface properties.

### 1. Introduction

In the current decade, technological developments in the material sector have increased rapidly because of the demand for environmentally friendly, lightweight, and strong materials. In looking for these characteristics' materials, researchers try to explore materials from natural sources such as natural fibers-based composites [1]. Some industry in the transportation field has applied natural fiber to replace synthetic fiber in building composites part [2] because the fiber is able to reduce the weight of the vehicle to 80% [3]. Natural fibers have offered many advantages compared to synthetic fibers, such as low density, low price, easily separated, abundant, renewable, and biodegradable [2]. Some of the examples have been applied in the polymer composite. *Sansevieria cylindrica* fiber (SCF) has potential as reinforcement in polymer-based composites because they exhibit a high strength (658 MPa), elastic modulus (7.6 GPa), and fiber elongation (10%) [4].

*S. cylindrica* are tropical and subtropical plants, easy to grow in Indonesia, and distributed in areas, with a distribution ranging from Africa to Southeast Asia and the islands of the Indian Ocean [5][6]. *Sansevieria* may thrive in a broad range of ecosystems as an aggressive invasive plant. It has a significant economic value since it is high in fiber, which is frequently utilized as a raw material in textiles. As a result, it is one of the most exported commodities [7]. Each stem on that same *S. cylindrica* plant is typically 10 to 20mm thick, and it may grow to a height of 1 to 2 meters. The stem is a

sandwich structure made up of around 5% fibers, 1% cuticle, 10% dry matter, and 84% water. SCF has superior mechanical qualities when compared to other natural fiber composites [8].

Most fiber treatment is conducted to ensure the fiber interface interaction between fiber and matrix in a better situation. Many chemical methods have been adopted to modify the interfaces in polymer-based composite materials, such as grafting [9], crosslinking [10], ethylation, acetylation [11], alkalization [12], and coupling agent (silane [13], maleic anhydride [14], diisocyanate[15]). Chemical reagents change the surface properties of the fibers, which choice depends on the type of polymer matrix used. The free and polar energy modification of fiber surface and dispersion components significantly increase fiber adhesion to the polymer matrix. Therefore, it supports wetting and impregnating during processing [16]. Functionalization of natural-based fiber surfaces is needed for increasing fiber reactivity and enhancing better psycho-chemical interaction. During the composite manufacturing process, functionalization using coupling agent was conducted in order to incorporate fiber interaction into the matrix. Controlling the texture and roughness of the fiber surface is significant in improving interface strength through the mechanical interlock mechanism. The compatibility of the natural fiber to synthetic matrix, such as resins, depends on the roughness or fineness of fiber's surface. Creating a rough fiber surface allows the matrix or resin to better interlock with natural fiber [17]. A rough surface texture improves the mechanical interlocking at the interface [18]. The mechanical interlock mechanism is ineffective in the presence of a surface that is too rough,

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suggesting the existence of ineffective bonding will reduce the natural fiber toughness [19]. This is accomplished by eliminating both natural and artificial impurities while also disrupting hydrogen bonding in the network structure, increasing the Interface Shear Strength (ISS) because of better compatibility [20]. Very few literature studies report the ISS of SCF fibers reinforced with epoxy composites. The present study evaluates the influence of silane coupling agent on ISS of SCF on epoxy composite. An optimal silane treatment is proposed to attain better reinforcing effectiveness, thereby providing a reference for the design of SCF-reinforced composite.

## 2. Material and Methods

Chemical materials for the composite matrix were Epoxy (Eposchon-A) and Hardener (Eposchon-B) were supplied by PT. Justus Kimia Raya, Indonesia. The chemical agents for treatment were NaOH (CV. Surya Techno Chemlab, Indonesia), ethanol (Merck, Germany), and Silane (KH 560, Sinoconvoy Materials, China). The study used SCF, obtained from the District of Batu, East Java, Indonesia, from a plant aged 6 - 8 months, and the stem length was about 50 cm.

