

# Effect of Silane Treatments and Fiber Loadings on Tensile Properties of Coir Fiber Reinforced Composites

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## ABSTRACT

*Nowadays, increasing awareness of replacing synthetic fiber by glass fiber has emerged due to environmental problems and pollution. Automotive manufacturers also seek new material especially biodegradable material to be used as non-load bearing application parts. This present work discusses the effect of silane treatments and fiber loadings on tensile properties of coir fiber reinforced polyester composites. From the experimental results, both silane treatments and fiber loadings increased the tensile properties. The presence of silane coupling agent on the fiber surfaces enhanced the compatibility between fiber and polymeric matrix. This compatibility promoted the capability of composites to sustain higher tensile loadings. From SEM observation pictured that longer fiber pull-out is a dominant factor for 5% fiber loading composites result lower tensile strength. Interfacial debonding also occurred for untreated fiber with silane coupling agent while silane treated fiber reinforced composites. Shorter pull-out fibers are observed and fibers are well gripped within the matrix showing better interfacial bonding and good tensile properties are achieved.*

*Keywords: Natural fiber, coir fiber, silane treatment, pullout fiber, interfacial bonding.*

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## INTRODUCTION

Coconut coir fibers are obtained from the fibrous cushion encapsulated the internal brittle shell of the fruit of the coconut palm. These fibrous cushions are the by-product of the copra extraction process and in the rural area these by-product are burned and become one of the environmental problems especially for developing countries. According to Goulart Silva G. *et al.* [1], coir fibers have high lignin but low cellulose content as a result of which the fibers are resilient, strong, and highly durable and they are used in textile industry due to the ability of coir fibers to tolerate water immersion for months without disintegrating the fiber integrity.

It is well known that, the performance of composites depends on the properties of the individual components and their interfacial compatibility. Cellulosic fibers like sisal, coconut fiber, jute, palm, bamboo and several wastes cellulosic products have

been used as reinforcement tools of different thermosetting and thermoplastic resins. One of the difficulties that have prevented an extended utilization of natural fibers in composites is the lack of good adhesion to most polymeric matrices. The hydrophilic matrix results in poor strength properties. To prevent this, the fiber surface has to be modified in order to promote adhesion [2]. Previous works [3 – 5] has shown that, a good correlation of the adhesion exist between natural fibers and polymeric resins when the natural fibers are modified by various silane coupling agents. While Rout J. *et al.* [6] conducted a work to investigate the surface characteristics of an untreated coir fiber and 5% alkali-treated coir fibers.

From fracture surface observations showed that, the fracture surfaces of the untreated coir-polyester composites with the fiber pullouts with many holes left after the fibers were pulled out of the matrix. The incompatibility of the untreated coir surface with the polyester matrix was also reflected, whereas an improved bonding with a lesser fiber pullout and better fiber dispersion was observed.

In order to develop composites with better mechanical properties and environmental performance, it is necessary to impart hydrophobicity to the natural fibres by chemical reaction with suitable coupling agents or by coating with appropriate resins. Such surface modification of fiber does not only decrease moisture adsorption, but also concomitantly increases wettability of fibers with resin and improve the interfacial bond strength, which are critical factors for obtaining better mechanical properties of composites.

Natural fiber is chemically treated with isopropyl triisostearoyl titanate (abbreviated as titanate), g-aminopropyl trimethoxy silane (abbreviated as silane), Sebacoyl Chloride (SC), and Toluene Di-Isocyanate (TDI). All these reagents are expected to block the hydroxy groups of jute thus making the fibers more hydrophobic. These surface modifiers penetrate and deposit into lumens of cell wall of fiber, minimizing the possible extent of moisture ingress [7]. Polymeric coatings of natural fiber with phenol-formaldehyde or resorcinol formaldehyde resins by different approaches are highly effective in enhancing the reinforcing character, giving as high as 20%-40% improvement in flexural strength and 40%-60% improvement in flexural modulus. These modifications improve the fiber-resin wettability and lead to enhanced bonding. Natural fiber such as jute can be graft co-polymerized with vinyl monomers such as methyl methacrylate, ethyl acrylate, styrene, vinyl acetate, acrylonitrile and acrylamide in presence of different redox initiator systems such as vanadium-cyclohexanol, vanadium-cyclohexanone etc. Grafting of poly-acrylonitrile (10%-25%) imparts 10%-30% improvement in flexural strength and flexural modulus of the composites. Grafting of polymethylmethacrylate is also effective in this respect, though to a lower degree [8].

