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RESEARCH ARTICLE

EFFECT OF TEOS CONCENTRATION ON SILICA COATING MORPHOLOGY AND THERMAL STABILITY OF ELAEIS GUINEENSIS FIBER FOR FIRE RETARDANT APPLICATIONS

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Abstract. Oil palm (Elaeis guineensis) empty fruit bunch (EGEFB) fiber, a prevalent agricultural byproduct in Malaysia, exhibits significant potential as an environmentally sustainable insulating material attributable to its thermal and acoustic resistive characteristics. Nonetheless, its pronounced flammability constrains its utilization, thereby necessitating the implementation of fire-retardant treatments. This study investigates the implications of varying concentrations of tetraethyl orthosilicate (TEOS) on the silica coating morphology of EGEFB fibers, employing a sol-gel approach with TEOS-ethanol ratios of 1:4, 1:7, and 1:10. The coated fibers underwent thermal curing at 80 °C for one hour. Characterization of the coated fibers was executed through X-ray diffraction (XRD) to ascertain the presence of silica, field emission scanning electron microscopy (FESEM) for the analysis of coating morphology, and thermogravimetric analysis (TGA) for the evaluation of thermal stability. The results elucidate that lower concentrations of TEOS yield more homogeneous silica coatings, resulting in enhanced adhesion and improved thermal stability, whereas higher TEOS concentrations lead to irregular coatings with suboptimal adhesion. These findings underscore the importance of optimizing TEOS concentration to attain effective, thermally stable coatings on EGEFB fibers. This paper offers valuable insights into the enhancement of fire resistance and thermal properties of EGEFB fibers, thereby contributing to the advancement of sustainable material development for sectors aiming for environmentally friendly, thermally stable and fire-retardant insulation materials.

Keywords: Oil palm, sol-gel, surface morphology, empty fruit bunch, thermal stability.

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1. INTRODUCTION

The silica coatings have emerged as pivotal methodologies for augmenting the functional properties of materials across diverse sectors, including but not limited to, glass, plastics, metals, ceramics, and natural fibers. The inherent stability, mechanical strength, and versatile surface characteristics of silica render it an exemplary candidate for incorporation into these substrates, thereby bestowing enhancements such as thermal resistance [1], ultraviolet protection [2], antimicrobial capabilities [3], and fire retardancy [4]. By establishing barriers against moisture penetration and degradation, silica coatings significantly enhance durability [5], which is particularly advantageous for applications pertaining to insulation and protection.

Elaeis Guineensis empty fruit bunch fiber (EGEFB), a prevalent agricultural byproduct in Southeast Asia [6], provides renewable and sustainable insulation attributes that align with environmentally conscious practices [7]. Nevertheless, challenges such as elevated flammability and hydrophilicity impede the industrial applicability of EGEFB [8]. The application of silica coatings to EGEFB fibers has the potential to mitigate these drawbacks, imparting fire resistance, water repellency, ultraviolet stability, and enhanced durability. Specifically, silica coatings confer hydrophobic characteristics and microbial resistance, thereby markedly augmenting the fibers' versatility for applications in sectors that necessitate durability.

Silica coatings improve the thermal stability of natural fibers, as demonstrated in cotton fabrics where the incorporation of silica and zinc oxide increased the time to ignite, and flame spread time significantly [9]. Although the use of silica coatings is well established, optimizing the coating process, improving coating morphology, and enhancing thermal stability specifically for EGEFB fibers remains vital.

Wood-based composite aerogels treated with silica show a decomposition temperature increase of approximately 45 °C and exhibit self-extinguishing behavior, maintaining structural integrity after combustion [10]. Organo-inorganic SiO_2 sol applied to cotton fabrics to prevent ignition and significantly reduce the area of damage upon exposure to fire [11].

However, achieving a uniform silica coating on EGEFB fibers is still challenging. Changes in the silica sol's concentration alter its viscosity, which in turn affects the deposition process and uniformity of the coating. If the silica sol is too concentrated, the coating's thickness can become inconsistent because the silica particles form unevenly (uneven nucleation) and vary in size, which weakens the coating's performance and reliability. This study examines how different amounts of tetraethyl orthosilicate (TEOS) in the silica sol-gel affect the coated fibers' structure (morphology) and their ability to withstand heat (thermal stability). By understanding these effects, the study aims to optimize the coating process and contribute to the development of fire-retardant, sustainable fiber materials for a wide range of industrial applications.

2. MATERIALS AND METHODS

2.1 Materials

Elaeis guineensis empty fruit bunch fiber (EGEFB) fiber used for this research work was obtained from Malaysian Palm Oil Board (MPOB), Bangi, Malaysia. The delignification procedure for the EGEFB fiber was taken and modified from published study by Rizal et al. [12] and Mohammad Al-Rajabi et al. [13]. The EGEFB was chemically treated by being immersed in 1%-concentrated NaOH for 2 hours with EGEFB fibre:alkaline ratio of 1:20 at room temperature. The EGEFB fiber was then washed with distilled water, oven-dried at 110 ± 5 °C for 30 minutes, grounded, and screened with mesh sieve (F1 < 10 mm) and transformed into short fiber.