### 2.1. Fiber Extraction Process

The fiber extraction was conducted by mechanical procedure by hitting *S. cylindrica* stem repeatedly until the stem was broken and SCF was separated from the fiber binding tissue. The destructed stem was cleaned by water SCF was immersed in water for 5 days. SCF was cleaned and dried in dry winds.

### 2.2. Alkali Pretreatment

Fiber pretreatment was conducted by soaking SCF in an alkali solution (5% NaOH). Pretreatment was conducted for 2 hours at 60°C. After the immersion process, SCF was washed with dH<sub>2</sub>O 5 times until pH neutral, then SCF was dried for 2 days in an oven at 60°C and put into a dry box 40% humidity.

### 2.3. Silane Treatment

Silane treatment was conducted by drip silane solution into the ethanol solution until the silane solution achieved concentrations of 2.5, 7.5, and 10.0%. SCF was soaked in each solution while conducting sonication (Ningbo Lawson, China) with a frequency of 20kHz, and power of 400W for 15 min. SCF was washed with dH<sub>2</sub>O and then put into an oven for drying at 75 °C for 2h.

### 2.4. Surface Morphology Observation

SCF was put in coater equipment (Emitech SC7-620) and then coated by 10 nm gold layers. SCF morphology was observed under Scanning Electron Microscope/SEM (FEI type Inspect S50, USA) at 15.00 kV.

### 2.5. Structural analysis

The SCF structure before and after SCA treatment was observed by X-Ray Diffractometers/XRD (PANalytical: Expert Pro) at ambient temperature. Scanning was conducted in a range of 10° - 60° at 30.0 mA and 40.0 kV. The crystalline degree (%Cr) and crystalline index (CI) were evaluated following the Segal formula (Eq. 1 and Eq. 2),

$$CI = \frac{I_{(002)} - I_{(am)}}{I_{(002)}} \times 100\% \quad (1)$$

$$Cr = \frac{I_{(002)}}{I_{(002)} + I_{(am)}} \times 100\% \quad \dots\dots\dots (2)$$

where  $I_{(002)}$  is the maximum intensity at  $2\theta$  from 22° to 23° represent crystalline substances, and  $I_{am}$  is the minimum intensity at  $2\theta$  about 18°, represents amorphous substances in the natural fiber.

### 2.6. Functional group analysis

The functional group of SCF before and after being treated by SCA was scanned using Fourier Transform Infrared Spectrometer (FTIR) (Prestige-21 type, Shimadzu, Japan). SCF samples of 0.1 mg in powder form were mixed with 1 mg KBr powder and pressed to form a pellet. The pellet was put into FTIR apparatus the spectrums were scanned at 4 cm<sup>-1</sup> resolution within the range from 400 to 4000 cm<sup>-1</sup>.

### 2.7. Interface Shear Strength (ISS) Analysis

ISS analysis was determined by the single fiber pull-out test, according to the previous study [21]. A pull-out test was done in the fiber tensile test equipment with 50.0 N maximum load. Samples were held by gluing fiber on the thick paper, as shown in Figure 1. Epoxy was added with a hardener with a ratio 2 : 1, then mixed and casted into molds. The treated SCF was embedded in the wet epoxy in molds slowly 200 μm in-depth, then left until the epoxy became hard. The pullout test was done with a rate of 3.50 mm/min. at 25°C with repetition five times for each sample.

