Effect of Stearic Acid Coating on the Properties of Bio-Epoxy Unidirectional Flax Fiber Composites

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1. Abstract

Natural fiber composites are increasingly investigated as sustainable alternatives to synthetic fiber systems, but their performance is often limited by poor fiber-matrix adhesion and moisture sensitivity. In this study, stearic acid surface treatments were used to modify the fiber-matrix interface. Flax fiber mats were soaked in a 6 wt. % stearic acid solution for 0.5, 1, and 2 h, then processed into 7-layered unidirectional laminates using a bio-based epoxy resin and vacuum-assisted resin infusion. Scanning electron microscopy (SEM) was used to observe the fiber surfaces before and after treatment. Fourier transform infrared spectroscopy (FTIR) was used to characterize the fibers with and without stearic acid treatment. Tensile tests were carried out to assess the influence of stearic acid soaking durations on the strength and stiffness of the composites. Water absorption tests were performed on untreated and stearic acid treated flax mats (without bio-epoxy). SEM showed the fiber surface was modified after stearic acid treatments. FTIR confirmed the chemical modification did not change the overall structure of the flax fibers and that stearic acid was successfully loaded onto the fibers. The untreated bio-epoxy/flax fiber composite had a tensile strength of 132 MPa and a tensile modulus of 4.2 GPa. Although the tensile strengths decreased for all stearic acid treatments, the tensile modulus reached a value of approximately 4.7 GPa with a 0.5 h treatment, but reduced to about 3.7 GPa at 2 h, suggesting that prolonged soaking may begin to over-modify or degrade the fiber surface. Untreated flax mats exhibited an average water absorption of approximately 589% and the lowest water uptake was observed at a 1 h soak duration reaching 312%. The study aims to clarify how stearic acid coating influences the mechanical and moisture absorption properties of unidirectional bio-epoxy/flax fiber composites.

2. Introduction

Fiber reinforced polymer composites (FRPC) containing synthetic fibers such as carbon, glass and aramid along with synthetic thermoset matrix (epoxy) are used in high performance applications

in the aerospace and automobile industries where high strength and stiffness are required. There has been an interest to use natural, sustainable materials in applications where low to moderate strengths are needed. Natural plant fibers offer a possible alternative, especially flax, as they are lightweight, biodegradable, with moderate mechanical properties. Bio-based epoxy can be derived from renewable resources such as vegetable oil, cardanol, rosin, sorbitol and lignin [1].

Unidirectional flax fiber reinforced bio-epoxy composites are particularly promising. Aligning the fibers in the load direction improves tensile and flexural performance, which makes them suitable for structural applications. However, one of the main limitations is poor bonding between the flax fibers and the epoxy matrix. Flax is naturally hydrophilic, while most epoxy resins are hydrophobic. This mismatch leads to weak adhesion, fiber pull-out, and poor load transfer [2].

To improve this interface, surface treatments have been explored on natural fibers. One option is stearic acid, a long-chain fatty acid that can reduce the hydrophilicity of the fiber surface and improve compatibility with the matrix. Tazwar et al. [3] highlighted that stearic acid treatment improved bonding and reduced moisture uptake, which led to better mechanical performance in thermoplastic based composites. These improvements are especially important when considering long-term durability. For example, Moudood et al. [4] showed that water saturation can reduce tensile and flexural properties of flax bio-epoxy composites by more than 50 %. This emphasizes the need to control moisture uptake through surface modifications. Stearic acid concentration and treatment immersion time are inter-related for achieving property improvements. For example, Kiattipanich et al. [5] treated bagasse fibers with 3, 5, 7 and 9 wt. % stearic acid for a 45 min treatment. They determined that the polypropylene composites containing 9 wt. % stearic acid had the least water absorption and the best tensile strengths, tensile modulus and impact strengths. More recently, Nayak et al. [6] soaked flax fibers in a 3 wt. % stearic acid ethanol solution for 24h and 36h. They found the tensile strength of the flax fibers significantly improved for the 36h treatment. Additional studies are needed for the use of stearic acid as a surface modification treatment of flax fibers to determine the optimum parameters for improved bio-epoxy composite properties.