In this study, coconut coir fibers are used as reinforcement tools in polyester resin. Dow Corning coupling agent has been used to cover the natural fiber surfaces to enhance the compatibility between polyester resin and fibers. This present work reports on the tensile properties of coir fiber reinforced composites containing different fiber volume fractions and coated with different silane concentration solutions. The discussion is carried out in order to study the effect of silane treatment on tensile properties and correlating with fracture mechanisms.



## EXPERIMENTAL METHODOLOGY

### Fiber and Composite Preparations

As received coconut fibers are prepared through several processes. Fibers are cut into 20mm length. Table 1 reveals the mechanical properties of coir fiber. Fiber surfaces are cleaned from any impurities and it is well known that the performance of composites depends on the properties of the individual components and their interfacial compatibility. Fibers experienced alkalization or purification process in 5% sodium to remove any unwanted fiber components on the fiber surfaces. After that, the purified fibers are washed with distilled water in room temperature and dried in furnace environment for 24 hours at 80°C. Then, these fibers are immersed into aqueous alkali solutions. The aqueous solutions contained 0%, 5%, 10% and 15% weight of silane coupling agent (Dow Corning Z-6040) mixed with methanol solution. The chopped coir fibers are immersed into these solutions while mechanical stirrer is used to stir the mixture to obtain uniform coating layer of silane on the natural fiber surfaces in room temperature for 1 hour. Then, the treated fibers are dried in furnace environment for 12 hour at temperature 50°C. Low heating temperature is used because methanol solution is easily evaporated event at room temperature. All fibers are placed in room condition with relative humidity 55% for not less than 24 hours prior to composite preparation. The treated fibers are placed in rectangular mould 200mm x 150mm in random orientations and polyester resin is poured into the mould contained the treated fibers. Three different fiber volume fractions are selected in this work; they are 5%, 10% and 15%. Polyester resin is carefully poured into the mould in order to avoid any pore formations. Then, male mould is pressed onto the wet composite to obtain 4mm thickness and to squeeze out the excessive resin. The composites are removed from the mould after 24 hours of curing process to obtain optimum hardness and shrinkage.

Table 1 Properties of coconut coir [1].

Density (g/cm <sup>3</sup> )	Tensile strength (MPa)	Young modulus (GPa)	Elongation at failure (%)	Moisture absorption (%)
1.25	600-700	6	15-25	10

### Tensile Test

Each composite board that contained different fiber volume fractions are shaped into tensile test specimen and all specimens are conditioned based on the standard procedure ASTM D618-99 before mechanical tests are conducted. Tensile tests are conducted in laboratory environment at temperature 24°C and at constant crosshead displacement 1.5mm/min. The specimens are carefully aligned in order to obtain pure uniaxial tensile loading and prevent any bending moment imposed onto the specimens.

This is important to produce better results. Extensometer is also used to measure specimens, elongation. This device is installed along the gauge length of tensile specimens. Stress/strain curves are automatically recorded by the universal testing machine GOTECH. Prior to the tensile test, specimens are pre-stressed lightly in order to avoid any results misinterpretation especially in the region of linear deformation.

### **Fracture Surface Observation**

The fracture surfaces of tensile test samples are investigated with a Leica Cambridge S-360 Scanning Electron Microscope (SEM). The specimens are sputter-coated with a thin layer of platinum to avoid electrostatic charging during investigation. During the observations, the emphasized sites are given, especially at the interfacial contact between fiber/matrix and to the whole fractured surface, to picture the toughening mechanism in order to strengthen the fiber reinforced composites.

