

## ***Surface modification of cellulose using silane coupling agent***

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## ABSTRACT

Recently there has been a growing interest in substituting traditional synthetic polymers with natural polymers for different applications. However, natural polymers such as cellulose suffer from few drawbacks. To become viable potential alternatives of synthetic polymers, cellulosic polymers must have comparable physico-chemical properties to that of synthetic polymers. So in the present work, cellulose polymer has been modified by a series of mercerization and silane functionalization to optimize the reaction conditions. Structural, thermal and morphological characterization of the cellulose has been done using FTIR, TGA and SEM, techniques. Surface modified cellulose polymers were further subjected to evaluation of their properties like swelling and chemical resistance behavior.

**Key Words:** *Natural cellulose; surface modification; thermal; morphological and physico-chemical properties.*

## 1. Introduction

Synthetic polymers have been found to play an important role in a number of applications(Zhou et al., 2013)(Ding et al., 2013)(Chen & Gao, 2007b). These polymer have been used as diferent components in the energy storage systems starting form electrolyte to dielectric capacitors for energy storage applications(Chen & Gao, 2007a)(Lin & Lee, 2013)(Lin, Thakur, Tan, & Lee, 2011a). However, during the last few decades, environmental friendly natural polymers have attracted greater attention all over the world due to the increasing environmental awareness as well as the depletion of petroleum based resources (Banerjee et al., 2013)(Bharti, Mishra, & Sen, 2013)( Thakur, Thakur, & Gupta, 2014)( Thakur, Thakur, & Gupta, 2013a). Due to their inherent properties such as renewable nature, biodegradability, economic/ low cost, easy availability, natural polymers are emerging as viable alternatives to traditional petroleum-based materials (G. U. Rani, Mishra, Sen, & Jha, 2012)( Thakur, Thakur, & Gupta, 2013c)(Basta, El-Saied, El-Hadi, & El-Dewiny, 2013). Among various types of natural polymers, cellulose based polymers are the most abundant natural materials available in earth(Mishra, Rani, & Sen, 2012)( Thakur, Thakur, & Gupta, 2013b). Different types of biopolymers ranging from algae to natural cellulosic fibers can be the best option to serve the current energy needs (Banerjee et al., 2013)(Jawaid & Khalil, 2011)(Sabo, Basta, & Winandy, 2013). The effective use of biorenewable cellulosic polymers, which have been shown to have some promising properties comparable to traditionally used synthetic polymers can successfully reduce the use of petroleum resources(P. Rani, Sen, Mishra, & Jha, 2012)(Dhakal, Zhang, Guthrie, MacMullen, & Bennett, 2013)(Thakur, Singha, & Thakur, 2011). Natural cellulosic polymers such as cellulosic fibers are typically derived from different types of plants, grasses and

various other resources(Thakur, Singha, & Thakur, 2013a). Cellulose fibers exhibit the potential to replace traditional synthetic fibers as reinforcement in the production of cellulose-fiber-reinforced green composites(Khoathane, Vorster, & Sadiku, 2008)(Thakur, Singha, & Thakur, 2013b)(Thakur, Singha, & Thakur, 2012). These cellulosic composites are being envisioned as one of the growing sectors in the composites industry due to their lower density, high specific modulus/ strength, and low economic cost than synthetic fibers reinforced composites(Dhakal, Zhang, Reis, Surip, & Bennett, 2012)(Khalil et al., 2013).

Among various cellulosic fibers *Eulaliopsis binata fibers* possess a strong potential to be used as reinforcement in composites(Thakur, Thakur, & Gupta, 2013c)(Thakur, Thakur, & Gupta, 2013e) (Thakur, Thakur, & Gupta, 2013f). These fibers can be used to prepare lightweight composites and in many other applications. However, the hydrophilic nature of these cellulosic fibers limits their applications as it results in poor chemical as well as moisture absorption resistance (Thakur, Thakur, & Gupta, 2013c). These properties in the long term, affect the overall properties of the composite materials. So in this preliminary work, we have performed the surface modification of cellulosic *Eulaliopsis binata fibers through a series of mercerization and silane functionalization*.

## 2. EXPERIMENTAL

### 2.1 Materials and methods

Natural Cellulosic *Eulaliopsis binata* fibers used in this preliminary work were obtained from local resources of Himalayan region. Prior to use, these fibers were purified as per standard method(Thakur, Thakur, & Gupta, 2013c)(Thakur, Thakur, & Gupta, 2013e). In brief, these fibers were initially soaked in a detergent solution for specific time intervals and subsequently extensively washed with tap water. Chemicals namely, ethanol, acetone, sodium hydroxide (NaOH), and vinyltrimethoxysilane were used as received.

The surface modification of the *Eulaliopsis binata* fibers was initiated by first carrying out the mercerization of these fibers(Singha et al., 2009). Mercerization is frequently referred as the pretreatment process in the modification of biopolymers(Singha & Thakur, 2009a). In the mercerization process, cellulosic *Eulaliopsis binata* fibers were immersed in pre-deminized (2%) concentration of NaOH solution for series of time intervals at room temperature for the optimization of time. Subsequently, these fibers were thoroughly washed with distilled water for removal of any NaOH content, followed by neutralization with 2 % acetic acid and excess washing with water(Singha & Thakur, 2009b). After the completion of this reaction step, these fibers were dried in an oven at 70 °C to a constant weight. After the optimization of time, the effect of different concentration of NaOH solutions (2-14%) at the optimized time period was also studied. The optimization of time and concentration of NaOH ensures that cellulosic fibers are ready for modification with silane treatment(Thakur, Singha, Kaur, Nagarajarao, & Liping, 2010).

Surface functionalization of the *Eulaliopsis binata* fibers was done using vinyltrimethoxysilane as the coupling agent. The mercerized *Eulaliopsis binata* fibers were dipped in an ethanol/water mixture in the ratio (60:40) with optimized concentration (5 %) of the coupling agent for 2 h. In order to ensure efficient coating of vinyltrimethoxysilane coupling agent on the fibers, the pH of the solution was maintained between 3.5 and 4, using the METREPAK Phydriion buffers(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b). After the predetermined time interval, the ethanol/water mixture was drained out. The *Eulaliopsis binata* fibres were then dried in air and then in an oven at 70 °C to a constant weight(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b).

