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A B S T R A C T

Biodegradable foams made from potato starch and natural fibres were obtained by extrusion. The effects of varying origins of these fibres on foam properties were studied, as well the relationships between their properties and the foam microstructure. The addition of fibres increased the expansion index and led to a significant reduction in water adsorption of starch foams, generally improving foam properties. The mechanical properties of the foams were affected by both relative humidity (RH) of storage and foam formulation. In general, as the RH increased, the foam strength decreased. The formulation presenting the best mechanical properties contained 10 wt% hemp fibre and had a maximal resistance of 4.14 MPa and a modulus of 228 MPa, corresponding to a more compact and dense microstructure.

Keywords:
Starch foam
Natural fibres
Microstructure
Mechanical properties
Biodegradation

1. Introduction

The difficulty for recycling the oil-derived synthetic plastics have promoted the development of biodegradable materials, made from agro-industrial polymers obtained from renewable, abundant and low cost sources (Gàspàr et al., 2005; Dogossy and Czigány, 2006; Davis and Song, 2006; Cinelli et al., 2006; Lui and Peng, 2005). Since about 41% of plastics production is used for packaging industry, and 47% of this is used for food packaging (Fomin and Guzeev, 2001), the use of biopolymers within this field appears as an excellent alternative for reducing current environmental problems.

Some studies have shown that it is possible to obtain food containers from mixtures of starch, fibres and water by processes such as thermopressing (Glenn and Orts, 2001; Glenn et al., 2001a; Schmidt, 2006; Shey et al., 2006; Shogren et al., 1998, 2002; Soykeabkaew et al., 2004), that can be an alternative to the extensively used expanded polystyrene foams.

The disadvantage of the resulting materials is their fragility and their high affinity for water (Glenn et al., 2001a,b). To improve these properties, the generation of these materials from modified starches or after addition of plasticizers, polymers, fibres and other additives has been reported. Some authors showed that starch foam tensile strength and density increased while foam flexibility decreased with increasing starch concentration, molecular weight and amylase content (Shogren et al., 1998). They reported that tuber starches, such as potato, produce trays with lower densities and higher flexibilities than those from cereals such as corn.

The tensile strength of starch foams and sometimes the deformation at break were also improved by the addition of different types of fibres, such as softwood, aspen, jute and flax fibres (Glenn et al., 2001b; Lawton et al., 2004; Shogren et al., 2002; Soykeabkaew et al., 2004) or sugarcane bagasse fibres (Mali et al., 2010).

Materials derived from agriculture are emerging as promising substitutes for conventional plastics. Thermoplastic starch has a low initial cost (US$ 0.25–0.6/kg) and wide availability; thus, it is the focus of a great interest in the production of disposable products. Starch foams with insulating properties similar to those of polystyrene foam have been industrially produced by the extrusion process (Chiellini et al., 2009). According to Guan and Hanna (2004), starch foams can be employed to substitute polystyrene products, especially for loose-fill packaging application. Loose-fill packaging materials provide cushioning, protection, and stabilization of articles packaged and have to present low density, good resilience, and compressibility (Tatarka and Cunningham, 1998). Starch foams have promising characteristics, but are susceptible to moisture when they are stored in high relative humidity. Water molecules attack the hydrogen bonds of starch, weakening them and decreasing the functional properties of the materials. The addition of natural fibres and other biodegradable polymers in the production of materials to improve moisture sensitivity has been

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reported (Lawton et al., 2004; Guan and Hanna, 2006; Salgado et al., 2008) as having good results.

In this work, different fibres from wheat or cotton were used as filler in starch foam to reduce moisture sensibility. Besides being inexpensive, non-toxic, and easily recycled, the use of this material contributes to environmental protection (Ruggiero et al., 2006). During biodegradation, the presence of fibres induces fast breakdown of foam due to the action of microorganisms attracted by its lignocellulosic components (Chiellini et al., 2009). Thus, the objectives of this work were to evaluate the effects of potato starch and different natural fibres on the mechanical properties, microstructure, density, expansion index, water adsorption of extruded foams, using a mixture design methodology.

