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Reinforcing of low-density polyethylene by cellulose extracted from agricultural wastes

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Abstract

Cellulosic materials were extracted from different agricultural wastes such as corn stalks, olive solid waste, and wood, by using a suitable extraction method. The extracted cellulosic materials were characterized using Fourier transform infrared spectroscopy. The produced cellulosic materials were used as reinforcements for low-density polyethylene to improve its tensile and thermal properties. A two-roll mill was used to mix the cellulosic materials (2.5–10 wt.%) with low-density polyethylene , and then the composite sheets were prepared by using a thermal press molding. The effects of filler type and its content on the mechanical and thermal properties were investigated by using the universal testing machine and differential scanning calorimeter, respectively. In general, with the increase of cellulosic materials content, there is an increase in the modulus of elasticity of the produced composites and a decrease of ductility. The ultimate tensile strength of the produced composites based on low-density polyethylene and cellulosic materials extracted from corn stalks and olive solid waste was found to be less than the tensile strength of low-density polyethylene, whereas the ultimate tensile strength of the composites based on low-density polyethylene and cellulose powder extracted from wood increased with increasing the cellulosic content. The addition of cellulosic materials was found to affect both the melting temperature of low-density polyethylene and its degree of crystallinity, depending on the cellulosic material source.

Keywords

Corn stalks, olive solid waste, wood, cellulose, elastic modulus, ultimate tensile strength, ductility, melting temperature, degree of crystallinity

Introduction

Agriculture produces significant amounts of wastes such as olive waste, wood and agricultural straws, which contain high quantities of organic matter. The agricultural wastes are causing pollution problems especially when they are burned or thrown in landfills.[1,2] Therefore, efficient utilization of such agricultural wastes is of great importance not only for minimizing the environmental impact but also for obtaining new materials that can be used in several applications. Recently, a more significant attention has been given to sustainable and biodegradable materials due to persistent discussions on the climate changes and regulatory demands underlining the need for clean environment and utilization of renewable resources.[1–4] Biomass includes a wide range of organic materials, which are composed of cellulose, hemicellulose, lignin and other compounds.[5]

Recently, cellulose-reinforced composites have received much attention because of their light weight, nonabrasive, combustible, nontoxic, low cost and biodegradable properties.[6] Several studies have been carried out to investigate the effects of addition of cellulosic materials on the properties of various types

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of polymeric matrices. Different types of natural fibers such as soybean, sisal, flax, hemp, jute and kenaf were used to improve the mechanical properties of different types of synthetic and natural polymers, the effects of fibers types and their contents, the mechanical and chemical treatments on the properties of the produced composites were investigated.[6–10]

Several researchers extracted cellulose from agricultural wastes such as olive solid waste, palm kernel cake, corn stalks, rice straw, wheat straw, sugarcane bagasse, sisal fibers, cotton stalks.[11–18] Hamed et al.[11] and Thaib et al.[12] extracted cellulose from olive solid waste by using suitable organic solvents, and after bleaching, they converted cellulosic materials to cellulose acetate. Cellulose was extracted and separated from non-cellulosic materials by using liquid phase oxidation process in which palm kernel was pretreated in hot water at 180°C and then 30% hydrogen peroxide was used in the oxidation step.[13] Cellulose was extracted from corn stalk, rice straw, wheat straw, sisal fibers and sugarcane bagasse, and the extracted cellulose was characterized using Fourier transform infrared spectroscopy (FTIR), transmission electron microscopy (TEM), thermogravimertic analysis (TGA), scanning electron microscope (SEM), X-ray diffraction (XRD), differential scanning calorimeter (DSC).[14–16] Cellulose fibers were extracted from rice straw by using alkaline treatment and thermal steam explosion methods, in which the dimensions of the extracted cellulose fibers were found to be affected by the extraction method.[17] Natural cellulose fibers were obtained from cotton stalks and then they were used to reinforce polypropylene.[18]

Graupner et al.[19] studied the mechanical properties of natural cellulosic fibers reinforced poly(lactic acid) (PLA) composites, and composites of different kinds of natural fibers like cotton, hemp, and kenaf were processed with fibers content of 40 wt.% and by using compression molding techniques.

Furthermore, Herrera-Franco and Valadez-González[20] studied the mechanical properties of short natural-fiber-reinforced composites and studied the degree of fiber–matrix adhesion and its effect on the mechanical reinforcement of short henequen fibers and a polyethylene matrix. Also, Reddy and Yang[21] studied the potential of using wheat straw as a source for long natural cellulose fibers for textile, composite and other fibrous applications.