This study evaluates how a stearic acid coating affects the mechanical and physical properties of unidirectional flax fiber reinforced bio-epoxy composites produced using the vacuum infusion process. The goal is to determine whether this surface treatment improves fiber–matrix adhesion, enhances tensile properties, water uptake and supports the development of sustainable composite materials for various applications.

3. Experimental

3.1. Materials

The resin was a bio-based infusion epoxy with a 28% renewable carbon content obtained from NERPA™ Polymers Inc. The unidirectional (UD) woven flax fiber fabric (300 g/m^2) was obtained from Bcomp®: AmpliTex™ 5009. Stearic acid (97% purity) flakes were purchased from Fisher Scientific. To manufacture the pure epoxy tensile specimens, a silicone-based rubber mold, Mold Max 10T from Sculpture Supply Canada was prepared for each specimen dimension.

3.2. Stearic acid coating preparation

Prior to stearic acid (SA) treatment, the flax fiber mats (300 mm x 250 mm) were dried in an oven at 60°C for 1 h. To prepare a 6 wt. % solution, about 25 g of stearic acid was dissolved in 500 ml of ethanol using a magnetic stirrer with hot plate set at 70°C. The flax fiber mats were deposited into a glass tray containing the ethanol/stearic acid mixture and allowed to soak for 0.5, 1 and 2 h (at 70°C). The treated mats were removed and carefully washed with distilled water to remove surplus stearic acid. The flax mats were oven dried at 80°C for 24 h.

3.3 Preparation of composites

Pure bio-epoxy samples had a mix resin:hardener ratio of 2.50:1 (by weight). The mixture was degassed under vacuum (>28 inHg) for 30 min to evacuate air bubbles generated due to mixing and slowly poured into silicone rubber molds. The composites were prepared by vacuum infusion as shown in Figure 1. To obtain a 3 mm thickness, 7 mats, $[0°]_7$ (300 mm x 250 mm) were layered with parallel fiber alignment using about 350 g bio-epoxy and 140 g of hardener. Both were cured for 48 h at 22°C and then post-cured at 82°C for 8 h. Tabs were bonded to the laminates with West System 610 adhesive and cut into rectangular geometries of 250 mm x 25 mm x 3 mm (l x w x t) for tensile testing.

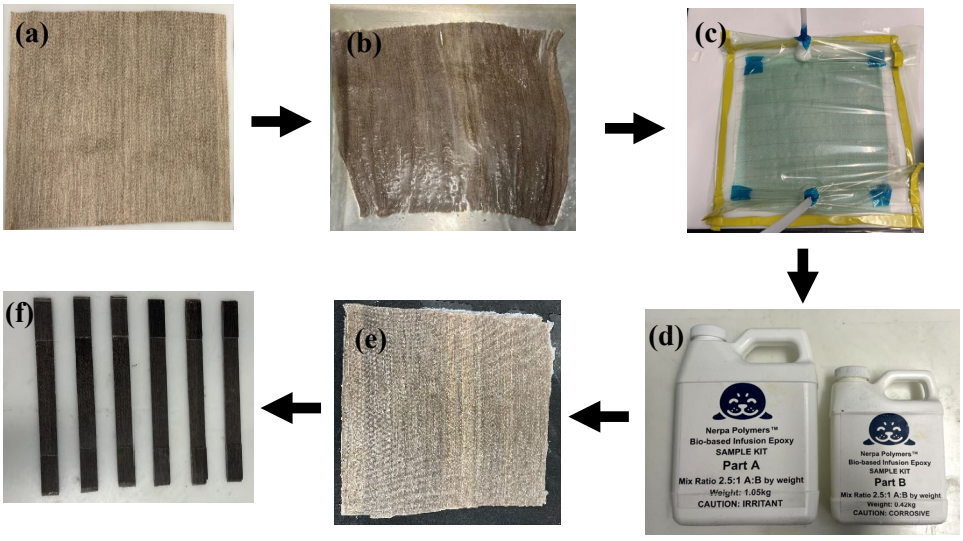

Figure 1: Digital image of (a) flax fiber fabric, (b) stearic acid treatment, (c) vacuum infusion process, (d) bio-epoxy and hardener, (e) cured tensile laminate and (f) tensile samples with tabs.