2. Experimental

2.1. Materials

Potato starch from Roquette society (France) contained between 20 and 25 wt% amylose, 75–80 wt% amylopectin and 0.05 wt% proteins based on dry weight.

Wheat straw fibres (l = 2.7 mm) from A.R. D society (France), hemp fibres (l = 15 mm) from Chanvrière de l'Aube (France), cotton linter fibres (l = 18 mm) from Maeda society (Brazil), cellulose fibres (l = 120 μm) from Rettenmaier & Söhne (Germany) have been used as reinforcement.

2.2. Sample preparation

Co-rotating twin-screw extruder (CLEXTRAL B21) was used for the foam extrusion. The general screw geometry was: 900 mm length, 25 mm diameter and a flat die (1.5 mm thick and 40 mm wide). The screw configuration included positive displacement screw elements with decreasing pitches. Screw speed was fixed at 300 rpm. Temperatures of the 12 heating zones from the feed end to the die of the extruder were: 30/30/50/60/70/80/90/90/100/120/120/160 °C. In general all formulations were processed with specific mechanical energy (SME) values between 60 and 90 Wh/kg. A previous work has shown that beyond SME value of 150 Wh/kg extrudates begun to degrade (Della Valle et al., 1995). The water was added with a peristaltic pump. Sheet shape samples were collected by calendaring the extruded foam.

2.3. Formulations

Different composites containing the same quantity of fibres were prepared. Cellulose, wheat straw, hemp and cotton linter fibre contents was 10 wt%. Regular expansion was achieved by adding 2 wt% of talc (Talc de Luzenac, France) and 2 wt% of additive (Hydrocerol, Clariant, France). The matrix (M) was prepared with: starch + 17 wt% of water + 2 wt% of talc + 2 wt% of additive. Different composites were produced afterwards by adding 10 wt% of fibres to this matrix.

2.4. Characterizations

The composites were characterized by their density, expansion ratio, mechanical properties, hygroscopicity, cellular structure and biodegradability.

The foam density was calculated as the ratio between weight and volume (Cha et al., 2001).

The expansion ratio was calculated as the ratio of the transversal sample surface to the die surface (Glenn et al., 2001a,b).

The mechanical properties (elastic modulus, maximal flexural strength and deformation at break) were determined by 3 points bending tests (Glenn et al., 2001a,b). Samples (95 mm × 22 mm × 4 mm) were equilibrated at 33, 56 and 75% relative humidity (RH) at 23 °C for 7 days. Flexural tests were performed using Adamel Lhomargy DY26 testing machine at a deformation rate of 2 mm/min.

In order to determine hygroscopicity of composites, pre-dried samples (48 h at 105 °C) were placed in sealable containers having relative humidity levels of 33, 56 and 75%. Moisture absorption of samples was calculated as the difference between the weight of a sample recorded after being exposed to humidity until saturation and the weight of a dried sample (Glenn et al., 2001a,b).

Cellular structure has been studied with help of Scanning Electron Microscopy (SEM). Foam samples for SEM were cut with a razor blade into 3 mm thick slices and mounted on aluminum stubs. The transversal cross-section area was examined by means of an Environmental Scanning Electron Microscope (ESEM, FEI 200 Quanta FEG, pressure 0.9 Torr).

Average diameters of about 100 cells were measured by image analysis software (AnalySYS 3.0). Plot of a cell size distribution enabled a comparison of cellular structure for different formulations of starch foams. Number (D_n) (Eq. (1)) and weighted (D_w also called D_{21}) (Eq. (2)) average of cell diameters were calculated (Trater et al., 2005).

$$\bar{D}_w = \frac{\sum(D_i^2 n_i)}{\sum(D_i n_i)} \tag{1}$$

$$\bar{D}_n = \frac{\sum(D_i n_i)}{\sum(D_i n_i)} \tag{2}$$

where n_i is the number of cells in class size i and D_i is the diameter of the cells in class size i.

