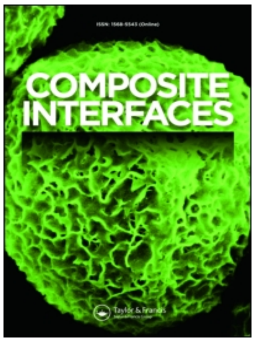

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Novel Jute/Polycardanol Biocomposites: Effect of Fiber Surface Treatment on Their Properties

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Abstract

In the present study, novel biocomposites with chopped jute fibers and thermosetting polycardanol were prepared using compression molding technique for the first time. Prior to biocomposite fabrication, jute fiber bundles were surface-treated at various concentrations using 3-glycidoxypropyltrimethoxy silane (GPS) and 3-aminopropyltriethoxy silane (APS), respectively. The interfacial shear strength, flexural properties and thermal properties of jute/polycardanol biocomposites reinforced with untreated and silane-treated jute fibers were investigated by means of single fiber microbonding test, three-point flexural test, dynamic mechanical analysis, thermogravimetric analysis and thermomechanical analysis. Both GPS and APS treatments played a role in improving the interfacial adhesion, reflecting that the organofunctional groups located at the end of silane coupling agents may contribute to linking between jute fibers and a polycardanol resin. As a result, it gave rise to increased interfacial shear strength of the biocomposites. Such interfacial improvement also led to increasing the flexural strength and modulus, storage modulus, thermal stability and thermomechanical stability.

Keywords

Jute fibers, polycardanol, biocomposites, silane treatment, interfacial shear strength, flexural properties, thermal properties

1. Introduction

Current researches on biocomposite materials are being directed at developing both natural fibers and polymer matrix materials, which can balance the performance and the cost due to increasing environmental concerns and increasing prices of petroleum-based chemical products [1–5].

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Jute (*Corchorus capsularis L.*) is an important tropical crop and agro-fiber in Bangladesh, India and some other countries. In general, natural fibers obtained from plants' bast like jute, kenaf and flax are inexpensive, non-abrasive, and have a good insulation advantage against heat and noise due to their cellular structure [6, 7]. Also, they have acceptable specific flexural and tensile properties. As a consequence of the fibers' low specific gravity, the specific modulus of jute fiber is comparable to that of glass fiber. In addition, there is no property deterioration due to fiber degradation when the processing temperature is below or at 200°C [8]. They also have economical and ecological advantages. Jute fibers have versatile applications with other thermoplastic and thermosetting resins to produce wood replacement panels, insulators and automotive components [7]. A number of studies [9–12] have been reported on fabricating jute/epoxy, jute/polyester, jute/phenol-formaldehyde, and jute/poly(lactic acid) composites and on characterizing their various properties without and with surface treatment of natural fibers.

However, because of the intrinsic polarity of natural fibers, the weak interfacial adhesion between untreated fibers and a polymeric matrix may cause insufficient mechanical properties of a composite [13]. To overcome this, appropriate surface treatment is necessary for natural fibers prior to composite fabrication [14–17]. Alkali treatment using sodium hydroxide has been frequently used as one of the methods to improve the interfacial adhesion [18, 19], whereas use of silane coupling agents has been an alternative [20, 21].

Nowadays, many studies on biocomposites have been made using thermosetting resins like unsaturated polyester, epoxy, and phenol-formaldehyde resins. Even though natural fibers are environmentally benign, most polymer resins are not. They may contain volatile organic compounds (VOCs), which is environmentally negative. One of the alternative ways to avoid such an environmental problem may be to introduce a resin without VOCs into a biocomposite system.

Cardanol, which can be obtained by thermal treatment of cashew nut shell liquid (CNSL), is a phenol derivative mainly composed of the *meta* substitute of a C15 unsaturated hydrocarbon chain with one to three double bonds [22, 23]. CNSL constitutes nearly one-third of the total nut weight. Thus, a large amount of CNSL is formed as a by-product of the mechanical processes used to render the cashew kernel edible and its total production approaches one million tons annually [24]. CNSL has industrially potential applications such as in resins, friction lining materials, and surface coatings.