## 2.2 Structural and physico-chemical characterization

FT-IR spectra of the pristine and vinyltrimethoxysilane treated *Eulaliopsis binata* fibers taken using KBr pellets on PERKIN ELMER RXI Spectrophotometer in the range of 400-4000  $\text{cm}^{-1}$ . The surface morphology of the pristine/ silane treated *Eulaliopsis binata* fibers was studied Scanning electron microscopic (SEM), while the thermal (TGA) behaviors was were studied with the help of a thermal analyzer (Perkin Elmer) at a heating rate of 10 °C/ min by taking 10mg weight for each sample in nitrogen atmosphere(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b). The effect of silane coupling agent on the swelling/ water uptake behavior of the pristine/ silane treated *Eulaliopsis binata* fibers was studied using the standard method in different solvents e.g. water, methanol, and isobutanol(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b). The percent swelling behaviors of the pristine/ vinyltrimethoxysilane treated *Eulaliopsis binata* fibers was determined in the following manner(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b):

$$\text{Percent swelling } (P_s) = \frac{W_f - W_i}{W_i} \times 100$$

Where  $W_f$  = Final weight of the *Eulaliopsis binata* fibers

$W_i$  = Initial weight of the *Eulaliopsis binata* fibers

The effect of silane coupling agent on the chemical resistance behavior of the pristine/ silane treated *Eulaliopsis binata* fibers was studied as per the method standard method in the following manner (Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b):

$$\text{Percent chemical resistance } (P_{cr}) = \frac{T_w - W_{aci}}{T_w} \times 100$$

Where  $T_w$  = total weight of the *Eulaliopsis binata* fibers

$W_{aci}$  = weight after certain interval *Eulaliopsis binata* fibers

### 3. Results and Discussion

Surface modification of materials procured from different natural and synthetic resources through different treatments is one of the facile method to obtain the materials with desired properties (Dhakal, Zhang, & Bennett, 2012) (Lin, Thakur, Tan, & Lee, 2011b) (Pilla, Gong, O'Neill, Rowell, & Krzysik, 2008). There is some existing literature available on the effect of surface treatment on the mechanical properties of polymer composites but limited information is available on the thorough study of effect of vinyltrimethoxysilane treatment on the

physicochemical properties of the *Eulaliopsis binata fibers* (Pilla, Gong, O'Neill, Yang, & Rowell, 2009) (Maheswari, Reddy, Muzenda, Shukla, & Rajulu, 2013). So in this work we have studied the effect of vinyltrimethoxysilane treatment on the physico-chemical properties such as water absorption, water uptake and chemical resistance of the *Eulaliopsis binata fibers*. The hydroxy groups present in *Eulaliopsis binata fibers* backbone have been found to responsible for the water/ moisture absorption (Thakur, Thakur, & Gupta, 2013c) (Thakur, Thakur, & Gupta, 2013e). These functional groups also play a key role in the surface modification of *Eulaliopsis binata fibers* as these are consist of linear glucan chains with repeating (1→4)  $\beta$ -glucopyranose units (Pinto, Chalivendra, Kim, & Lewis, 2014) (Pinto, Chalivendra, Kim, & Lewis, 2013) (Thakur, Thakur, & Gupta, 2013c) (Thakur, Thakur, & Gupta, 2013e). Mercerization is one of the best method to remove some of the impurities from the surface of the natural cellulosic fibers. In the present work, the activation of functional groups on *Eulaliopsis binata fibers* have been carried out using mercerization of these fibers with different concentration of NaOH for selected time intervals using the standard method reported in the literature (Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b). These mercerized *Eulaliopsis binata fibers* were subsequently subjected to surface functionalization with the vinyl silane coupling agent.

### **3.1 Effect of reaction time on mercerization of *Eulaliopsis binata fibers***

The effect of reaction time in the mercerization of *Eulaliopsis binata fibers* was studied in terms of percentage weight loss using the standard method (Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b). Figure 1 shows the effect of the mercerization (selected % of NaOH was used in all the experiments). The maximum mercerization of *Eulaliopsis binata fibers* was found to occur at 270 minutes and after which it nearly saturates due to the trapping of



sodium ions and other side reactions as these did not favor the further weight loss in the *Eulaliopsis binata* fibers. The mercerization of *Eulaliopsis binata* fibers facilitates the removal of impurities as well as definite amount of wax and lignin. After the optimization of time, mercerization of *Eulaliopsis binata* fibers was done using different concentration of NaOH starting from 3-15 %. The concentration of NaOH was varied in the range of 3-15 % because *Eulaliopsis binata* fibers are full of cellulose, hemicellulose and lignin along with various other constituents. These different components are strongly associated with each other and are difficult to separate/ remove using mild concentration of NaOH. An appropriate amount of NaOH helps in the removal of undesired components from the cellulosic fibers and facilitates the modification through silane treatment/ graft copolymerization. In this work the concentration was carried using different % of NaOH and was optimized by studying the % swelling behavior of *Eulaliopsis binata* fibers in different solvents; % water uptake behavior of *Eulaliopsis binata* fibers and % weight loss (in different chemicals) after treatment with fixed amount of silane as discussed in the following section.

### **3.2 Silane treatment on mercerized *Eulaliopsis binata* fibers**

Vinyltrimethoxysilane treatment of different mercerized *Eulaliopsis binata* fibers was done as per the method reported in experimental section. Vinyltrimethoxysilane induced functionalization of *Eulaliopsis binata* fibers is a facile route to alter the surface characteristics of the raw fibers. However it is not as much effective as the graft copolymerization techniques where hydrophobic polymers are grafted onto the cellulosic fibers(Thakur, Singha, & Thakur, 2014)(Thakur, Thakur, & Singha, 2013). Functionalization of *Eulaliopsis binata* fibers with vinyltrimethoxysilane involves hydrolysis, condensation and bond formation stages, similar to

the one reported for other natural cellulosic fibers with the difference in the structure of the silane coupling agent(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b).

### **3.3 Physico - chemical properties of Silane functionalized *Eulaliopsis binata* fibers**

Effect of vinyltrimethoxysilane treatment on the swelling behaviour, water uptake and chemical resistance of the pristine and functionalized *Eulaliopsis binata* fibers has been shown in the figure 2 to 5 respectively. It is obvious from the figure 2 that the raw *Eulaliopsis binata* fibers follows the trend in the swelling as  $H_2O > CH_3OH > iso-BuOH$ . This trend in the swelling behavior of raw fibers can be attributed the fact that raw cellulosic *Eulaliopsis binata* fibers contain abundant hydroxyl (-OH) groups and water molecules have greater affinity for these groups compared to the  $CH_3OH$  &  $iso-BuOH$ . As a result of the greater affinity of water towards the hydroxyl groups of *Eulaliopsis binata* fibers, there is more penetration of water into these fibers rather than the other alcoholic solvents.

However after the silane functionalization, the *Eulaliopsis binata* fibers show less swelling due to the blockade of active functional groups cited by the vinyltrimethoxysilane(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b). It is also evident from the figure that the 12% mercerized *Eulaliopsis binata* fibers followed by treatment with the vinyltrimethoxysilane show the lowest swelling behaviour. Similar behaviour was also observed in case of water uptake demonstrating that 12% mercerized fibers are most efficiently functionalized by vinyltrimethoxysilane coupling agents (figure 3). These functionalized *Eulaliopsis binata* fibers were also subjected to the study of chemical resistance behavior in the solutions of 1N HCl and 1N NaOH. The resistance of the *Eulaliopsis binata* fibers towards chemicals was also found to be enhanced after silane functionalization (Figure 4-

5). This behavior was also attributed to the blockage of active functional groups on the *Eulaliopsis binata* fibers vulnerable to the chemical attacks(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b).