Ali et al.[22] studied the effect of mixing conditions in an internal mixer on the viscoelastic and mechanical properties of biodegradable composites based on mater Bi-Z, a commercial polymer blend contains starch and polylactone, and sisal fibers. The properties of the produced composites were found to be affected by the mixing temperature, time of mixing and speed of rotation.

In this study, three different abundant agricultural wastes (olive waste, wood and corn stalks) were used as precursors for cellulose extraction; the selection for these wastes was because large amounts of such wastes are produced in Palestine which are not utilized in useful way causing harms to environment especially when thrown into lands or burned illegally. The extracted cellulose was used as a reinforcement for LDPE. Thermal and tensile properties of the produced composites were tested in order to investigate the effect of reinforcement type and its content on the properties of produced composite materials.

Materials and methods

Materials

Low-density polyethylene (LDPE) IPethen ®323 LDPE supplied by Carmel olefins Company with melt flow reference (190; 2.16) of 2 g/10 min was used as a polymeric matrix.

Ethylene glycol dimethyl ether ≥90%, hydrochloric acid 37%, methanol (anhydrous 99.8%), ethyl acetate (anhydrous 99.8%), sodium hydroxide ≥98%, sodium sulfide ≥90%, sodium hypochlorite 13%, hydrogen peroxide 30%, formic acid ≥95% and magnesium sulfate heptahydrate ≥98% were purchased from Sigma Aldrich and Peroxyformic acid was purchased from PubChem; all chemicals and reagents were used as supplied with further purification in extraction of cellulosic materials. Ground wood, olive industry solid waste, corn stalks were obtained from local market and used as precursors of cellulosic products.

Extraction methods

Extraction of cellulose from wood. Firstly, ground wood was dried in an oven at 105°C for 3 h; the samples (0.15–0.25 g) of oven-dried ground wood were weighed and placed in labeled 15 ml vials. Next, 2.0 ml of ethylene glycol dimethyl ether and 0.5 ml of 10 M hydrochloric acid were added to each vial, which was closed with an aluminum seal fitted with Teflon-butyl liners. The vials were shaken for 1 h in a water bath at 90°C. The seals were then removed from the vials and the residue collected by gravity filtration on a preweighed and labeled filter paper. The residue was placed into a 100-ml Erlenmeyer flask, and 20 ml of methanol was used to rinse the vials and wash the residue on the filter paper, then the residue was dried in an oven at 85°C for 90 min.[23]

Extraction of cellulose from olive solid waste. Olive solid waste was added to a round bottom flask (1 L) of Soxhlet extractor and extracted with ethyl acetate.

Then ethyl acetate was removed using the rotary evaporator. Kraft pulping was conducted in a high Parr reactor of 1 L capacity. In this process, white liquor, which is a mixture of sodium hydroxide and sodium sulfide, was used to release cellulose by breaking lignin into small molecules. White liquor was cooked in the reactor to 160°C, and then the pulp was washed.

Bleaching of the obtained pulp was carried out using a multistage process; firstly, olive solid waste was treated with 0.5% of sodium hypochlorite at 10% consistency at 60°C for 1 h. The pH of the solution was adjusted to 10; the pulp was separated by suction filtration and washed with water. In the second stage, the pulp was treated with a solution of 1% sodium hydroxide and 0.5% hydrogen peroxide to produce a solution of 10% consistency. The sample was kept at 70°C for 90 min, then the pulp was separated by suction filtration and washed with water until a neutral filtrate was obtained. Next, an aqueous solution of 2% hydrogen peroxide containing 0.5% magnesium sulfate heptahydrate and 3% sodium hydroxide was added to the pulp to produce a solution of 10% consistency which was kept at 60°C for 1 h. Later, the pulp was separated by suction filtration and washed with water. Finally, an aqueous solution of 1% sodium hypochlorite and 0.15% triethylamine was added to the pulp to produce a solution of 10% consistency which was kept at 60°C for 1 h. The pulp was separated by suction filtration and washed with water, and then it was dried in an oven at 85°C for 90 min.[11,12]

Extraction of cellulose from corn stalks. The corn stalks were refluxed with 90% (v/v) formic acid concentration on a hot plate for 2 h at boiling temperature, and the volume of liquor to fiber ratio was 8. On completion of the reaction time, the fibers were filtered in a Buchner funnel and then washed with fresh formic acid, followed by hot distilled water. The formic acid treated mass was further treated with peroxyformic acid, which was prepared by mixing 90% formic acid with 4% hydrogen peroxide, at 80°C in a thermostatic water bath for 2 h. Bleaching of unbleached pulp was carried out by using 4% hydrogen peroxide at 80°C for 1 h; the pulp concentration was 10%, and the pH of the solution was adjusted to 11 by adding the required amount of sodium hydroxide.[14] After filtration and washing with water, the pulp was dried in an oven at 85°C for 90 min.