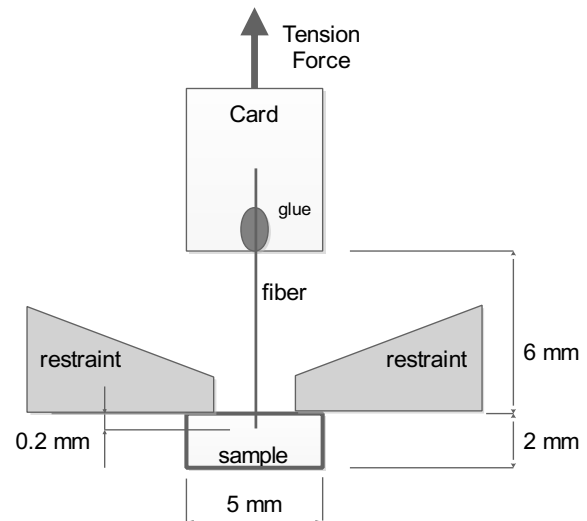


Fig. 1. Single Fiber Pullout Test

## 3. Result and Discussions

### 3.1. Surface Morphology

Figure 2 illustrates the surface of SCF taken from the SEM. After SCA treatment, the SCF surface becomes smoother (Figure 2A, 2B, and 2C). The higher the silane concentration in treatment, the rougher SCF surface. The SCF surface is rougher starting at SCA concentration of 7.5% (Figure 2D) due to the amorphous substance in the fiber surface removed by SCA, which causes the rough surface fiber [22].

Figure 2A-2C shows that silane can remove impurities, lignin, and hemicellulose in the fiber [23]. The smoother natural fiber was caused by the siloxane layer established on the surface of SCF and, as a result of the condensation of the silane groups, which somewhat decreased the surface roughness of the treated CSF and therefore provided a slightly

regular and residue-free surface [24]. In samples 2D and 2E, there is a significant increase in roughness due to the addition of too much silane causing the breakage of lignin and hemicellulose bonds which causes discontinuities between fibers so that changes in fiber surface roughness become significant. These results are in line with the XRD test shown in Figure 3, which shows the intensity value continues to increase.

In composite interface, the interaction between polymer matrix and fiber reinforcement has a role in transferring load from matrix to fiber. The morphology of SCF and resin matrix affects the interface interaction area, so it greatly determines the structural properties of the composite. Micromechanics of stress transfer and the composites' strength depends on the interface area's direct interaction capability. The fiber-matrix interface interaction act as an indicator of the micromechanics of composite stress transfer. A better stress transfer ability results in higher ISS [25].

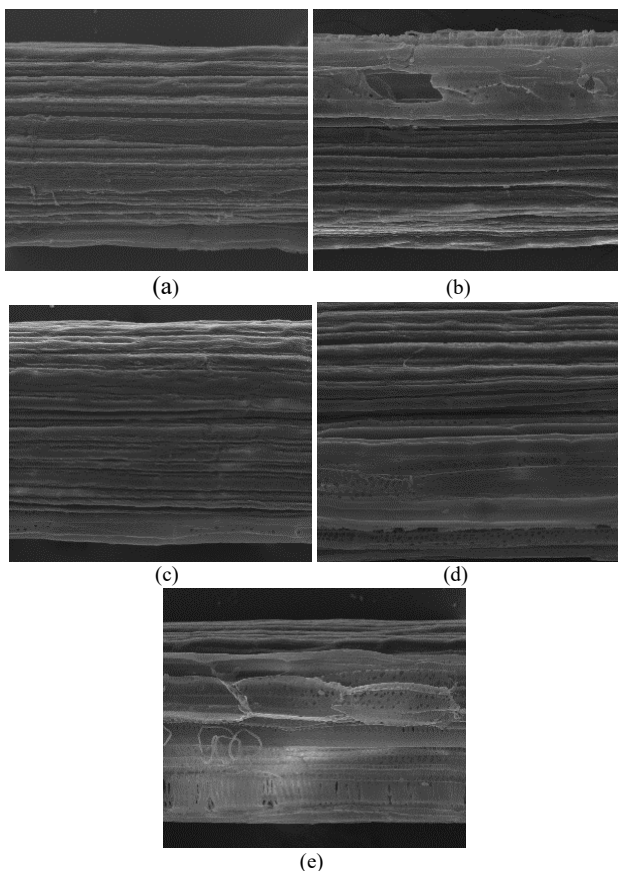


Fig. 2. Surface Morphology of SCF After Silane Treatment at Concentration of (a) 0.0 (control), (b) 2.5, (c) 5.0, (d) 7.5, and (e) 10.0%

The compatibility of the surface of fiber and resins depends on the surface fineness or roughness. However, there are optimum conditions for surface roughness to increase the shear stress. In the silica glass and resin composite system, the optimum roughness conditions reach the range of 40-60 nm, after which the shear strength will decrease. This indicates the mechanical interlock mechanism is ineffective in the presence of too rough a surface, indicating surface damage [26].

