Differential Thermal Analysis of Argon and Oxygen Plasma Treated Jute

Md. Masroor Anwer, Md. Mahbubul Hoque

Abstract— **Low temperature plasma (LTP) treatment, a kind of environmentally friendly surface modification technique, was applied to biodegradable and ligno-cellulosic jute fibre with the use of two nonpolymerizing gases, namely argon (Ar) and oxygen (O2) at various discharge power levels of 50, 75 and 100 W and exposure times 5, 10, 15 and 20 min. with a flow rate of 0.2 L/min. Differential thermal properties of both raw and low temperature Ar and O2 plasma treated jute were studied at various discharge power levels and exposure times. From Differential Thermal Analysis (DTA), it is seen that a broad endothermic peak was observed in the temperature range of 60–120 □C in both raw jute and LTP treated jute. Degradation temperature of celluloses increases slightly with the increment of both discharge powers and exposure times when LTP treatment done by the Ar or by the O2. In addition, degradation temperature of hemicellulose remain constant when treatment was done by the Ar plasma but for the case of O2 plasma, degradation temperature increases with the increase of discharge power as well as treatment time. From the DTA thermogram, it is seen that the fibres degradation temperatures for both the celluloses and hemicelluloses were unstabled.**

Index Terms— **Differential Thermal Analysis, Discharge Power, Exposure Time, Jute and Plasma**

I. INTRODUCTION

Thermal analysis comprises a group of techniques in which a physical property of a substance is measured as a function of temperature, while the substance is subjected to a controlled temperature programme. In differential thermal analysis, the temperature difference that develops between a sample and an inert reference material is measured, when both are subjected to identical heat treatments. Differential Thermal Analysis (DTA) is a thermo analytic technique [1]. DTA is applied to study structural and phase change that occurs during heating a polymeric sample. In DTA, the material under study and an inert reference are made to undergo identical thermal cycles, while recording any temperature difference between sample and reference. This differential temperature is then plotted against time, or against temperature (DTA curve or thermo gram). Changes in the sample, either exothermic or endothermic, can be detected relative to the inert reference. Thus, a DTA curve provides data on the transformations that have occurred, such as glass transitions, crystallization, melting and sublimation. The area under a DTA peak is the enthalpy change and is not affected by the heat capacity of the sample [2]. In Chemistry, endothermic is a reaction that absorbs energy, while in thermodynamics, the word endothermic describes a process or reaction in which the system absorbs energy from the surroundings in the form of heat or in other words endothermic reactions are the chemical reactions that must absorb energy in order to proceed. Endothermic reactions cannot occur spontaneously. Work must be done in order to get these reactions to occur. When endothermic reactions absorb energy, a temperature drop is measured during the reaction. Endothermic reactions are characterized by positive heat flow (into the reaction) and an increase in enthalpy (+ΔH). Photosynthesis is an example of an endothermic chemical reaction. In this process, plants use the energy from the sun to convert carbon dioxide and water into glucose and oxygen. Many chemical reactions release energy in the form of heat, light, or sound. These are exothermic reactions. Exothermic reactions may occur spontaneously and result in higher randomness or entropy ($\Delta S > 0$) of the system. They are denoted by a negative heat flow (heat is lost to the surroundings) and decrease in enthalpy ($\Delta H < 0$). In the lab, exothermic reactions produce heat or may even be explosive. An example of an exothermic reaction is the mixture of sodium and chlorine to yield table salt [3].

Plasmas are ionized gases. An ionized gas consists mainly of positively charged molecules or atoms and negatively charged electrons [4]. A gaseous complex that may be composed of electrons, ions of both polarity, gas atoms and molecules in the ground or any higher state of any form of excitation as well as of light quanta is referred to as plasma [5]. The ionization degree can vary from 100 % (fully ionized gases) to very low values (partially ionized gases). The presence of a non-negligible number of charge carriers makes the plasma electrically conductive so that it responds strongly to electromagnetic fields. Plasma therefore has properties quite unlike those of solids, liquids or gases and is considered to be a distinct state of matter [6].