## **RESULTS AND DISCUSSION**

### **Effect of Silane Treatments on Tensile Properties**

Obviously, the presence of silane coupling agent affected the tensile properties of the natural fiber reinforced composites. Silane also contributed to the interfacial compatibility between fiber and matrix. Purification process alone not significantly affected the tensile properties of this composite. Figure 1 and 2 show the interrelationship between silane concentrations coated on the fiber surface with Young modulus and ultimate tensile strength of the composites at different fiber loadings, as expected these properties increase with the fiber volume fractions and silane concentrations. Generally, silane coupling agent coated on the fiber surfaces increased the Young modulus for all composites containing different fiber loadings. Higher silane concentration contributes to the uniform distribution of silane around the fiber surface therefore, improving the interfacial bonding between fiber and matrix. Possible explanation with low silane density, this coupling agent is not distributed uniformly to the whole fiber surface. This behavior will produces weak bonding at the interfacial contact between matrix and fiber. At the same, stress will concentrates at this location reducing the composite integrity to sustain higher tensile loading.

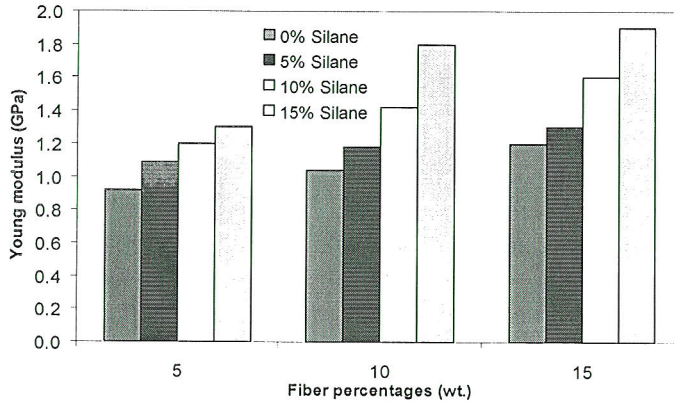


Figure 1. Effect of fiber percentages on modulus young at different silane treatment.

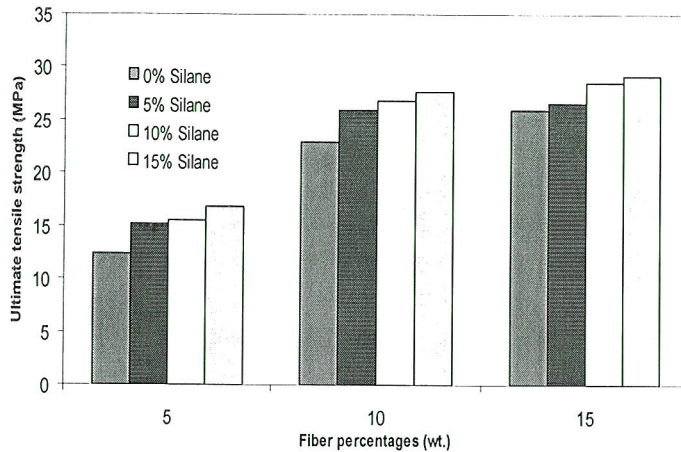


Figure 2. Effect of fiber percentages on ultimate tensile strength at different silane treatment.

Theoretical calculations of Young's modulus in the longitudinal direction are calculated using eq. (1):

$$E_c = E_f V_f + E_m V_m \quad (1)$$

where,  $E_c$  is composite Young's modulus,  $E_f$  is fiber Young's modulus,  $E_m$  is matrix Young's modulus,  $V_f$  is fiber volume fraction,  $V_m$  is matrix volume fraction. A comparison of experimental and theoretical tensile modulus versus volume fraction is carried out.