Mechanically, the adhesion theory is associated with adherence to porous and rough surfaces. The rough surface can range from microns to nanoscale. When a load is applied, the rough surface redistributes it and strengthens the interface resulting from the increased roughness. This has the potential to shift the way the fracture occurs, moving it from a less

intense mode to a more energetic one. If the two polymers do not mix well, the mechanism shifts from a pull-out process to a different type of plastic deformation [27].

### 3.2. Crystallinity analysis

Figure 3 shows a diffraction pattern of SCF after SCA treatment. Diffractogram indicates 3 peaks at a  $2\theta$  of 16.6°, 22.5°, and 34.7° representing the crystalline fraction of SCF correlated to the crystal plane of [011], [002], and [400], respectively [28]. These peaks have been associated with the structure of cellulose I [29]. The various SCA concentration of 0.0, 2.5, 5.0, 7.5, and 10.0% produces peaks at  $2\theta$  of 22.3°; 22.4°; 22.5°; 22.6°; and 22.5°, with the intensity of 231, 380, 400, 414, and 425 a.u., respectively. The minimum peak intensity at about 18° represents an amorphous substance, and the diffraction angle from 22° to 23° represents the crystalline substance in cellulose fibers [30].

The CI and Cr of the control specimen are 49.0% and 66.0%, respectively, as shown in Table 1. The CI and Cr with the highest value are present in the SCA treatment with a concentration of 10.0%, having crystalline percentages of 66.0% and 75.0%, respectively. This value is caused by the chemical treatment in the SCF surface reducing the non-crystalline fraction, such as hemicellulose and lignin in lignocellulosic fibers, so that it can increase the crystalline fraction [31].

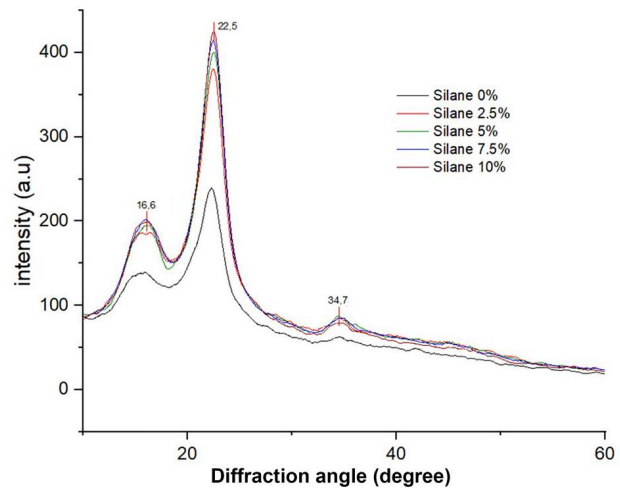


Fig. 3. Diffractogram of SCF After being Treated by SCA with Various Concentrations

Table 1. Quantification of Degree of Crystallinity (Cr) and Crystalline Index (CI) of Silane Treated SCF

Silane concentration (%)	$I_{am}$	$I_{(002)}$	CI (%)	Cr (%)
0.0	18.4	22.3	49.0%	66.0%
2.5	18.3	22.4	59.0%	71.0%
5.0	18.2	22.5	64.0%	73.0%
7.5	18.7	22.6	64.0%	73.0%
10.0	18.6	22.5	66.0%	75.0%

### 3.3. Functional Group Analysis

Natural fibers are lignocellulosic materials that show a wave pattern with relatively sharp. The SCA treatment caused the fiber to swell and removed some components like hemicellulose, lignin, and impurities from the surface. The shift in the functional group of fiber caused by silane treatment was able to analyze with FTIR.

