

## **EFFECT OF FIRE RETARDANT TREATMENT ON THERMAL PROPERTIES OF JUTE FIBER**

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### **ABSTRACT**

To keep the world green for future generation the consciousness on natural fiber increases. However, the idea of producing long lasting composite materials made of jute and natural fiber has not been considered in the past because of their swelling and burning properties. Moreover, the major weakness of natural fiber is that they can easily burn due cellulosic materials. In contrast, the long cellulosic fiber come in contact with fire and ignition has occurred as well as shows poor mechanical properties. Due to this fire retardant treatment has been done on the jute fiber. Following the trend, jute fiber was made fire retardant by using a modified method. With the help of ammonium salt fire retardant treatment has been established. The experimental investigation of fire retardant treated jute fibers were carried out by means of FTIR, DSC and TGA. After fire retardant treatment, the jute fiber became more oxidation resistant compared to the raw jute fiber. This concludes that the thermal stability increase but the rate of pyrolysis decreased after fire retardant treatment compared to the raw jute fiber. The crystallization properties of the fire retardant treated fiber increased with the chemical concentrations.

**Keywords:** Jute fiber, Fire retardant, FTIR, DSC, TGA, Crystallization.

### **1. INTRODUCTION**

Natural fibers are biodegradable, non toxic, recyclable and energy efficient. Due to their eco-friendly nature, natural fibers reduce the issue of waste disposal as seen in the case of synthetic fibers such as glass, carbon and Kevlar. Jute is a natural fiber which offers a number of benefits as reinforcement in synthetic polymers. It is the cheapest lignocellulosic long vegetable bast fiber and abundantly available in South Asia, especially in Bangladesh. Natural fibers are amenable to chemical modification due to the presence of hydroxyl groups. The interfacial properties can be improved by giving appropriate modifications to the components, which gives rise to changes in physical and chemical interactions at the interface. Most of the research done so far is concentrated on the fiber surface treatment. An enormous amount of work has been conducted in the field of fiber modification [1-4]. Thermal properties of water retardant jute fiber [5] and other natural fiber

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[6-8] were evaluated by different researchers. To our knowledge, no work has been reported on fire retardant treated jute fiber. With a view to investigate new fire retardant treated jute fiber, present work deals fire retardant treatment of jute fiber, structural as well a thermal study of the treated jute fiber.

## 2. MATERIALS AND METHOD

The jute fibers were collected from the Bangladesh Jute Research Institute (BJRI), Faridpur Regional Station, Bangladesh. The supplied jute fiber was CVL-1 (*Corchorus capsularis* L.). The middle portions of the whole jute fiber were cut for fire retardant treatment. Di-ammonium hydrogen phosphate and lissapol were used for fire retardant (FR) treatment. Raw and treated fiber was characterized by FT-IR using a Digital Fourier Transform Infrared spectrophotometer, Model Nicolet-380, USA using a technique of Attenuated Total Reflectance (ATR). The transmittance range of scan was 370 to 4000  $\text{cm}^{-1}$ . The thermal (decomposition) behavior of 20%, 25% and 30% fire retardant treated jute fibers were investigated by means of Differential Scanning Calorimetry (DSC) and Thermo Gravimetric Analysis (TGA). The DSC measurement involved DSC Q10 in nitrogen atmosphere. Control and treated jute fiber samples weighing between 5 and 10 mg were placed in an aluminum capsule sealed and punctured to allow gases to escape during heating. The heating scheme ranged between 30 to 500°C and heating rate was 10°C/min. The thermographs were analyzed for any changes in the thermal behavior of jute and fire retardant (FR) treated fibers. The TGA measurements were done in TA Instrument SDT Q50 computer-controlled thermo-gravimetric analyzer. Fiber samples cut to 1 cm lengths weighing 50-70 mg were used for the analysis. The samples were heated from ambient to 500°C under nitrogen purging in all experiments. A heating rate of 10°C/ minute was maintained. Computer software was used to obtain data for derivative thermo gravimetric (DTGA) analysis from the TGA experiments.