2.2 Coating and Sample Preparation

Sol with three concentrations were prepared with TEOS ($Si(OC_2H_5)_4$) to ethanol ratio 1:4, 1:7, 1:10. With 30-minute-constant stirring, ethanol-water with 1:1 ratio was added to each TEOS-ethanol concentration after HCl drops were added and clear solution was obtained. PH of each sol mixture was adjusted to 2-4 pH at the end of the process by adding NaOH or HCl. The pH of the sol-gel formulation utilized for the silica coating of natural fibers was meticulously regulated within the range of 2 to 4 to modulate the hydrolysis and condensation processes, thereby guaranteeing the stability of the sol, averting the degradation of the fibers, and facilitating the attainment of a homogenous silica coating on the natural fibers.

About 0.5 g EGEFB fibers were immersed in 10 ml of the prepared sol for 30 minutes, whereby gelling occurred. The condensation reaction of NaOH-treated EGEFB fiber (cellulose-OH) and the hydrolyzed TEOS ($Si(OH)_4$) formed siloxane bond Si-O-C during immersion resulted in cellulose-O-$Si(OH)_3$. Continues condensation formed more siloxane bond, establishing silica network Si-O-Si via crosslinking. The reactions were depicted in Figure 1. The samples with TEOS to ethanol ratio 1:4, 1:7, 1:10 were denoted as S14, S17 and S110, respectively. The control sample (without sol immersion) was labelled as S00. The gel-coated EGEFB fibres were oven-dried at 80 ± 5 °C for 1 hour. The produced coated fiber samples were cooled at ambient temperature and kept for one week in individual plastic bottle before being processed for tests and characterization.

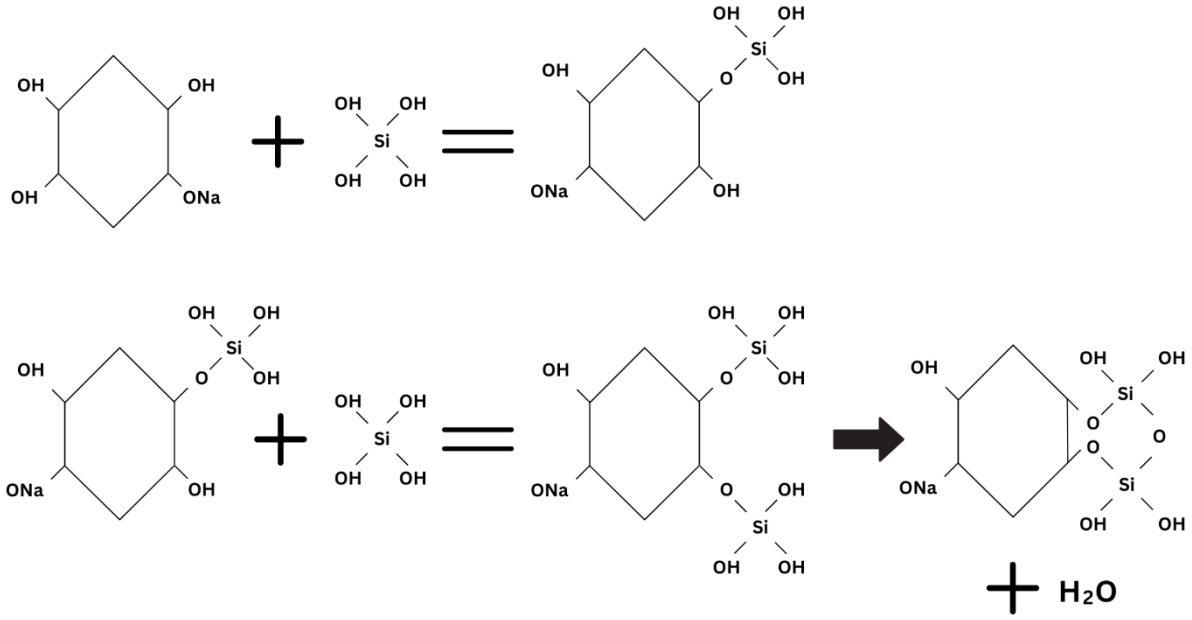

Figure 1: The reactions of the hydrolyzed TEOS and the NaOH-treated EGEFB fiber during immersion

2.3 Characterization and Testing

The samples were characterized by X-ray diffraction analysis by using Miniflex XRD, Rigaku, Japan to confirm the existence of silicon dioxide-, SiO_2 or known as silica coating on the EGEFB fiber. Energy Dispersive X-ray Spectroscopy (EDX) was employed to determine the elemental composition of the samples. The analysis was conducted using an EDX instrument, (EDAX AMETEK Elect Super 31285, USA), integrated with a FE-SEM instrument (Hitachi SU-5000, Nara, Japan). The three different concentration-TEOS coated EGEFB fiber samples, S14, S17, S110 were investigated. A thin gold layer was applied to the samples to supply electricity before scanning. The determination of silica amount and dispersion were carried out with EDX area analysis. The characteristic X-ray peaks corresponding

to different elements were identified and quantified using APEX. Four samples, (S00, S14, S17 and S110) of approximately 6 mg were used for thermogravimetric analysis by using Shimadzu (TGA-50, Japan) and were heated up in nitrogen atmosphere with flow rate of 100 ml/min and heating rate of 20 °C/min.