Polydispersity index (PDI) was determined in order to indicate uniformity of cellular structure.

$$\mathrm{PDI} = \frac{\bar{D}_n}{\bar{D}_w} \tag{3}$$

To calculate the index of sphericity (Eq. (4)) the lengths of the minor axis (b) and the major axis (a) cells, treated as ellipses were measured

$$\bar{I}_s = \frac{\bar{b}}{\bar{a}} \tag{4}$$

To determine cell wall thickness we assumed that cells were circular, with centers of circles being vertices of hexagon (in one plane) and this circle inside of the hexagon. In terms of density, it was calculated that coefficient of space fulfilment for such formation would be 0.74.

$$\rho_{\mathrm{foam}} = \frac{m_{\mathrm{starch}}}{V_{\mathrm{foam}}} = \frac{\rho_{\mathrm{foam}} 4/3\pi(R^3 - r^3)}{4/3\pi R^3/0.74}$$

$$= 0.74 \times \rho_{\mathrm{starch}} \times \left(1 - \frac{(R-e)^3}{R^3}\right) \tag{5}$$

with: ρ_{foam} is the density of foam, ρ_{starch} is the density of starch (Knutson, 1998), R-radius, r-radius and e-cell wall thickness ($r = R - e$).

Biodegradability of composites was investigated by determining Biochemical Oxygen Demand (BOD). For these aerobic tests we used standard ISO 14851 procedure (AFNOR, 2004). Tests were carried out in closed OxyTop® respirometer. Samples, inorganic medium and inoculum are stirred in closed bottles of respirometer. The oxygen consumption was determined by measuring pressure changes inside closed bottles while CO_2 produced by microorganisms is absorbed by sodium hydroxide solution in headspace of the bottle. The pressure variations were then converted to BOD values.

Tests were performed in bottles containing approximately 100 mg of samples (weight calculated according to standard), 97 mL of inorganic medium and 3 mL of inoculum.

Inorganic medium was prepared according to standard and was composed of different solutions:

- Solution A: KH_2PO_4 (8.5 g/L), K_2HPO_4 (22.5 g/L), Na_2HPO_4, $2H_2O$ (33.4 g/L), NH_4Cl (0.5 g/L)
- Solution B: $MgSO_4$, $7H_2O$ (22.5 g/L)
- Solution C: $CaCl_2$, $2H_2O$ (36.4 g/L)
- Solution D: $FeCl_3$, $6H_2O$ (0.25 g/L)

1 L of test medium contained 10 mL of solution A, 1 mL of each solution B, C and D and distilled water. pH of test medium was approximately 6.8.

The inoculum used was microbial inoculum extracted from activated sludge taken from wastewater treatment plant.

Bottles were placed in a dark room and temperature of the room was maintained at $20 \pm 1\,^{\circ}C$ during 31 days. Samples were tested in triplicate and BODs of inorganic medium and inoculum containing no sample (BOD_b) were also determined. Material's biochemical oxygen demand (BOD_m) was then calculated as:

$$BOD_m(mg/L) = BOD_{tot} - BOD_b \quad (6)$$

where BOD_{tot} were values measured for bottles containing samples.

Percentage of biodegradation (D_t) was then calculated as ratio of specific biochemical oxygen demand (BOD_s) and theoretical oxygen demand (ThOD) (Mezzanotte et al., 2005).

BOD_s was determined as:

$$BOD_s = \frac{BOD_m}{\rho_m} \quad (7)$$

where ρ_m is material concentration in test medium mg/L.

Theoretical oxygen demand (ThOD) of $C_cH_hCl_{cl}N_nS_sP_pNa_{na}O_o$ with molecular mass M_r, can be calculated as:

$$ThOD = \frac{16\,[2c + 0.5 \times (h - cl - 3n) + 3s + 2.5p + 0.5na - o]}{M_r} \quad (eq.8)$$

And percentage of biodegradation (D_t) is then calculated as (Eq. (9)):

$$D_t = \frac{BOD_s}{ThOD} \times 100 \quad (9)$$

3. Results and discussion

3.1. Water absorption

The maximum quantities of water absorbed by the matrix and the samples containing different types of fibres are represented, for different formulations (Fig. 1).

