



## Development of sustainable biodegradable lignocellulosic hemp fiber/polycaprolactone biocomposites for light weight applications

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### ABSTRACT

Biocomposites with poly( $\epsilon$ -caprolactone) (PCL) as matrix and lignocellulosic hemp fiber with varying average aspect ratios (19, 26, 30 and 38) as reinforcement were prepared using twin extrusion process. The influence of fiber aspect ratio on the water absorption behavior and mechanical properties are investigated. The percentage of moisture uptake increased with the aspect ratio, following Fickian behavior. The hemp fiber/PCL biocomposites showed enhanced properties (tensile, flexural and low-velocity impact). The biocomposite with 26 aspect ratio showed the optimal properties, with flexural strength and modulus of 169% and 285% respectively, higher than those of neat PCL. However, a clear reduction on the mechanical properties was observed for water-immersed samples, with reduction in tensile and flexural moduli for the aspect ratio of 26 by 90% and 62%, respectively than those of dry samples. Summarily, the optimal sample provides an eco-friendly alternative to conventional, petroleum-based and non-renewable composites for various applications.

### 1. Introduction

The growing interest in the use of natural plant fiber as reinforcements in composites is due to their high strength-to-weight ratio, abundant, recoverable and biodegradable nature after the end of their service life. Lignocellulosic fiber comes from renewable resources and they require less energy to produce compared to their conventional counterparts. Cellulose characterised many organic materials, as it supports the advent of green products in materials manufacturing. Its wide applications can be traced to attributes such as versatility, abundance and compatibility [1]. These properties make natural lignocellulosic fiber attractive reinforcements for polymer matrix composites [2–9]. Comparatively, among some commonly used bast or natural plant fibers such as flax, jute and date palm, hemp fiber has an outstanding physical and mechanical properties, especially in terms of specific tensile strength at break and tensile modulus [4,10–13].

Lignocellulosic fiber (such as hemp, flax and jute) reinforced composites have been successfully used for light-weight applications in recent years, especially in the automotive and construction industries. This is required in order to reduce an estimated 75% of energy consumption by automobiles, mainly caused by the weight of vehicles [14].

However, significant barriers for structural applications of these composites still exist [15–19]. The full acceptance of natural fiber composites for structural components has been further limited by their inherent complex morphological structure and variation with regard to chemical composition, crystallinity, thermo-mechanical properties, surface roughness and profile.

Conventional polymer matrices such as epoxy, vinyl ester, polyester are attractive for several structural applications as these polymers provide good mechanical properties compared to biodegradable polymers. However, these polymers are non-recyclable and non-biodegradable and as a result they cause serious environmental problems [20,21]. In this context, the development and use of biodegradable polymers for composites are considered as one of the important strategies for reducing the environmental challenges from the use of petroleum-based and non-biodegradable polymers.

Among the biodegradable matrices such as poly (lactic acid) (PLA), poly (hydroxyalkanoates) (PHA), poly ( $\epsilon$ -caprolactone) (PCL) [22,23], PCL has attracted considerable attention as matrix material in manufacturing lignocellulosic biocomposite materials. PCL is one of the relative hydrophobic polymers that are widely used in electronics, packaging, among other day-to-day life applications [24]. Among the

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biobased matrices, an investigation into the falling weight impact response of jute/methacrylated soybean oil biocomposites under low-velocity impact loading was conducted [15]. Similarly, mechanical performances such as impact strength of PLA-based composite materials have been enhanced when reinforced with addition of carefully designed core-shell and inversed core-shell particles, increased twice than neat PLA matrix [25]. Furthermore, a recent comprehensive review undertaken by Pappu et al. highlighted the industrial potential of renewable and biodegradable banana reinforcements. Their review paper further concluded that banana possesses an excellent mechanical properties such as tensile strength and modulus as well as flexural properties, impact energy, to mention but a few [26].

Additionally, there are several reported works on the development and properties of natural fiber reinforced thermoplastic composites and biodegradable biocomposites for various engineering applications. These properties include, but are not limited to, thermal degradation kinetics [27], mechanical (elastic modulus, tensile strength and modulus), thermo-physical (degradability) and structural [28], among others as well as cyclic and creep [29] and mechanical (3-point flexural and monotonic tensile) [30]. For examples, the experimental work carried out by Talla et al. [31] highlighted the effects of hemp fiber reinforcement on the mechanical and structural properties of polyethylene terephthalate (PET)-hemp fiber composite samples. They concluded that addition of additives increased the mechanical properties (elastic modulus and strain at break) of the concerned composite samples. Similarly, Talla et al. [32] reported the effect of heating rates on thermostability of the same composite sample. They suggested the possibility of effective melt process of natural fibers/high-melting thermoplastic composites with limited thermal degradation of the reinforcements.

There are not reported works which have systematically and comprehensively investigated the influence of hemp fiber aspect ratio on the mechanical properties and water absorption behavior of lignocellulosic PCL biocomposites.

The objective of the present study was to investigate the reinforcement efficiency of hemp fiber with different fiber aspect ratios on PCL-based biocomposites and compare the influence of aspect ratio on moisture absorption behavior, tensile, flexural and impact properties of hemp fiber/PCL biocomposites.