3. RESULTS AND DISCUSSION

3.1 Silica Coating's Existence

The X-Ray diffractogram illustrated in Figure 2 elucidated the presence of amorphous silica (SiO_2) across all three coated samples, as evidenced by the peak at 2 θ of 22.5°. This finding was in concordance with the conventional silica characteristics (JCPDS card no. 29-0085). Nonetheless, the samples revealed variations in silica intensity, with the highest weight percentage ratio of TEOS associated with the minimal presence of silica. Sample S14, which incorporated the greatest weight percentage of TEOS in the synthesized sol, exhibited the least quantity of silica on the fiber, followed in order by S17 and S110. The inverse correlation between TEOS concentration and silica presence indicated that an elevated TEOS weight percentage resulted in diminished adherence of the silica coating to the sample fibers. This phenomenon was ascribed to the formation of larger silica particles at higher TEOS concentrations, which consequently decreased the adherence of silica to the EGEFB fiber due to a reduced silica-fiber contact angle [14,15].

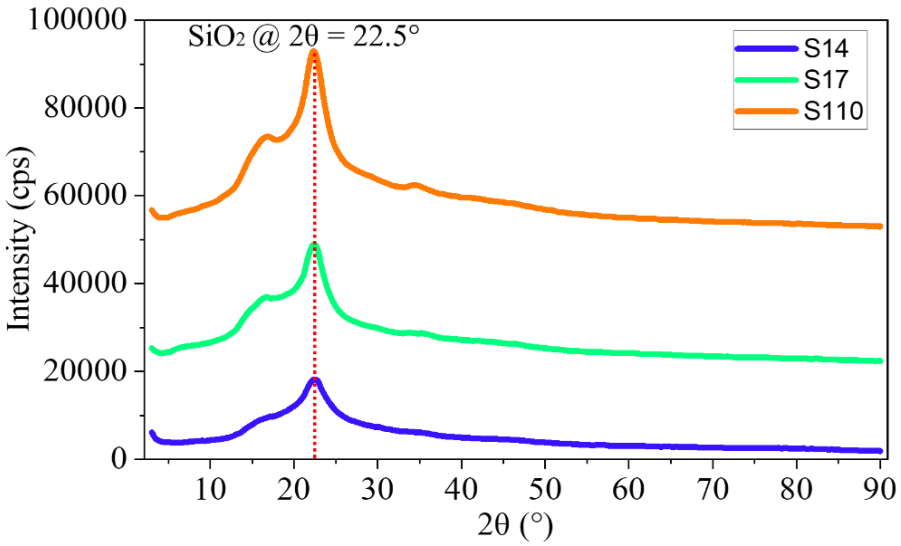

Figure 2: XRD pattern of samples's silica coating

3.2 Silica Coating's Morphology

The specimens showed in Figure 3 elucidate the morphology of the silica coating on EGEFB fibers. A decrease in the proportion of TEOS within the TEOS-ethanol mixture resulted in the acquisition of more stable silica coatings. The reference sample, designated as S00 in Figure 3(a) exhibited EGEFB fibers devoid of any coating. Sample S14 in Figure 3(b) demonstrated a resemblance to the reference sample; however, it presented salt-like silica crystals adorning its surface. As the concentration of TEOS was diminished, the formation of silica coating on the fibers became evident, as illustrated by S17 in Figure 3(c) though it contained cracks on both the fiber body and the pores. A

further reduction in TEOS concentration to a TEOS:ethanol ratio of 1:10 resulted in a smoother, crack-free coating, as depicted in Figure 3(d).

This phenomenon was ascribed to the elevated presence of larger particles in concentrated sol, which may have exhibited diminished adhesion to the substrate, consequently leading to ineffective bonding. This result is consistent with the finding of Xia et. al, which stated that decrease in TEOS concentration can lead to a smoother and more stable coating, as larger particles in concentrated sols may not adhere well to the substrate [16].