Values of starch foam hygroscopicity at 33, 56 and 75% RH (relative humidity) are equal to 9, 12.5 and 17%, respectively. Soykeabkaew et al. (2004) reported equivalent for starch foam (7.9, 11.1 and 16.6% at 33, 53 and 75% RH, respectively). Thus, our results are close to those obtained by these authors. For all starch/fibres formulations, a slightly lower hygroscopicities are observed.

These results suggest that the presence of fibres modifies the water sensitivity of starch. The fibres, which may be absorbed less water, are responsible of this water sensitivity reduction for composites.Composites reinforced by hemp and cellulose fibres are those that absorb less water. Composites with cotton or wheat fibres absorb more water than starch. These results may explain better mechanical properties obtained for starch foam reinforced by hemp fibres. In all cases, water absorption increases with a higher moisture content.

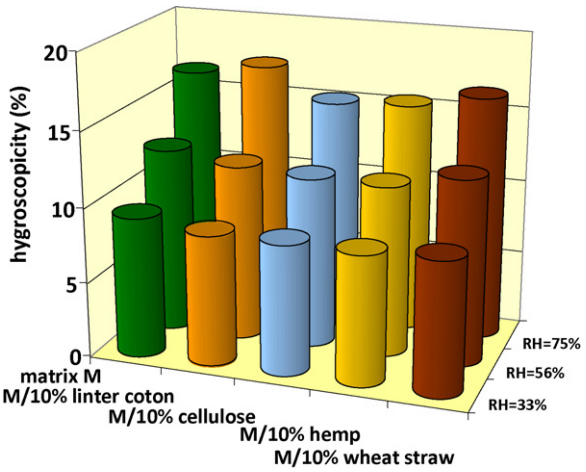

Fig. 1. Hygroscopicity of the composite formulations containing 10 wt% fibres.

The water absorption of isolated fibres was measured (Fig. 2). It can be concluded that even the fibres absorb water, this absorption is less important than that of starch (about 10% instead of 17% for foamed starch at 75% RH). This lower fibre absorption may explain the slight decrease in sensitivity to water of these composites.

Nevertheless intrinsic lower water sensibility of fibres may not explain alone the water of composites regardless to the law of mixtures. Indeed interactions between fibres and matrix (formation of hydrogen bonds) may be also altered in presence of RH. Furthermore, differences in the microstructure such as wall thickness and cell size, may induce variation in hygroscopicity of composites.

3.2. Mechanical properties

Mechanical properties of expanded starch–fibre composites (Table 1) were influenced by the fibre nature and by the storage conditions (relative humidity). Among the various tested fibres, at a constant fibre content (10 wt%), hemp and cellulose fibres confer a significant reinforcement effect to the starch foam. In the case of the hemp, the reinforcement was assigned to the highest rigidity and length of fibres.

An increase in relative humidity level results in a decrease in the mechanical properties. This is related to the plasticizing effect of water with respect to starch. It has been shown (Figs. 1 and 2) that natural fibres are less water sensitive than starch and that the incorporation of fibres in starch foam may imply a reduction of

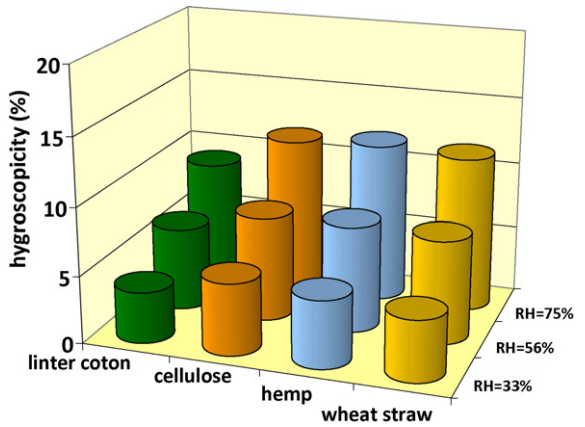

Fig. 2. Hygroscopicity of different isolated fibres.

