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# Stone apple reinforced clay composite

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# ABSTRACT

Natural Fibers in biocomposites have grown because they are light weight, combustible, non toxic, low cost, and easy to recycle. Lack of good interfacial adhesion, poor resistance to moisture absorption, and poor thermal stability, make the use of natural fiber reinforced composites less attractive. In this paper, these drawbacks have been tried to overcome by using stone apple as natural fiber for reinforced biocomposites which show remarkable thermal stability and interfacial adhesion. Thermal Stability has been studied using DSC (Differential Scanning Calorimeter) while SEM (Sacnning Electron Micrograph) and FTIR (Fourier Transform IR spectroscopy) show strong interfacial adhesion between fibers of stone apple and clay polymer matrix. Thus this paper aims to make a green material.

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#### **1. INTRODUCTION**

Now a days utilization and recycling of plastics have become a global environmental problem. Incineration of plastics emits large amount of carbon-dioxide which creates global warming and pollution and recycling involves expenditure of labour and energy. Need of the hour is to develop a green polymeric material that would not involve the use of toxic components in their manufacture and could be degraded in the natural environment products<sup>[1]</sup>. Due to such severe environmental concerns, the commercial utilization of biological polymers has become an active research area due to low cost availability and biodegradable properties<sup>[2]</sup>.

PLA (polylactide) and polysaccharides are the most promising candidates because they are made or come from naturally abundant products and are readily biodegradable<sup>[3]</sup>.

Renewable resource based biodegradable polymers

include cellulosic plastic (plastic made from wood), PLA corn derived plastics and PHA (polyhydroxyalkanoate) bacterial polyesters. Methylcellulose is well known for its use as environmentally friendly products, especially as coating or mulching film, low cost and easy process ability<sup>[4]</sup>.

In order to render the biopolymers able to compete with stronger polymers such as PE (polyethylene) or PP (polypropylene), it is needed to improve their properties such as thermal stability, mechanical properties, and barrier properties. Polymer layer silicate nanocomposites technology has already proven to be a good way to improve these properties significantly<sup>[3]</sup>.

The final composites often exhibit a desired enhancement of physical/ or chemical properties relative to the virgin polymer matrix, even at very low clay content (< or = 5 wt%) which results in high aspect ratio and high surface area. MMT (montmorillonite) is one of the most important natural clays and consists a

# **KEYWORDS**

Nanoclays; Hybrid composites; Recycling; Fiber/matrix bond; Thermal properties.

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stacked structure of parallel silicate layers. Depending on the extent of compatibility between clay and polymer matrix, conventional composites, intercalated or exfoliated morphologies can be obtained<sup>[5]</sup>. Though clay is an inexpensive natural mineral, used as filler for rubber and plastics, yet its reinforcing ability is poor. Reinforcing ability of clay polymer matrix can be strengthened by use of natural fibers.

Natural fibers such as bamboo<sup>[6]</sup>, sisal<sup>[7]</sup>, switchgrass<sup>[8]</sup>, soybean oil<sup>[9]</sup>, biodegradable resins<sup>[10]</sup> in biocomposites has grown because they are light weight, combustible, non toxic, low cost and easy to recycle<sup>[11]</sup>.

Vegetable fibers used with cement mortar can produce high performance fireboard, which can be used as a substitute for asbestos cement. They can also be used with soil to form load bearing structures<sup>[7]</sup>.

The major problem lies in natural fibers being used as reinforcement is that they readily absorb moisture because they contain polar hydroxide groups which result in a high moisture sorption level of natural fibers reinforced polymer matrix composites, thus preventing extensive applications of these materials. Another limiting factor is that natural fibers are thermally unstable at elevated temperatures and are thermally weakened at about 160°C<sup>[12]</sup>. Lack of good interfacial adhesion also makes the use of natural fiber reinforced composites less attractive<sup>[13]</sup>.

Previous works have described the use of perox-



Figure 1: Cross section of stone apple fiber

ide to improve adhesion in cellulose-fiber reinforced thermoplastic composites leading to improved mechanical properties<sup>[14]</sup>.

Though much attention is given to polymer clay hybrids, little attention has been given to biopolymer clay hybrid materials.

In this paper, we focus our attention in preparing a green composite reinforced with natural fiber using clay (dispersed by natural dispersant), a biodegradable polymer CMC (carboxy methylcellulose) and fibers of stone apple. The characterization of this composite is done by DSC, SEM, and FTIR. Cross section of stone apple fruit (Aegle Marmalos) has been shown in figure 1.