## 3. RESULTS AND DISCUSSION

The jute fiber chars and burns without melting until heating up to high temperature. Even with the supply of sufficient heat energy, the long chain molecules bound by numerous hydrogen bonds cannot flow past one another to exhibit plasticity. With increasing heat the chain molecules vibrate more and ultimately disintegrate leading to violent chemical reactions observed as fiber combustion [9].

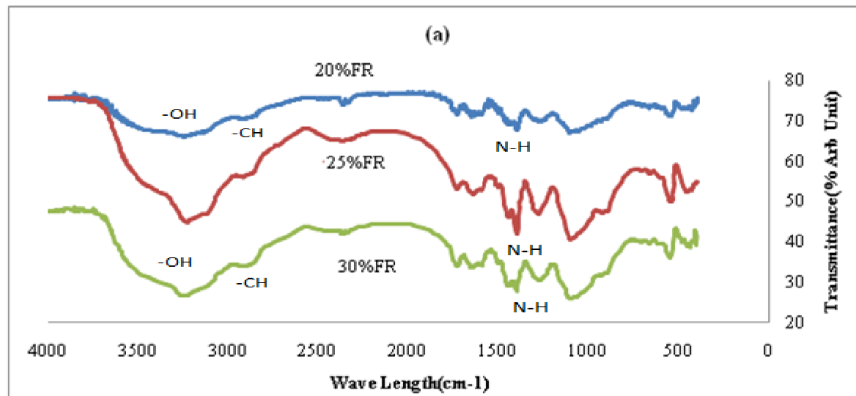
Figure 1 shows a decrease in the intensity of the O-H absorption band at 3450–3400  $\text{cm}^{-1}$  indicating that the hydroxyl group contents in jute were reduced after fire retardant reaction of top, middle and bottom portions jute fiber. However after the FR treatment, this absorption bands occurred at higher wave lengths. There is one thing that the peak of the treated fiber became sharper. Absorption in the first region is obtained from O-H stretching vibrations in hydroxyl, phenol and carboxyl groups. The intensity of the O-H peak also increased due to more hydroxyl group present in fire retardant treated jute fiber. After fire retardant treatment, the C-H peak is clearly abolished. The intensity of C-H stretching decreased with the fire retardant chemical concentration. Approximately around 1430  $\text{cm}^{-1}$ , N-H bending observed due to fire retardant chemical i.e.  $\text{NH}_4$ . There are two new peaks observed after fire retardant treatment at 400 and 500  $\text{cm}^{-1}$ [10]. The absorption at the lower band (1300-800  $\text{cm}^{-1}$ ) is also present may be due to esterification [11].

### 3.1. Thermo Gravimetric Analysis

Weight change in different temperature, 10% and 50% weight loss are shown in Table 1, which were higher than the control. Residue was also higher compared to the control jute fiber that means flammability of FR treated jute fibers were lower compared to the control jute fiber. The residue at 600°C temperature of FR jute fiber is higher due to volatile material [12] coated by FR treatment that made them non volatile. The mass loss (Figure 2 (a)) and derivative mass loss (Figure 2 (b)) due the heat was lower compared to the control. The control fiber degraded earlier than the fire retardant treated jute fiber. It means that FR treatment occurred in the fiber surface. It can be also concluded that the thermal effect gradually changed with the fire retardant chemical concentration.

**Table 1.** TGA data of control and FR treated jute fiber.

| Sample    | Weight change between 50-150°C (%) | Weight change between 150-300°C (%) | Weight change between 300-700°C (%) | Derivative weight change temp (%/°C) | Residual weight (%) | 50% Weight loss temp (°C) | 10% Weight loss temp (°C) |
|-----------|------------------------------------|-------------------------------------|-------------------------------------|--------------------------------------|---------------------|---------------------------|---------------------------|
| 20%FRM    | 5.451                              | 39.65                               | 22.24                               | 263.29                               | 30.48               | 336.35                    | 208.51                    |
| 25%FRM    | 4.339                              | 38.84                               | 21.27                               | 262.23                               | 33.53               | 358.50                    | 208.59                    |
| 30%FRM    | 2.028                              | 39.06                               | 19.61                               | 268.61                               | 37.71               | 464.60                    | 281.00                    |
| Control M | 5.471                              | 17.83                               | 55.82                               | 332.85                               | 13.96               | 327.10                    | 76.31                     |