Recently, Kim *et al.* [25, 26] reported that the enzymatic oxidative polymerization of thermally treated CNSL using peroxidase secreted from the basidiomycete fungus, *Coprinus cinereus*, produced very efficiently the oily soluble polymer, which is called polycardanol. It has no volatile organic compounds (VOCs) so that it has potential as a glossy formaldehyde-free coating material, a finish, and a composite matrix resin [22, 25]. Polycardanol is normally dark brown, highly viscous, and thermosetting. Based on the chemical structure of polycardanol with unsatu-

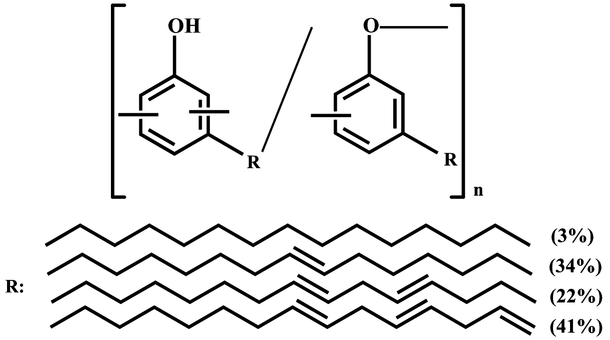


Figure 1. Chemical structure of polycardanol.

rated double bonds (Fig. 1), it may be thermally cured with peroxides and/or cobalt naphthenate as similarly in a typical unsaturated polyester resin [27–29].

A composite material consisting of polycardanol and plant-based natural fibers is now an interesting topic because of the use of all environmentally friendly constituents without VOCs in the composite. Although a large number of papers on biocomposite materials have been dealing with a variety of plant-based natural fibers and polymer matrices, only a few papers on natural fiber composites with CNSL have been reported [30, 31]. No report has used polycardanol as biocomposite matrix. The ultimate aim of our research is to first develop natural fiber-reinforced polycardanol matrix biocomposites. Consequently, the objective of this study is primarily to prepare novel jute/polycardanol biocomposites and explore the effect of silane-based coupling agents on the interfacial, flexural and thermal properties of the biocomposites. The result will be discussed in terms of interfacial shear strength, flexural strength and modulus, dynamic storage modulus, thermal stability, and thermal expansion of untreated and silane-treated jute/polycardanol biocomposites.

2. Experimental

2.1. Materials

Jute was cultivated in Bangladesh and jute fibers in the 30–40 cm long filament form were supplied from Bangladesh Jute Institute, Bangladesh. The jute fiber bundles were chopped to 10 mm long in average and the fiber contents were 40 wt% for fabricating randomly oriented jute/polycardanol biocomposites.

Polycardanol, which was prepared by enzyme-catalyzed oxidative polymerization, was kindly supplied by Korea Research Institute of Chemical Technology, Korea. Figure 1 depicts the chemical structure of polycardanol used in this work. The as-received polycardanol was of pre-polymer and thermally curable. Its monomer-to-polymer conversion was 97.7%. Methyl ethyl ketone peroxide (MEKP) used as initiator was supplied from Sewon Chemical Co., Korea. Cobalt naphthenate (Co-Naph) used as accelerator was purchased from Strem Chemicals, Inc., USA.

2.2. Fiber Surface Treatment

Two different types of silanes were used as coupling agents: 3-glycidoxypropyltrimethoxy silane (GPS) and 3-aminopropyltriethoxy silane (APS). Figure 2 shows the chemical structures of silanes used in the present work. For the surface treatment of the 'as-received' jute fiber bundles, 0.5, 1, and 3 wt% of each silane compound were dissolved for hydrolysis in a mixture of water–ethanol (50:50 v/v) and the jute fibers were soaked in each solution for 2 h at room temperature by agitating continuously [14]. Finally, the fibers were washed with distilled water and dried in an oven at 100°C for 12 h.

2.3. Jute/Polycardanol Microdroplet Formation

In order to form a resin microdroplet on a single natural fiber surface, a very tiny amount of polycardanol containing 1 wt% MEKP and 0.2 wt% Co-Naph was dropped onto a single jute fiber. The polycardanol resin microdroplet of an ellipsoidal shape surrounding the jute fiber surface was fully cured in a conventional oven at 180°C for 3 h. Thirty specimens were prepared by this procedure to form a polycardanol resin microdroplet on a single jute fiber treated with GPS and APS, respectively.

2.4. Jute/Polycardanol Biocomposite Fabrication

All the untreated and 1 wt% silane-treated natural fibers bundles were chopped to 10 mm long and sufficiently impregnated in the polycardanol resin that 1 wt% MEKP and 0.2 wt% Co-Naph were mixed uniformly. Then, the mixture was placed in a conventional oven for 10 min at 110°C for B-staging polycardanol. Next, the B-staged molding compound was placed in a stainless steel mold with the dimensions of 50 mm × 50 mm and then compression-molded using a hot-press (Carver 2518).