# 2. EXPERIMENTAL

# 2.1. Materials

Clay used in this experiment was mainly Bikaner Bentonite. Natural dispersant (soap stone powder) was used to disperse clay and was obtained from natural source. Fibers of stone apple were obtained from natural source. CMC (carboxy methylcellulose) was obtained from Loba Chemie. PVA (polyvinyl alcohol) was obtained from S.D. Fine Chemicals Limited.

# 2.2. Modification of clay

Clay was made organophilic by natural dispersant. To 1 gm clay, 0.1% dispersant solution (0.01 gm of dispersant in 10 ml water) was mixed under constant stirring for 4 hours so that clay gets well dispersed in it. 1 drop of clove oil was added to the natural dispersed clay suspension to keep it bacteria free. The resulting suspension was centrifuged and dried at room temperature.

# **2.3. Preparation of stone apple reinforced clay biocomposite**

5 wt% clay (dispersed by natural dispersant) was taken and suspended in water. To it 1% CMC solution was added and stirred till homogenous suspension was obtained. Fibers of stone apple were then added to clay CMC suspension and stirred well till all the fibers have dispersed in the suspension. The resulting suspension was then spread as a film on a glass slide of uniform thickness and allowed to dry at room temperature. The same method was carried out using PVA as

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Figure 2: IR spectra of stone apple clay hybrid



Temperature (°C) Figure 3 : DSC of stone apple clay hybrid



Figure 4: SEM of stone apple clay network

binder in place of CMC. It was observed that fibers of stone apple did not interact with PVA and was immiscible.

# 2.4. Instrumentation

DSC of stone apple-clay hybrid was performed on Differential Scanning Calorimeter Perkin Elmer DSC-7 under nitrogen atmosphere at a scan rate of 20°C/ min. SEM of stone apple- clay hybrids was taken using



Figure 5: SEM of stone apple powder

Hitachi S-3400N Japan using 3 KV. IR was done on Perkin Elmer FTIR spectrometer Spectrum 2000.

# **3. RESULTS AND DISCUSSION**

# 3.1. IR analysis

IR spectrum of stone apple indicates the presence of saturated esters. The three group wave numbers correspond to the above indication, C=O stretch peak at 1750-1735 cm<sup>-1</sup>, C-C-O stretch at 1210-1160 cm<sup>-1</sup>, and O-C-C stretch at 1100-1030 cm<sup>-1</sup>. In the IR spectrum of stone apple clay film, Si- O- Si asymmetric stretch at 1040 cm<sup>-1</sup> (peak due to swelling) and Si-O symmetric stretch peaks are very much reduced, C-C-O stretch peaks at 1210-1160 cm<sup>-1</sup>, and O-C-C stretch peaks at 1100-1030 cm<sup>-1</sup> are also lost, indicating interaction of clay matrix with stone apple as can be seen in figure 2. Some bond formation might have taken place between stone apple and clay matrix.

# 3.2. DSC analysis

In figure 3, Stone apple gives one endothermic peak at 90°C and another at 210°C. Stone apple clay film gives one major broad endothermic peak at 90°C only indicating moisture loss. Intercalation may have taken place between clay matrix and stone apple fibers. Film without stone apple gives one endothermic peak at 90°C and decomposition peak at 270°C. It can thus be said that stone apple fibers provide thermal stability to clay film.

# 3.3. SEM analysis

SEM samples of stone apple reinforced clay hybrid reveal that stone apple and clay form a mesh network as can be seen in figure 4. Fibers of stone apple

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Figure 6: SEM showing adherence of stone apple fibers on clay platelets

are shown in figure 5. Adherence of stone apple on clay platelets can be seen in figure 6.

#### 4. CONCLUSION

Based on our results, the following can be concluded:

- Thermal stability of stone apple reinforced clay hybrid is quite enhanced and the hybrid material shows stability till 300°C.
- Since fibers of stone apple contain saturated esters, moisture absorption is to a less extent.
- Stone apple fibers bind well with clay polymer matrix as can be seen from SEM images.
- Thus the main drawbacks of natural fiber reinforced clay hybrid, that of moisture absorption due to presence of hydroxide groups, lack of interfacial adhesion and poor thermal stability have been tried to overcome in this work to a certain extent.

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