3.4 Treated and untreated flax fiber assessment

Chemical composition (cellulose, hemicellulose, and lignin contents) of the flax fibers were determined using Ankom 200 fiber analyzer (Macedon, NY, USA) in triplicates and averaged. The acid detergent fiber (ADF) and neutral detergent fiber (NDF) was determined using Ankom Method 5 [7] and Ankom Method 6 [8], respectively. The % lignin was determined using acid detergent (ADL) ANKOM Method 8 [9], equation 1. The % cellulose and % hemicellulose were calculated using equation 2 and 3, respectively.

$$\%\ lignin\ =\ ADL \tag{1}$$

$$\%\ cellulose\ =\ ADF\ -\ ADL \tag{2}$$

$$\%\ hemicellulose\ =\ NDF - ADF \tag{3}$$

3.5 Scanning electron microscopy

To view the effect of stearic acid treatment on the fiber surfaces, an environmental scanning electron microscope (ESEM) (model Quattro, Thermo Scientific, Waltham, MA, USA) was used. A thin layer of gold was applied to prevent charging.

3.6 Fourier infrared spectroscopy

Surface functional groups were identified by Fourier transform infrared (FTIR) spectroscopy (PerkinElmer Spectrum Two, Massachusetts, USA) in the range 4000-500 cm^{-1} with a resolution of 4 cm^{-1} and 32 scans.

3.7 Mechanical tests

Pure bio-epoxy tensile test were conducted according to ASTM D638-14, Type I with dimensions of 165 x 13 x 3.2 mm^3 (l x w x t). Flax fiber and bio-epoxy flax composites were tested using the guidance of ASTM D3039-17 and measured 250 x 25 x 3 mm^3 (l x w x t). Both tests were carried out using an Instron (model 5967, Canton MA, USA) equipped with a 30 kN load cell and a cross-head speed of 2 mm/min. Five samples of each were tested and averaged.

3.8 Water absorption

Untreated and SA treated flax fiber mats with a geometry of 20 mm x 20 mm x 3 mm (l x w x t) were submitted to a 28 day water absorption test in distilled water at 22°C until equilibrium was reached using the guidance of ASTM D570-22. The samples were initially dried (w_2) and after the 28 day period immersion time, the mats were removed from the beaker, blotted with paper towel to remove surface moisture and weight was taken (w_1). The weight of water absorption (%) was calculated using equation 4. Three replicates were measured and averaged for each batch.

$$Water\ absorption\ (\%) = \frac{w_1 - w_2}{w_2}\ x\ 100 \tag{4}$$

4 Results and discussion

4.3 Flax fiber chemical analysis

The flax fiber on a dry basis consisted mainly of cellulose (70.4 %) with minor amounts of hemicellulose (12.1 %) and lignin (5.6 %). This is similar to the results of flax fibers where the content of cellulose, hemicellulose and lignin was reported to be 75.1 %, 7.1% and 1.9 % [10]. The difference could be due to the plant growing conditions such as water, sunlight, soil type and the variety of plant species.

4.4 Scanning electron microscopy (SEM)

The surface morphology of the flax fiber surface before and after SA treatment was observed using the scanning electron microscope (SEM). As shown in Figure 2a, the surface of the untreated fibers had smooth surfaces which contains a waxy layer as has been observed elsewhere [6]. Figure 2b, c and d show a progressive roughness of the fiber surface and additional stearic acid on the fibers as the stearic acid soak time was increased. After the stearic acid treatments, the weight percentage gain was calculated based on the dry weight of starting untreated fabrics and the dry weight of the post treated fabrics. The flax fibers soaked in a 6 wt. % SA solution for 0.5, 1 and 2 h increased in weight by 6.37, 8.16 and 16.92 wt. %, respectively.