The molding was conducted at 150°C for 15 min with a pressure of 500 psi and then the molding temperature was increased to 190°C for 10 min with a pressure

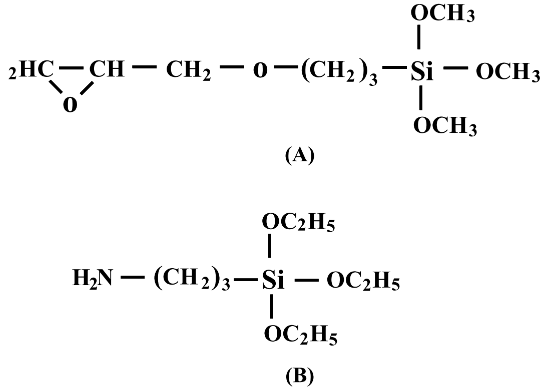


Figure 2. Chemical structures of silane coupling agents used. (A) 3-glycidoxypropyltrimethoxy silane (GPS) and (B) 3-aminopropyltriethoxy silane (APS).

of 1500 psi. Finally, the mold was naturally cooled down to ambient temperature. After demolding the composite plaque, it was further exposed to 180°C for 24 h for long-term cure. The dimensions of the obtained jute/polycardanol biocomposites were 50 mm × 50 mm × 2 mm (length × width × thickness).

2.5. Characterization

2.5.1. Single Fiber Microbonding Test

A single fiber microbonding test using a polycardanol resin microdroplet formed on a single jute fiber was performed by means of a tailor-made test apparatus equipped with a universal testing machine (UTM, Instron 4467), as shown in Fig. 3. A load cell of 100 N was used and the crosshead speed was 2 mm/min. The grip distance was 20 mm.

Prior to the resin microdroplet formation, jute fibers with a relatively uniform fiber diameter were selected for the single fiber microbonding test because cellulose-based natural fibers may, in general, have different fiber diameters at different locations due to irregular fiber surfaces. The average diameter of a single jute fiber was 22–25 μm and the average sizes of an ellipsoidal-shaped polycardanol microdroplet were 266–268 μm in length and 175–185 μm in width, as indicated in Fig. 3.

The average interfacial shear strength (IFSS) value was obtained from 30 specimens of each biocomposite. The IFSS was calculated using the following equation:

$$\tau = F/(\pi \cdot D_\mathrm{f} \cdot L_\mathrm{e}),$$

where τ is the interfacial shear strength; F is the force required for debonding the polycardanol microdroplet from the single fiber filament while tensile loads are applied; D_f is the diameter of the measuring jute fiber; and L_e is the jute fiber

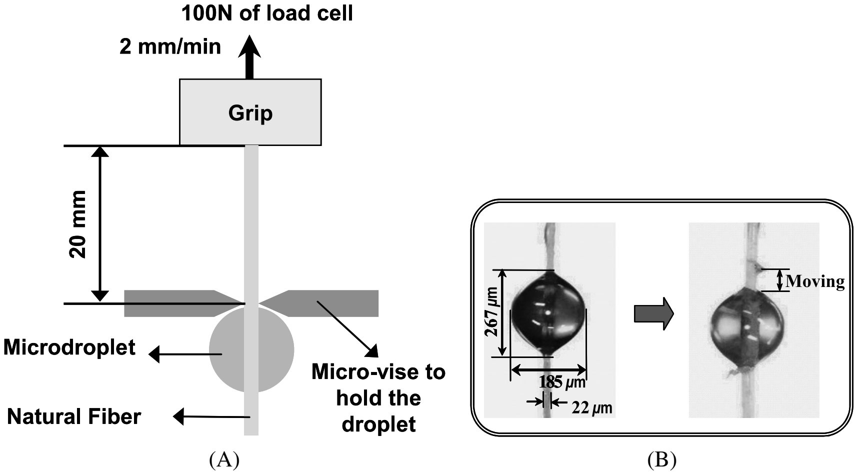


Figure 3. Schematic illustration of (A) a single fiber microbonding test using a jute/polycardanol biocomposite model specimen and (B) optical microscopic views of the specimens before and after the test.

length embedded in the polycardanol microdroplet. The formed jute/polycardanol microdroplets were observed with an optical microscope (Nikon, ECLIPSE E200) before and after the single fiber microbonding test, as shown in Fig. 3.