Jute is a golden fibre as well as a major cash crop of Bangladesh. A great advantage of jute fibre is that, it is environment friendly natural fibre. This natural fibre earns a lot of foreign currency by its export and its various products. Jute plays a very important role in the socio-economic activities of Bangladesh [7], [8]. Prospect for producing a wide variety of jute products and thus maximum utilisation of jute in the possible fields of textile sectors as well as thermal sectors are very encouraging. At present jute is facing tough competition from the convenient and competitive synthetics counter parts in the world market. The only way to save jute is through its uses in various diversified ways. Hence for better performability and to explore diverse use of jute, study of thermal properties of jute fibre is very important.

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II. MATERIALS AND METHODS

A. *Low Temperature Plasma Treatment*

Jute fibres (Corchorus Olitorius or Tossa jute) were collected from the local market in Bangladesh. The fibres were introduced into a bell jar type capacitively coupled glow discharge reactor as shown in figure 1

Fig. 1 Schematic diagram of jute fibre and position of it in the glow discharge reactor

To sustain a glow discharge i.e. for getting proper and uniform plasma, the conductive electrodes are separated 0.035 m apart from each other. In order to exposed all through uniform LTP treatment on the samples surface, the fibres (length of each fibre: 0.08 m) were inserted in between the two metallic electrodes by a carrier. After placing jute fibres between pair of electrodes, the glow discharge chamber was evacuated by a rotary pump at a pressure of 1.33 Pa. Ar was considered as plasma gas for treating the jute fibre. In all treatments, both process gases were introduced separately into the reaction chamber by a flowmeter at a flow rate of 0.2 L/min. which is maintained by a needle valve. The discharge powers were adjusted at 50, 75 and 100 W at a line frequency of 50 Hz with the duration of exposure times of LTP treatment of fibres were 5, 10, 15 and 20 min. Figure 2 shows a flow chart of a plasma treatment system which was used in this experiment.

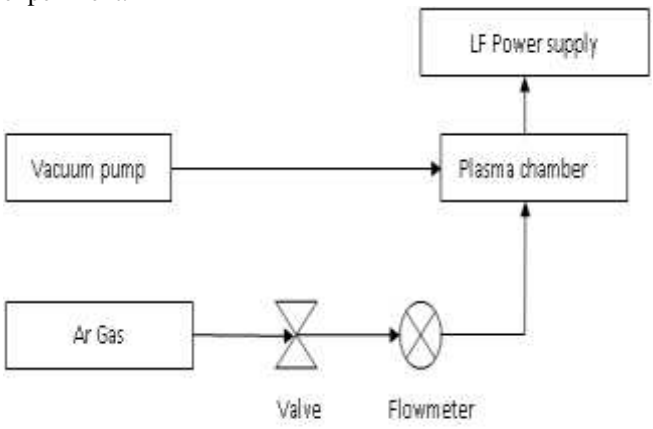

Fig. 2 Flow chart of the plasma treatment set-up

After plasma treatment has been finished, and the vacuum chamber was vented, jute samples were then removed and handled carefully in order to avoid possible surface contamination to the fibres. Later, the plasma treated fibres were immediately placed into a desiccator with the silica gel.

B. *Sample Preparation*

In preparing the samples, both raw and plasma treated jute fibres were cut into small pieces of sizes of about 1.0-2.0 mm. By mortar and pestle these small pieces of jute were ground, crushed and mixed in order to convert into powder form. Finally, the jute powders were seived by a very fine and thin net to make the powder finer. The powdered form jute of about 200 mg. was then put in a specially prepared high-pressure die. In order to make the tablets from jute powder, a high pressure (14000 psi) was applied by a hydraulic press (Model: X30659, 0-16000 psi, Mold Pressure, P.S.I: 1" and 5/4" Mold, Will Corporation, NY, USA). The diameter and the thickness of each equipped tablet was 13.5 and 1.5 mm respectively. In this way twenty five types tablets (one tablet was for raw jute and another twelve were for LTP treated jute) were prepared with treated jute samples of different discharge powers and exposure times. All the tablets were oven-dried at 100 °C for 20 minutes before characterization of the samples.

