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Original Research Paper

Effect of filler load and high-energy ball milling process on properties of plasticized wheat gluten/olive pomace biocomposite

Asma Boudria [a], Yasmina Hammoui [a,b,*], Nawel Adjeroud [a], Nabila Djerrada [a], Khodir Madani [a]

[a] Laboratoire de Biomathématiques, Biophysique, Biochimie, et Scientométrie (L3BS), Faculté des Sciences de la Nature et de la Vie, Université de Bejaia, 06000 Bejaia, Algeria
[b] Département de microbiologie et de biochimie, Faculté des Sciences, Université de M'sila, 28000 M'sila, Algeria

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A B S T R A C T

The increase of particles surface area can optimize the dispersion state of biocomposite components and enhance their properties. First in this paper, we aimed to elaborate a novel biocomposite without any treatments. Plasticized wheat gluten (WG), was filled with 0–20% of olive pomace (OP) powder. The second objective was the improvement of biocomposite properties using physical treatment. High-energy ball milling process was applied on the blend of wheat gluten and olive pomace powders (MPs). The grinding effect of particle shape, size and distribution in biocomposite was characterised by particle size distribution using a laser-light diffraction and by SEM analysis. The cryo-fractured surface of selected films, mechanical properties, moisture absorption and thermal properties of both biocomposites were described in details. It was found that the sensitivity of biocomposites to moisture absorption was reduced with the increase of filler content after the applying of high-energy ball milling process. The thermal stability of OP biocomposite decreased with the increase of loading, while that of MPs was unaffected by high-energy ball milling process. This process affects the physical and morphological characteristics of the powders. The mechanical properties were improved by grinding process at filler content lower than15%.

1. Introduction

Given growing environmental concerns, biodegradable polymers have received much attention in academia and industry in the past two decades [1]. Moreover, governments in many countries are supporting usage of green products [2] and renewable resources such as agricultural byproducts. Wheat gluten is an interesting candidate because it is a low-cost raw material, renewable and available [3]. It has remarkable viscoelastic properties, ability to cross-link upon heating and low water solubility. From the chemical standpoint, gluten is composed of two storage proteins, gliadin and glutenin. Wheat gluten-based materials can be obtained by thermoplastic processing, which consists in mixing proteins and plasticizer by a combination of heat and shear [4]; followed by a thermo-mechanical treatments (e.g. compression moulding) [5]. Protein-based materials have been explored as potential materials because of their good barrier properties against oxygen and aroma compounds [4]. However, wheat gluten-based materials have drawbacks that can limit their applications, such as their brittleness and their high moisture sensitivity [6]. This requires an improvement of theirs properties by reinforcement of plasticized wheat gluten to produce a novel biocomposite with characteristics that hold great promise [7].

Raw fibres/particles have been largely exploited as reinforcements into polymer matrices as a substitute to the used synthetic fillers. The natural fillers can be obtained from both forestry and agricultural resources [8], among them, olive pomace which is a by-product of olive oil production industry. Considerable amounts of these wastes are produced and present an environmental hazard in olive oil producing countries. Therefore, there is an urgent need to treat these materials safely [9]. In Algeria, huge amounts of olive pomace are generated; it represents 10^5 t per year, this amount of agrowastes is usually burned [10], however, they can be an important source of renewable fillers since they give bio-based composites unique properties by improving their mechanical properties and water resistance [11]. It contains a great amount of cellulose, hemicelluloses, and lignin. To produce biocomposites with good mechanical properties, a strong adhesion has to be obtained by interfacial interactions, including mechanical interlocking,

* Corresponding author at: Laboratoire de Biomathématiques, Biophysique, Biochimie, et Scientométrie (L3BS), Faculté des Sciences de la Nature et de la Vie, Université de Bejaia, 06000 Bejaia, Algeria.
E-mail address: yasmina.hammoui@univ-bejaia.dz (Y. Hammoui).

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chemical bonding and physical adhesion [12]. There has been a lot of research on various methods to improve adhesion between biocomposite components [13]. High-energy ball milling process is a cost-effective and eco-friendly physical technique [14]. Materials with novel microstructures and properties have been synthesised via this process, using planetary ball mill [15]. High-energy ball milling process combined friction, collision and shear resulting from the grinding balls and the container wall [16]. This particle-size reduction technique can increase the surface area and improves interface adhesion in the materials by particle distribution enhancement in the biocomposite [17]. However, the particle size can be expected to influence a range of mechanical properties [18]. The present work aims to elaborate a biocomposite based on plasticized wheat gluten with glycerol containing 0–20% of OP. To improve OP biocomposite properties, high-energy ball milling process was applied to produce MPs biocomposite. The properties of boths biocomposites, in function of filler contents, were studied. Grinding effect on shape, size and distribution of particles in the biocomposite were characterised by particle size distribution (PSD) using a laser-light diffraction and SEM analysis. The cryo-fractured surface of selected biocomposites was observed using SEM analysis. The particle-size reduction effect on mechanical properties, moisture absorption, mass loss, micropores ratio and thermal properties of biocomposites were also investigated.