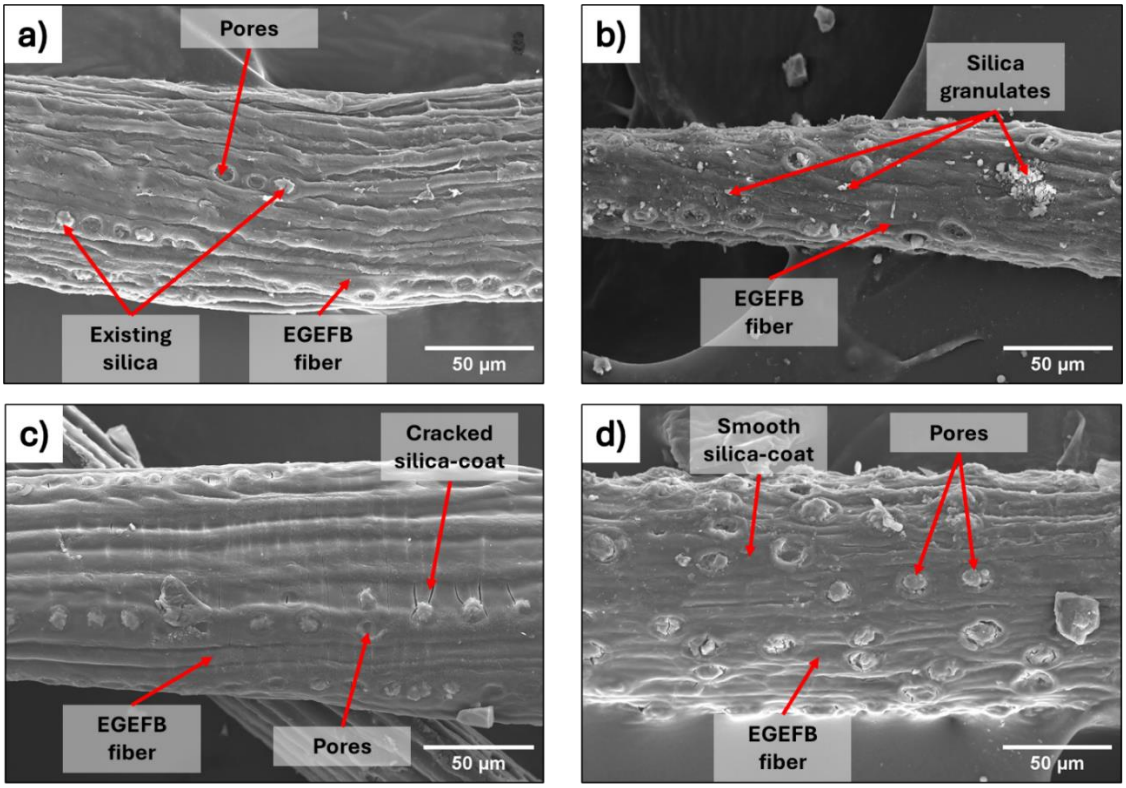

Figure 3: FESEM images of samples's silica coating (a) S00, (b) S14, (c) S17 and (d) S110

3.3 Silica Coating's Smoothness and Interaction with EGEFB Fiber

Figure 4 exhibited silica coating on the EGEFB fiber and interaction of silica-EGEFB fiber at pores. It showed the higher the TEOS concentration, the weaker the silica-EGEFB fiber interaction and the lower the silica spread. The EGEFB fiber exhibited retention of silica within certain pores, signifying that the alkaline treatment did not eliminate all silica components. This observation was corroborated by the control sample illustrated in Figure 4(a). Concurrently, Figure 4(b) illustrates the presence of silica crystals on the surface of the EGEFB fiber, which demonstrated minimal adherence to the substrate's surface, particularly at the elevated TEOS concentration. Analysis of Figure 4(c)indicated that with a TEOS: ethanol ratio of 1:7, the resulting coating exhibited enhanced smoothness, yielding a superior silica-coated EGEFB fiber in comparison to that of S14. This finding implied a moderate interaction between the coating and substrate under conditions of moderate TEOS concentration. Nonetheless, the coating was observed to exhibit cracking at the pores. At the lowest TEOS concentration, the highest degree of smoothness without cracks at existing filled and empty pores was evident in Figure 4(d) Figure 4(e), with the strongest silica-EGEFB fiber interaction observed in the S110 sample. The findings revealed that the sol at elevated TEOS concentrations displayed inadequate adherence to the EGEFB fiber, likely attributable to the poor adhesion characteristics of the larger silica particles to the fiber substrate. Furthermore, the elevated interfacial tension inherent in

more concentrated sol may have further contributed to the diminished adherence of the silica coating. The observed phenomenon of 'cracking' could be ascribed to significant shrinkage of the sol with higher TEOS concentration throughout the drying and curing phases [17].

