

# Accelerated hydrothermal aging of biocarbon reinforced nylon biocomposites



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## ABSTRACT

Nylon/biocarbon biocomposites show great potential for semi-structural auto-part applications due to their enhanced mechanical and physical properties. However, the long term durability of these biocomposites has never been studied before. The durability of the biocomposite was compared to that of talc reinforced nylon composite at 20 wt. % for both composites. They were subjected to accelerated and aggressive conditioning by complete immersion in water at 85 °C for prolonged periods of time up to 28 days. The water uptake of the nylon/biocarbon biocomposite was reduced in comparison to that of the nylon suggesting that biocarbon particles act as a barrier while also concentrating the absorbed moisture within its pores and around the interface with nylon. Thermal properties revealed little changes in the crystallinity of the nylon/biocarbon composites. However, the glass transition temperature was observed to decrease significantly after conditioning due to the presence of bound water molecules within the amorphous phase of nylon. The impact strength remained mostly unchanged even after conditioning, suggesting interaction between biocarbon and nylon to restrict the chain mobility and thereby annulling the effect of moisture on the nylon. The morphology of the impact fractured surface of the conditioned sample revealed two distinct phases. One was significantly affected by moisture, revealed swelling and debonding of biocarbon particles from the nylon matrix while the other remained unchanged when compared to that of the unconditioned sample.

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## 1. Introduction

High performance parts, whether for machinery, construction or electronics, require materials with enhanced properties. In the automotive industry especially, where light weighting and mechanical performance go hand in hand, the use of materials such as engineering polymer blends and composites have become increasingly sort after. Engineering polymer composites especially, has seen increased use as replacements for heavier and more expensive metallic parts such as fuel caps and lines, brake lines, air intake manifolds and fan blades. These composites are typically

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made from nylon 6 and nylon 6,6 because of their high strength and stiffness as well as their resistance to corrosion and high temperatures. Therefore, nylons have become the dominant engineering polymer used in the automotive industry today. However, these polymers come with drawbacks; one of which is the inferior durability compared to those of metals in aggressive environments. Consequently, despite the outstanding and improved properties that these composites might possess, the long term effect of the surroundings is crucial in determining their application scope. Examples of nylon composites designed for specific applications are nylon/talc composites with improved moisture barrier and stiffness [1], nylon/glass fiber composites with improved strength, stiffness and heat deflection temperature [2] and hybrid composites consisting of nylon/secondary matrix/rubber for balanced strength, stiffness and impact properties while providing reduced moisture absorption as well [3]. These composites may have excellent tailored properties but may also have poor long term durability.

Nylons are well known for their hygroscopicity; they absorb moisture from the surrounding environment [4]. They are also

vulnerable to degradation in the presence of moisture at elevated temperatures through the hydrolysis of the amide linkages in its backbone. The dissociation of the amide bonds lead to shorter chains and subsequently to a lower molecular weight. This results to a reduction in both mechanical and thermal properties. Fig. 1 illustrates the hydrolysis of nylon in the presence of moisture and heat. The water molecules break up into hydrogen ( $H^+$ ) and hydroxide ( $OH^-$ ) ions, which then react with and break the amide bond in the presence of heat [5]. For this reason, it is of utmost importance that nylon be dried prior to processing to prevent the hydrolysis of its chains. As a result, nylon and its composites are mostly tailored for dry under hood parts where their high resistance to distortion is required. However, they still get exposed to humid conditions and undergo long term hydrolysis as a result. It has been established that the durability of nylon composites in presence of moisture are diminished resulting from the plasticization of the chains and through debonding of the filler-matrix interface by swelling and weakening of the phase boundary [6,7].

In recent years, the goal to reduce the use of petroleum based nylons and high density fillers in nylon composites has resulted to the use of lower density and more thermally stable fillers. Thermally pretreated biomass is one example. These fillers, known as biocarbons, show potential to be utilized as reinforcement in nylon and yet stable enough to withstand the high processing temperatures of nylons up to 290 °C.

Several investigations into the use of biocarbon as reinforcement have been done [8–18]. However, only a few have dealt with using nylon as the matrix. In our recent publication, we studied the effect of biocarbon particle size ranges on the mechanical and thermal properties of polyamide 6, 10 [19]. It was found that smaller biocarbon particles improved the impact strength in comparison to biocarbon particles with a wider size range. It was also observed that the biocarbon particles were well embedded in the nylon matrix, indicating a good bonding between them. The interaction between biocarbon and nylon was suggested as to be due to polar-polar interactions and possibly hydrogen bonding. Recently, a study on the interactions of biocarbon and nylon showed that the presence of functional groups on the biocarbon surface is necessary for bonding to occur with the nylon chains [20]. Biocarbons with little to no functional groups exhibited voids at the interface with nylon when used. Similarly, a hybrid composite system involving biocarbon, epoxidized natural rubber (ENR) and nylon 6 was studied [21]. It was shown that the functional groups on the biocarbon surface interacted with the epoxide groups on the ENR through to form ester bonds. It was also shown that the biocarbon particles were well bonded to the nylon.