2. Experimental work

2.1. Chemical

Analytical grade glycerol (≥99%) was purchased from Sigma Aldrich (Saint-Louis, United States).

2.2. Raw materials

Wheat gluten was obtained from Tereos Syral (Marckolsheim, France). Chemlal olive pomace (OP) was obtained from a local olive refinery in the area of Fenaia-Ilmaten (Bejaia, North-east of Algeria), and washed with hot tap water to remove all water-soluble impurities, followed by drying at room temperature. The product was ground using an electrical grinder (IKA model-A11, Staufen, Germany) and was sieved using standard 125 µm sieve.

2.3. Particle size distribution (PSD)

The particle size distribution of wheat gluten, OP and MPs powders were determined using a laser-light diffraction unit (Mastersizer S, Malvern Instruments Ltd., Worcestershire, UK) equipped with 300 RF lens. The diameters D (v, 0.10), D (v, 0.50) and D (v, 0.9) at 10% (small particles), 50% (medium size particle), 90% (large coarse particle), respectively and volume mean diameter (D [4.3]) were computed.

2.4. High-energy ball milling process

A planetary ball mill (model PM400, Fritsch, Haan, Germany) was used to grind the dry blend of this study. Using 250 mL capacity stainless steel milling jars and lids, charged with spherical zirconia (ZrO) balls (10 mm diameter) as grinding media. The powder (g) to media (g) ratio was maintained at 1:10 for all milling experiments. The speed of ball milling was set at 150 rpm. The duration of milling was 10 h. Grinding was performed as follows: 35 g of the blend of powders (wheat gluten and olive pomace) labelled MP_s, and 47 spherical zirconia balls were placed in the jars, which was filled with 80 vol% of ZrO balls and powders. The rate of each blend compounds was shown in Table 1. After treatment, the milled blend of olive pomace and wheat gluten powders, named Milled powders (MPs) was collected after removing the balls, then mixed with a plasticizer in order to manufacture the biocomposites.

Table 1
Composition of plasticized Wheat Gluten (WG) and biocomposites, all percentages were calculated on a dry weight basis.

Biocomposite names[a]		Sample compositions (%, w/w)	
		Wheat gluten	Powder (filler)
WG		65	0
OP_5	**MPs_5**	60	5
OP_10	**MPs_10**	55	10
OP_15	**MPs_15**	50	15
OP_20	**MPs_20**	45	20

[a] **WG:** plasticized Wheat Gluten. All materials were plasticized with 35 (%, w/w) of glycerol, and the composition of WG and biocomposites were calculated on a dry weight. The indexes 5, 10, 15, 20 represent the percentage of powder. **OP:** Olive Pomace powder, **MPs:** Milled Powders.

2.5. Preparation of biocomposites

Processing of [19,20] was adopted to elaborate biocomposites, wheat gluten and dried OP powder were firstly hand mixed to the desired proportions (Table 1). Then, the resulting powder was mixed with glycerol (35%, based on total dry weight) in a two-blade counter-rotating batch mixer, turning at 3:2 differential speed (Brabender, Duisburg, Germany). For the milled powders, glycerol (35%, based on total dry weight) was directly mixed with MPs powder to the desired proportions (Table 1). The mixtures were performed by mixing at a speed of 100 rpm during 15 min at 70 °C. The blends were then thermo-moulded in a heated press (Carver hot press model-2629, Wabash, United States) at 120 °C. Approximately, 4 g of the blends were placed between two aluminium sheets in a rectangular mould (80 × 40 mm) for 10 min without pressure, followed by 3 min under a pressure of 15 MPa. Then they were removed from the mould and cooled at room temperature. The thickness of the resulting films was approximatively 0.5 mm. Prior to the tensile test, the films were conditioned into a desiccator producing 43% relative humidity at 24 °C for one week.

2.6. Scanning electron microscopy (SEM)

The micrographs of morphology observation of MPs powder contains 5, 10, 15 and 20 (%, w/w) of OP powder (Table 2), WG films, selected biocomposites (OP_10 and MPs_10) (Table 3), were obtained using 8 kV secondary electrons microscopy (JEOL JSM-6100, Tokyo, Japan). Each material sample was frozen in liquid nitrogen and fractured. All samples (MP_s powders and cryo-fractured biocomposites) were coated with gold/palladium on a JEOL JFC-1100E ion sputter coater (Tokyo, Japan) before observation.

2.7. Micropore ratio estimation

The micropore ratio (%) of biocomposites (Table 3) were derived from SEM images. To investigate the repeatability of the results, a minimum set of five similar images in term of magnifications (×300), and contrast for each films were processed by ImageJ software (ver. 1.49, NIH, Maryland, USA), using manual threshold-based segmentation algorithm. The results were reported as mean ± standard deviations (S.D).

Before ImageJ analysis steps [21,22], which are detailed below, the SEM images needs to be calibrated to their scales:

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