



Processing and characterization of reinforced polyethylene composites made with lignocellulosic fibres isolated from waste plant biomass such as hemp [☆]



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ABSTRACT

This study was conducted to evaluate the suitability of using residual plant fibres from agricultural waste streams as reinforcement in thermoplastic composites. Three groups of plant fibres evaluated included: hemp straw, hemp pomace and cellulose fibres isolated from hemp straw. The chemical composition of these fibres was determined. Composite materials were produced from these natural lignocellulosic fibres using high density polyethylene and the aminosilane or maleated polyethylene (MA-g-PE) as compatibilizer. Chemical modification of the cellulose fillers has been performed to allow a good compatibilization between hydrophilic fibres and non-polar polymers and also to improve dispersion of fillers. The composite samples were manufactured with 30%, 40% and 60% of fibre fillers. The samples were extruded, subsequently injection-moulding processed into standard dog-bone specimens, and finally tested for physico-mechanical properties. The thermal and mechanical properties were studied by thermogravimetric analysis (TGA) and tensile tests, respectively. FT-IR spectroscopy has been employed to understand the origin of the observed changes in the structure of polymers due to the chemical modification of the fibres. The chemical composition of fibres, in terms of lignin, cellulose and hemicelluloses contents, was found to have a strong influence on the mechanical properties of the composites. Better compatibility and enhanced mechanical properties were obtained for cellulose fibres reinforced composites than for waste biomass reinforced composites.

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1. Introduction

In recent years, there has been a resurgence of interest for the use of renewable materials such as plant fibres (PF), also called “lignocellulosic fibres” (LCF), due to increasing environmental concerns along with the unique characteristics of these fibres. These include abundant availability, renewability, biodegradability, as well as reduced weight, increased flexibility, greater moldability, reduced cost and sound insulation. Lignocellulosic fibres if compared with today’s most used synthetic fibres offer the advantage of lesser health hazards and of course lower cost. Biocomposites have future commercial application that would unlock the potential of these underutilized renewable materials and provide a non-food-based market for agricultural industry [1–3]. Agricultural

crops’ residues and lignocellulose waste materials (LCW) can be a valuable source of lignocellulosic fibres, which can be used to reinforce plastics [4–6]. The LCW may be grouped into different categories such as wood residues (including sawdust and paper mill discards), grasses, waste paper, agricultural residues (including straw, stover, peelings, cobs, stalks, nutshells, non-food seeds, bagasse, domestic wastes lignocellulose garbage and sewage), food industry residues, municipal solid wastes and the like [8].

Lignocellulosic biomass mainly consists of three polymeric components hemicellulose, cellulose and lignin (see Fig. 1). Cellulose is a long chain polysaccharide formed by D-glucose units, linked by β -1,4 glycosidic bonds: its structure has crystalline parts and amorphous ones [7–11]. Each cellulose chain is stabilized by intermolecular hydrogen bonds formed between the oxygen atom in the pyranose ring of one anhydroglucose residue and the hydrogen atom in the hydroxyl group in the next anhydroglucose residue [7–11]. The hydrogen bonds cause the chains to group together in highly ordered crystal-like structure and hold the network flat. The individual cellulose chains are combined with each other to form

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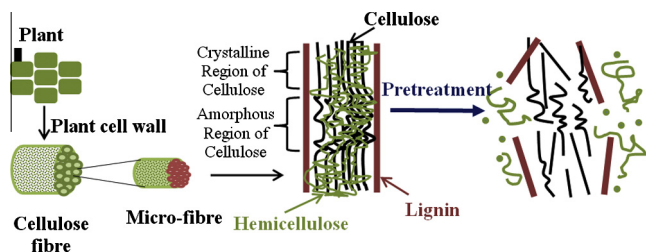


Fig. 1. Structure of lignocellulosic plant biomass.

the micro-fibrils or elementary fibrils with a diameter of 3.5 nm, which, in turn, are arranged in bundles of 20–50 nm in diameter, forming a structure known as micro-fibrillar cellulose. These bundles are the component parts of the cellulose fibres. In these fibres, cellulose is normally associated and mixed with other substances such as lignin, pectin, hemicelluloses, fats, and proteins.

