Materials and Design 54 (2014) 425-429

Contents lists available at ScienceDirect

Materials and Design

journal homepage: www.elsevier.com/locate/matdes

# Effect of ammonium polyphosphate on flame retardancy, thermal stability and mechanical properties of alkali treated kenaf fiber filled PLA biocomposites

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#### ARTICLE INFO

Article history: Received 11 April 2013 Accepted 29 July 2013 Available online 6 August 2013

Keywords: Ammonium polyphosphate Alkali treatment Kenaf fiber Polylactic acid Biocomposite

#### ABSTRACT

In present research polylactic acid (PLA) biocomposites were prepared from PLA and kenaf fiber using dry blending, twin screw extrusion and compression molding techniques. PLA was blended with kenaf core fiber, polyethylene glycol (PEG) and ammonium polyphosphate (APP). Kenaf fiber was treated with 3%, 6% and 9% NaOH solution separately. Both raw and treated kenaf along with 10, 15 and 20 phr APP was utilized during composite preparation. The effects of APP content and alkali treatment on flammability, thermal and mechanical properties of kenaf fiber filled PLA biocomposites were investigated. APP is shown to be very effective in improving flame retardancy properties according to limiting oxygen index measurement due to increased char residue at high temperatures. However addition of APP decreased the compatibility between PLA and kenaf fiber, resulting in significant reduction of the mechanical properties of PLA biocomposites. Thermogravimetric analysis (TGA) showed that NaOH treatment improved the thermal stability of PLA biocomposites and decreased carbonaceous char formation.

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#### 1. Introduction

Natural fiber reinforced composite is an emerging area in polymer science. At present, natural fibers are used in combination with plastics. The purpose of reinforcement is to provide strength and rigidity, helping to sustain structural load. Position and orientation of the reinforcement is maintained by the matrix or binder (organic or inorganic). Using natural fibers with polymer based on renewable resources will allow many environmental issues to be solved. These natural fibers are of low cost with low density and high specific properties. These are biodegradable and non-abrasive. Using biodegradable polymers as matrices, natural fiber-reinforced plastics become the most environmental friendly materials; because they can be composed at the end of their life cycle [1].

The main target of natural fiber reinforced green biocomposites is to achieve a good combination of properties and processability at a moderate cost as natural fibers has increasingly gained importance in the thermoplastic industry [2]. Biofiber/natural composites (bio-composites) are rising as a viable option to glass fiber reinforced composites especially in building product and automotive applications. By embedding biofibers with renewable resource-based biopolymers; the so-called green biocomposites are constantly being developed [3]. Among all biopolymers, polylactid acid (PLA) has the greatest potential to replace the petroleum based synthetic polymers. Some of the environmental benefits of PLA are its biodegradability, low production energy and reduction of greenhouse gas production. Because of its ability to be thermally crystallized, impact modified, stress crystallized, filled, copolymerized and processed in most polymer processing equipment, it may be the best polymer with the broadest range of applications including plastic bags and planting cups [4]. However, lower ability in resisting thermal deformation, lower water vapor and gas barrier properties as well as brittleness are the deficiencies of PLA that limit its extensive application [5]. Consequently there is great interest to incorporate natural filler into the PLA matrix, which will improve the overall properties of PLA matrix.

Polyethylene glycol (PEG) is a polyether compound with many applications from industrial manufacturing to medicine [6]. In this study, PEG was used as a plasticizer to improve the flexibility and ductility of glassy polymers. Improvement of PLA in terms of flexibility may widen its application as a film material and biodegradable packaging. Addition of plasticizer is also to improve the fragility and increase the elongation of break or elasticity of PLA.

Flammability is one of the very important parameters, limiting often the application of a composite to a given area. APP 760 was used in this study to preserve the environmentally friendly character of the biocomposites. It is an intumescent halogen-free flame retardant, which does not generate additional quantities of smoke.





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<sup>0261-3069/\$ -</sup> see front matter  $\odot$  2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.matdes.2013.07.095

APP was used in previous research to increase thermal stability and flame resistance properties of composites [7,8]. In present research, a systematic study was carried out to determine the effects of APP content on alkali treated PLA/kenaf/PEG/APP biocomposites and compare it with untreated ones in terms of mechanical, thermal and flammability properties.