Table 1
Mechanical properties of foamed starch and biocomposites at different relative humidities.

Relative humidity	Modulus (MPa)			Flexural strength (MPa)			Deformation at break (%)		
	33%	56%	75%	33%	56%	75%	33%	56%	75%
Matrix (*M*)	126 ± 27	106 ± 13	106 ± 15	2.81 ± 0.38	2.19 ± 0.21	2.10 ± 0.29	3.45 ± 0.56	2.87 ± 0.44	2.87 ± 0.26
M + 10 wt% cellulose fibres	199 ± 13	176 ± 9	156 ± 10	3.92 ± 0.36	3.38 ± 0.34	2.91 ± 0.17	3.04 ± 0.32	2.73 ± 0.47	2.74 ± 0.28
M + 10 wt% hemp fibres	228 ± 16	208 ± 16	172 ± 8	4.14 ± 0.45	3.76 ± 0.51	3.53 ± 0.33	2.33 ± 0.48	2.27 ± 0.43	2.69 ± 0.45
M + 10 wt% linter coton fibres	107 ± 9	96 ± 12	91 ± 16	2.47 ± 0.20	2.15 ± 0.17	1.77 ± 0.20	3.66 ± 0.80	3.71 ± 1.09	2.77 ± 0.31
M + 10 wt% wheat straw fibres	151 ± 17	116 ± 16	105 ± 11	3.02 ± 0.40	2.52 ± 0.19	2.17 ± 0.31	2.82 ± 0.45	3.18 ± 0.44	3.08 ± 0.65

hygroscopicity and thus an improvement of mechanical properties in some cases especially for hemp fibres.

3.3. Density, expansion ratio and foam microstructure

Density data are presented in Table 2. It is noticeable that the addition of fibres contributes to lower the composite density except for hemp fibre. Composite containing linter cotton fibres gave foam with the lowest density. It was also observed that the presence of fibres reduces the expansion ratio of starch foam for cellulose and hemp fibres (Table 2). During extrusion process two main mechanisms may occur. On one side, fibres may increase the viscosity of the moulded starch so that cell growing ability is lowered. On the other side, fibres may act as nucleating agents providing surfaces for cell growth increasing therefore the number of cells.

The study of the physico-chemical and mechanical properties of different types of composites showed that these properties change depending on the nature of fibre in the composite. Generally, an improvement of mechanical properties of expanded starch with the addition of fibres was observed. This has highlighted a reinforcing fibre and a good adhesion between fibre and matrix (interface). It was shown (Table 2) that the density of the expanded starch was reduced with the addition of fibres. It was assumed that a nucleation effect of fibre was the cause of this phenomenon. However, differences between the functional properties of the composites containing different fibres and nucleating agents have been observed.

One hypothesis assumes that these differences can be attributed to variations in composite morphologies.

The microscopy of the surface cross-sections of the matrix and composites containing 10 wt% of the various types of fibres are shown in Figs. 3 and 4. Table 3 lists the values of the cell size, the wall thickness, the polydispersity index and the sphericity of the cells.

For all composites, a decrease of cell size with the decrease of density is observed (Table 2). This decrease could lead to a reduction in the wall thickness of walls. All this is only available with an increase of the cell number.

Similarly, it was difficult to establish a relationship between changes in mechanical properties and morphology of composites. The cell size and the thickness of cell walls are the two most important parameters when discussing the morphology of composites. Only one of these two parameters cannot be set because at the same time changing which makes it difficult to evaluate the mechanical properties depending on a single parameter.

Moreover, the polydispersity index (PDI, Table 3) of composites reinforced by linter cotton fibres is the highest. This microstructure reflects more cell homogeneity in this composite. Sphericity is close to 0.70 for all composites because of the use of the same process.

3.4. Biodegradability

The curves of the Biochemical Oxygen Demand (BOD) analysis show (Fig. 5) differences between the BOD of the final plateau.