### 3.4 Characterization of raw and grafted *Eulaliopsis binata* fibers

The characterization of the *Eulaliopsis binata* fibers was carried out using FT-IR Spectroscopy, SEM and TGA analysis to ascertain the vinyltrimethoxysilane induced surface functionalization of these fibers. The FT-IR spectrum of the raw *cellulosic Eulaliopsis binata* fibers and vinyltrimethoxysilane functionalized fibers are shown in the figure 6(a-b). Figure 6 a shows the spectrum of raw *Eulaliopsis binata* fibers. The peaks at  $3415\text{ cm}^{-1}$ ,  $1746\text{ cm}^{-1}$  and  $1244\text{ cm}^{-1}$  in the spectrum of the *Eulaliopsis binata* fibers can be assigned to the bonded OH groups and the C-O ring of lignin and carboxylic groups of pectin and hemicellulose(Thakur, Thakur, & Gupta, 2013c)(Thakur, Thakur, & Gupta, 2013e). Several other peaks in the spectrum of the *Eulaliopsis binata* fibers can be assigned to the basic constituents of any lignocellulosic fibers. However, after the functionalization of the *Eulaliopsis binata* fibers, some of the basic peaks of the fibers are disappeared while some due to silane groups are appeared. The peaks at  $1746\text{ cm}^{-1}$  and  $1244\text{ cm}^{-1}$  were found to be disappeared as a result of mercerization of these fibers. However, some new peaks as a result of the silane incorporation onto the *Eulaliopsis binata* fibers also appeared in the region from  $750\text{-}1800\text{ cm}^{-1}$  further confirming the successful functionalization of these fibers through silane treatment(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b). The peaks at  $1575\text{ cm}^{-1}$  and  $1490\text{ cm}^{-1}$  strongly demonstrated the presence of the silane functional groups on the *Eulaliopsis binata* fibers(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b).

Figure 7 (a-b) shows the SEM images of the raw and vinyltrimethoxysilane functionalized *Eulaliopsis binata* fibers. Both the images show the clear cut difference in the surface morphology of the fibers. It is observed from these image that the fibers surface changes significantly after the silane treatment and become almost rough. The changes in the morphology can be attributed to the removal of the impurities, wax, hemicellulose etc. from these fibers as well formation of silane coating the *Eulaliopsis binata* fibers surface.

Figure 8 (a-b) shows the thermograms of the raw and vinyltrimethoxysilane functionalized *Eulaliopsis binata* fibers surface. It is evident from the thermograms that the thermal degradation of *Eulaliopsis binata* fibers prior to and after silane functionalization covers a wide range of temperatures. For the raw *Eulaliopsis binata* fibers, the initial decomposition (IDT) temperature was found to be of 208 °C with a percent weight loss of 11 %. The final decomposition temperature (FDT) for these fibers was found to be 480 °C with a percent weight loss of 75 %. This thermal behaviour is similar to the other cellulosic fibers(Singha et al., 2009) (Singha & Thakur, 2009a) (Singha & Thakur, 2009b). After silane functionalization the modified fibers showed little variation in the thermal stability. The FDT of the vinyltrimethoxysilane functionalized *Eulaliopsis binata* fibers was found to be 491 °C. Thus both the SEM and TGA study further supports the FTIR data reconfirming the successful functionalization of the *Eulaliopsis binata* fibers through vinyltrimethoxysilane coupling agent.

#### 4. Conclusions

*Eulaliopsis binata* fibers are one of the unexplored lignocellulosic materials having huge potential to be used in a number of applications. In this work, the surface functionalization of

lignocellulosic *Eulaliopsis binata* fibers was carried out using mercerization followed by silane treatment using vinyltrimethoxysilane as a coupling agent. Subsequently, the effect of silane functionalization onto the physico-chemical behavior of the *Eulaliopsis binata* fibers was studied. It was observed from the study that the vinyltrimethoxysilane induced functionalization significantly affects the physico-chemical properties of the *Eulaliopsis binata* fibers. These surface modified fibers can be subsequently used as *one* of the component in the preparation of the green polymer composites.