The data indicate that experimental values are lower than theoretical values. The differences between these values may be attributed to the difference of interfacial bonding between matrix and fiber. In equation (1), the interfacial bonding between matrix/fiber interfaces are not taken into account therefore, equation (1) alone cannot be used to estimate the Young's modulus. For fiber reinforced composites, interfacial bonding plays an important role in determining the load-bearing performance of the composites. Figure 3, shows the differences between theoretical and experimental values. Similar research report may be found in [9]. Theoretical values are increased linearly as fiber loadings increase, these values actually are overestimated because the fibers are purified and treated with and without silane. Figure 3 also reveal that, the experimental values are always lowered that theoretical values.

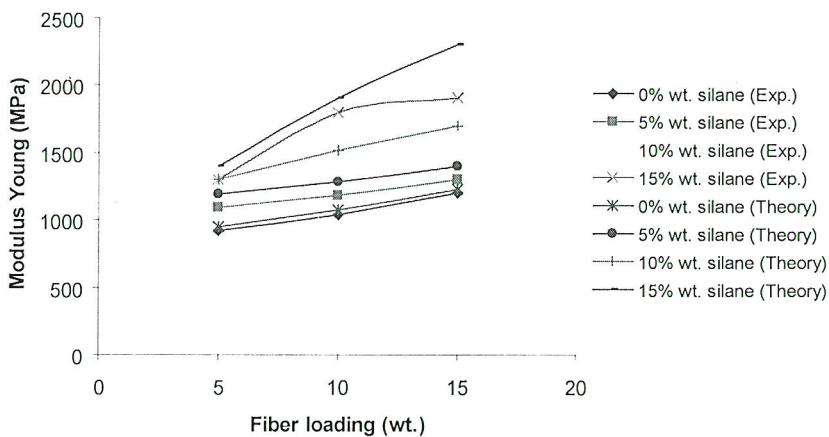


Figure 3. Theoretical and experimental values of composite contained different fiber loadings and treated with different silane concentrations.

### Effect of Fiber Loading on Tensile Properties

The tensile properties are significantly affected by fiber loadings (Figures 1 and 2), where increasing fiber loadings have strengthened the fiber reinforced composites. Therefore, increasing the effectiveness of load transferring from the matrix to the fibers. But higher fiber content will also produce the bonding imperfections especially at interface between matrix/fiber. According to [9, 10] reported that, composites failed at the interface because of the weak interfacial bonding. The weak interfacial bonding also affects Young's modulus and tensile strength of the composites. Figure 3 also shows that, 15% fiber loading does not contribute to increase the ultimate tensile strength significantly. In this work, maximum elongation of fibers is lower than matrix material, therefore increasing fiber loadings lead to weaken the composite because the fiber is unable to sustain higher tensile loadings. Figure 4 depicts the influence of silane treatments on strain at failure at different fiber loadings. It is seen that, the presence of

silane alters the strain at failure of composites. Higher failure strain occurs for 10% and 15% fiber loading composites. These behaviors may be attributed to interfacial bonding between fiber and matrix. Higher fiber contents more effective the performance of load-bearing of the composites. Strain at failure indicates the composite capability to sustain higher tensile loading through different failure mechanisms.

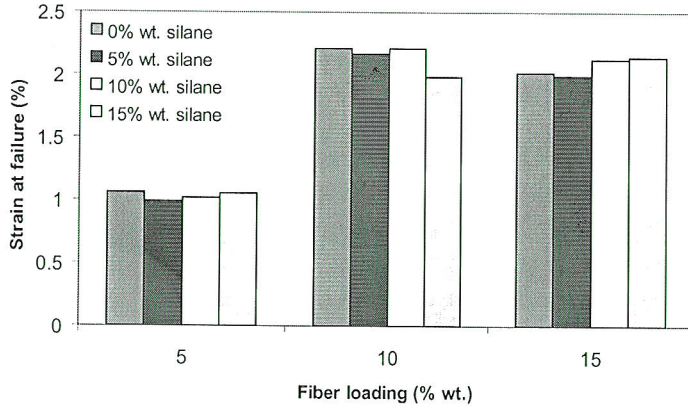


Figure 4. Effect of fiber loading on strain at failure at different silane concentrations.