#### 2. Experimental details

#### 2.1. Materials

Polylactide (NatureWork<sup>TM</sup> PLA 300ID) in pellet form, used as the matrix, was obtained from NatureWork<sup>®</sup> LLC, Minnetonka, MN USA. It has a specific gravity 1.24 g/cm<sup>3</sup> and melt flow index (MFI) around 15 g/10 min (190 °C/2.16 kg). The kenaf core fiber was approximately 2 mm in size. It was obtained from Innovative Pultrusion Sdn Bhd at untreated condition. It was then sieved to a size range of 150–300 µm. Polyethylene glycol (PEG) was added to the composite formulations in present research to plasticize PLA. PEG with a weight-average of 600 g/mol and density of 1.126 g/cm<sup>3</sup> was obtained from Acros Organics, Belgium. Flame retardant used in this study was ammonium polyphosphate (APP 760) obtained from Carliant. Kenaf was treated with sodium hydroxide (NaOH) with a molecular weight of 40.0 g/ mol and density of 2.13 g/cm<sup>3</sup> obtained from Merck Chemicals, Germany.

#### 2.2. Preparation of PLA biocomposites

Samples of different PLA/kenef/APP/PEG ratios were prepared using a co-rotating twin screw extruder (Brabender Plasticoder PL 2000, Germany) according to Table 1. Kenaf fiber was initially dried at 50 °C for 24 h before treatment with 3%, 6% and 9% NaOH separately. Then the fiber was washed with water and dried at 60 °C for 24 h. Before melt compounding of composite, different components were mixed in a high speed rotor mixer for 15 min to achieve homogeneous mixing. The extrusion was conducted at a speed of 50 rpm. The temperature profile adopted during compounding were 180 °C at the feed section and increased to 200 °C at the die head. The extruded strands were then air-dried and pelletized. After that the samples were molded at 190 °C using compression molding machine.

#### 2.3. Flammability testing

The limiting oxygen index (LOI) test was done as per ASTM: D 2863. In the test, the sample was held vertically in the transparent chimney, where the flow of oxygen and nitrogen were controlled. The test was repeated under various concentrations of oxygen and nitrogen to determine the minimum concentration of oxygen needed for burning the sample in 3 min.

Table 1
Designation and formulations of PLA/kenaf/PEG/APP biocomposites.

Designation	PLA (wt%)	Kenaf (wt%)	PEG (wt%)	APP (phr)	Concentration of NaOH (%)
A0	60	25	15	-	-
A10	60	25	15	10	-
A15	60	25	15	15	-
A20	60	25	15	20	-
A10-N3	60	25	15	10	3
A10-N6	60	25	15	10	6
A10-N9	60	25	15	10	9

#### 2.4. Thermogravimetric analysis

PLA/kenaf biocomposite samples were characterized using a thermogravimetric analyzer (TGA) model 2050 (TA Instruments, New Castle, DE) in order to determine their thermal stability. The specimens were scanned from 30 °C to 800 °C at the rate of 10 °C/min and measurements were performed under a nitrogen gas flow.

#### 2.5. Mechanical testing

Flexural test of kenaf/PLA biocomposites was carried out by using Lloyd Universal Tester according to ASTM: D 790 with a cross head speed of 3 mm/min at room temperature. In order to determine impact strength, notched Izod impact testing was done using an Izod Impact Tester LS-22005 according to ASTM: D 256. Five samples were tested for each batch and the average results was taken and reported.

#### 3. Results and discussion

#### 3.1. Effect of APP content

#### 3.1.1. Flame retardancy properties

LOI measurement test is widely used to evaluate flammability of materials. Effect of APP content on flame retardancy properties of PLA/kenaf/PEG/APP biocomposites is presented in Table 2. It can be seen that LOI values obviously increased from 27.6 to 31.6 with the incorporation of 20 phr APP into PLA/kenaf/PEG biocomposites. These results confirmed the fact that increasing amounts of APP in the mixtures had improved the fire retardant properties. The concept of APP 760 as an intumescent flame retardant relies upon the formation of an expanded carbonized layer on the surface of the polymer during thermal degradation. This layer acted as an insulating barrier, reducing heat transfer from the polymer towards the flame as well as the diffusion of oxygen into the material.

 Table 2

 Effect of APP content on flame retardancy properties of PLA/kenaf/PEG/APP biocomposites.

Designation	APP (phr)	LOI (%)
A0	0	27.6
A10	10	29.4
A15	15	30.3
A20	20	31.6

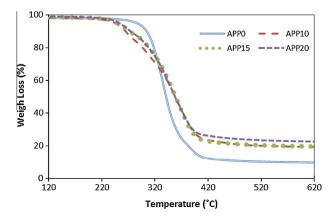


Fig. 1. TGA curves of A0, A10, A15 and to A20 biocomposites.

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