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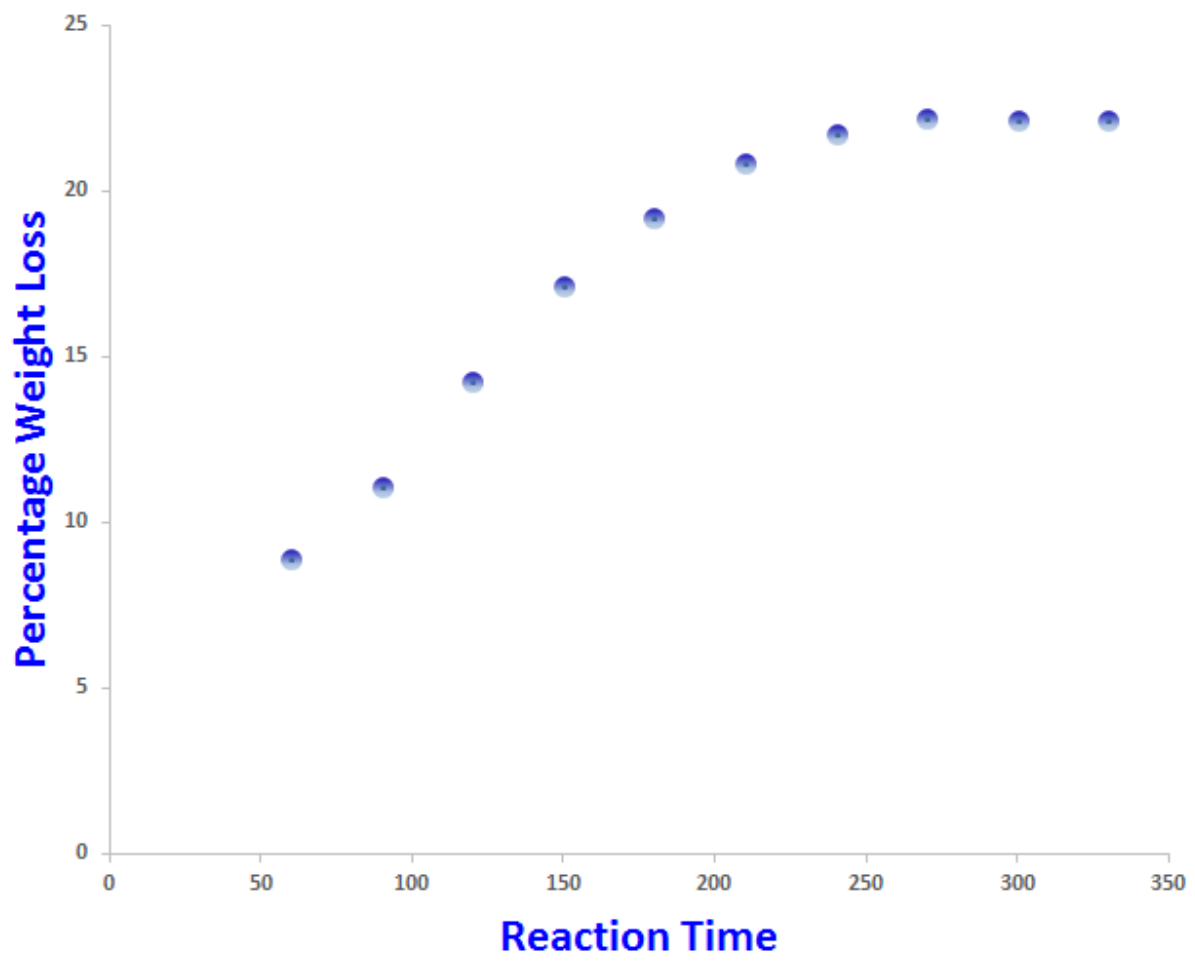
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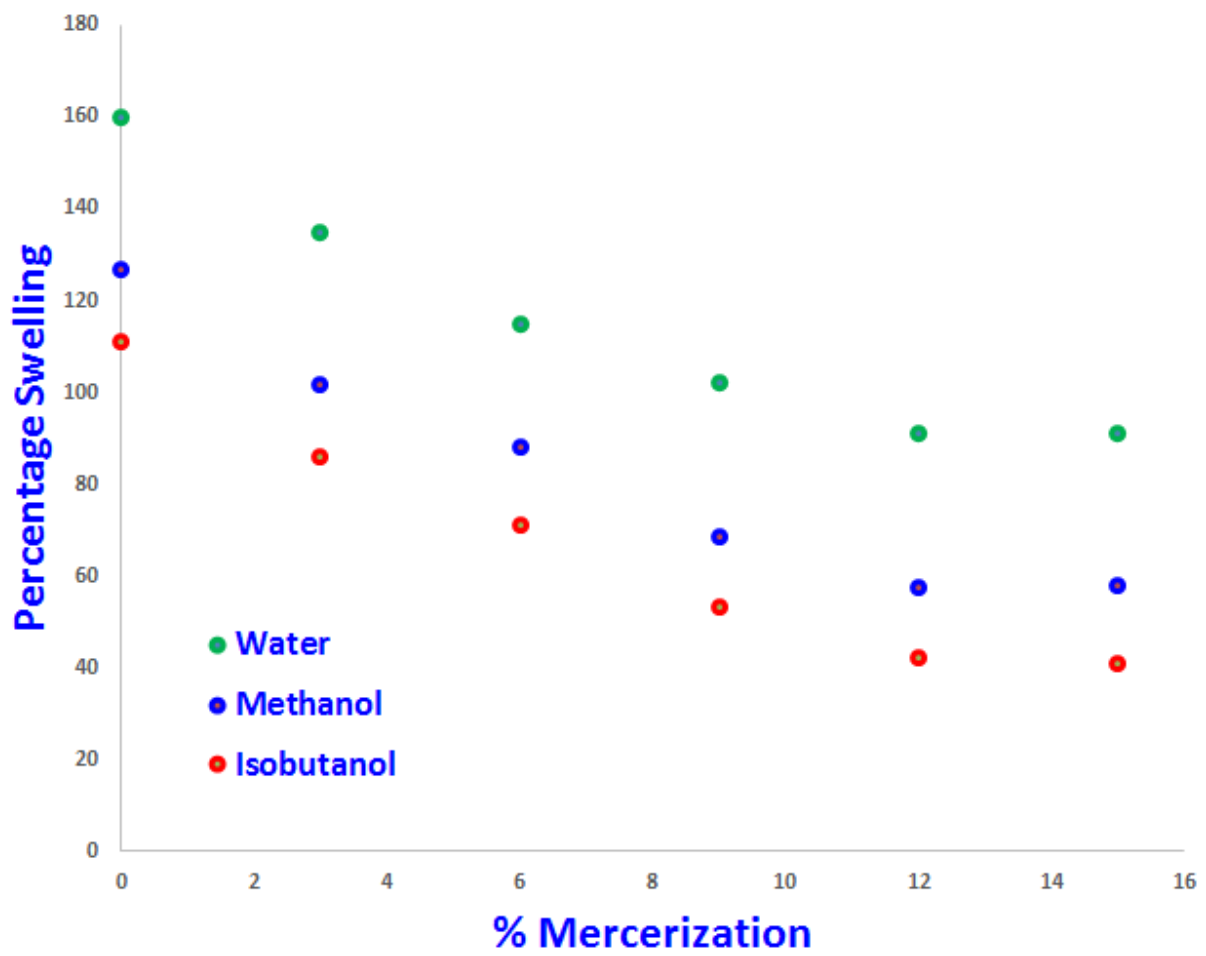
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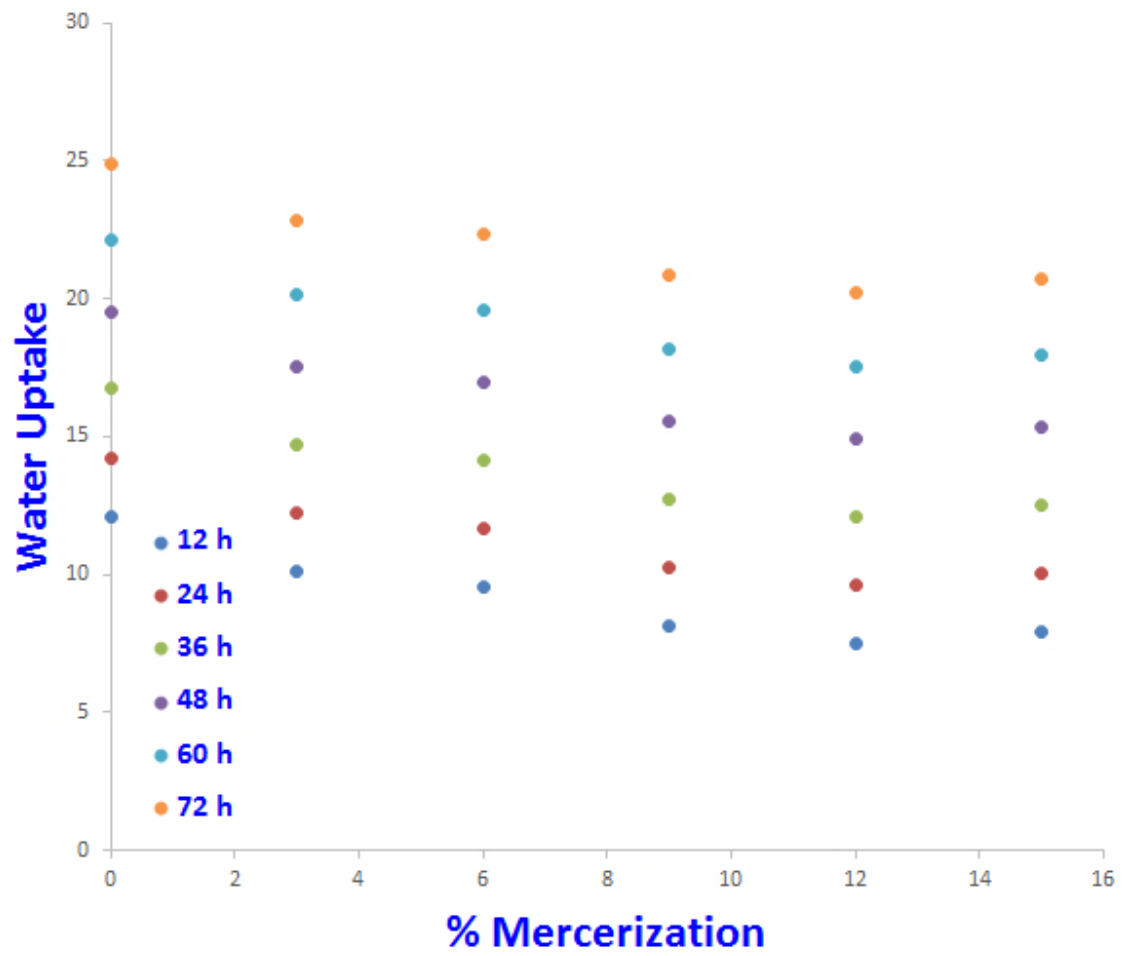
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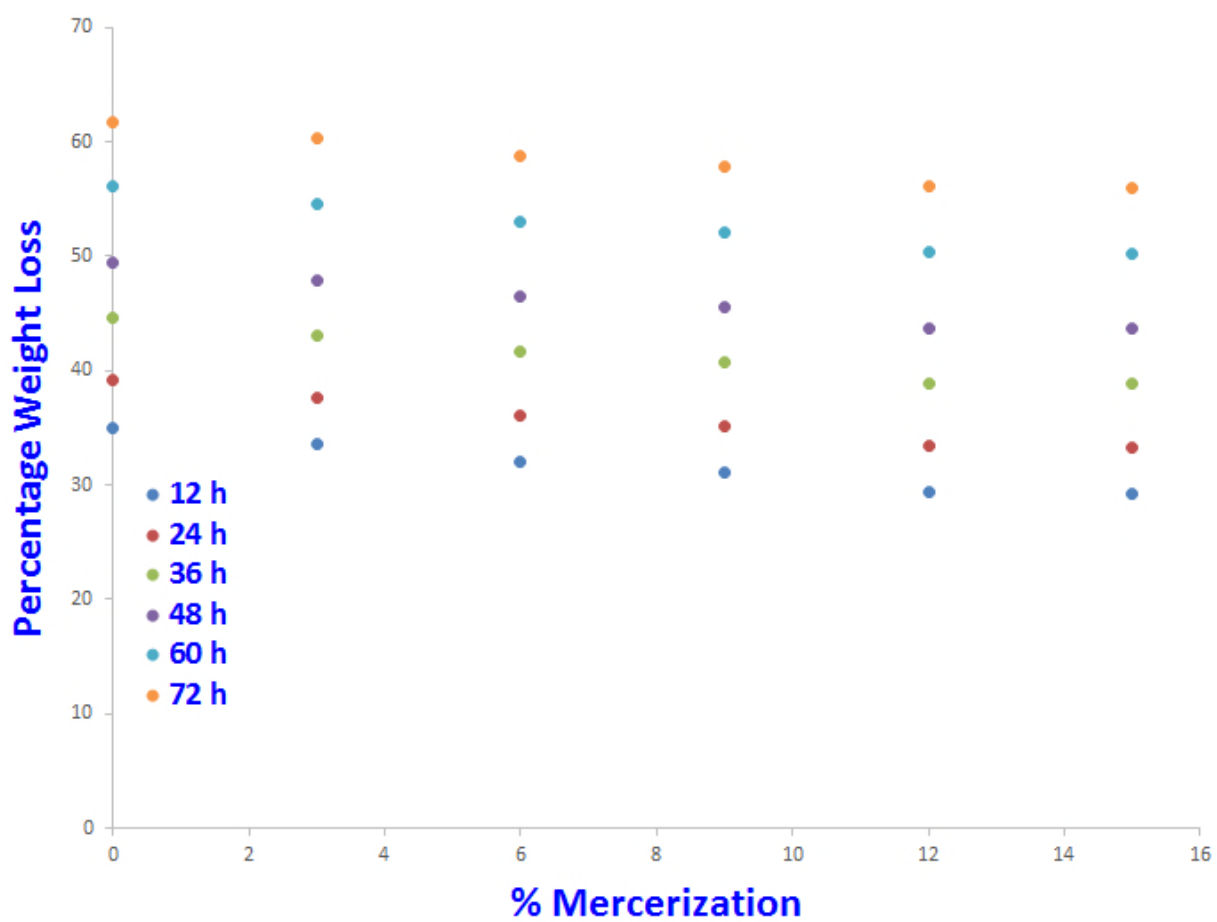
**Figure 1-** Effect of reaction time on mercerization of *Cellulosic Eulaliopsis binata* fibers