2.5.2. Flexural Test

The flexural properties of jute/polycardanol biocomposites were measured utilizing a three-point bending method according to ASTM D790M-86 using a universal testing machine (UTM, Instron 4467). The specimen dimensions were 50 mm × 25 mm × 2 mm. The span-to-depth ratio was 16. A load cell of 30 kN was used. A crosshead speed of 0.85 mm/min was used. The average values of flexural strength and modulus of each biocomposite were obtained from 10 specimens.

2.5.3. Thermogravimetric Analysis

Thermogravimetric analysis (TGA) is a useful method for the quantitative determination of the degradation behavior and the composition of the fiber and the matrix in a composite. The magnitude and location of peaks found in the derivative thermogravimetric (DTG) curve also provide information on the component and the mutual effect of the composite components on the temperature scale. Thermogravimetric analysis (TGA Q500, TA Instruments) was utilized to examine the thermal stability of jute, polycardanol, and untreated and silane-treated jute/polycardanol biocomposites. The measurements were performed with a dynamic scan from ambient temperature to 550°C. A heating rate of 20°C/min was used with a purging nitrogen gas. About 20–30 mg of each specimen was loaded for each measurement. Derivative thermogravimetric (DTG) thermograms were also recorded.

2.5.4. Thermomechanical Analysis

Thermomechanical analysis (TMA 2940, TA Instruments) was used to examine the thermomechanical stability including the coefficient of linear thermal expansion of each biocomposite. The temperature range was from ambient temperature to 150°C. The heating rate of 2°C/min was used with a purging nitrogen gas. The expansion mode to monitor the thermomechanical behavior was used. The dimensions of each specimen were 5 mm × 5 mm × 5 mm.

2.5.5. Dynamic Mechanical Analysis

Dynamic mechanical analysis (DMA) is a powerful technique to study the effect of temperature on the mechanical properties of polymer composite materials exposed to oscillating sinusoidal deformation at a given frequency. Dynamic mechanical analysis (DMA Q800, TA Instruments) was performed to examine the dynamic storage modulus of untreated and silane-treated jute/polycardanol biocomposite. Before measurement, the instrument was calibrated to have correct clamp position and clamp compliance. A fixed frequency of 1 Hz and the oscillation amplitude of 0.2 mm were used throughout this work. A single cantilever mode was used. The measuring temperature range was from 40°C to 240°C with a purging liquid nitrogen gas and the heating rate was 5°C/min. The specimen dimensions were 35 mm × 12 mm × 2 mm.

3. Results and Discussion

3.1. Interfacial Properties

In the single fiber microbonding test, biocomposite model specimens composed of a single jute fiber as reinforcement and a polycardanol resin as matrix were used to mimic a composite system composed of fibers and matrix. The test provides useful information on predicting the interfacial adhesion between the fibers and the matrix of a composite material [32–34].

Table 1 lists the interfacial shear strength (IFSS) results obtained from jute/polycardanol biocomposites with jute fibers untreated and treated at 0.5, 1.0, and 3.0 wt% of GPS and APS, respectively. The IFSS of the untreated one was significantly increased by both GPS and APS treatments, indicating the greatest value at 1 wt% of GPS. The greatest improvement of the IFSS was about 52% compared to the untreated. The GPS treatment contributes, with a greater extent, to increasing the IFSS value of the biocomposite than the APS.

The variation of IFSS between untreated and silane-treated biocomposites may be explained based mainly on the chemical reaction occurring in silane-treated natural fibers [35, 36]. In general, the co-reactivity between silane-treated reinforcing fibers and a polymer resin may provide some covalent bonds at their interfaces [37]. It has been found that bifunctional silane molecules significantly contribute to chemically linking between natural fibers and a polymer resin in a biocomposite system, especially in a thermosetting resin [21, 38]. The contribution depends on the organofunctional group located at the end of silane coupling agents due to their different reactivity and interaction with polycardanol, as proposed in Fig. 4.