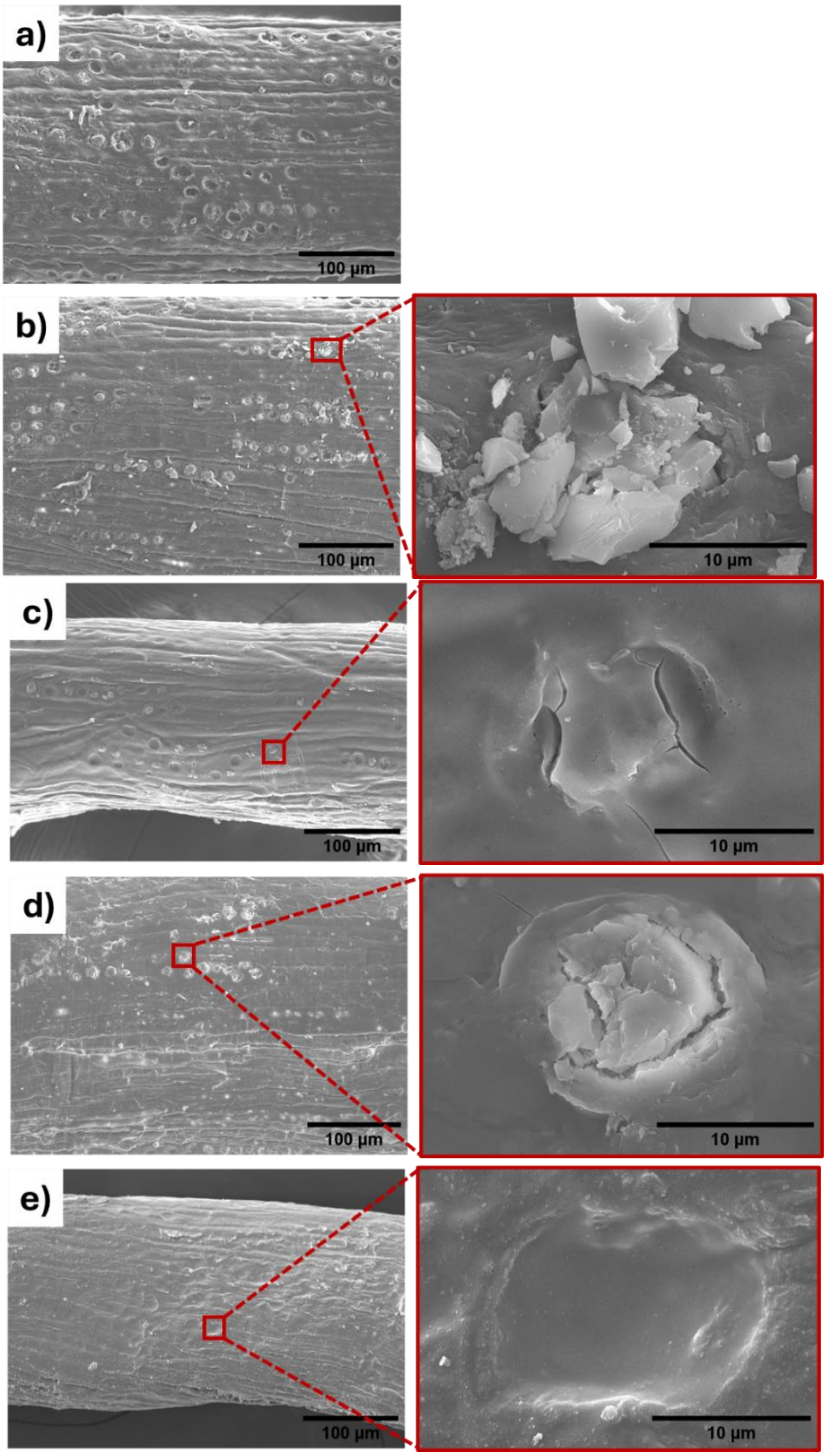

Figure 4: Silica coating smoothness and interaction with the EGEFB fiber (a) S00 - Substrate without coating (control sample), (b) S14 – Substrate with the highest concentration of TEOS, (c) S17 – Substrate with moderate concentration of TEOS, (d) S110a – Substrate with the lowest concentration of TEOS at filled pore and (e) S110b – Substrate with the lowest concentration of TEOS at empty pore

3.4 Silica Distribution on the EGEFB Fiber

Figure 5 demonstrates that the silica distribution on the substrate increased as the amount of TEOS decreased. This indicated that a lower concentration of TEOS resulted in higher silica coating dispersion on the substrate. In Figure 5(b), it can be extracted that at a TEOS-ethanol ratio of 1:4 in S14, the amount and spread of silica were identical to the naturally occurred silica at pores in uncoated sample in Figure 5(a), showing that silica did not adhere to the substrate at high TEOS concentrations. As the concentration of TEOS was reduced in S17, the silica dispersal on the substrate increased as showed in Figure 5 (c). In Figure 5 (d), S110 exhibited the highest silica adhesion at 21 wt.% with the most uniform scattering.

