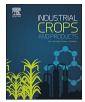
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Effect of formulation and process on the extrudability of starch-based foam cushions



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ABSTRACT

Solid foams produced from petroleum sources have a number of environmental and sustainability issues. Using renewable resources to produce foams has great perspectives but those kinds of foams are technically difficult to process. Starch foams from wheat, pea and potato starches were prepared using reactive extrusion with different concentrations of starch/glycerol/gluten/sodium bicarbonate. The density, the expansion ratio, the elasticity and the dynamic damping properties of such foams were determined. Foams made from wheat starch exhibited the best mechanical properties, especially during ageing. Using the ratios of wheat starch/glycerol/gluten/sodium bicarbonate that gave the best results (100/46/25/1), extrusion parameters were adjusted and higher and more stable expansion (Expansion ratio = 9.1) was obtained when the screw speed was as low (300 rpm) as the highest input rate could afford (21 kg/h).

1. Introduction

Synthetic plastic cushioning foams are usually used as foamed particles. They come from oil, a non-renewable resource and are nonbiodegradable. These foams are obtained from the injection or the in situ creation of gas into the macromolecular matrix of a polymer. They are generally used as protective material for fragile products during transport and storage. The most commonly used cushioning foams in the market are expanded polystyrene foams to protect large and light products; expanded polyethylene foams for less bulky, heavy and fragile products; expanded polypropylene foams to provide additional benefits such as high heat resistance and good insulation properties; and polyurethane foams for fragile and light products. Cushion foams coming from agricultural resources are part of the family of foams produced from bioplastics. The development of extrusion of starchbased foams is the most commonly used substitute the traditional petrochemical foams, which are mainly manufactured by extrusion. The expansion created by injected gas or in situ formed gas during extrusion causes high pressure at the exit of the die. In contact with the ambient air, the pressure suddenly drops and this results in the foaming of the material. The gas bubbles formed in the starch paste are generally molecules of carbon dioxide (CO_2) , but in some cases they are nitrogen molecules (N₂). In order to obtain trays or solid block in starch foams, the most common process is the baking process using compressed molds (Bergel et al., 2017; Kaisangsri et al., 2012; Machado et al., 2017; Kanlaya Pornsuksomboon et al., 2016; Sanhawong et al., 2017; Shey et al., 2006). Starch foams could also be produced by a microwave heating process. It leads to rigid foams for both structural or packaging applications (Lopez-Gil et al., 2015). Soykeabkaew et al. (2015) presented a large overview of starch-based composite foams. They compared different starch foaming process (extrusion, baking/compression, microwave heating, freeze-drying/solvent exchange and supercritical fluid extrusion). They concluded that extrusion process led to large cell size foams with low density so this process was well suitable for packaging transportation. The development of extrusion starch-based foam was studied by other authors. Indeed, Bhatnagar and Hanna (1995) studied the properties of extruded starch-based foams. Chinnaswamy and Hanna (1987), Lee et al. (2009) and Robin et al. (2010) added a blowing agent such as water or sodium bicarbonate in the formulation that decomposes at high temperature and under pressure to form CO₂ gas bubbles thus expanding foams. While other authors, like Alavi et al. (1999, 2003), Cho and Rizvi (2009), Goel and Beckman (1995), Jeong and Toledo (2004) and Muljana et al. (2009) preferred to inject the carbon dioxide or the nitrogen into the form of supercritical fluid at low temperature directly during the extrusion, thus causing the formation of gas bubbles in the material. Della Valle et al. (1997) determined the relationship between the rheological properties of molten starches and their expansion upon extrusion. They found that the expansion was dependent on the extrusion temperature, the water content of the starch and its concentration in amylose.

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Although the method of manufacturing starch-based foams appears simple, the complexity lies in the stability of the process and the properties of the resulting foams, especially for extrusion process.

Concerning the ingredients to add to starch to improve the properties of the foam, several studies have been performed to find the best formulation and the ideal process for developing starch-based foams having mechanical properties comparable to those of petrochemical foams. Among these works, Lin et al. (1995) studied the mechanical properties of extruded starch foam cushioning particles that contains high level of amylose. Pushpadass et al. (2008) examined the effect of temperature, moisture and talc on the physical properties of the extruded foam particles based on starch. However, the cushion foams based on extruded starch still present technological barriers in terms of processability and final properties. Most previous works have studied loose fill chips for protective packaging (Pushpadass et al., 2008; Heartwin et al., 2010). In order to enlarge the range of starch foams in packaging, this paper is devoted to the development of starch-based foams as strips rather than loose-fill particles (chips) to overcome the lack of control during processing.

The main objective of this paper is to understand and optimize the process of manufacturing starch foam plates by extrusion to ensure stability and reproducibility of the process. In the first part, we compare the properties of starch foams with different additives (plasticizer, gluten, yeast) and from different sources of starch (wheat, pea and potato) and the impact of the formulation on the expansion ratio, rheological properties. In the second part, extrusion parameters are studied in order to obtain a stable starch foam with best expansion ratio and dynamic damping properties.