Production of composite materials

Composites were prepared by mixing the cellulosic reinforcements with the LDPE by using a two-roll mill at 130°C for 3 min, and then the mixture was cooled to room temperature. The mixture then introduced into a homemade thermal press to form composite sheets, the press is equipped with two metallic electrically heated plates, the pressure was applied onto plates by the mean of compressed air. The thermal press is also furnished with a water cooling system. Each sample was pressed into 2.0 mm thick sheets using an 8×10 cm mold by using a homemade thermal press molding apparatus at a temperature of 130°C for 10 min under a pressure of 5–6 bar. After that, the produced sheet was cooled to room temperature by using cooling water system.

Characterization of extracted cellulose and prepared composite samples

FTIR. FTIR spectra in the range of 400 to 4000 cm^{-1} at 4 cm^{-1} resolution were acquired using a PerkinElmer Spectrum One instrument equipped with an ATR sampling apparatus. The spectral resolution used for all samples was 4 cm^{-1} and a total of 32 interferograms were acquired. FTIR test was acquired to examine the success of cellulose extraction and the purity and constituents of extracted product.

Tensile test. Tensile test was carried out by using Universal testing machine (Gunt Hamburg apparatus WP 310 machine) at a constant speed of 4 mm/min at room temperature. For each composite, five specimens of 70 mm gauge length, 20 mm width and 2.0 mm thickness were tested. The test was carried out according to the standard test method for tensile properties of plastic ASTM D638-14

Thermal test. Thermal properties of produced samples were measured by using DSC model Pyrix-6, PerkinElmer Corporation, U.K. A mass of 5 to 8 mg from each sample was placed in a sealed aluminum pan. The same temperature profile (from room temperature to 200°C), heated at 10°C/ min was applied to all the samples. Melting temperature and heat of fusion of the samples were obtained from the maximum peak and area under the peak, respectively. The latter was essential to estimate the percentage of crystalline regions. DSC test was done with regard to ASTM D3418-15 (standard test method for transition temperatures and enthalpies of fusion and crystallization of polymers by differential scanning calorimetry).

Results and discussion

FTIR results

The curves a, b and c in Figure 1 show the spectra of cellulose product synthesized from different precursors: corn, olive solid waste and wood, respectively. It is

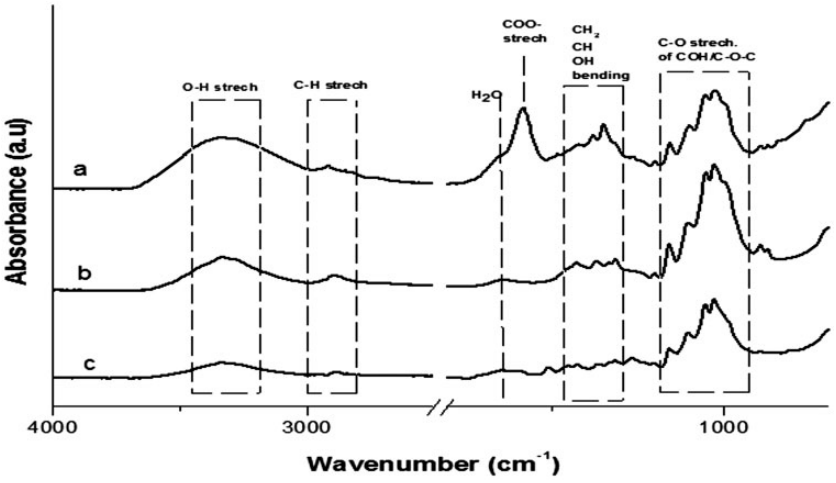

Figure 1. FTIR spectra of cellulosic samples prepared from different precursors: corn stalks (a), olive solid waste (b) and wood (c). FTIR: Fourier transform infrared spectroscopy.

Table 1. Tensile properties of composites based on LDPE and cellulose extracted from olive solid waste.

Reinforcement content (wt.%)	Ultimate tensile strength (MPa)	Tensile modulus (MPa)	Ductility (%EL)
0	8.1 ± 0.2	86.6 ± 17.2	78.3 ± 3.3
2.5	6.9 ± 0.7	141.3 ± 14.8	34.4 ± 3.4
5.0	6.4 ± 0.3	157.0 ± 4.4	20.7 ± 4.4
7.5	6.4 ± 0.5	152.6 ± 14.2	22.8 ± 2.8

LDPE: low-density polyethylene.