It can explain by the fact that the degradation of starch may occur before the degradation of fibres. In fact, the activated sludge was used for this experiment came from a wastewater treatment. Such sludge contains bacteria that can more easily produce enzymes for degradation of starch. These bacteria degrade first the starch depending on the acclimation time (the time required to produce specific enzymes). The degradation of fibres will occur then. The time of acclimation of bacteria can be very variable. A time of fibre degradation exceeding 31 days could explain this variation in the final plateau of the BOD.The foam containing only starch has the highest biodegradation. The composites with cellulose or hemp fibres have a higher degradation rate than composites made from wheat or cotton for a given degradation time.

Table 2
Densities of starch based composites as a function of fibre nature.

Biocomposite	Density (g/cm^3)	Expansion ratio
Matrix (*M*)	0.236 ± 0.017	2.919 ± 0.213
M + 10 wt% cellulose fibres	0.191 ± 0.010	2.847 ± 0.075
M + 10 wt% hemp fibres	0.242 ± 0.010	2.844 ± 0.171
M + 10 wt% linter cotton fibres	0.175 ± 0.008	3.360 ± 0.169
M + 10 wt% wheat straw fibres	0.210 ± 0.008	3.470 ± 0.113

Table 3
Size (D) and wall thickness (e) of cells, polydispersity index (PDI) and sphericity ($\bar{I}_s$) of the matrix and foamed starch based composites containing 10 wt% fibres.

Composites	$\bar{D}_n$(μm)	$\bar{D}_w$(μm)	PDI	e (μm)	$\bar{I}_s$
Matrix *M*	875.5	1046.6	0.84	21.52	0.72
M + 10% cellulose fibres	577.6	730.7	0.79	12.54	0.70
M + 10% wheat straw fibres	653.8	812.7	0.80	18.61	0.70
M + 10% hemp fibres	784.1	966.9	0.81	17.39	0.70
M + 10% linter coton fibres	648.9	734.1	0.88	15.12	0.70

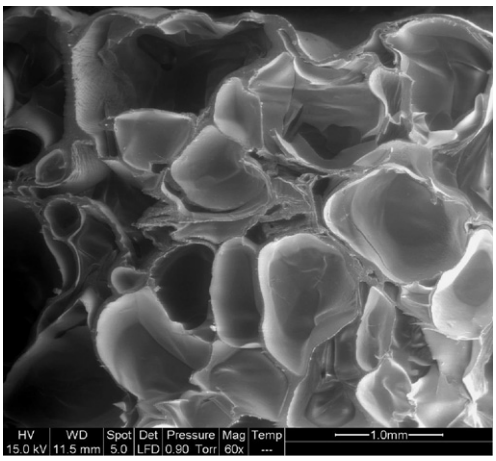

Fig. 3. Microscopy of the surface of cross section of the matrix (magnification 60).