In the case of GPS-treated jute fibers, with increasing silane concentration, the epoxide groups in the GPS may react with polycardanol resin by substitution reaction, generating hydrogen bonds to form the network. A self-condensation reaction may also occur to form moderate intermolecular interlocking. The greatest value of IFSS was obtained at 1 wt% of GPS and the lowest value at 3 wt%. The reason for

Table 1.
A comparison of the interfacial shear strengths for jute/polycardanol biocomposites untreated and treated with GPS and APS at different concentrations

Biocomposite	Silane conc. (wt%)	IFSS (MPa)
Untreated jute/polycardanol	0	48.4 ± 3.7
GPS-treated jute/polycardanol	0.5	65.4 ± 4.6
	1.0	78.9 ± 3.0
	3.0	62.3 ± 3.6
APS-treated jute/polycardanol	0.5	56.6 ± 4.6
	1.0	63.5 ± 3.8
	3.0	53.4 ± 4.2

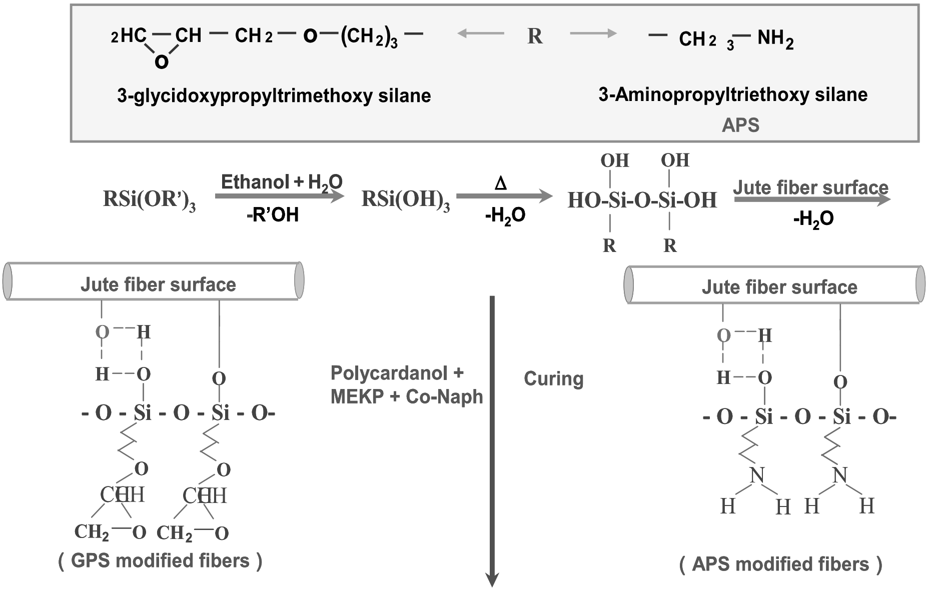


Figure 4. A schematic illustration of jute fiber surfaces chemically modified by silane treatment.

the lowest value is that the self-condensation reaction may take place between superfluous silanol groups. This may lead to the formation of dimeric and oligomeric structures and influence the reaction to the matrix, resulting in the lowering of the interfacial adhesion between the fibers and the matrix.

Similar behavior may also be occurring in the case of APS treatment. The difference between GPS and APS may be the chemical reaction with the natural fiber. The –OH and –CH groups formed after ring opening of the epoxide group in the GPS may react with –OH and –O groups in the polycardanol *via* hydrogen substitution reaction and hydrogen bonding. On the other hand, $-NH_2$ groups in the APS may react with –OH groups in the polycardanol *via* hydrogen bonding. Figure 5 illustrates a proposed reaction scheme occurring between the jute fibers and the polycardanol resin at the interfaces, based on the earlier work with cellulose-based natural fiber-unsaturated polyester and –vinyl ester composite systems by Abdelmouleh *et al.* [39] and Ray *et al.* [40].

Since the greatest IFSS value was obtained with 1 wt% for both GPS and APS, the effect of silane treatment on the mechanical and thermal properties of jute/polycardanol biocomposites was investigated with the concentration in the following work.