Table 2. Tensile properties of composites based on LDPE and cellulose fibers extracted from corn stalks.

Reinforcement content (wt.%)	Ultimate tensile strength (MPa)	Tensile modulus (MPa)	Ductility (%EL)
0	8.1 ± 0.2	86.6 ± 17.2	78.3 ± 3.3
2.5	7.3 ± 0.6	216.2 ± 24.9	26.6 ± 6.5
5.0	7.5 ± 0.3	219.4 ± 19.9	40.3 ± 1.0
7.5	7.2 ± 0.9	227.2 ± 24.6	18.4 ± 5.8
10	6.4 ± 0.5	221.2 ± 34.0	10.5 ± 1.7

LDPE: low-density polyethylene.

evident from the spectra that the existence of stretching and bending bands of CH, OH and C-O groups are the main constituents of the cellulosic structure. The existence of COO– stretching band in the corn spectrum may be related to the formation of carboxymethyl cellulose or hemicellulose which may be related to the lack of sufficient treatment or purification.[24] The spectra of wood's extract indicate several bands in the range of nearly 1510 to 1209 cm^{-1} which all ascribe to the existence of CH_2, CH and OH bending bands which are evidence of better formation of cellulose than that for olive waste and corn waste.

Tensile properties

Composites based on LDPE and cellulose powder extracted from olive solid waste. Table 1 shows the tensile properties of the produced composites based on LDPE and cellulose powder extracted from olive solid waste at different composition, and the modulus of elasticity of the composites was increased by increasing the composition of the cellulose powder, but both the ultimate tensile strength and ductility of the produced composite decreased with increasing the cellulose content. The decrease in the ultimate tensile strength may be related to poor adhesion and compatibility between LDPE because the latter is hydrophobic and cellulosic reinforcements are hydrophilic, and another reason could be related to poor mixing between cellulose and high viscous polymer melt, which results in poor distribution and dispersion of cellulosic powder within the polymeric matrix and affect adversely the role of reinforcement in enhancing the tensile strength of the produced composite due to unfilled regions.

Composites based on LDPE and cellulose fibers extracted from corn stalks. The tensile properties of the produced composites using LDPE and cellulose fibers extracted from corn stalks are shown in Table 2.

It can be seen from Table 2 that the modulus of elasticity of the composites was increased with increasing the reinforcement content which reached 227.2 MPa at a cellulosic content of 7.5 wt.%. The ultimate tensile

Table 3. Tensile properties of composites based on LDPE and cellulose powder extracted from wood.

Reinforcement content (wt.%)	Tensile strength (MPa)	Tensile modulus (MPa)	Ductility (%EL)
0	8.1 ± 0.2	86.6 ± 17.2	78.3 ± 3.3
2.5	8.9 ± 0.7	215.8 ± 14.8	19.5 ± 8.3
5.0	10.4 ± 0.8	276.3 ± 12.9	18.7 ± 5.8
7.5	10.7 ± 1.1	296.4 ± 29.9	12.9 ± 2.7
10	10.8 ± 0.3	331.6 ± 36.8	12.0 ± 2.7

LDPE: low-density polyethylene.

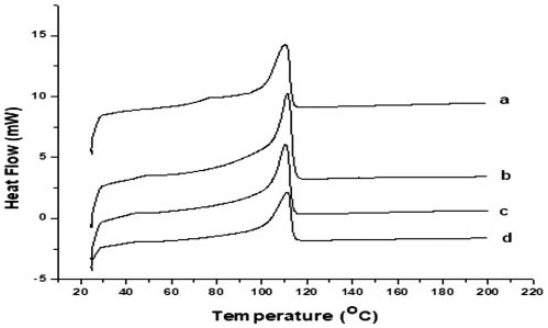

Figure 2. DSC results for (a) LDPE and LDPE containing 5 wt.% cellulose extracted from Corn (b), wood (c) and olive solid waste (d). LDPE: low-density polyethylene.

strength of the produced composites is less than the tensile strength of LDPE. The decrease in ultimate tensile strength may be related to poor adhesion between the reinforcement and the matrix and due to the presence of traces of hemicellulose and/or carboxymethyl cellulose as observed from FTIR results, the existance of COO– streching band in corn spectrum was observed (Figure 1). In addition, at low-fiber content, there was poor distribution and voids were observed in the produced composite sheets until 5 wt.%. A maximum ultimate tensile strength of 7.5 MPa was obtained by the addition of 5 wt.% cellulose fibers. The ductility of the composite materials decreased from 78.3 to 40.3 with the addition of 5 wt.% cellulose.

