Thermal Analysis Studies Regarding the Eco-Composites Based on Jute by Applying Salinity Treatment

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Jute fibers as reinforced material in unsaturated polyester resin were obtained after immersing in a saline solution with a concentration of 5%NaCl at the room temperature for 3 months (2160 hours). The work presents some aspects concerning thermal properties of composite materials examined by the dilatometer analysis (DIL-75/1400) and by differential scanning calorimetry analysis (DSC 200 F3 Maia) under nitrogen atmosphere to reveal the variation of the physical dilatation coefficient versus temperature and the glass transition temperature. In other words, the paper aims are to present moisture absorption due time, coefficient thermal expansion, and glass transition temperature between treated and untreated samples. Thermogravimetric analysis (TGA-DTA) was used to study the thermal decomposition behavior of treated and untreated jute/polyester composites from room temperature up to 600°C.

Keywords: natural fiber, eco-composites, polymer matrices, thermal analysis

Jute is one of the most affordable natural fibers and is second only to cotton in amount produced and variety of uses of vegetable fibers. Jute fibers are composed primarily of the plant materials cellulose and lignin. It falls into the bast fiber category (fiber collected from bast, the phloem of the plant, sometimes called the *skin*) along with kenaf, industrial hemp, flax (linen), ramie, etc. The industrial term for jute fiber is raw jute. The fibers are off-white to brown, and 1-4 meters long. Jute is also called the golden fiber for its color and high cash value.

In the past few decades, environmental issues concerning global scale pollution and climate changes renewed the interest for natural ecological materials [1]. Environmental awareness, new rules and legislations are forcing industries to seek new materials which are more environmental friendly [2]. This fact has given rise to what is commonly called *Green Composites*, namely those obtained from lower environmental impact raw materials from biodegradable or renewable sources [3]. The use of natural fibers instead of traditional reinforcement materials such as glass fibers and carbon provides several advantages of bio based materials and cellulose is an abundant and naturally occurring polymer that can be obtained from numerous resources and cellulose micro-fibrils are the basic structural unit of all plants [4]. The development of environment-friendly green materials is because of natural fiber's biodegradability, light weight, low cost, emissions to the atmosphere, low density, high specific strength compared to glass and carbon, recycling and renewing natural sources [5, 6].

Jute as a natural fiber is eco-friendly, low cost, versatile in textile fields and has moderate mechanical properties, which replaced several synthetic fibers in development of many composite materials [7, 8].

Polymer matrices are most commonly used because of cost efficient, easy of fabricating complex parts with less tooling cost and also having excellent room temperature

properties when compared to other matrices. Thermoset matrices are formed due to an irreversible chemical transformation of resin into an amorphous cross-linked polymer matrix [9]. The latter is due to its hydrophilic nature that produces a harmful effect on almost all properties, including the dimensional stability. This problem can be imperative if the composite is used with high moisture environments. Moisture uptake in polymer matrix composites has a deleterious effect on their mechanical properties [10]. Therefore, it is important to study in detail the water absorption behavior in order to estimate not only the consequences that the water absorbed may have, but also the durability of natural fiber composites aged under water [11]. These new natural reinforcements must be investigated to determine their thermo-mechanical properties. In our study, saline treatments were performed on the jute material and NESTRAPOL 455-60 unsaturated polyester resin composite for 90 days, which increase or decrease the thermic properties.

Cellulosic fibers have low thermal stability that results in the exclusion of some manufacturing processes, and also limits the use of the composites at low temperature applications. The low thermal stability increases the possibility of cellulosic degradation and the possibility of emissions of volatile materials that could adversely affect the composite properties. Processing temperatures are thus limited to about 200°C, although it is possible to use higher temperature for short periods of time [12]. Dilatometry analysis was performed to analyses the thermal coefficient expansion with DIL – 75/1400 equipment

These analyses reveal the variation of the physical dilatation coefficient versus temperature. Thermal expansion of polymers is influenced by an isotropic building structure and by the large free volume between weakly bound molecular chains and primary and secondary

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phases of glass transition which also contribute to thermal

expansion [13].

The fundamental knowledge on the thermal coefficient expansion (TCE) material properties define and use 3 different types of thermal coefficient expansion; linear, superficial and volumetric. These TCE coefficients are constant only over specific temperature intervals and are defined function of this temperature [14].

The thermal coefficient expansion is useful for understanding dimensional changes as well as thermal stresses caused by thermal variation. In general, the TCE analysis of a fiber reinforced polymer composite may be lower than that of a pure polymer material, because polymer's thermally expansion with a greater extent than that of most reinforcing fibers likes glass and carbon. Lowering the TCE value is desirable in minimizing thermosdimensional changes in composite exposed to temperature changes during composite use [15].

Differential scanning calorimetry (DSC) was applied to determine the glass transition temperature. The glass transition temperature (T_a) is the temperature at which the material undergoes a structural transition from an amorphous solid state (glassy state) to a more viscous (rubbery) state [16]. Before DIL, TG/DTG, DSC thermal analysis the treated samples were kiln-dried at controlled temperature of 30°C for seven days.

Experimental part

Materials and methods

For this research, commercial jute as material and unsaturated polyester resin were purchased.

Ten specimens with dimensions: 80 mm x 15 mm were obtained. All were left for drying several days in order to obtain the optimum mechanical properties. The cured composite plates were cut using the electrical fretwork Proxxon.

Water uptake test of jute/resin composites were left to soak for 90 days in salt water with 5% NaCl at room temperature (22°C). In order to measure the water absorption, the specimens were took out and periodically weighed with a precise electronic balance, for 12 days every 24 h, for monitoring the variation of the sample mass during the ageing process. The absorption process was expressed with the relation (1):

Water uptake =
$$(\frac{P_{w}-P_{0}}{P_{0}}) * 100$$
 [%] (1)

where:

 $P_{_{o}}^{^{^{\prime}}}$ - is the wet weight, $P_{_{o}}^{^{w}}$ - is the dry weight of the specimen.

