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Original Article

Thermogravimetric characterization of polyester matrix composites reinforced with eucalyptus fibers☆

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ABSTRACT

The substitution of natural fibers for synthetic ones as reinforcement of polymer matrix composites is today not only the subject of investigation but also engineering applications. Natural fibers display environmental advantages in association with economic benefits related to comparatively lower cost as well as less energy consumption. Several natural lignocellulosic fibers (LCF's) extracted from worldwide cultivated plants, such as sisal, coir, cotton, flax, among others, are successfully being used in composites. A great number of other LCF's, especially from wood species, has a reinforcement potential waiting to be explored. Thus, the objective of this short communication is to evaluate the thermogravimetric (TG/DTG) behavior of polyester matrix composites reinforced with relatively higher volume fractions, 30, 40 and 50 vol%, of eucalyptus fibers. The incorporation of eucalyptus fibers slightly reduces the thermal stability of the polyester matrix by a small decrease in the onset of thermal degradation and the DTG peak temperature as compared to neat polyester. The limit for practical application of these composites could be set as 300 °C, before the onset of major weight loss.

1. Introduction

Nowadays there is a growing concern in our modern society regarding environmental issues such as climate changes and worldwide pollution caused by industrial activities [1]. In parallel, sustainable actions emphasizes the search for effective solutions based on energy saving and global warming control. One of these actions is the substitution of natural materials for synthetic ones. In this respect, natural lignocellulosic

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fibers (LCFs), obtained from plants, become a promising alternative as reinforcement of polymer matrix composites. Indeed common synthetic fiber composites like fiberglass, which is still extensively used in several engineering sectors [2,3], are steadily being replaced by environmentally friendly, cheaper, lighter and tougher LCF composites [4,5]. Since past decades, an escalating number of research works [6–12] has been dedicated to LCFs and related composites. These review articles demonstrated that the relative success of LCFs over synthetic fibers is the fact that natural fibers are also biodegradable, renewable and less abrasive to processing equipment.

Among the many investigated LCFs, those extracted from wood only recently are attracting attention for their potential as polymer composite reinforcement due to the high cellulose (~60%) content [13]. In particular, trees that are extensively cultivated such as pine and eucalyptus, for both building construction and paper production, always have left over (bark and small branches) pieces from which fibers could be extracted to be used in composites. This is the case of eucalyptus, a tree native from Australia and cultivated in many tropical and tempered countries.

Recent works on eucalyptus fibers (EUF) incorporated into different polymer matrices revealed an effective reinforcement behavior [14–22]. However, for uses in engineering systems, not only mechanical properties but also thermal stability are required. This latter is important in order to define the limit temperature for practical application.

A basic evaluation of the thermal stability of a material is usually performed by thermogravimetric (TG) and its derivative (DTG) analysis. A first review mentioning the thermal stability of polymer composites reinforced with common LCFs [23] indicated that degradation of the fiber inside the matrix generates volatiles at temperatures above 200 °C, resulting in porosity and impairing the mechanical properties. Another review [24] concluded that processes and applications of natural fiber composites should be restricted to 250 °C. In spite of references related to several common as well as less common LCFs composites, no wood fiber composite was mentioned in these review articles [23,24]. Therefore, the objective of this short work is to present TG/DTG analyses of polyester matrix composites incorporated with relatively high amounts, 30–50 vol% of eucalyptus fibers.

2. Experimental procedure

Continuous eucalyptus fibers (EUFs) were extracted from bark pieces, Fig. 1(a), of a tree trunk. Fibers longitudinally cut with a sharp knife, Fig. 1(b), present equivalent diameter around 1 mm and 9 mm in length.

After cleaning in running water and drying at 60 °C the EUFs were placed, separately, in amounts of zero (neat polyester for control), 30, 40 and 50 vol% inside a cylindrical steel mold with 5.5 mm in diameter and 10 mm in length. Still fluid unsaturated orthophtalic polyester resin mixed with 0.5 wt% of methyl-ethyl-ketone catalyst/hardener was poured into the mold. A pressure of 3 Mpa was applied onto the mold lid to improve the fiber resin contact and the composite was cured at room temperature (RT) for 24 h. After removing the composite rod from its cylindrical mold, 1 mm thick discs were cut with approximately 2 mg in weight for thermal analysis. This procedure ensures that a uniform composition is obtained in every small sample. TG/DTG analyses were performed in three samples for each EUF composition using a model TGA Q 500 V 2010 Build 36 TA Instrument System. The mass variation as function of temperature was carried out in air at a heating rate of 10 °C/min from RT to 800 °C.