This phenomenon was explained by the lowest viscosity of the least concentrated TEOS, which led to even sol spreading over the EGEFB fiber, resulting in good adhesion. Additionally, the higher wettability of the lower TEOS concentration contributed to the maximum silica adherence observed in S110. The results aligned well with the findings of Watanabe et al. [18]. The process of sol spreading over a substrate was governed by the equilibrium of interfacial tensions, encapsulated in Young's equation:

$$\gamma SL - \gamma SV + \gamma LV\cos\theta = 0 \quad (1)$$

where:
γSL is the solid–liquid interfacial tension,
γSV is the solid–vapor interfacial tension,
γLV is the liquid–vapor interfacial tension, and
θ is the contact angle.

The contact angle (θ) served as an indicator of wettability. A smaller value of θ signified enhanced wetting. If the solid-liquid interfacial tension of the sol, γSL, was elevated, it implied that the sol did not spread efficiently on the substrate. This resulted in an increased contact angle, thereby indicating reduced wetting.

Therefore, a higher interfacial tension of the sol corresponded to diminished wetting over the substrate, as the sol found it more energetically favorable to minimize its contact with the substrate, leading to a larger contact angle and less spreading [17,18].

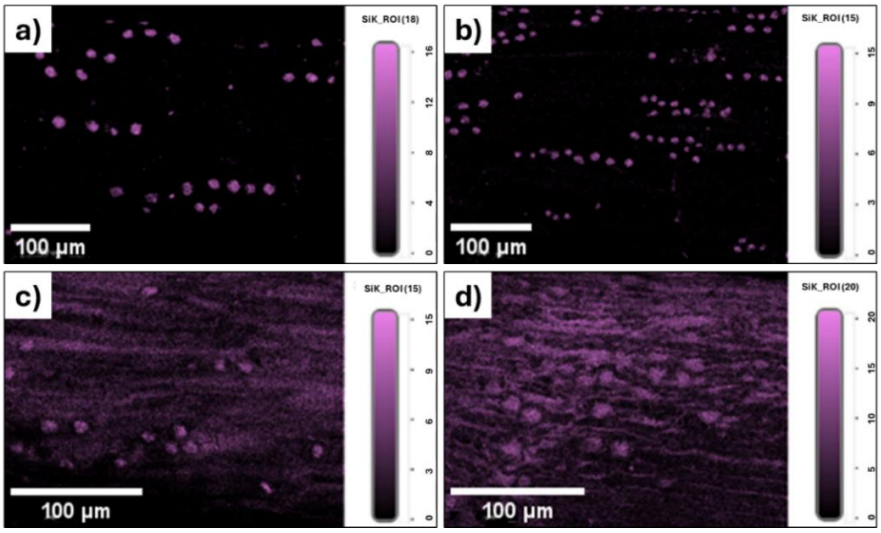

Figure 5: EDX image mapping of silica distribution on the substrates (a) S00, (b) S14, (c) S17 and (d) S110

Figure 6 denotes the silica distribution on EGEFB fibers' body at three distinct locations. The trend indicated that the presence of silica was higher and more consistent with a decrease in TEOS concentration. The silica content of the control sample, S00, was almost zero on its body. The insignificant silica amount on S00 reflected that the detected silica was the residual silica remaining after the alkaline treatment. While S14 and S17 exhibited different proportions of silica at various locations, denoting uneven silica distribution on the fiber, S110 demonstrated the highest consistency of silica weight percentage at different points. The inconsistent silica distribution of S14 and S17 were reflected by their large error bars.

The results were evident that a lower concentration of TEOS allowed the sol to spread more evenly and enabled smaller silica particles to adhere more strongly to the fiber. The lower viscosity reduced interfacial tension, and higher wettability of the lower-concentration sol, contributed to the stronger adhesion phenomenon [18].

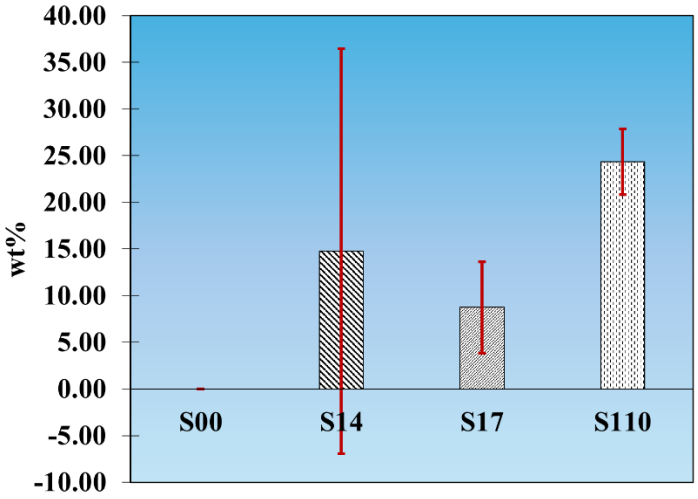

Figure 6: Average silica distribution at EGEFB fiber body of three different locations