Figure 4 shows that the broadband at 3000 to 4000  $cm^{-1}$  was referred to O-H stretching of cellulose. The changes that

occur in O-H group undergo a strain and shift of peaks at the wavenumber of 3100 to 3600. The fiber surface shows a difference in intensity depending on the concentration variation of SCA due to the hydrolysis reaction of SCA to the surface in SCF [32].

The higher the concentration of SCA, the intensity of the frequency value will decrease. The broadband of 2850 to 3000  $\text{cm}^{-1}$  indicated a functional group of C-H from alkane compounds. The peaks in spectral 1450 to 1650  $\text{cm}^{-1}$  are attributed to alcohol compounds. Peak 1600  $\text{cm}^{-1}$  was referred to OH vibration of water absorbed by the fiber. The peaks at 900 to 1100  $\text{cm}^{-1}$  were referred to C-O stretching from cellulose compound and C-C stretching of alkenes function. The peaks at 700 to 900  $\text{cm}^{-1}$  were referred to C-H group of aromatic hydrogen compounds. The peak in spectral wave from 890 to 900  $\text{cm}^{-1}$  was referred to  $\beta$ -glycosidic linkage from both hemicellulose and cellulose of fiber [2].

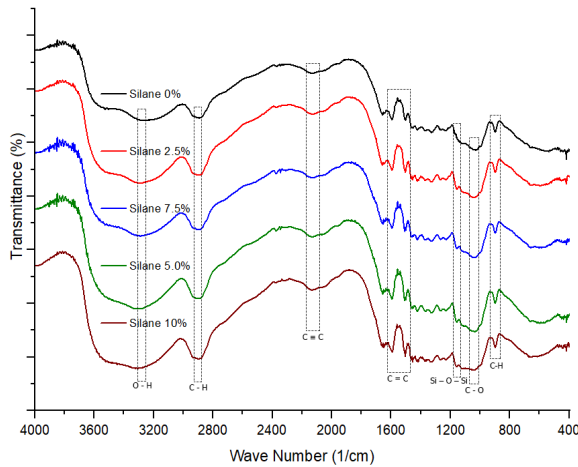


Fig. 4. FTIR analysis of SCF Treated with SCA at a concentration of 0.0, 2.5, 5.0, 7.5, and 10.0%

The decrease in hydroxyl groups within these fibers occurs due to the hydrolysis of SCA, leading to the emergence of a new peak in the fiber's spectrum, approximately at the peak value of 1157 (Si-O-Si). The untreated fiber does not exhibit a similar peak. The presence of Si-O-Si groups indicates the occurrence of a condensation reaction between SCA and the fiber, facilitated by the reaction with silane [33].

The presence of reactive functional groups in both synthetic fibers and resins is critical for high interfacial energies. The effect of surface roughness and functional groups on ISS of PE fiber that received corona discharge treatment and untreated HDPE film was analyzed by multivariate regression analysis divided into 2 groups, namely group I with low radiation energy showed an increase in ISS due to the influence of functional groups which was more dominant compared to surface roughness. Another group exposed to higher radiation energy showed an increase in ISS due to increased surface roughness and contribution between functional groups (-COOH) with a contribution of 50% [34]. The performance of composite materials with natural fiber reinforcement depends on the coherent surface bond between the matrix and fiber. The incompatibility of hydrophilic fiber and hydrophobic matrix causes a poor interfacial adhesion of composite.

### 3.4. Interface Shear Strength (ISS) Analysis

The SCA concentration variation used to treat SCF greatly affects the ISS between the epoxy and fiber. The pullout test results indicate that the highest ISS is obtained at the SCA treatment of 10.0% with an ISS value of 91.3 Ma, but the

lowest ISS is obtained at non-treated SCF with an IFS value of 39.9 MPa. The increased concentration of SCA treatment affects the epoxy and SCF bonding, increasing the ISS of SCF in epoxy matrix composite.