3. Results and discussion

Fig. 2 shows typical TG curves for samples of neat polyester, 30, 40 and 50 vol% eucalyptus fiber (EUF) composites. In these curves tangent lines as well as numerical values, temperature and weight loss, are indicated for the interpretation of thermal stability. As seen in Fig. 2(a) the onset of weight loss occurred at 99 °C for the neat polyester while below 50 °C for the composite, Fig. 2(b)–(d). This is probably due to release of surface moisture that exists in hydrophilic natural fibers [6–12]. Above 100 °C constitution water is released and 2% weight loss occurs at a slightly higher temperature (175 °C) for the neat polyester. The onset of major weight loss, defined by the intersection of two tangent lines, also occurs at slightly higher temperature (330 °C) for the neat polyester. The end of this major weight loss (93–94%) occurs at about 10 °C lower temperature for the neat polyester. The remaining ashes (6–7%), corresponding to inert material after complete thermal degradation, is stable up to the investigated finishing temperature of 800 °C.

The results if Fig. 2 revealed that the thermal degradation of EUFs reinforced polyester composites, Fig. 2, despite the relatively high amount of fibers, is only slightly different than that of neat polyester. This suggested that fiber has limited effect on both the onset and the major stages (315–395 °C) of thermal degradation. Moreover, since the amount of remaining ashes is the same, one might infer that EUFs do not practically contribute to the final inert residues.

Fig. 3 shows DTG curves for samples of neat polyester as well as 30, 40 and 50 vol% EUF composites. As in the case of TG curves in Fig. 2, only minor differences are noted between the DTG curve of neat polyester, Fig. 3(a), and those of the composites, Fig. 3(b)–(d). Indeed, the main DTG peak, corresponding to the maximum thermal degradation rate, occurs around 365 and 369 °C in all case in Fig. 3. This clearly indicates that there is little effect caused by the EUFs to the stability of polyester composites. Worth mentioning are the small shoulders peaks, before (~200 °C) and above (~500 °C) the main peak, existing only in the composites, Fig. 3(b)–(d). These shoulders peaks are commonly found in natural fiber composites with higher amplitude (peak height) than those in the present work [24]. The shoulders around 200 °C are attributed to hemicellulose while those around 500 °C could be related to lignin decomposition [25,26]. Since most cellulose decomposition of natural fibers coincides in temperature (~370 °C) with the breaking and depolymerization of polyester molecular chains, one might expect coincident DTG main peaks for both fiber and matrix. This is apparently the case in Fig. 3.

As a final remark, the reader should notice the similarity between the neat polyester and EUF composites for both TG (Fig. 2) and DTG (Fig. 3) curves. Consequently, the polyester matrix and not the EUF is what characterizes the stability of

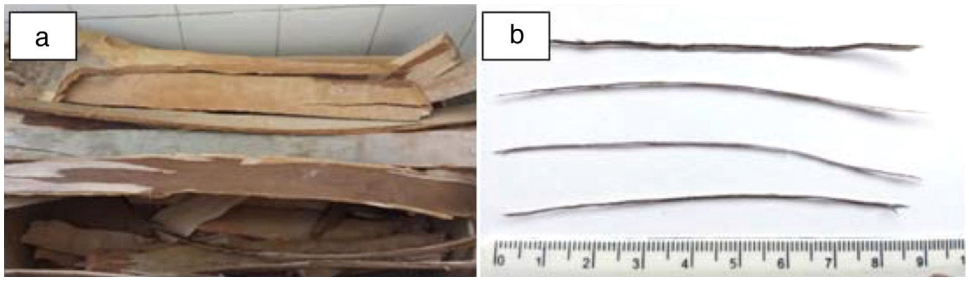

Fig. 1 – Eucalyptus: (a) bark pieces extracted from tree trunk; (b) longitudinally cut fibers.