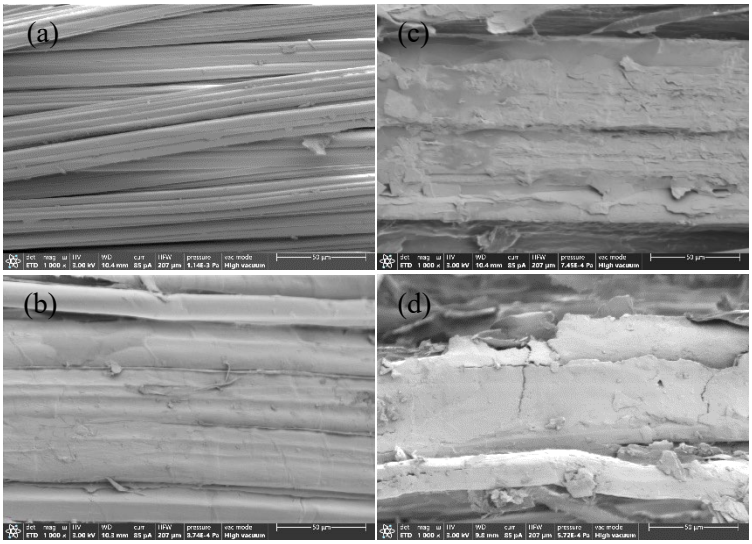

Figure 2: SEM of the flax fiber fabric (a) untreated flax fiber, (b) flax fibers treated with 6% SA-0.5 h, (c) flax fibers treated with 6% SA-1 h and (d) flax fibers treated with 6% SA-2 h, at 1000 x.

4.3 Fourier transform infrared (FTIR) spectroscopy

Figure 3 shows the FTIR spectra of stearic acid (SA), untreated (FF) and stearic acid treated flax fibers (SA 6 wt. % for 0.5 h, 1 h and 2 h.). For stearic acid, distinct peaks are observed at 2954, 2914 and 2847 cm^{-1} and belong to methylene (CH_2) and methyl (CH_3) groups found in stearic acid. The peak at 1472 cm^{-1} is associated to CH_2. The peaks at 1699, 1297 and 941 cm^{-1} are attributed to the carboxylic acid (-COOH) group from stearic acid while 719 cm^{-1} represents a long-chain linear methylene structure in stearic acid [11]. The untreated flax has a broad band at 3334-3290 cm^{-1} which represents cellulose, hemicellulose, lignin and pectin which decreased as the stearic acid treatment time increased and eventually disappeared. The small peaks for untreated flax around 2851-1918 cm^{-1} are associated with cellulose, hemicellulose, lignin, pectins, waxes, and fats, while the peaks detected at 1186-917 cm^{-1} are also for cellulose, hemicellulose and pectin [12]. The peaks at 1186-917 cm^{-1} are reduced in magnitude as the stearic acid hold time increased. However, the peaks at 2954, 2914, 2847, 1699, 1472, 1297, 941 and 719 cm^{-1} increased in their intensity for the flax fiber treated with stearic acid as the soak time increased. FTIR tests confirmed that the chemical modification did not change the overall structure of the flax fibers and that stearic

acid were successfully loaded onto the fiber. This is also corroborated by the SEM results in Figure 2.