2. Materials and methods

Wheat starch (25–28% amylose content) and wheat gluten were obtained from *Chamtor*, France. Pea starch (35% amylose content) was obtained from *Cosucra*, Belgium and potato starch (25% amylose content) was from *Haussimont*, France. Glycerol, used as a plasticizer, was provided from by *A.R.D* society, France. Sodium bicarbonate, used as a blowing agent, was coming *Ecodis*, France. Petrochemical foams were used to compare their behavior with ours: one polyethylene foam (Etafoam 220, 35 kg/m³, from Dow Chemical) and a polyether foam and an expanded polystyrene cushion foam (from our laboratory).

The foams were compounded using a Clextral BC21 co-rotating twin-screw extruder machine, with an optimized screw configuration and temperature profile and with a ribbon die (20 mm width, 2 mm thickness). Plasticizer was first introduced by a liquid pump, just before the kneading zone of the barrel. After extrusion, samples were cut into 15 cm strips, identified and conditioned for one week at 23 ± 2 °C and $50 \pm 10\%$ relative humidity according to NF EN ISO 7214 prior characterization.

Each formulation is denoted by a nomenclature which refers to its constituents by abbreviations followed by their percentage rate relative to the starch. These classifications are obtained as follows:

The first letter of the nomenclature defines the nature of the starch used, for example the letter "B" refers to wheat starch, "P" to pea starch and "PDT" to potato starch. The second letter defines the plasticizer used. In our case a big "G" refers to glycerol. Then the third letter uses a small "g" to refer to the wheat gluten and the fourth letter defines the blowing agent (in our case sodium bicarbonate) and is designated by a large "L". For example, the formulation |BG46g25L1| contains wheat starch as the main resin, with 46 parts of glycerol as a plasticizer, 25 parts wheat gluten and 1 sodium bicarbonate, per 100 parts of starch (pcs).

Table A (in Appendix A) shows the different formulations used in this paper.

2.1. Foam density

In accordance with the NF ISO 845 standard, foam density was calculated in kg/m^3 by dividing the weight of the sample in kg into its volume in m^3 . Five samples from each formulation were tailored into a rectangular shape and were weighed. The length, width and thickness of each sample were measured three times with a graduated Vernier caliper.

2.2. Expansion ratio (ER)

The expansion ratio of the foam was calculated by dividing the cross-sectional area of the foam by the cross-sectional area of the die in mm^2 . The result was the mean of five samples of each formulation.

2.3. Foam microstructure

The structural properties were determined by using a dnt^{*} Digital Micro Capture Pro microscope. 5 mm thick slices were cut using a double blade cutter. The surface of each sample was colored using blue ink in order to show the contrast of the cellular structure. The cell size, which is the cross-sectional area of each cell in mm², and the cell wall thickness, which is the cell wall thickness of the cells in mm, were calculated using ImageJ 1.43 software. The cell density of each specimen in cell/mm² was then determined by dividing the numbers of cells of the cross-sectional area of the foam by the area of this foam section. The average values and standard deviation of all the measurements were calculated and sorted in the results and discussion section.

2.4. Differential thermal analysis (DSC)

The melting properties of the starch based materials were determined using differential scanning calorimetry. The raw materials and the mixtures were analyzed by a measuring apparatus DSC "NETZSCH Thermal Analysis", after 48 h conditioning at a temperature of 23 °C and 65% relative humidity. Samples of 10–20 mg are placed and sealed in aluminum seals to prevent loss of water and are heated under pure nitrogen and controlled by a thermocouple at temperatures ranging from 25 to 200 °C with a heating rate of 2 °C/min.

2.5. Moisture content (MC)

The moisture content of the different foams was determined by using an "OHAUS moisture analyzer". This thermogravimetric analyzer calculates the weight change between the value of the foam dry sample and the wet weight of that sample ($20 \times 20 \times 15 \text{ mm}^3$) at 80 °C.

2.6. Viscoelastic properties

The viscoelastic properties of our foams were examined on the resulting foams using a stress-controlled rheometer (AR2000Ex, TA Instruments), with torsion geometry. The samples were cut into a 12 mm wide, 50 mm long rectangular solid. The thickness varied with the sample but was around 10 mm. The tests were performed at 25 $^{\circ}$ C and at a strain of 0.005 Pa (such a strain was chosen within the linear viscoelasticity range).

2.7. Creep tests

The compressive creep tests were performed on the resulted foams using a dynamic mechanical analysis instrument (DMA) designed by TA Instruments, with a thermal relative humidity accessory. This allowed mechanical properties of the foam sample to be determined under standard conditioning atmosphere of 23 ± 1 °C and 50 ± 2 % relative humidity. The creep/recovery test was performed using parallel

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