3.2. Flexural Properties

In many applications, the flexural test is more relevant to the advantages for material design or specification purposes than a tensile test [41]. Table 2 demonstrates that the flexural strength and modulus of jute/polycardanol biocomposite were im-

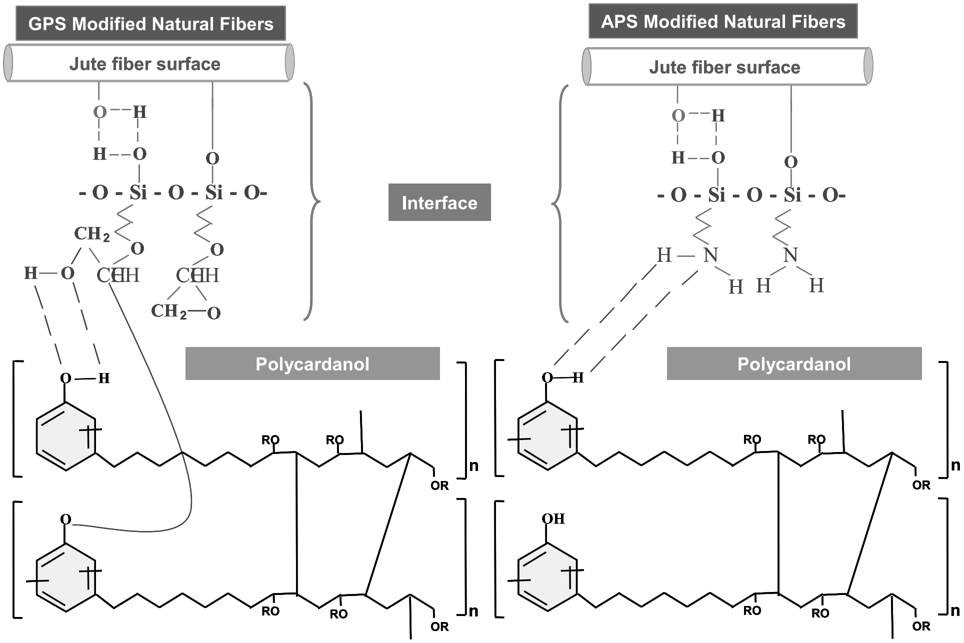


Figure 5. A proposed scheme of chemical reaction occurring at the interfaces between silane-treated jute fibers and a polycardanol resin.

Table 2.
Flexural strength and modulus of jute/polycardanol biocomposites with untreated and 1 wt% silane-treated jute fibers

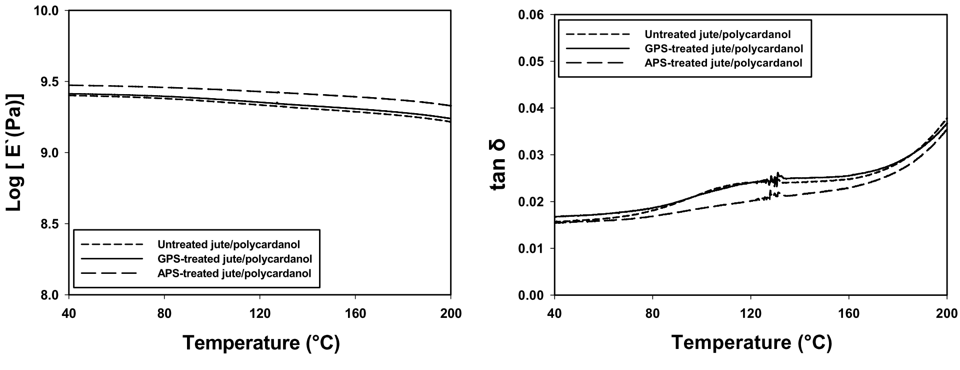
Biocomposite	Flexural strength (MPa)	Flexural modulus (GPa)
Untreated jute/polycardanol	33.8 ± 5.2	2.8 ± 0.9
GPS-treated jute/polycardanol	35.9 ± 3.8	3.2 ± 1.0
APS-treated jute/polycardanol	39.1 ± 5.4	3.4 ± 0.5

proved by the silane treatment. The flexural properties were slightly dependent on the type of silane coupling agents used. The biocomposites reinforced with APS-treated jute fibers exhibited slightly higher flexural strength and modulus than those with GPS-treated jute fibers. The improvement of the flexural properties in the presence of GPS and APS was attributed to the chemical bonds formed between the silane-treated jute fibers and the polycardanol resin. In the case of APS treatment, the relatively higher value of the flexural strength was due probably to a larger number of hydrogen bonds formed between the polycardanol molecules and the $-NH_2$ groups in the APS-treated jute fiber surfaces during composite processing. This can contribute to increase in the efficiency of stress transfer between the natural fibers and the matrix. This may also lead to a modest enhancement of the interfacial adhesion between the jute fibers and the polycardanol.