3.5 Silica Coating's Thickness

The silica coating thickness for all samples was recorded in Figure 7. It illustrated how coating thickness responded to the TEOS concentration. A lower concentration resulted in a more homogeneous silica coating with similar thicknesses at various points. The cross-section of the control sample S00, which was uncoated EGEFB fiber, was shown in Figure 7(a).Even though it was uncoated, naturally occurring silica existed at the fiber body, which was partially removed via 1%-concentrated NaOH treatment. Meanwhile, S14 in Figure 7(b), which used the highest TEOS ratio, did not form a coating and instead formed silica crystals. With a lower TEOS ratio than S14, S17 created a thin layer of silica coating, as depicted in Figure 7(c), however, the coating cracked and did not adhere well to the fiber's surface, indicating weak coating-substrate interaction. Conversely, S110 in Figure 7(d) not only formed a silica coating with consistent thicknesses but also established stronger bonding with the irregular EGEFB fiber surface through mechanical interlocking, making the TEOS-to-ethanol ratio of 1:10 a favorable concentration.

The larger silica particle size of the S14 sol at the highest TEOS concentration led to very weak to zero adhesion of the silica particles to the EGEFB fiber, leaving large silica crystals as independent flakes on the fiber's surface. This finding was consistent with results published by Bang Zhou et al., which indicated that highly concentrated TEOS caused a rapid increase in coating thickness, leading to agglomeration in flake and floc form [19]. At a moderate TEOS concentration, S17 exhibited cracking and low adherence, possibly due to coating shrinkage during drying and curing. This behavior was attributed to the non-uniform surface tension of the silica coating during the process. The result agreed

with findings from Cheng Zhang et al. [20] who stated that the degree of cracking increased with higher TEOS concentrations. Conversely, the silica coating on S110, with the lowest TEOS concentration, locked onto the EGEFB surface mechanically due to low sol-substrate interfacial tension, increasing adhesion at a homogenous thickness approximately 3 µm. The homogeneous silica coating thickness on S110 was attributed to the low viscosity of its sol, which enabled even spreading over the substrate and effective EGEFB fiber wetting, resulting in strong coating-fiber bonding.

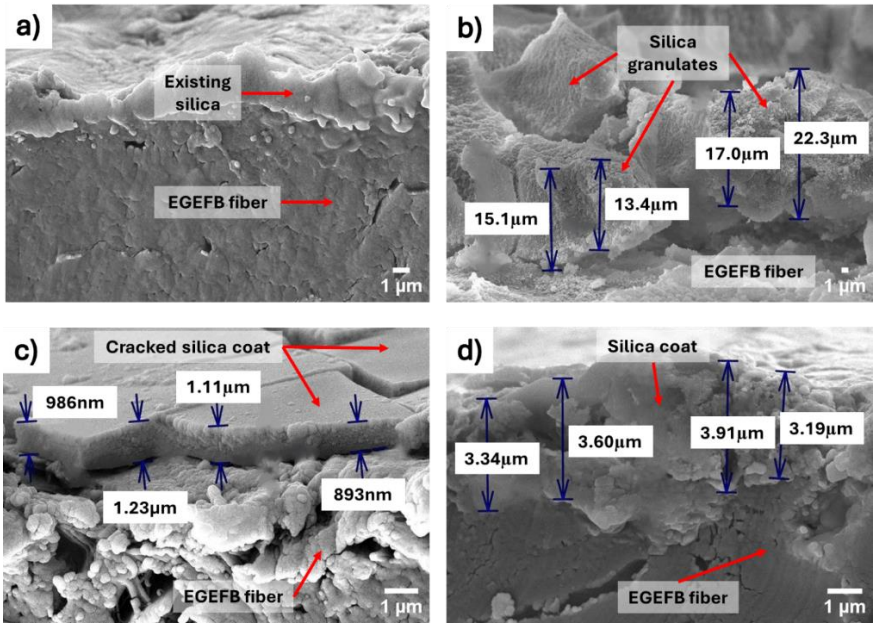

Figure 7: Silica coat thickness on the substrates (a) S00, (b) S14, (c) S17 and (d) S110

3.6 Silica Coating's Thermal Stability

Thermal stability of silica-coated EFB fiber is shown in Figure 8, whereby weight percentage reduction over time of silica-coated and -uncoated EFB-fiber at temperature rate 20 °C/min and nitrogen gas flow rate 100ml/min is depicted. From Figure 8, it can be extracted that sample S14 shows the fastest weight percentage reduction rate, followed by S17 and S110. Even though S17 and S110 show a similar slow weight decrease trend, sample S110 demonstrated a lower weight percentage reduction rate at 900 seconds and beyond. Sample S00 is the benchmarked sample of EFB fiber without silica coating.

At a rate of temperature of 20 °C/min and a nitrogen gas flow rate of 100 ml/min, silica-coated EFB fibers exhibit slower and more gradual weight reduction compared to uncoated fibers. This is due to the thermal insulation properties of silica, which delays the onset of pyrolysis and protects the organic fibers from rapid degradation. The uncoated fibers, on the other hand, degrade faster due to the absence of this protective barrier, experiencing significant weight loss as the temperature approaches the cellulose and lignin decomposition range. The final residual weight is expected to be higher for the silica-coated fibers due to the non-degradable nature of the silica at typical thermal analysis temperatures.