Composites based on LDPE and cellulose powder extracted from wood. Table 3 shows the tensile properties of the produced composites using LDPE and cellulose powder extracted from wood at different compositions. The ultimate tensile strength of the produced composites increased dramatically from 8.1 MPa to 10.8 MPa with the addition of 10 wt.% cellulose powder. Also, the modulus was largely increased from 86.6 MPa to 331.6 MPa with the addition of 10 wt.% cellulose fibers. On the other hand, the ductility decreased with the increase of cellulose powder content. The enhancement achieved in both tensile strength and modulus could be ascribed to the purity of the cellulose produced from wood precursor and its liberation from non-cellulosic components such as CMC or hemicellulose as shown in Figure 1. This purity of reinforcement contributed in increasing the degree of adhesion with the polymeric matrix.

Thermal properties

Pure LDPE and samples containing 5 wt.% of each type of cellulose were scanned by a DSC from room temperature to 200°C, and the heating curve of each sample is shown in Figure 2.

Table 4. Thermal properties of 5 wt.% composite materials & pure LDPE.

Reinforcement type	Melting temperature (°C)	ΔH^0_f (J/g)	%Degree of crystallinity
Pure LDPE	110.2	52.1	18.0
Olive solid waste	111.3	50.6	18.4
Corn stalks	111.4	40.9	14.8
Wood	110.5	64.6	23.4

LDPE: low-density polyethylene.

The effects of reinforcement type at constant reinforcement content (5 wt.%) on the melting point and degree of crystallization of LDPE are shown in Table 4. The percentage of crystalline regions can be calculated from the following equation

$$x_c = \frac{\Delta H_f}{\Delta H_f^0 X \varphi} \times 100\%$$

where x_C is percent of crystalline regions, ΔH_f is heat of fusion of the sample; ΔH°_f is heat of fusion for 100% crystalline polyethylene which is found to be 290 J/g[25] and φ is weight percent of polyethylene in the produced composite.

Table 4 shows the melting point and the percentage of crystallinity of each produced composite at a cellulose content of 5 wt.%. The increase in the melting point of LDPE was varied from 0.3°C to 1.2°C, and it is obvious that there is no clear effect on melting temperature which in turn will not adversely affect the ease of processing of these composite materials. Furthermore, the percentage of crystallinity was increased for all the composites except the one that contains cellulose fibers extracted from the corn stalks. The composite which contains LDPE and cellulose that was extracted from wood had the highest percentage of crystallinity (24.2%). The increase in both melting point and degree of crystallinity

by the addition of reinforcement may be related to the fact that the reinforcement acts as nucleating agent.

The degree of crystallinity affects the tensile strength and modulus of elasticity of the produced composites, and it can be seen from Tables 1 to 3 that the tensile strength and modulus of elasticity of composites containing 5 wt.% cellulosic powder extracted from wood are higher than those produced by using other reinforcements. This can also be attributed to the increase in the degree of crystallinity of LDPE when cellulosic reinforcement extracted from wood was used. This result emphasizes the existence of more cellulose in the extract of wood because cellulose has a crystallinity between 50 and 81% depending on the source,[26] and when it is present in the composite it will attribute to the resultant crystallinity and meanwhile enhancing all related properties.

Conclusion

Cellulose reinforcements were extracted from corn stalks, olive solid wastes, and wood. It was found that the cellulose is the main constituent of wood and olive solid waste, but corn-stalk wastes contain cellulose and hemicellulose. Tensile properties of the produced composites using LDPE and cellulose reinforcement extracted from wood have better tensile properties. It was observed that the modulus of elasticity of the produced composite increases from 86.6 to 331.6 MPa and its ultimate tensile strength increases from 8.1 to 10.8 MPa when 10 wt.% cellulose extracted from wood was used. The degree of crystallinity of LDPE was found to be affected by cellulosic material source, and it increased from 18.0% to 23.4% when 10 wt.% cellulose extracted from wood was added. The enhancement in both modulus and tensile strength of composite samples containing cellulose from wood and olive waste could be a positive indication toward the use of these composites in different applications such as floor panels, interior house decorate parts, food packages, etc., but the noticed drop in ductility (which is a normal relation with modulus; an increase in modulus leads to decrease in ductility) might restrict their application in certain fields.

Finally, it is concluded that the utilization of these wastes could minimize the harmful effect on environment besides the enhancement of mechanical and thermal properties of LDPE polymer.

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Declaration of Conflicting Interests

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