Although it was obvious that the silane treatment of jute fibers increased the interfacial shear strength of jute/polycardanol biocomposites, the flexural result was not quite consistent with the fact that the GPS-treated jute/polycardanol biocomposite resulted in a higher interfacial shear strength than the APS-treated counterpart. It has been reported previously that the topological changes of natural fiber surfaces due to silane treatment may influence the mechanical properties of a biocomposite system [21, 36, 42]. It is expected that the silane-treated jute surfaces may be affected by the physical interaction as well as the chemical reaction with the polycardanol resin upon composite processing up to 190°C. There may also be some additional causes, such as differences in the wetting and debonding behavior and the methodology between the single fiber microbonding test and flexural test. However, the reason for this difference has not been unequivocally established yet. Therefore, further study is needed to elucidate the reason, by considering the physical and processing effects on silane-treated jute/polycardanol biocomposite system.

3.3. Dynamic Mechanical Properties

Figure 6 shows the variations of the storage modulus E' and $\tan\delta$ as a function of the temperature observed for jute/polycardanol biocomposites fabricated using untreated and silane-treated jute fibers, respectively. The storage modulus and $\tan\delta$ of untreated biocomposite were slightly enhanced by the fiber treatment. The APS-treated jute/polycardanol biocomposite exhibits a greater storage modulus than the GPS-treated one. A damping property of a composite material may also be explored by means of DMA, because $\tan\delta$ is the ratio of the energy dissipated to the energy stored during a dynamic loading cycle. It has been reported that incorporation of stiff natural fibers into a polymer matrix restricts the movement of the molecular motion of polymer matrix. As seen in Fig. 6, the $\tan\delta$ peak height of APS-treated



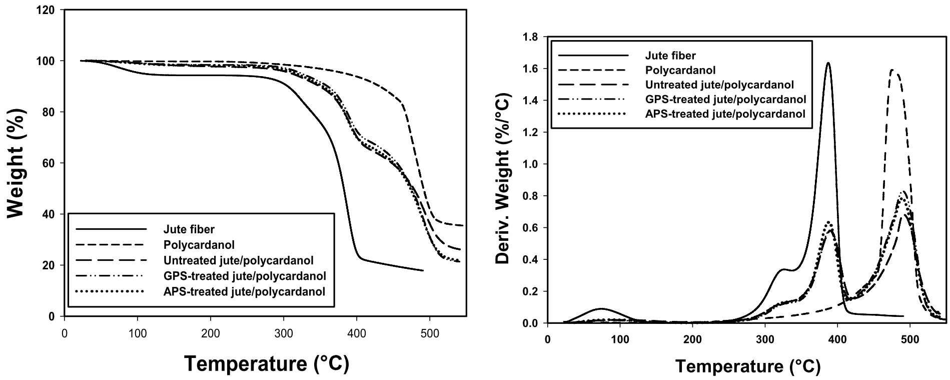
(A) (B)

Figure 6. Variations of (A) dynamic storage modulus and (B) $\tan\delta$ measured for jute/polycardanol biocomposites with untreated and silane-treated jute fibers.

jute/polycardanol biocomposites was reduced due to a fiber reinforcing effect in comparison with the untreated and GPS-treated counterparts.

3.4. Thermal Stability

Figure 7 shows the thermal stabilities of jute, polycardanol and untreated and 1 wt% silane-treated jute/polycardanol biocomposites. It was found from the DTG curves that there were two distinguishable peak temperatures, indicating the fastest weight losses occurring in the biocomposites. One in the lower temperature region is from jute and the other in the higher temperature is from polycardanol. The thermograms from the biocomposites reflect the two peaks. This demonstrates that the biocomposites have two distinguishable decomposition stages, depending on the constituent. It was likely that the first stage was associated with the degradation of jute and the second stage with that of polycardanol. The initial weight loss of about 7%–8% starting below 100°C was ascribed to the removal of intrinsically bound water molecules in the jute fibers. The silane-treated jute/polycardanol biocomposite specimens exhibited slightly higher thermal stability than the untreated counterpart. This was due probably to the increased interfacial adhesion between the jute fibers and the polycardanol matrix by the silane treatment and also to the removed waxes and weakly bound layers on the surfaces. However, both primary and secondary weight loss temperatures of the biocomposites were not changed meaningfully with the surface treatment that was carried out or with the silane type. Near 270°C, the jute fiber and the untreated and treated biocomposite specimens began to lose weight significantly due to the decomposition of cellulose and hemicellulose components therein. The shoulder at about 310°C was due to the thermal decomposition of hemicellulose and the glucosic linkage of cellulose, and the main peak at about 380°C to the decomposition of α-cellulose [12, 43]. The result indicates that the hemicellulose component was not removed by the silane treatment.