## 2. Experimental section

### 2.1. Materials and methods

Matrix material used was polycaprolactone, which is a semi crystalline polymer and it has a molecular weight of 80,000 g/mol. The PCL was used because of its biodegradability and low melting temperature (60 °C), which is desirable in the compounding of lignocellulosic fiber, such as hemp and it was supplied by Perstop (UK) (Capa© 6800). The hemp (*Cannabis sativa L.*) used was Fedora 17, which was harvested in Aube, France and supplied by FRD©. The aspect ratio plays an important role in the properties such as tensile, flexural and water absorption behaviour of composites. The samples were fabricated alongside with others to determine the influence of aspect ratio on water absorption and mechanical and the performance of a hemp fiber/polycaprolactone (HF/PCL) biocomposites. This was a combination of a natural hemp fiber and innocuous biodegradable PCL polymer in order to produce a completely biodegradable “green composite”, which is environmentally friendly, sustainable and renewable [14,25,33,34].

Fig. 1(a) shows the raw hemp fiber fraction, while Fig. 1(b) is the focus of the Fig. 1(a) where large bundles are dominants. The fiber aspect ratio is the ratio of the mean hemp fiber element length,  $l$  ( $\mu\text{m}$ ) to the mean fiber element diameter,  $d$  ( $\mu\text{m}$ ) ( $l/d$ ). Therefore, using lengths of 432, 568, 708 and 845  $\mu\text{m}$  and diameters of 22.4, 21.7, 23.6 and 22.5  $\mu\text{m}$ , respectively to produce aspect ratios of 19, 26, 30 and 38, respectively. Fig. 1(c) and (d) are illustrations of the extremes average

fibers aspect ratio,  $l/d$  values of 19 and 38 respectively, obtained after processing and after extraction from the PCL matrix. Diameter reduction of the bundles are clearly evidenced, and fragmented bundles and damaged are also observed, as depicted in Fig. 1(c), with the average  $l/d$  of 19. In Fig. 1(d), the increasing proportion of small bundle diameter or individual fibers is illustrated (average  $l/d$  of 38).

Fig. 1(b)–(d) are from a tabletop microscope scanning electron microscope (TM-1000, Hitachi, Tokyo, Japan). There were no metal coatings of the samples necessary for observing the samples at the magnification presented. The scale bars in Fig. 1(b), (c) and (d) are 100  $\mu\text{m}$ .

### 2.2. Fabrication of neat PCL and hemp fiber/PCL composite samples

To ensure homogeneity within the fiber, before being later processed, small bundles of scutched bast hemp fibers were first copped manually to length of  $1.4 \pm 0.34$  cm measured by scanner assisted image analysis (Simpalab, Techpap, Grenoble France). The composites were prepared using a laboratory-scale twin screw extrusion (TSE) Clextal BC 21 (Firminy, France). The samples were fabricated using a Clextal BC 21 laboratory-scale twin screw extrusion machine. The extruder had a length of 900 mm and a diameter of 20 mm and two profiles varying in severity were used as reported by Beaugrand and Berzin [35]. Also the hemp and PCL were introduced into the extrusion machine at two different hopper locations. For the average aspect ratio ( $l/d$ ) of 19 and 26, the profile “1” was used, a more severe screw profile than the less severe profile “2” that results in the average of 30 and 38  $l/d$ . Once the PCL was melted, the hemp was then added further down in the barrel, resulting in much less severe extrusion conditions than if they were introduced into the hopper zone simultaneously giving the average  $l/d$  of 19, 26, 30 and 38.

To prevent irregular feeding which usually occurs with long fibres when using either volumetric or gravimetric feeders. In this study, the fibres were therefore fed manually. To do so, during the extrusion the fibres were poured manually at a constant rate into the feeder or the barrel opening having the provision of beakers with 2 g prepared temporarily.

### 2.3. Water absorption test

The water absorption tests of hemp fiber/PCL biocomposites were carried out in accordance to the EN ISO 62:1999 [4]. Samples along with neat PCL, (dimension of 70 mm  $\times$  70 mm) of four different average aspect ratios: 19, 26, 30 and 38 were dried in a fan-assisted oven at 50 °C, then cooled to room temperature in a desiccators before weighting them to the nearest 0.1 mg prior to immersion into water bath. This process was repeated until the mass of water immersion specimens were reached constant [4]. Water absorption tests were conducted by immersing the composite samples in a de-ionised water bath at 25 °C, until the samples reached near saturation. The percentage of water absorption in the neat PCL and hemp fiber/PCL composites was calculated by weight difference between the samples immersed in water and the dry samples.

### 2.4. Tensile test

Tensile test samples were individually cut from the composite slabs into dumbbell shaped (6 mm thickness, 25 mm gauge length) at room temperature, using water jet cutting, the test was performed on Zwick/Roell Z030 machine. A total of four samples were tested from each type of sample with a crosshead speed of 10 mm/min.

### 2.5. Flexural test

The PCL and hemp fiber/PCL composites were tested for determining flexural strength and modulus under three-point bending test

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