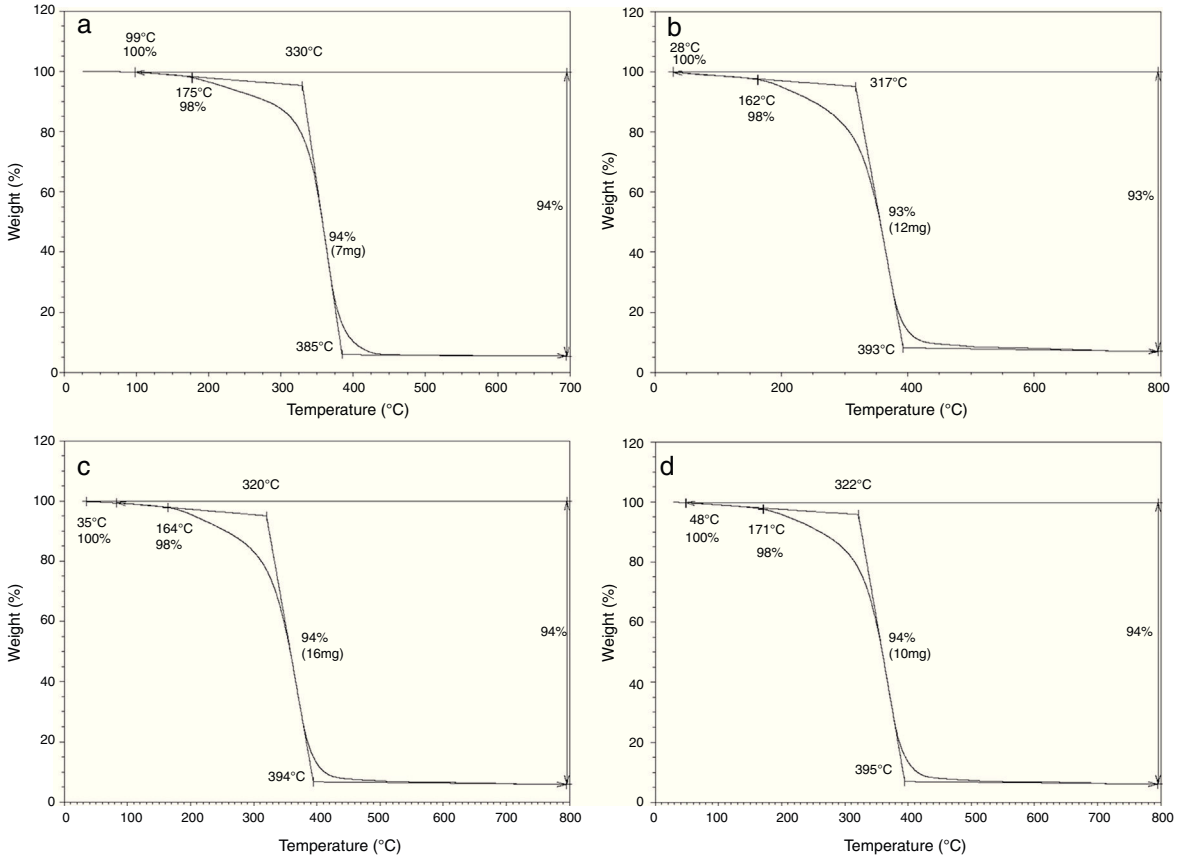

Fig. 2 – TG curves for: (a) neat polyester; (b) 30; (c) 40; and (d) 50 vol% eucalyptus fiber polyester matrix composites.

the composite. In other words, the thermal insulation behavior of EUF composites up to 50 vol% is mainly due to the insulating capacity of the polyester matrix. Slightly decrease in the onset of water release temperature as well as the initial thermal degradation (hemicellulose in EUF) associated with shoulder peak around 200 °C are the only noticeable EUF effects on the composites thermal stability.