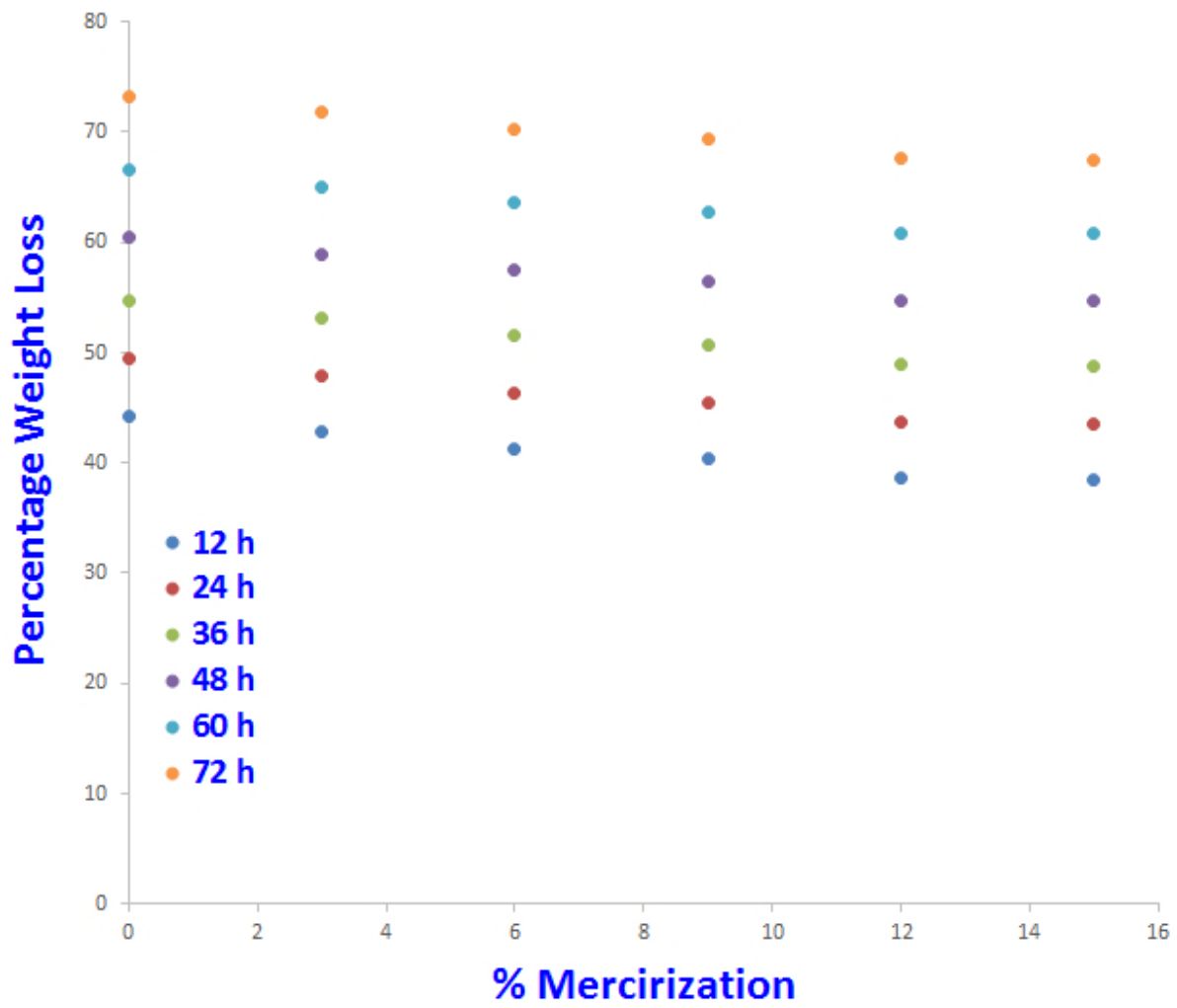
**Figure 2** Effect of silane treatment on % swelling behavior of *Eulaliopsis binata* fibers in different solvents