The correlation between thermal stability and fire-retardancy is significant, as enhanced thermal stability often contributes to improved fire-retardant properties in materials. Various studies indicate that the incorporation of specific additives can lead to both increased thermal stability and fire resistance, demonstrating a synergistic relationship between these two properties [21,22].

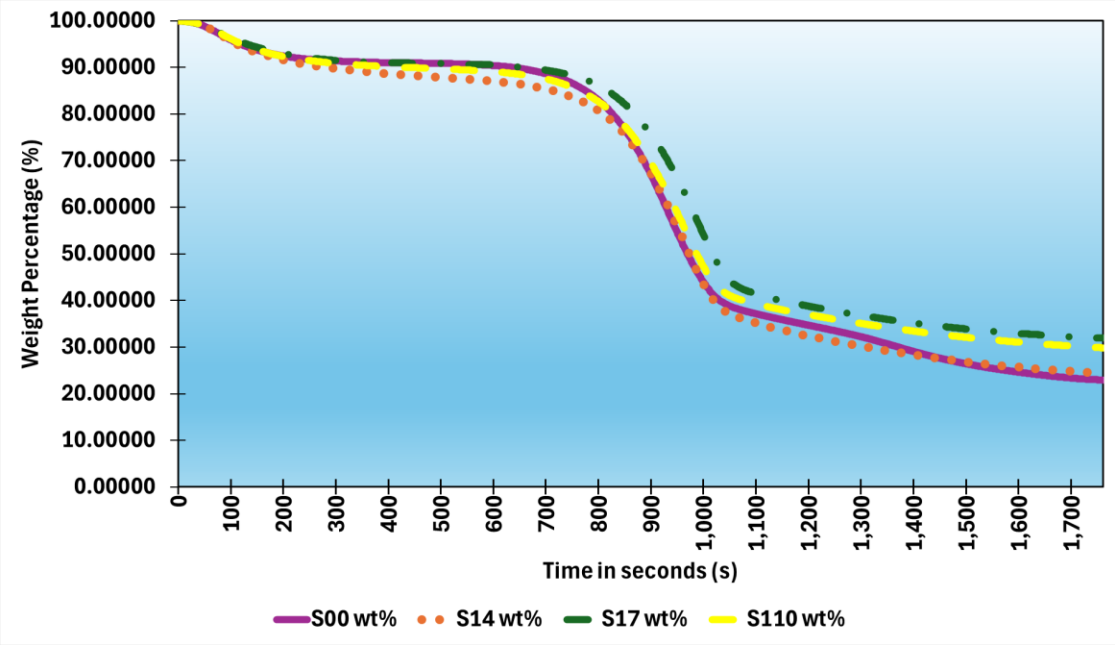

Figure 8: Weight percentage reduction over time of silica-coated and -uncoated EFB-fiber at temperature rate 20 °C/min and nitrogen gas flow rate 100 ml/min

4. CONCLUSIONS

In this study, the smoothness of the silica coating, its interfacial interaction with the fiber, the uniformity of silica distribution across different points, and the coating thickness were identified as key factors influencing the overall morphology of the silica layer on EGEFB fibers. A well-distributed silica coating enhanced the thermal stability of EGEFB fiber, thereby increasing its fire resistance for broader industrial applications. Higher TEOS concentrations in S14 and S17 resulted in increased sol viscosity, leading to higher interfacial tension, a higher contact angle, and lower wettability. Additionally, the higher TEOS content in S14 produced larger sol particles. Although some areas of the EGEFB fiber were coated in S17, the coating exhibited cracks due to shrinkage gaps during the drying and curing process. As the TEOS concentration increased, sol viscosity and interfacial tension rose, the contact angle became larger, wettability decreased, and coating adhesion weakened. In summary, a high concentration of TEOS in the coating sol led to poor silica adhesion to the EGEFB fiber surface. The optimized TEOS concentration was achieved with a TEOS-to-ethanol ratio of 1:10, which resulted in well-distributed silica on the EGEFB surface, consistent thickness, and strong silica-fiber interaction, even at the EGEFB fiber pores. This work also found that an effective silica coating ensures higher thermal stability of EGEFB fiber, as demonstrated by S110. The TEOS-to-ethanol ratio of 1:10 was identified as suitable for sol-gel preparation of silica coating, as it established the highest thermal stability amongst all samples, hence enhancing the fire-resistance potential application of EGEFB fiber for industrial use.

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Author Contributions

All authors contributed toward data analysis, drafting and critically revising this paper and agree to be accountable for all aspects of the work.

Disclosure of Conflict of Interest

The authors have no disclosures to declare.

Compliance with Ethical Standards

The work is compliant with ethical standards.

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