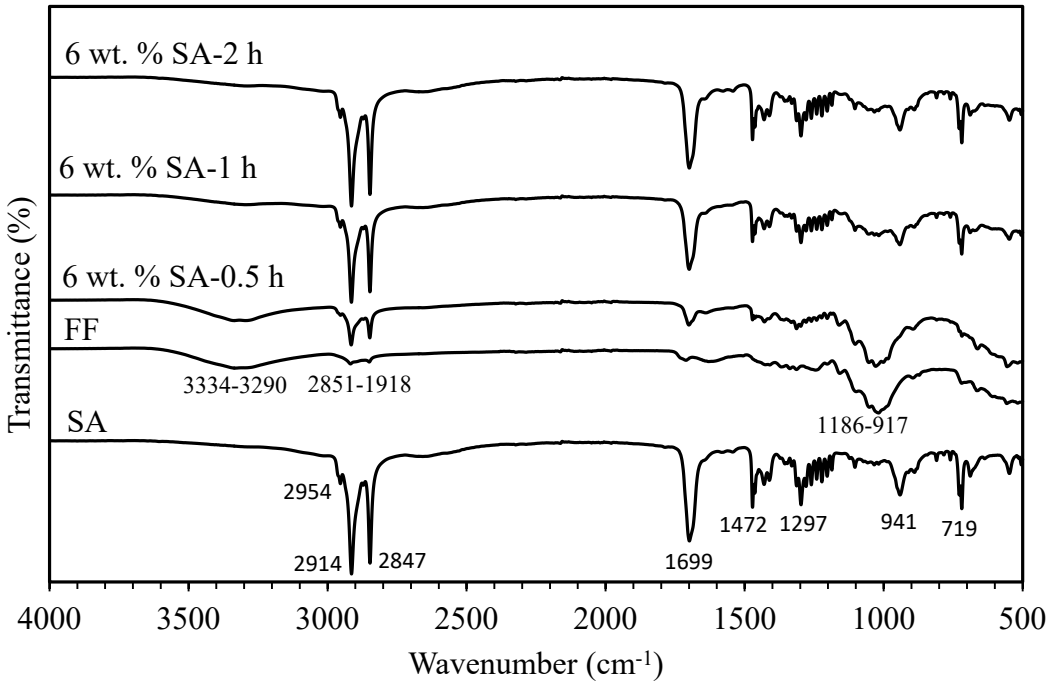

Figure 3: FTIR spectra for (a) stearic acid (SA), (b) flax fiber fabric (FF), (c) flax fiber fabric treated with 6 wt. % SA-0.5 h, (d) flax fiber fabric treated with 6 wt. % SA-1 h and (e) flax fiber fabric treated with 6 wt. % SA-2 h.

4.4 Mechanical properties

Representative stress-strain curves for the pure bio-epoxy (BE), bio-epoxy untreated flax fabric (BE/FF) composites and bio-epoxy composites containing flax fibers treated with 6 wt. % stearic acid for 0.5, 1 and 2 h soak time are shown in Figure 4. All composites contained 7-layers $[0°]_7$. The composites showed a non-linear behaviour with two distinct slopes as has been previously reported for an epoxy unidirectional flax fiber composite [13]. The first slope occurs at the initial phase of deformation from 0% to about 0.1% strain and a transition to a second slope at a range of about 0.1% to failure. At the beginning, there is elastic stretching of both the cured bio-epoxy matrix and the flax fibers [14]. As the load is increased, reorientation of the helically aligned cellulose flax plant microfibrils with the tensile axis occurs [15]. Further increasing the load, results in matrix yielding [14] and elastic response of the aligned microfibrils [15] and finally fracture. The second slope is lower than the first slope. This phenomenon is not observed for pure bio-epoxy.

The tensile property results of pure bio-epoxy (BE), untreated flax fabric (FF) bio-epoxy untreated flax fabric composites (BE/FF) and the bio-epoxy composites containing 6 wt. % SA treated flax fibers for 0.5, 1 and 2 h soak time are shown in Figure 5. The pure bio-epoxy tensile property results were slightly lower possibly due to porosity in the samples. Compared to the untreated BE/FF composites, the flax fiber fabric treated with stearic acid for 0.5, 1 and 2 h decreased the composite tensile strengths by 18, 25 and 51%, respectfully. However, the modulus improved by 13% and 7% for 0.5 and 1 h soaking, respectively but thereafter decreased by 10% for the 2 h soak