(A) (B)

Figure 7. (A) TGA and (B) DTG thermograms measured for jute, polycardanol, and jute/polycardanol biocomposites with untreated and silane-treated jute fibers.

3.5. Thermomechanical Stability

Thermomechanical analysis (TMA) measuring the coefficient of linear thermal expansion (CTE) is a powerful tool for understanding of the dimensional changes by thermal energy. In general, the CTE of a fiber-reinforced polymer matrix composite may be lower than that of a neat polymer because most polymers thermally expand to a greater extent compared to reinforcing fibers. Reducing the CTE is desirable in order to minimize the thermo-dimensional changes of a composite exposed to temperature changes that occur during composite processing or in use.

Figure 8 depicts the thermal expansion behavior of untreated and silane-treated jute/polycardanol biocomposites. As can be seen, the dimensional change of untreated biocomposite was reduced over the whole temperature range by the silane treatment done to jute fibers. It is also noticed that the biocomposites exhibited a thermal contraction behavior due to shrinkage of cellulose-based natural fibers in the range of 35°C to 100°C. Table 3 summarizes the CTE values measured in the two temperature ranges: 35°C–100°C and 100°C–150°C. The CTE values obtained in the range of 35°C to 100°C were negative, demonstrating the shrinkage behavior of jute fibers. Cellulose-based natural fibers consist of a large number

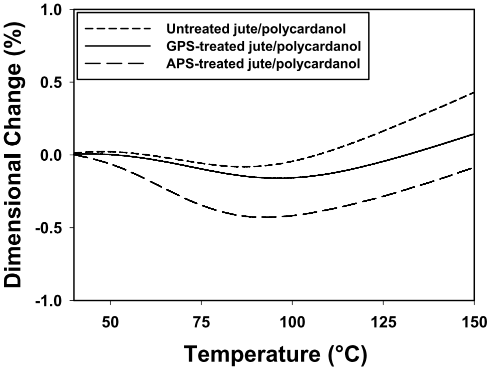


Figure 8. TMA thermograms showing the dimensional changes measured for jute/polycardanol biocomposites with untreated and silane-treated jute fibers.

Table 3.
A comparison of the coefficients of linear thermal expansion measured for untreated and 1 wt% silane-treated jute/polycardanol biocomposites

Biocomposite specimen	35°C–100°C (μm/m/°C)	100°C–150°C (μm/m/°C)
Untreated jute/polycardanol	−14.4	97.4
GPS-treated jute/polycardanol	−28.0	68.4
APS-treated jute/polycardanol	−84.2	63.3

of cells, which are thermally sensitive. The CTE value in the range of 100°C to 150°C was markedly decreased from 97.4 μm/m/°C to 63.3 μm/m/°C due to the silane-treatment done to jute fibers. This may be ascribed to the improved interfacial adhesion between the jute fibers and the polycardanol matrix by the silane treatment. Also, it may be said that the enhanced thermomechanical stability of silane-treated jute/polycardanol biocomposites was consistent with the increased flexural and dynamic mechanical properties of the biocomposites due to the surface treatment.

4. Conclusions

In this study, novel chopped jute fiber-reinforced polycardanol biocomposites have been successfully prepared for the first time. In order to improve the interfacial adhesion between jute fibers and a thermosetting polycardanol resin, two silane coupling agents, GPS and APS were utilized to treat the jute fiber surfaces. Use of 1 wt% GPS and APS played a role in enhancing not only the interfacial shear strength between jute fibers and a polycardanol resin, but also the flexural and thermal properties of the biocomposites.

The result showed the increased interfacial, flexural, dynamic mechanical properties and thermomechanical stability of jute/polycardanol biocomposites with the silane treatment of jute fibers before processing. The flexural strength and modulus, dynamic storage modulus, thermal stability and thermomechanical stability of the biocomposites with APS-treated jute fibers were greater than those with GPS-treated counterparts. Their results agreed with each other, showing a similar tendency. This study suggests that the silane treatment done to jute fibers was effective at 1 wt% to improve the various properties of jute/polycardanol biocomposites.

Acknowledgement

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