So far to date, no study has been made to determine and/or investigate the durability of biocarbon reinforced nylons biocomposites. In this study, we investigate for the first time, the influence of biocarbon on the durability of nylon under the complete immersion of water and at an elevated temperature. Consequently,

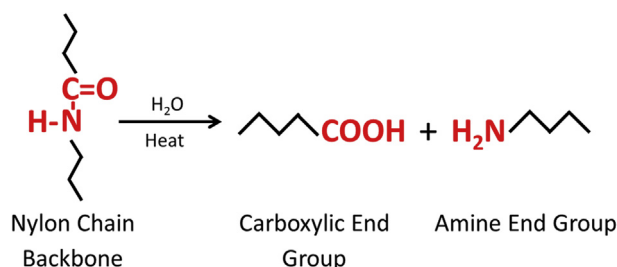


Fig. 1. Hydrolysis mechanism of nylon in the presence of moisture and heat.

the goal was to investigate and compare the mechanical, thermal and morphological property changes of biocarbon reinforced nylon to that of nylon and talc reinforced nylon by subjecting them to accelerated simulated environments similar to those found in the under hood of automobiles. Talc reinforced nylon was chosen for comparison and possible replacement due to their wide spread use in the automotive industry in applications where enhanced stiffness, impact strength and moisture barrier properties are required. Evaluation of the aforementioned properties were done by Fourier transform infrared spectroscopy, differential scanning calorimetry, dynamic mechanical analysis, tensile and flexural tests and scanning electron microscopy.

## 2. Materials and methods

### 2.1. Materials

In this study, heat stabilized nylon 6 (Ulramid B3K) was purchased from Entec Polymers, FL, USA. Chopped miscanthus plant fibers were obtained from New Energy Farms (Leamington, Ontario, Canada), then pyrolyzed at 650 °C and hammer milled to  $\leq 397 \mu\text{m}$  thereafter by Competitive Green Technologies (Leamington, Ontario, Canada). Talcum powder was purchased from Imerys Talc, USA under the trade name of Artic Mist with a platelet median diameter of 2.2  $\mu\text{m}$ .

### 2.2. Sample preparation and hydrothermal conditioning

Prior to processing, nylon and talc were dried in a conventional oven over night at 80 °C while the biocarbon was dried until constant weight at 105 °C. Three sets of samples were fabricated; neat nylon, nylon/biocarbon and nylon/talc composites. Both composites were fabricated at 20 wt. % filler loading. The talc was used as received while the biocarbon was milled again by placing 25 g of biocarbon in a 500 mL stainless steel ball mill container and milled for 1 h at 300 rpm using a milling machine (Retsch PM100, Germany). A combination of 65 zirconium oxide balls and 2 stainless steel balls were used as milling media. The zirconium oxide balls were 10 mm in diameter and weighed 3.34 g each, while the stainless steel balls were 40 mm in diameter and weighed 256 g. The milled biocarbon was collected and dried at 105 °C for 24 h prior to further use. After milling and drying, the biocarbon was placed in a stack of series of sieves ranging from 216 to 20  $\mu\text{m}$ . The same sieve shaker and procedure was used according to Nagarajan et al. [22]. The biocarbon particles conveniently passed through the 20  $\mu\text{m}$  sieve, therefore indicating that the particle size was  $\leq 20 \mu\text{m}$ . Composites were extruded in a twin-screw extruder (Labtech Engineering Company LTD, Thailand) with L/D ratio of 48, processing temperature of 250 °C and at a screw speed of 250 rpm. The extrudates were cooled through a water bath, pelletized and dried at 80 °C in a conventional oven over night. For comparison purposes and elimination of the effects of processing conditions, neat nylon was also processed in a similar manner. The dried pellets were made into test samples using an injection molding machine (ARBURG allrounder 370C with model number 370 S 700-290/70, Germany) with a 35 mm screw diameter. Injection molding was carried out at 250 °C and with a mold temperature of 70 °C.

Conditioning of the samples was performed by complete immersion in water at 85 °C for durations of up to 28 days. A hot water dispenser manufactured by SUNPENTOWN INT'L INC with model number SP-5020 was used to condition the samples. At different time intervals of 1, 3, 7, 14, 21 and 28 days, the samples were removed, wiped dry and placed in a conventional oven at 80 °C for 12 h to dry. After drying, they were placed in a desiccator under vacuum to cool. All tests were conducted thereafter except for those

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