**Figure 3** Effect of silane treatment on % water uptake behavior of *Eulaliopsis binata* fibers.

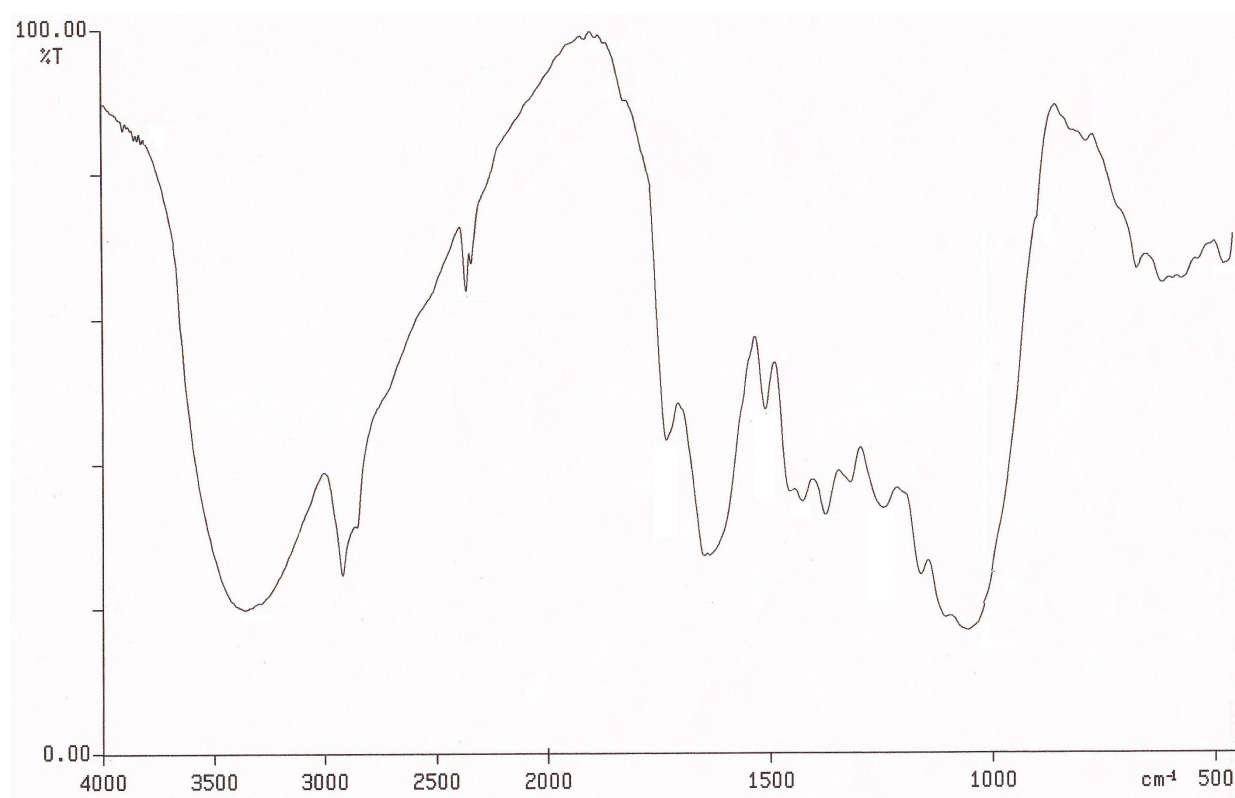


**Figure 4** Effect of silane treatment on acid resistance behavior of raw and grafted *Cellulosic Eulaliopsis binata* fibers in 1 N HCl at different time intervals