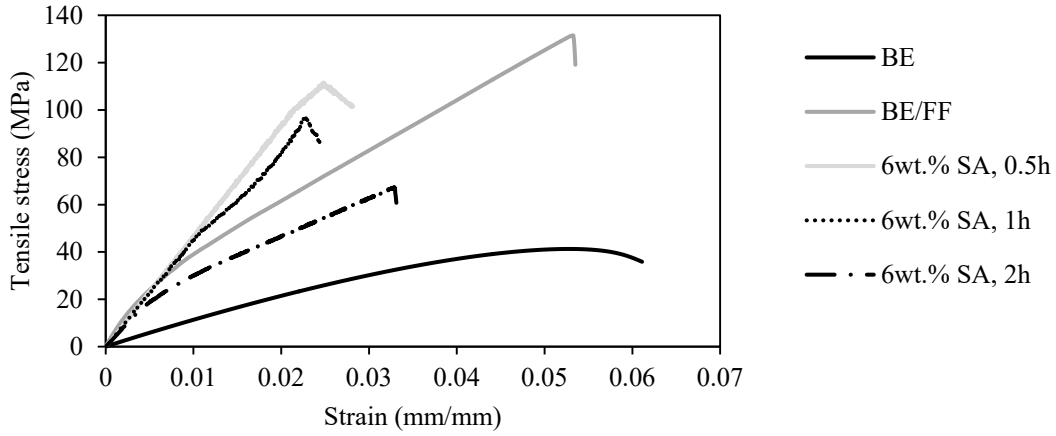

Figure 4: Typical stress-strain curves for BE, seven layers $[0°]_7$ BE/FF, and seven layers$[0°]_7$ flax fabric treated with 6 wt. % SA for 0.5 h, 1 h and 2 h soak time.

time. The composites containing flax fibers treated with 6 wt. % for 0.5 h demonstrated a higher tensile strength and modulus compared to the other treatment times. The improvement may be due to an enhanced stress transfer from the bio-epoxy matrix to the stearic acid treated flax fiber. However, a decrease in properties may be ascribed to the limitations of the cohesive strength of the stearic acid coating itself on the fiber surface as a result of longer treatment times [16]. A thicker, flakier coating is observed in Figure 2d where the soaking time was for 2 h. According to the FTIR spectra, the peak intensity was highest for the flax fiber fabrics held in the stearic acid solution for 2 h. Longer soak times at 6 wt. % concentrations led to a reduction in the benefit of a SA coating. However, longer soak times at lower concentrations may have positive outcomes. For example, Nayak et al. [6] highlighted that since stearic acid is a weak acid and does not degrade the fiber surface to a great extent, when using a 3 wt. % stearic acid solution a longer treatment (36 h compared to 24 h) maybe be required to improve the tensile strengths of flax fibers.

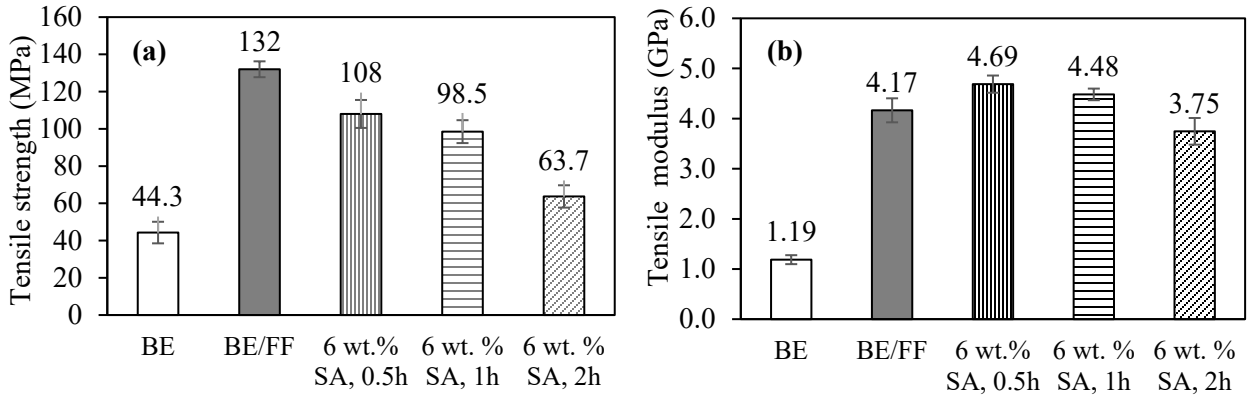

Figure 5. Effect of stearic acid (SA) fiber surface treatment on (a) tensile strength and (b) tensile modulus of bio-epoxy and bio-epoxy composites.