In figure 1 it's shown the weight gain due the moisture uptake of saline water absorption samples jute/resin composite for 90 days. It is observed that the samples have had significant linear sharp in the first 12 days, initially there it is a very rapid gain of solution and the absorption becomes slower and static in time and these samples reached their saturation state after 30 days.

The minimum percentage is 3.64% in the first 12 days and the maximum moisture content is 4.35 %. Jute is mainly built up of cellulose, which is a hydrophilic glycan polymer. The elementary unit of jute is anhydro-d-glucose,

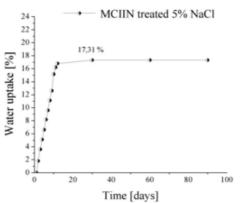


Fig. 1. Median water uptake (%) for jute/resin composite immersed in salt water

which contains three hydroxyl (-OH) groups. This hydroxyl groups in the cellulose structure account for the strong hydrophilic nature of jute as a result [19].

Dilatometry analysis

The thermal coefficient expansion (TCE) was performed using DIL-75/1400 equipment. The samples used for dilatometry were solid cylindrical specimens measuring 6 mm in diameter and length of 10 mm presented in figure 2. In the dilatometry experiments, the samples were heated (one thermal cycle) at a rate of 10⁰ C/min from room temperature up to 250° C. Before thermal analysis the treated samples were kiln-dried at controlled temperature of 30° C for seven days.

In figure 3 are presented the dilatation curves in function of the temperature for one heating cycle. The solid lines noted with 1 and 2 are the dilatation curves measured and the solid lines noted with 3 and 4 are the coefficients of thermal expansion between untreated and treated jute/ polyester composites.



Fig. 2. Dimensions of tested specimens

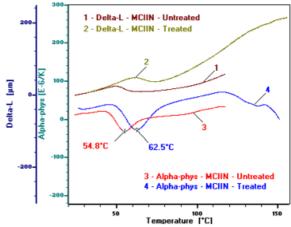


Fig. 3. The coefficients of thermal expansion analysis for untreated and treated composites

Table 1 CTE VALUES BETWEEN UNTREATED AND TREATED MCIIN COMPOSITES

Samples	T (°C)	CTE (E-6/K)			
MCIIN untreated	55.2	- 29.6			
MCIIN treated in salinity of	(5%NaCl) 64.25	-25.5			

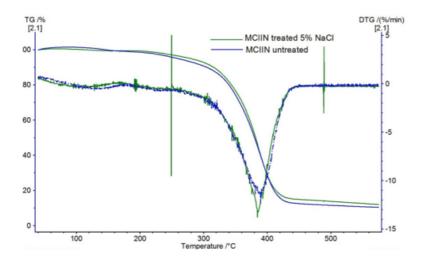


Fig. 4.TG/DTG curves of untreated and treated jute/ unsaturated polyester (MCIIN)

 Table 2

 MASS DEGRADATION AND TEMPERATURE OF TREATED AND UNTREATED MCIIN COMPOSITES

Sample	MCIIN											
	Treated with 5% NaCl Maximum					Untreated						
							Maximum					
Mass loss (%)	10	30	50	70	86,09		10	30	50	70	90,66	
Temperature (°C)	323,5	363	383	400	575		303	358	380	398	575	

As it can be seen the TCE, which is reflected by the dimensional changes for jute/polyester composite untreated and treated, exhibit an almost linear expansion over the temperature range examined. The differences between the curves are not higher and TCE values of untreated composite have a better stability compared with treated composite and are given in table 1.

Thermogravimetry analysis

A STA 449 F3 JUPITER thermo-gravimetric analyzer instrument was used to analyze the thermal decomposition of the untreated and treated 5% NaCl jute/unsaturated polyester composite. Samples with weight of around 15 mg were heated from room temperature to 600° C at heating rate of 10° C min⁻¹ under nitrogen atmosphere at a gas flow rate of 20 mL/min as purge gas to determine the residual limited to thermal stability results associated with mass loss with temperature.

The TG/DTG curves of jute fibers reinforced in polymer matrix are shown in figure 4 and the results of mass degradation versus temperature are presented in table 2.

Conclusions

The TG/DTG curves for untreated samples have three steps for mass degradation. The initial weight loss is observed in a range of 50 – 150 °C that belongs to water evaporation below to 100°C. Total evaporation of water in a natural fiber is impossible, even with oven – drying prior to fabrication.

The second stage of mass degradation is between 170 - 300°C, with a decomposition of constituent of natural fiber and volatiles with 9.35 % mass loss. The third stage of degradation is for unsaturated polymer between 300 - 500°C with 88.48 % mass loss. The TG/DTG curves for treated samples are presented in figure 2 were are shown the mass degradation in range of 50 -200°C by water evaporation and volatiles and begin with a larger peak of DTG from 250 to 400°C.

The natural constituent's degradation processes is small at 300°C with 6.85 % mass loss and big at 500°C with a percentage of loss mass of 74.32 %.

The thermal stability on composites was found to be different. By comparing the temperature and mass loss between treated and untreated composites, Table 2 shows that the mass degradation for untreated is faster than that of treated composites. The treated jute/unsaturated polyester composite present a thermal stability as that of against untreated. M. R. Ishak [23] affirms that the salt water treatment improves the characteristics of a natural fiber removal of outer layer of hemicellulose and pectin. So, it is possible that the treatment with salt water to decompose the constituents of jute fibers and delay the degradation.

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