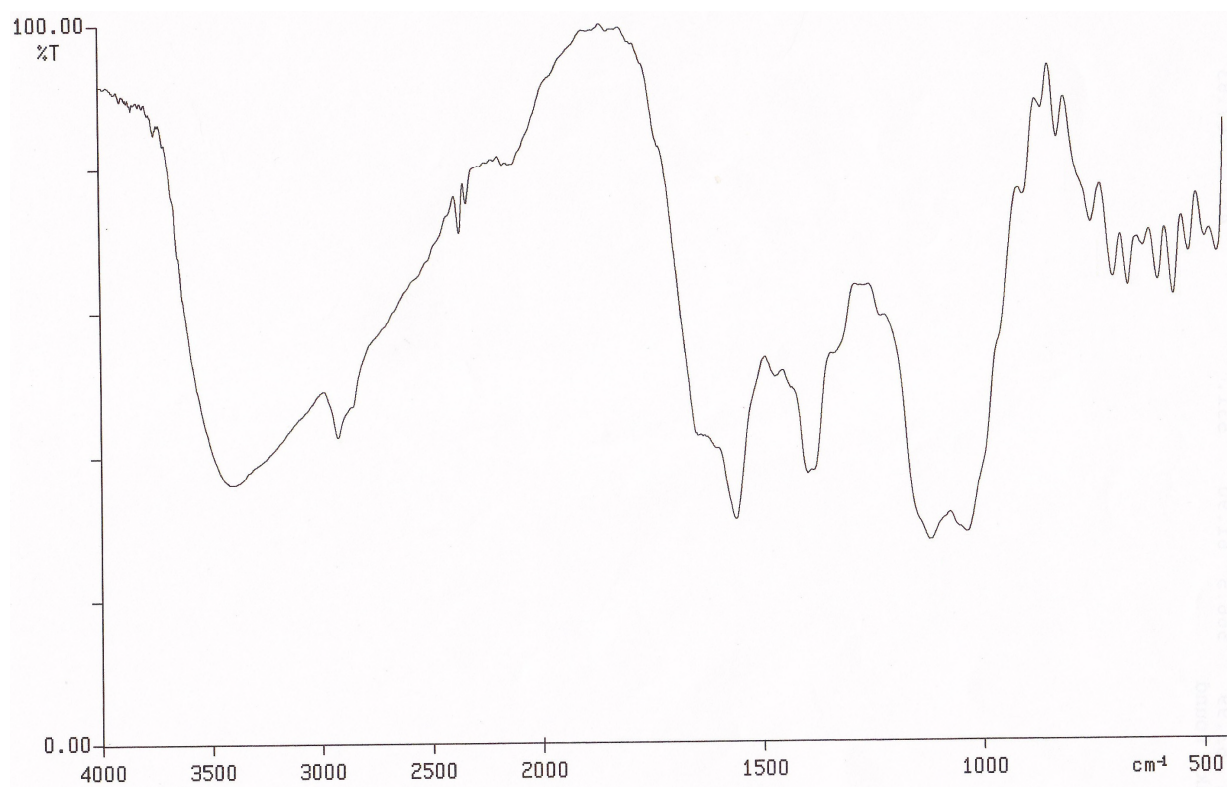


**Figure 5** Effect of silane treatment on base resistance behavior of raw and grafted *Cellulosic Eulaliopsis binata* fibers in 1 N NaOH at different time intervals.

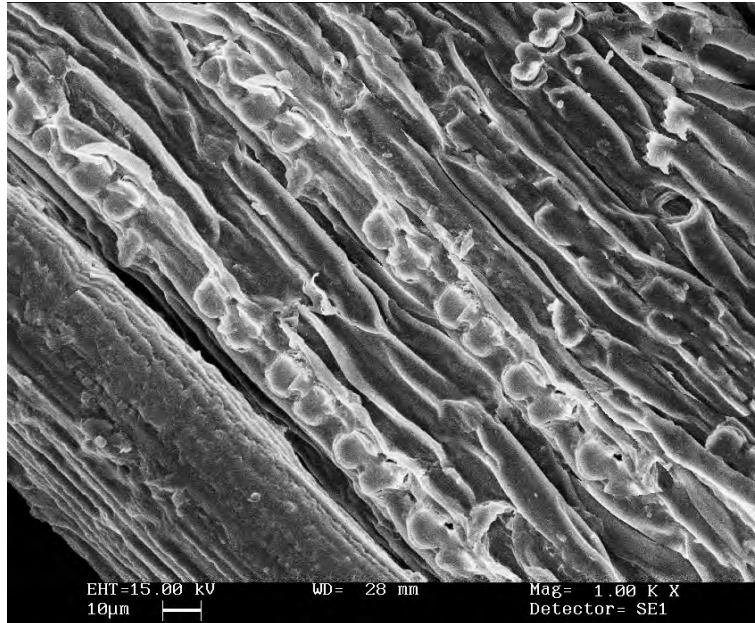




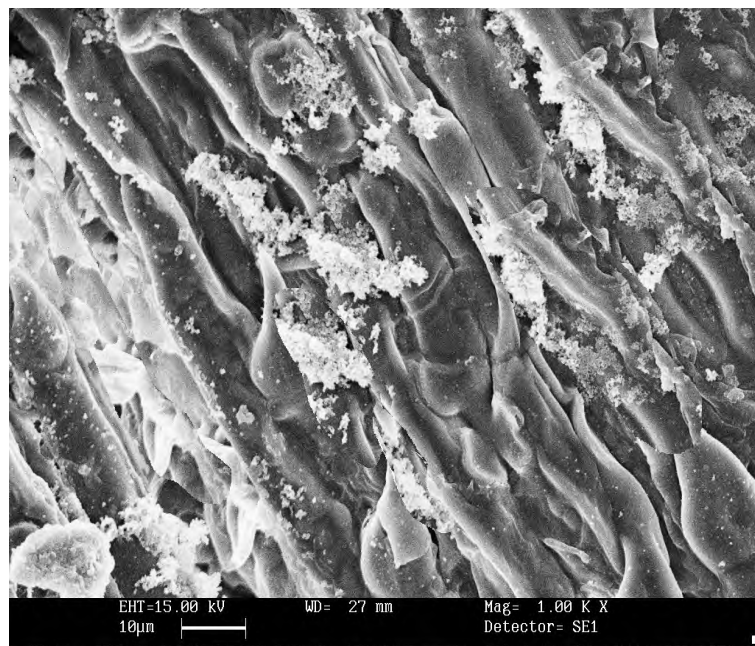
**Figure 6(a)** FTIR spectrum of raw *cellulosic Eulaliopsis binata* fibers.