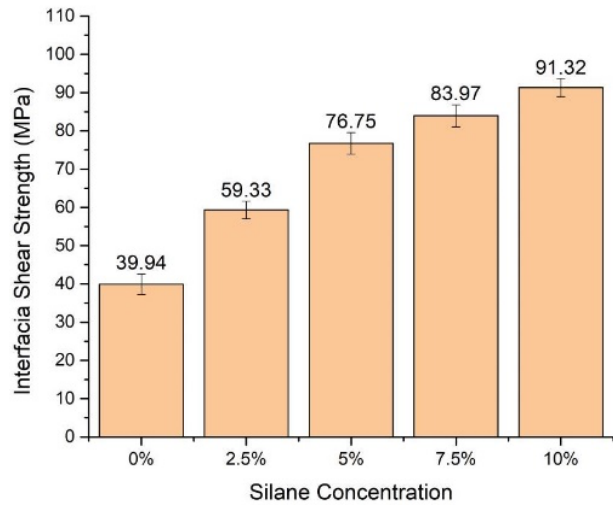


Fig. 5. ISS of SCF in Epoxy Matrix Composite in Various SCA Concentrations

Rough surface fiber is identical to the wider area due to contour factors. The rougher surface has intrinsically higher surface energy per unit area. It causes effective adhesion of polymer into the rough surface fiber. A rough surface can redistribute the tensile load and increase the ISS because the rough surface can shift the fracture mechanism. If the surface of two polymers has an incompatible condition, the fracture mechanism shifts from chains to plastic deformation [35].

ISS is an effective indicator of the bond between fibers and matrix. SCF without treatment was resulting ISS epoxy composite by 39.94 MPa. After the silane treatment, ISS of SCF-epoxy matrix was increased to 59.33, 76.75, 83.97, and 91.32 MPa with percentages of 48, 92, 110, and 129% by SCA concentration of 2.5, 5.0, 7.5, and 10.0%, respectively. The treatment using silane coupling agent can strengthen fiber-matrix interactions through chemical bonding mechanisms and activation of chemical functional groups [36]. Surface polarity due to chemical treatment also has an influence on fiber/matrix interactions in the composite, where a decrease in fiber polarity due to chemical treatment causes better compatibility of fibers in nonpolar matrix so that the mechanical strength increases [37]. In this study, increasing the crystalline phase improves the ISS of SCF-epoxy composite. The increase in crystallinity can also strengthen the bond adhesion and improve the stress transfer efficiency at the interface.

The finding is correlated to the efficiency of stress transfer from matrix to fiber which implies providing alternative structural materials in the composite industry. It answers the primary challenge in composite design, ensuring optimum stress transfer between the fiber and matrix to maximize the composite's strength and stiffness as essential factors to be fulfilled for composite materials [38] to ensure the materials meet the desired performance and requirements. ISS also implies the critical fiber length that denotes the minimal fiber length necessary for load transfer until a fracture occurs. They act as an indicator of the interphase's ability to transmit stress in fibrous composites. When shorter fibers are incorporated into the matrix while retaining the ISS, the load transfer within the composite becomes more effective. It is interesting because, normally, fibrous composite uses short fibers as reinforcement [39]. In application, some researchers have

used Sansevieria fiber in automotive industries [40] as interior and exterior materials [41] and brake pads [42], composite panels for furniture [43], building [44] and sound-absorbing materials [45]. Till now, Sansevieria fiber has been studied as being composite reinforcement. Therefore, it is essential to comprehend its behavior when combined with other resins, particularly concerning the fabrication of biocomposites and hybrid composites, to expand its potential applications in various existing products.

#### 4. Conclusions

Treatment of various concentrations of SCA to SCF was studied to evaluate fiber characteristics and ISS composite. SCA treatment on SCF with 10.0% concentration results in a rougher surface and increases ISS fiber to the matrix. SCF structure after SCA treatment indicates three peaks at a  $2\theta$  of

16.6°, 22.5°, and 34.7°, strongly associated with the structure of cellulose I. The higher SCA concentration increases the crystallinity of SCF. The addition of SCA concentration increases the ISS of SCF in epoxy matrix composite with the highest ISS value of 91.32 MPa, obtained from SCA with a concentration of 10.0%. In the future, this treatment can develop as a major treatment to get high-quality natural fiber epoxy composite materials.

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