C. *Differential Thermal Analysis*

The DTA and TGA traces are taken in the temperature range of 25 to 500 °C at a scan rate of 10 °C/min. using model TG/DTA 6300, SII, SEIKO INSTRUMENTS Inc., JAPAN which is shown in figure 4.6 to investigate the thermal behavior of the raw jute and LTP treated jute.

Fig. 3 A photograph of a TG/DTA 6300 machine

III. RESULTS

The DTA were performed on the raw jute and LTP treated jute at the heat rate of 10°C/min. Figures 4.1 to 4.4 show the DTA traces of raw jute and LTP treated jute at 50 W, 15 min.; 75 W, 15 min.; 100 W, 10 min. and 100 W, 20 min. Table 1 shows the degradation temperatures of raw jute and LTP treated jute

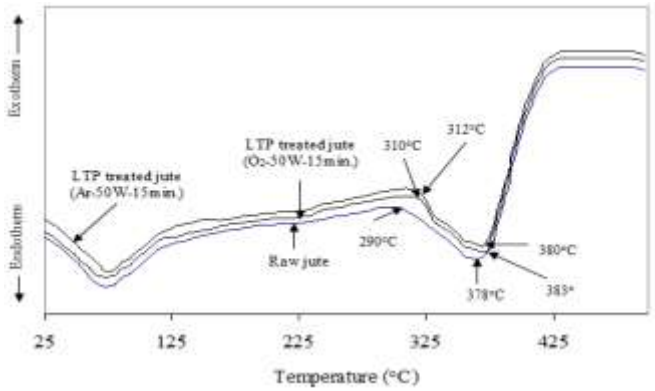

Fig. 4.1 DTA traces of raw jute and LTP treated jute at 50W discharge power and 15 min. treatment time of Ar and O_2 plasmas

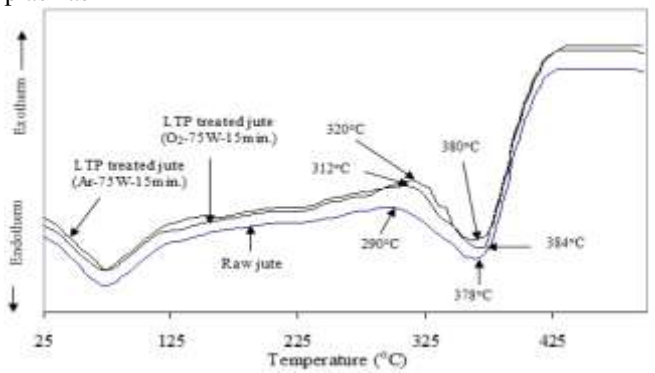

Fig. 4.2 DTA traces of raw jute and LTP treated jute at 75W discharge power and 15 min. treatment time of Ar and O_2 plasmas

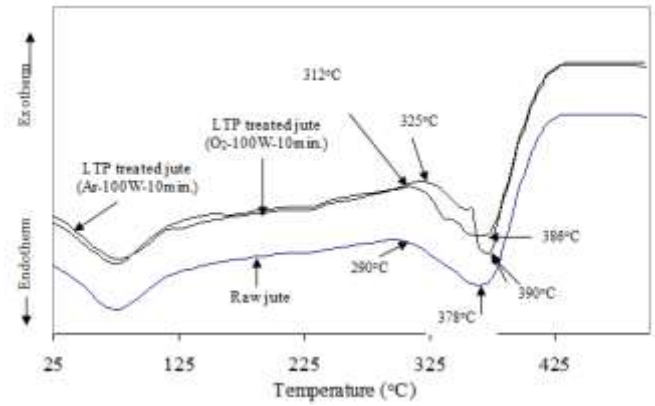

Fig. 4.3 DTA traces of raw jute and LTP treated jute at 100W discharge power and 10 min. treatment time of Ar and O_2 plasmas.