### Fracture Mechanisms of Composites Under Tensile Loadings

The interfacial adhesion characteristics have a significant effect on the load carrying capacity of a natural fiber reinforced composite. From [11] stated that the average interfacial shear strength can be calculated with the following equation.

$$\tau_c = k \frac{1}{2} \sigma_c \left( \frac{d_f}{l_c} \right) \quad (2)$$

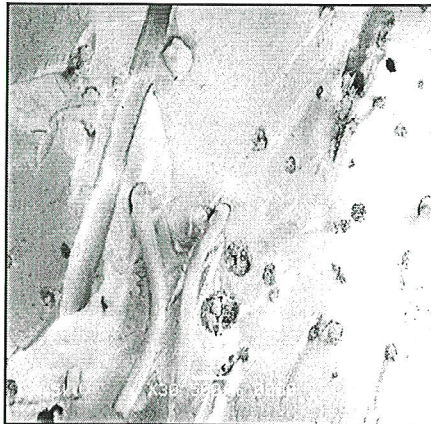
Where,

- $\tau$  Interfacial shear strength between fiber and matrix.
- $\sigma_c$  Fiber tensile strength (MPa)
- $d_f$  Fiber mean diameter
- $l$  Average fragment length
- $k$  Statistical correction factor ( $k = 0.889$ )

Therefore, longer pull-out fiber lowers interfacial shear strength. This is due to poor interfacial bonding between fiber and matrix. Figure 5(a) depicts the fracture mechanism of 5% fiber loading reinforced composite under tensile loading. Longer pull-out fibers

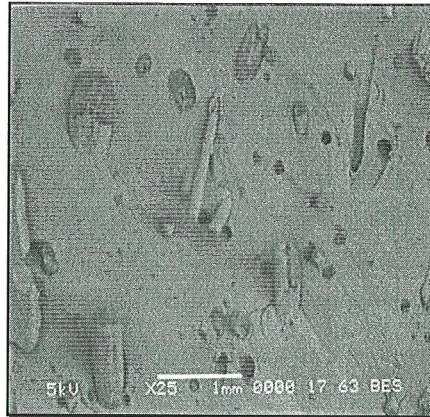
are observed about 2mm accompany with interfacial debonding. At the left side of Figure 5(a) shows a groove of fiber pushed-out during the stress mode indicating that the contact bonding between these fiber and matrix materials are weak. For this type of composite also found that the matrix is well gripping the fiber indicating that purification process is responsible to strengthen the composite but not significantly affected. 5% fiber loading composites results the lowest tensile properties. For comparison with Figure 5(a), Figure 5(b) reveals that 5% fiber loading composite treated with silane coupling agent and there is no interfacial debonding occurs and shorter pull-out fibers are detected showing this composite is tougher than untreated fiber reinforced composite. This result is well agreed with Figures 1 and 2.

Figure 6 shows the fracture mechanisms of 15% fiber loading composites. Each fiber is treated with 10% and 15% silane coupling agent, respectively. Both Figures 6a and 6b have shorter pull-out fibers than in Figure 5, indicating that the composite integrity is stronger under tensile load. But for 10% silane treated fibers, interfacial debonding occurs and weakening the composite. Prior to fracture, interfacial debondings play an important role in concentrating the stress at those locations and degrading the fiber strength. In this fracture surface, interfacial debonding is not a dominant factor in degrading the composite integrity because most of pull-out fibers still are intact within the matrix.