**Fig. 1.** FTIR of the raw and treated jute fiber.

The derivative weight change of the fire retardant treated fiber decreased compared to the control jute fiber due to the fire retardant chemical ( $\text{NH}_4$ ) property. Thus the oxidation resistance increased with the fire retardant chemical concentrations. Due to coating property of the  $\text{NH}_4$  salt, which acts as a fire retardant element, the FR treated fiber became thermally stable at higher temperature. The first weight decay is related to moisture loss [13] with a peak observed at 50°C for all samples. It is

higher for raw jute compared to the treated jute fiber, because the coating inhibits the moisture to vaporize. The burning temperature of cellulose is about 230°C, whereas afterglow temperature is approximately 345°C. Although the cellulose start burning before 230°C in case of treated jute fiber but the cellulose does not fully burn due to fire retardant coating. The afterglow temperature is higher and mass changed is lower of treated jute fiber due to phosphorus coating materials [13].

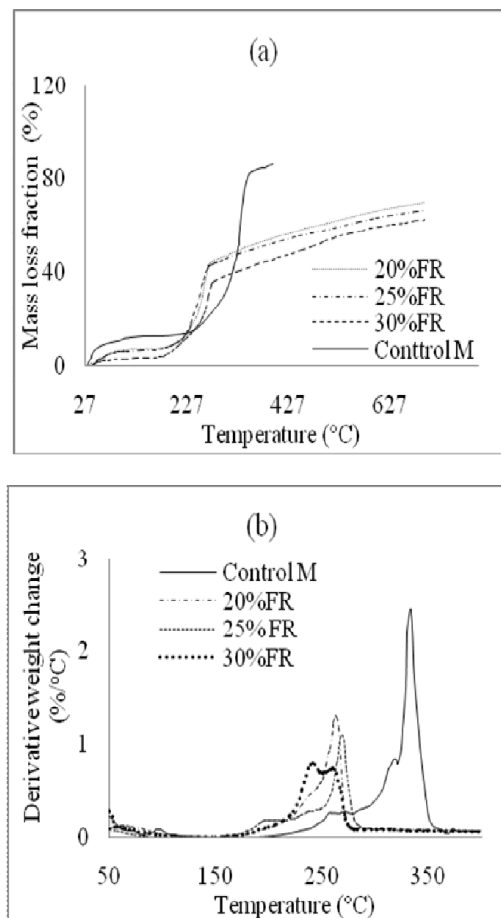


Fig. 2. (a) Mass loss and (b) DTGA thermograph of fire retardant jute fiber.

### 3.2. Digital Scanning Calorimetric Analysis

Crystallinity and first exothermic peak temperature are a function of chemical concentration [14-15]. The 1<sup>st</sup> endothermic peaks temperature for the fire retardant jute fiber increased with the fire retardant chemical concentration (Tables 2). The first exothermic peak temperature was maximum for 20%FR and decreased with the increase of chemical concentration (Table 2 and Figure 3) and this changed due to the presence of reactive groups in the fire retardant jute fiber. A broad

endothermic peak in the temperature range of 60-140°C in both treated and raw jute fiber corresponds to the heat of vaporization of water absorbed in the fiber. It is reported [16] that in cellulose fiber lignin degrades at around 200°C whereas the other polysaccharides such as cellulose degrade at higher temperature. Therefore, the peak which is at higher temperature than 200°C indicates the decomposition of cellulose in the fiber. The first exothermic peak in the raw jute fiber is about 290°C due to thermal degradation of cellulose and the glycosidic linkages of cellulose. The very strong 2nd endothermic peak indicates the degradation of cellulose, leading to the formation of char. In addition, there is one more step in the peak at around 431°C. In this step the rest of the char is oxidized and the rest of the mass consumed. The appearance of both endo and exothermic peaks (Table 2) could be related to the morphological changes possibly occurred by the decomposition of hemi-cellulose and cellulose respectively. The peaks are sharper more than raw jute fiber may be due to  $\text{NH}_4$  coating. The slope of the heat flow increased with the chemical concentration as shown in Figures 3. Due to coating property of the  $\text{NH}_4$  salt, which is a fire retardant element, the FR treated fiber heat flow decreased compared to the control jute fiber.