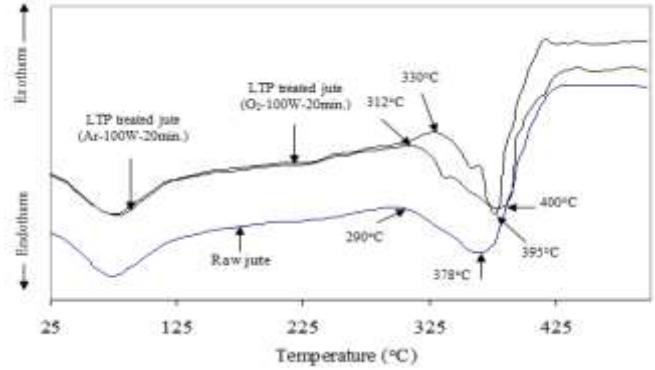

Fig. 4.4 DTA traces of raw jute and LTP treated jute at 100W discharge power and 20 min. treatment time of Ar and O_2 plasmas

Name of samples	Reported degradation temperature of hemicellulose (°C) [9, 10]	Degradation temperature of hemicellulose (°C)	Reported degradation temperature of cellulose (°C) [9, 10]	Degradation temperature of cellulose (°C)
Raw jute	290	290	386	378
Ar - 50 W - 15 min.	-	312	-	380
O_2 - 50 W - 15 min.	-	310	-	383
Ar - 75 W - 15 min.	-	312	-	380
O_2 - 75 W - 15 min.	-	320	-	384
Ar - 100 W - 10 min.	310	312	388	386
O_2 - 100 W - 10 min.	-	325	-	390
Ar - 100 W - 20 min.	318	312	398	400
O_2 - 100 W - 20 min.	-	330	-	395

IV. DISCUSSION

From the DTA traces, it is seen that these have a broad endothermic peak observed in the temperature range of 60–120 °C in both raw jute and LTP treated jute, corresponds to the heat of vaporization of water absorbed in the jute fibres. From the plot and table 1 it is seen that the degradation temperatures of hemicellulose lowers from to 312 and 310 °C for Ar and O_2 plasmas respectively when discharge power and exposure time were at 50 W, 15 min. Moreover, from the table 1 it is seen that the reported degradation temperature for raw jute also was 290 °C [9, 10]. Similarly, the values of the degradation temperature of hemicelluloses were near about at 312 and 320 °C when discharge power and exposure time were at 75 W, 15 min.; 312 (reported 310) [9, 10] and 325 °C when discharge power and exposure time were 100 W, 10 min. and 312 (reported 318) [9, 10], 330 °C when discharge power and exposure time were at 100 W, 20 min. treated jute by the Ar and O_2 plasmas respectively.

From the plot the cellulose degradation temperatures are found to increase from 378 (reported 386) [9, 10] for raw jute to 380 and 383 °C for Ar and O_2 plasmas respectively when discharge power and exposure time were 50 W, 15 min. Similarly, the values of the degradation temperature of celluloses were near about 380 and 384 °C when discharge power and exposure time were at 75 W, 15 min.; 386 (reported 388) [9, 10] and 390 °C when discharge power and exposure time were 100 W, 10 min.; 400 (reported 398) [9, 10] and 395 °C when discharge power and exposure time were at 100 W, 20 min. treated jute by Ar and O_2 plasmas respectively which are shown in table 1.

It is seen from the above analyses that the degradation temperature of hemicellulose treated by the Ar plasma remain constant but the degradation temperature increases with the increase of both discharge power and exposure time for the case of O_2 plasma. Moreover, it is seen from the above analyses that the degradation temperature of celluloses treated by the Ar and O_2 plasmas increases slightly with the increase of both discharge powers and exposure times.

The fibre degradation temperatures for both celluloses and hemicelluloses becoming unstable may be due to the endothermic and exothermic reactions by which the polymeric bonds of jute fibre break and undergo decomposition. This may also due to the formation of new intermonomeric bonds in them in the polymeric compounds of jute after the LTP treatment.

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