The cellulose fibres obtained from waste materials can be used in the preparation of micro- and nano-cellulose fibres. The size of the obtained cellulose nano-fibres depends on several factors, a particular importance having the source of cellulose and the applied treatment [8,11]. In literature nano-fibres are often referred as “nano-whiskers” or “nano-crystals” [9–11]. Despite their dimensions in the nano-scale level these crystallites have been named “micro-fibrils” or “micro-crystallites”. The term “nano-fibres” can be defined as long flexible particles consisting of elementary assemblies of distinct polymer units that have diameters in the order of tens of nanometres, whereas the term “whiskers” is used to designate elongated crystalline rod-like nanoparticles [9–11]. The cell wall of wood fibres consists of repeated crystalline structures resulting from the aggregation of cellulose chains (micro-fibrils). These micro-fibrils are surrounded by an amorphous matrix of hemicelluloses and lignin (Fig. 1). The main reason for using cellulose nano-fibres in composite materials is their potentially high stiffness that is necessary for the reinforcement. This property can be achieved with the reducing amount of amorphous material by breaking the structure of the plant into individualized nano-fibres with high crystallinity. Nano- and micro cellulose have generated a great deal of interest as a source of nanometre-sized fillers because of their sustainability, easy availability, and the related characteristics such as a very large surface-to-volume ratio, outstanding mechanical, electrical, optical, magnetic, ferromagnetic, dielectric and thermal properties [10]. The fact that almost any cellulose material could be considered as a potential source for the isolation of nano-cellulose structures represents another important advantage in using cellulose elements as the reinforcing phase in polymeric matrix composites. However, to obtain a significant increase in material properties the nano-fibres should be well separated and evenly distributed in the matrix material. Different processing methods aided with a variety of chemicals (compatibilizers, surfactants, etc.) have been explored to fulfil these requirements [13]. The most important and widely used methods for cellulose nano-fibres isolation are mechanical, chemical, physical and biological ones [10–12,14–16]. Despite of these attractive characteristics, cellulose fibres have also a few disadvantages due to their polar and hydrophilic character [13]. These limitations make them poorly compatible with non-polar matrices, such as polyolefin, and induce loss of mechanical properties of composite material upon moisture adsorption.

The main goal of this research is to obtain polymer composites that will contain acceptable and assimilable components in the natural environment. Producing such a polymer composites will allow for using of raw materials, which are available in the given region, as well as recycling waste deriving from plastics disposal. The reduction of synthetic polymer content to the benefit of

natural components was the fundamental assumption while designing polymer composites. In this respect, the primary objective of the present work is to characterize lignocellulosic fibres from agricultural waste biomass in order to evaluate their suitability as reinforcement for biocomposite application. The final objective of this study is to obtain the highly filled polymer that will be friendlier to the nature. During this study three groups of plant fibres have been studied: lignocellulosic fibres isolated from hemp straw and hemp pomace by mechanical grinding as well as micro-cellulose fibres isolated from hemp straw by thermal/mechanical/chemical and enzymatic processing.

2. Experimental

2.1. Materials

2.1.1. Agricultural crops' residues

The raw materials used in the study were hemp straw and hemp pomace obtained after separation from bast fibres of hemp plants. These materials were obtained from the Agricultural University in Cracow under the Innovative Economy Operational Programme Project No. 01.01.02.-10-123/09 BIOMASS.

2.1.2. Matrix

The high-density polyethylene (HDPE M300054 supplied by Sabic) was used as matrix for the preparation of lignocellulosic fibres reinforced composites.

2.1.3. Coupling agents and additives

For coupling the cellulose fibres to the PE matrix, maleic anhydride grafted polyethylene designated as Licocene PEMA 4351 (supplied by Clariant), γ -aminopropyltriethoxysilane (APS) designated as A-3648 (supplied by Sigma-Aldrich). Non-metal amide wax (Licowax C) (supplied by Clariant) as a modifier of hemp straw fibres (HSF) was applied.

2.2. Preparation of plant fibres

Lignocellulosic fibres were obtained through surface pulping as a result of mechanical grinding of the hemp straw (HSF) and hemp pomace materials (HPF) into a high-speed mill. In the following step, lignocellulosic fibres (HSF, HPF) or micro-cellulose fibres isolated from the hemp straw (MCF-HS) were intensively mixed with plasticizers or aminosilane in the high-speed backward mixer Eirich R02. LCF were palletized with plasticizer in order to obtain easily deliverable pellets and to increase the bulk density of fibres.

2.2.1. Silane treatment

Micro/nano-fibres (MCF-HS) were sprayed by 5 wt.% aminosilane-ethanol solution and then mechanically mixed. The solution volume added was equivalent to 1% silane with respect to the filler mass [13].

2.3. Preparation of micro or nano-cellulose fibres (MCF/NCF-HS)

Micro/nano-cellulose fibres were obtained from the hemp straw by thermal/mechanical/chemical and enzymatic processing.

2.3.1. Purification of biomass from non-cellulosic components (extraction of fibres)

Thermal treatment of the biomass was carried out in a pulp digester with a capacity of 15 dm³. Weighed sample of biomass in an amount of 0.5–1 kg was placed in a digester and subjected to steam treatment at a pressure of 2 atm. for 10 min. Next, the biomass was three times ground in a Sprout-Waldron mill with

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