The theoretical volume fraction of the BE/FF composites were calculated for one laminate thickness based on the rule of mixtures (ROM) using equation 5 which assumes a pore free composite.

$$V_f = \frac{(\rho_m \cdot W_f)}{(\rho_m \cdot W_f + \rho_f \cdot W_m)} \quad (5)$$

where, V_f is the volume fraction of fibers, ρ_m is the density of matrix, W_f is the weight of dry fibers, ρ_f is the density of fibers, and W_m is the weight of matrix. For a fiber weight fraction (mass of the fibers/total mass of the composite) of 0.52, the volume fraction obtained was 0.41. This is an estimated value since flax fibers generally have a hollow core known as the lumen which does not fill with epoxy. Therefore, the weight of the dry fibers is not exact. In a related study, epoxy unidirectional flax fiber composites were manufactured using the resin transfer molding (RTM). Four fiber weight fractions of 0.26, 0.37, 0.36 and 0.54 were evaluated where the calculated volume fractions obtained were 0.21, 0.32, 0.42 and 0.47, respectively [17]. Flax fiber walls tend to absorb resin which reduces the amount of resin available to fill the spaces between the fibers resulting in reduced packing and lower volume fiber fractions in the range of 10-40%. At low fiber volume fractions, the composites exhibited a reduced strength and stiffness as the load-carrying capacity is controlled by the weaker matrix rather than the high-strength fibers.

4.5 Water absorption

As shown in Figure 6, the untreated flax fabric (FF) had the highest water absorption due to their hydrophilic (affinity for water) nature in part from the high cellulose content of 70.4%. The cellulose contains hydroxyl groups (-OH) which interact with water. The water absorption decreased for flax fibers treated with stearic acid at time intervals of 0.5 and 1 h where the least amount of water absorption was obtained for the 1 h. When the soak time increased to 2 h, a higher absorption was observed possibly due to a non-uniform coating on the fiber surface. A study showed a thicker stearic acid coating formed a less homogeneous layer [18] which can be less effective at sealing the fibers as can be seen in Figure 2d. An alternative stearic acid coating technique would use a vapor deposition method which can produce a more uniform coating than the solvent method. Overall, the flax fibers treated with stearic acid retained less water compared to the untreated fiber. This can be attributed to the stearic acid treatment as it is hydrophobic (repels water) as well as reduces the number of hydroxyl groups available for bonding with water molecules.

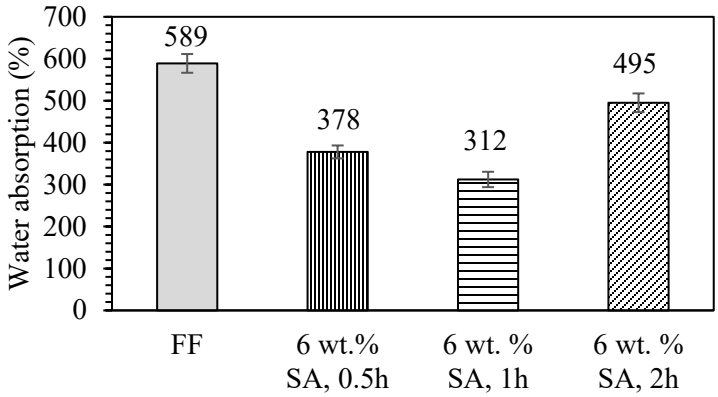

Figure 6: Effect of stearic acid treatment of flax fiber fabric on water absorption.

5 Conclusions

Stearic acid was deposited onto unidirectional flax fiber fabrics before adding them to a bio-epoxy resin. From SEM, stearic acid weight percentage gains and FTIR results it can be concluded that stearic acid was present as a thin layer on the flax fiber surfaces. The stearic acid concentration and treatment time limited tensile strengths, but improved the tensile modulus for the flax fibers treated with 6 wt. % SA for 0.5h. The stearic acid treatment reduced the water absorption the most for flax fibers treated with 6 wt. % SA for 1 h, and was slightly higher for flax fibers treated with 6 wt. % SA for 0.5h. Future work is to decrease the stearic acid concentration to between 3-4 wt. % and determine the appropriate stearic acid/ethanol treatment soak time.

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