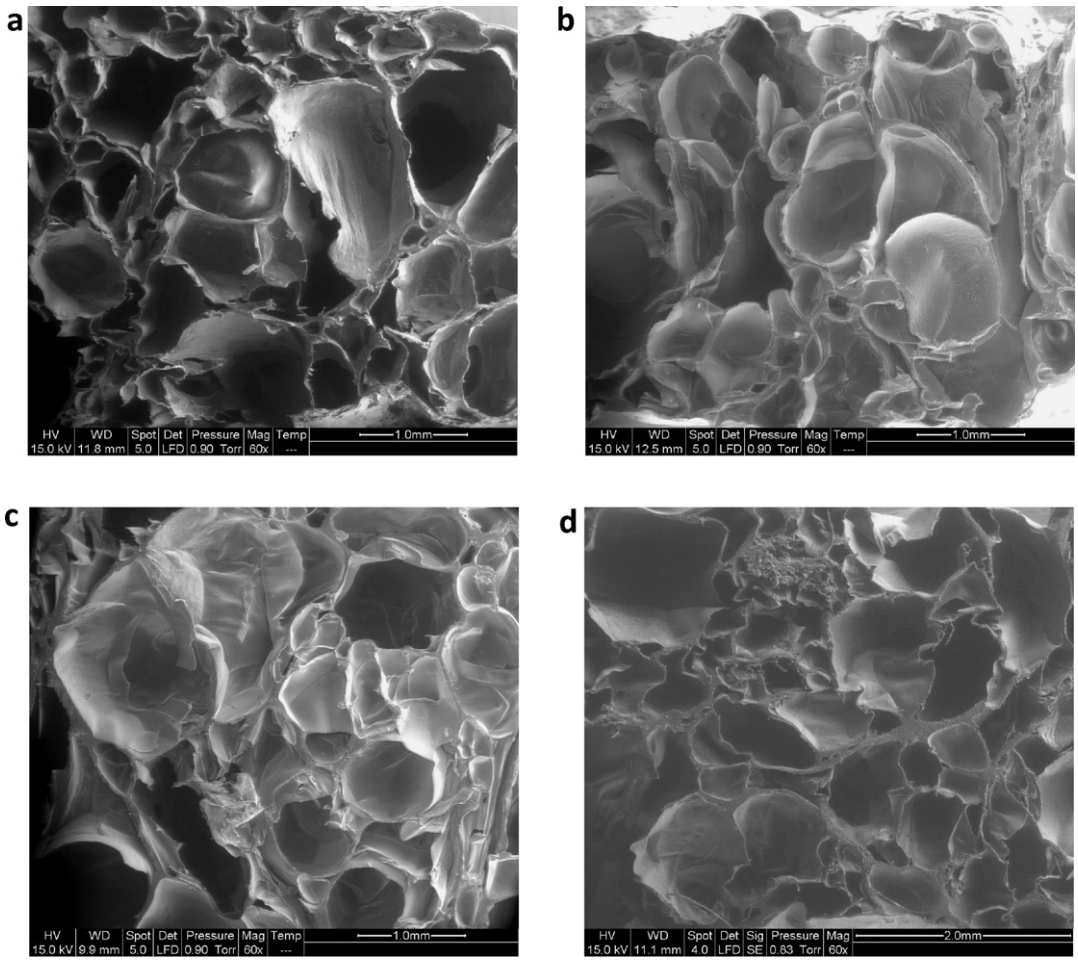

Fig. 4. Microscopy of the surface of cross section of composites containing 10 wt% fibre (a) cellulose, (b) wheat straw, (c) hemp, (d) cotton (magnifications 60).

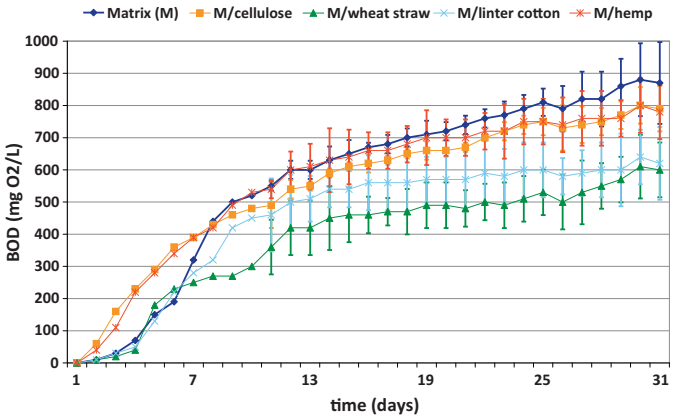

Fig. 5. Evolution of the composite biodegradation (BOD).

4. Conclusion

In this work, biodegradable foams were produced by extrusion of raw materials, such as potato starch and natural fibres and their functional properties (density, expansion, mechanical properties) were characterised. The presence of fibres may affect the mechanical properties of the foam. Fibre nature as well as water absorption rate are the main factors modifying the mechanical strength of the composites. The mechanical properties were affected by both relative humidity (RH) of storage and foam formulation.

The formulation containing starch and 10 wt% hemp fibres exhibited the best properties, including maximal resistance and a notably reduced water absorption.

Results reported in this work showed that these materials may be considered as alternatives to the expanded polystyrene trays. But the use of these materials as loose-fill packaging requires further researches, mainly to improve their expansion and to reduce their water sensibility.

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