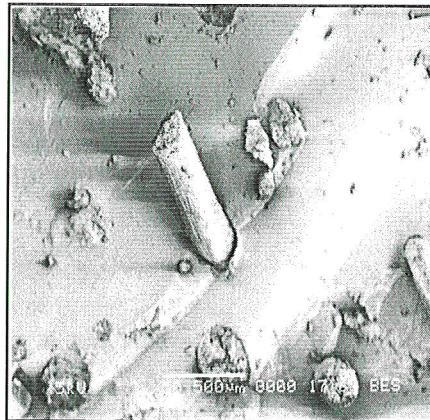
(a)





(b)

Figure 5. Fracture mechanisms of (a) untreated fibers with silane 5% fiber loading composite and (b) treated fibers with silane 5% fiber loading composite.



(a)



(b)

Figure 6. Fracture mechanisms of 15% fiber loadings and treated with (a) 10% and (b) 15% of silane coupling agent.

## CONCLUSION

The results from this study shows that, the purification process alone is not effective enough to improved the wetability process between polymeric resin and cellulose or natural fibers. The presence of silane coupling agent on coir fiber surfaces improved the tensile properties of composites and at the same time, fiber loadings also significantly affected the tensile properties. Both increasing silane concentration coated on fiber surfaces and fiber loadings increased the mechanical properties of natural fiber reinforced composites. The lower values of tensile properties especially for untreated fiber with silane may be attribute to poor interaction between matrix and fiber through ineffective load-bearing application. From SEM observation, longer pull-out fibers are observed for untreated fiber with interfacial debonding dominated the fracture surfaces while for treated fibers, shorter pull-out fibers are detected and interfacial debonding also occur but not significantly dominated the fracture surfaces.

## REFERENCES

1. Goulart Silva G et al. (2000). Mechanical and thermal characterization of native Brazilian coir fiber. *J. Appl. Poly. Sci.* 22:468-475.
2. Pedro JHF, Alex VG. (2005). *Fiber-matrix Adhesion in natural fiber composites*. Chapter in *Natural fibers, biopolymers and biocomposites*, edited by Amar KM et al. Tylor and Francis, pp. 177-230.

3. Miller AC, Berg JC. (2003). Effect of silane coupling agent adsorbate structure on adhesion performance with a polymeric matrix. *Composites 34A*:327–332.
4. Hanafi Ismail et al. (2001). The effect of a compatibilizer on the mechanical properties and mass swell of white rice husk ash filled natural rubber/linear low density polyethylene blends. *Polymer Testing 20*:125 – 133.
5. Valadez-Gonzalez A et al. (1999). Effect of fiber surface treatment on the fiber – matrix bond strength of natural fiber reinforced composites. *Composites 30B*:309 – 320.
6. Rout J. et al. (2001). The influence of fiber treatment on the performance of coir-polyester composites. *Polym. Compos 61*:1303 – 1310.
7. Roger M. Rowell, Anand Sanadi, Rod Jacobson, and Dan Caulfield. (2005), In: *Kenaf Properties, Processing and Products*; Mississippi State University, Ag. and BioEngineering. ISBN 0-9670559-0-3. Chapter 32, pp. 381-392.
8. X. Li, S.A. Panigrahi, L.G. Tabil, W.J. Crerar. (2004). *Flax Fiber-reinforced Composites and the Effect of Chemical Treatments on their Properties*. North Central ASAE/CSAE Conference Sponsored by the Manitoba Section of CSAE Winnipeg, Manitoba, Canada September 24-25.
9. R.M.N. Arib, S.M. Sapuan, M.M.H.M. Ahmad, M.T. Paridah, H.M.D. Khairul Zaman. (2006). Mechanical properties of pineapple leaf fibre reinforced polypropylene composites. *Materials and Design 27*:391–396.
10. Luo S, Netravali N. (1999). Mechanical and thermal properties of environment-friendly “green” composites made from pineapple leaf fibres and poly (hydroxybutyrate-co-valerate) resin. *Polym Compos 20*(3):367–78.
11. Torres FG, Cubillas. (2005). Study of the interfacial properties of natural fiber reinforced polyethylene. *Polymer Testing 24*:694–698.