4. Conclusions

- Thermogravimetric analysis based on TG/DTG curves of both neat polyester and polyester matrix composites incorporated with 30, 40 and 50 vol% of eucalyptus fibers revealed only minor effects caused by the fibers.
- About 50 °C decrease in the onset of water release in the composites as compared to the neat polyester is assigned to surface adsorbed moisture in hydrophilic eucalyptus fiber.
- The onset of TG major weight loss stage is less than 15 °C lower in the composites as compared to the neat polyester.
- The final amount of inert ashes, around 6–7 wt%, has no apparent contribution from the eucalyptus fibers and might be residues from polyester degradation.
- Small shoulder peaks below (~200 °C) and above (~500 °C) the major DTG peak, around 365 °C, are attributed to decomposition of hemicellulose and lignin, respectively, in the eucalyptus fiber.
- The thermal stability and insulation capacity of eucalyptus fiber composites can be practically considered similar to that of neat polyester with only slight reduction in

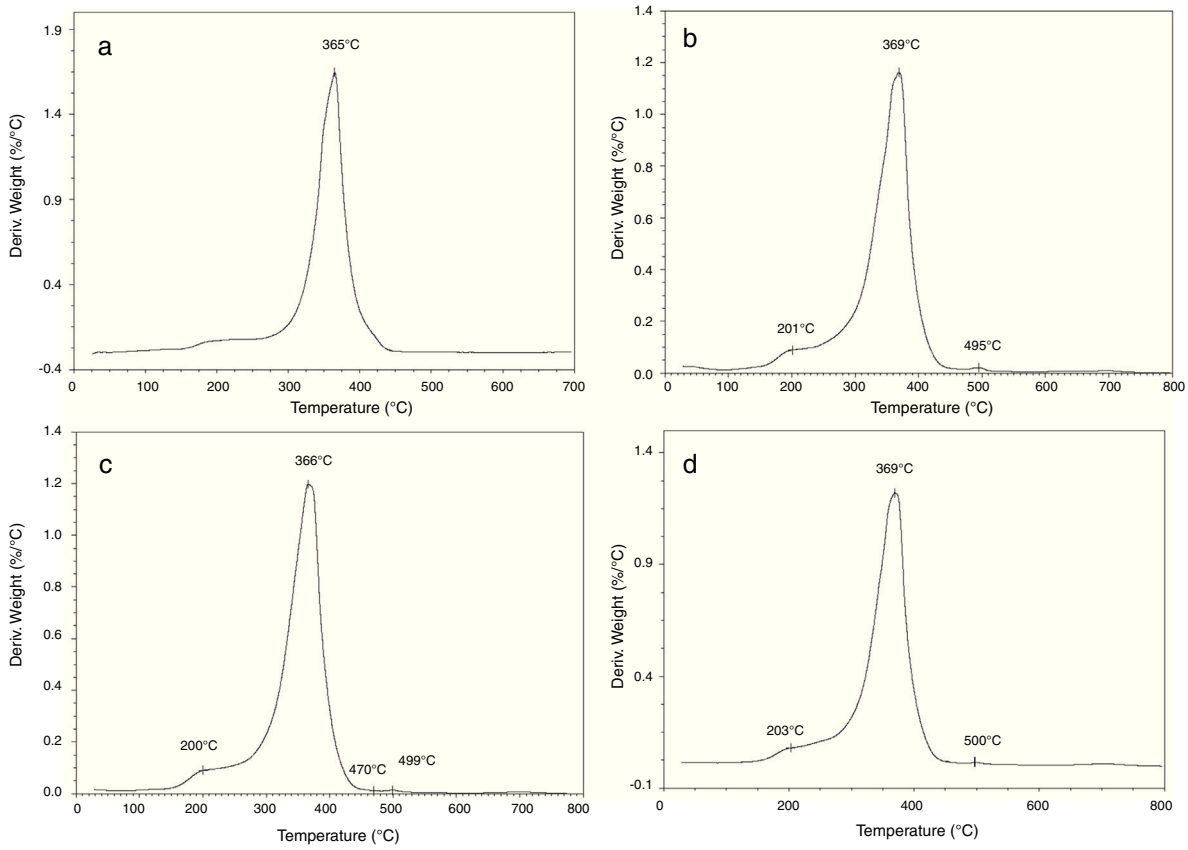

Fig. 3 – DTG curves for: (a) neat polyester; (b) 30; (c) 40; and (d) 50 vol% eucalyptus fiber polyester matrix composites.

degradation temperature. A maximum temperature of 300 °C, before the onset of major weight loss, could be considered the limit for practical use of eucalyptus fiber polyester matrix composites.

Conflicts of interest

The authors declare no conflicts of interest.

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