**Table 2.** Peak temperature and nature of peak of FR jute fiber from DSC data.

| Sample    | 1 <sup>st</sup> peak temp (°C) | 2 <sup>nd</sup> peak temp (°C) | 3 <sup>rd</sup> peak temp (°C) | 4 <sup>th</sup> peak temp (°C) | Nature of 1 <sup>st</sup> peak | Nature of 2 <sup>nd</sup> peak | Nature of 3 <sup>rd</sup> peak | Nature of 4 <sup>th</sup> peak |
|-----------|--------------------------------|--------------------------------|--------------------------------|--------------------------------|--------------------------------|--------------------------------|--------------------------------|--------------------------------|
| 20% FRM   | 76.53                          | 202.64                         | 282.18                         | 293.64                         | endo                           | endo                           | exo                            | exo                            |
| 25% FRM   | 73.25                          | 203.42                         | 266.71                         | 290.60                         | endo                           | endo                           | exo                            | exo                            |
| 30% FRM   | 66.36                          | 200.87                         | 275.39                         | 189.10                         | endo                           | endo                           | exo                            | exo                            |
| Control M | 65.07                          | 353.83                         | -                              | 396.82                         | endo                           | endo                           | -                              | exo                            |

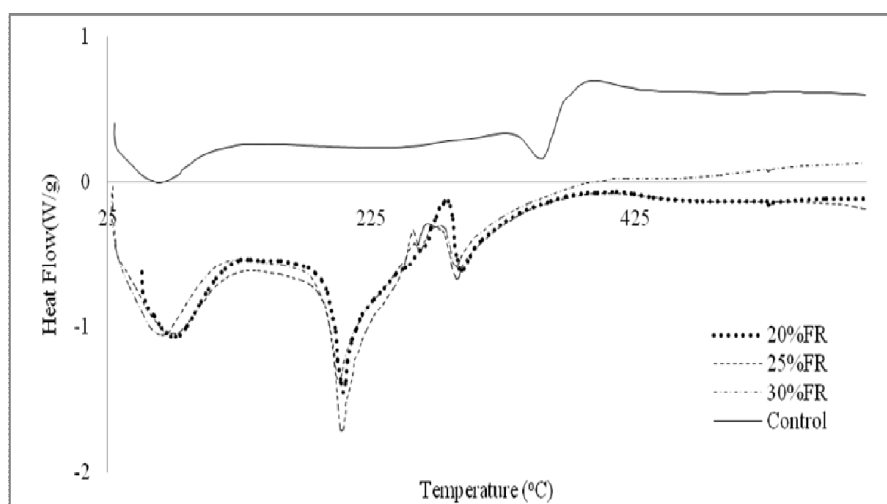


Fig. 3. DSC thermograph of fire retardant jute fibers.

#### 4. CONCLUSIONS

Fire retardant treatment of jute fibers effectively changes the surface topography of the fibers and their crystallographic structure. However care must be taken in selecting the concentration of diammonium hydrogen phosphate for fire retardant as results show that some fibers at certain chemical concentration level enhance thermal resistance as elucidated by the DSC method. The coating impurities on plant fibers may be an advantage for fiber to matrix adhesion as it may facilitate both mechanical interlocking and the bonding reaction due to the exposure of the hydroxyl groups to chemicals such as resins and dyes. TGA (both non-oxidative or oxidative), on the basis of the presented results, can serve as a standard procedure for determining the influence of fire retardant chemicals on thermal stability. The results indicate that fire retardant chemicals have an influence on the thermal stability of the FR treated jute fiber. After fire retardant treatment, the jute fiber became more oxidation resistant compared to the raw jute fiber. Thermal stability and residue of fire retardant treated fiber increased above temperature of 325°C compared to the control jute fiber. The barrier mechanism is the most widely proposed mechanism by which composite formation imparts fire retardancy to polymers.

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