**Figure 6 (b)** FTIR spectrum of silane treated *cellulosic Eulaliopsis binata* fibers

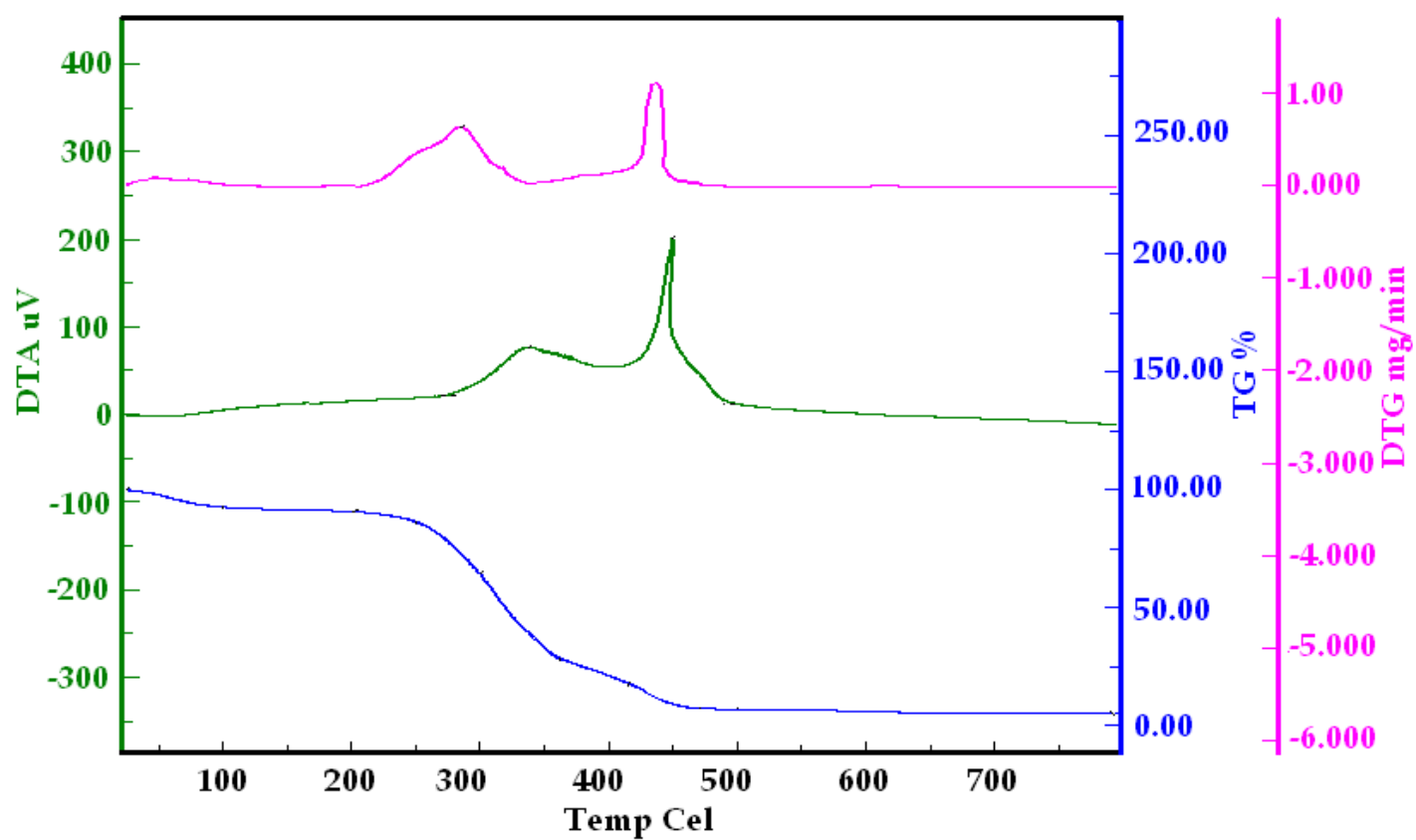


(a)



(b)

**Figure 7-** Scanning electron micrographs of (a) raw *cellulosic Eulaliopsis binata* fibers (b) *silane treated cellulosic Eulaliopsis binata* fibers.



**Figure 8 (a)** - Thermogravimetric analysis of raw *cellulosic Eulaliopsis binata* fibers

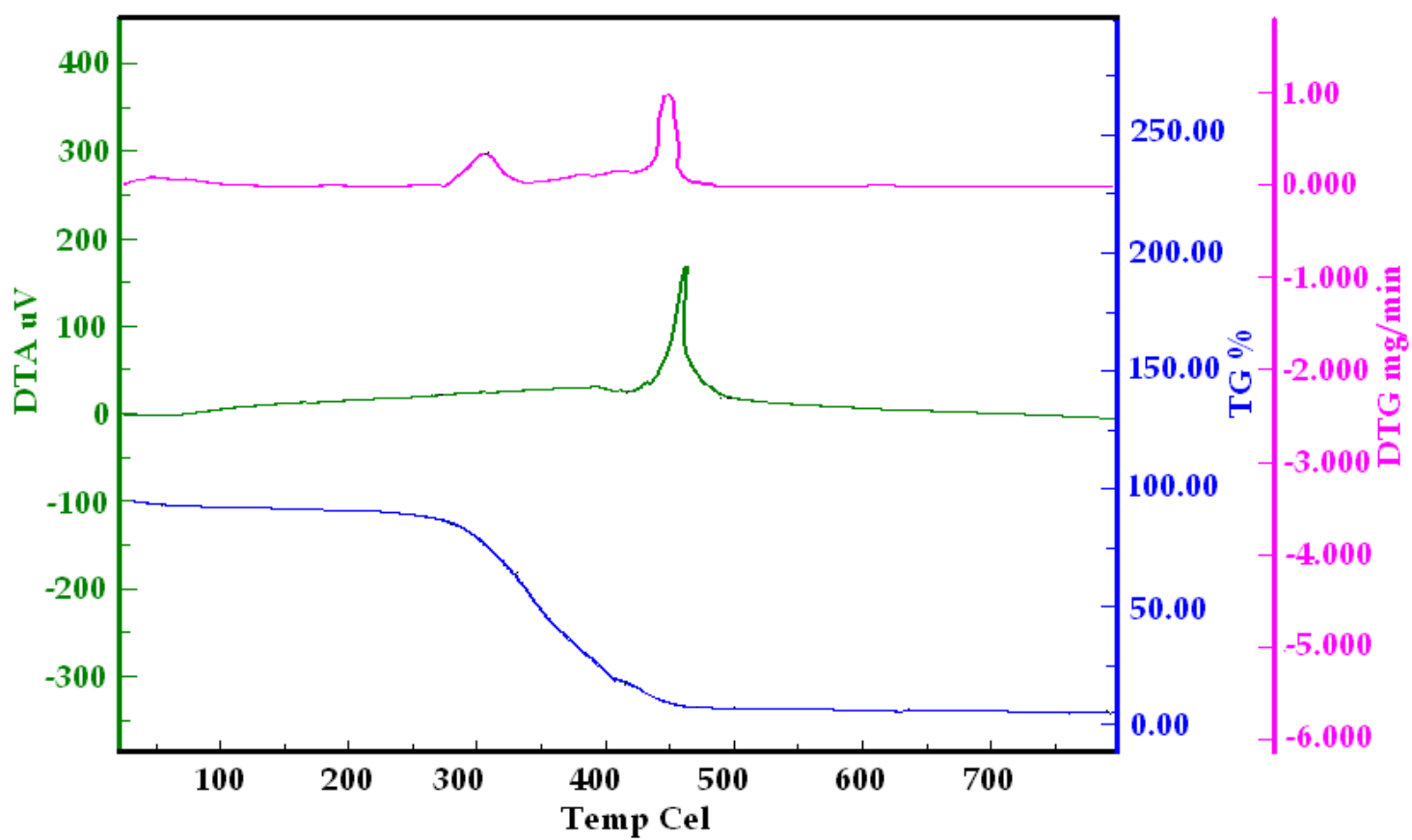


Figure 8 (b) - Thermogravimetric analysis of *silane treated cellulosic Eulaliopsis binata fibers*

# Surface modification of cellulose using silane coupling agent

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