Biodegradable foams based on extracted fractions from sorghum by-products

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Abstract. Agricultural by-products comprise abundant, renewable sources of value-added compounds for the food and packaging industries. The production of biodegradable foams can be focused on replacement of polystyrene-foam with biodegradable materials, which are regarded as environmentally friendly. In this study, biodegradable food trays were developed via thermopressing, using modified and unmodified corn starches with additional extracted fractions (celluloses, hemicelluloses, and lignin) from sorghum stalk and bran. The effect of several formulations of extracted fractions from sorghum by-products on bio-foam properties was studied. All trays presented thicknesses between 3 and 4.17 mm, density between 0.16 and 0.64 g cm⁻³, and moisture content between 5 and 12% (dry basis). The foam trays made from chemically modified starch had lower densities, higher tensile strength and elongation at break than unmodified starch. The increment of cellulose fraction from 2% to 5% and hemicellulose fraction from 1% to 10% had a significant effect on the trays’ physical and mechanical properties. All trays exhibited no color and structural changes during storage at 25 °C and 4 °C. The formulation presenting the best properties contained 2% of cellulose and 1% of hemicellulose, had a maximal resistance of 0.77 MPa and 9.46 mJ of total work, attributes which corresponded to a compact, homogenous, and dense microstructure.

Keywords: biodegradable foam, thermopressing, corn starch, cellulose, hemicellulose, lignin.

1. Introduction

The valorisation of agricultural by-products comprises an ongoing field of research, aiming to identify novel pathways of their exploitation as abundant renewable and cheap sources. One such pathway represents their utilisation as food packaging material, targeting the replacement of petroleum-based materials with environmentally friendly and biodegradable materials. Sorghum (Sorghum bicolor L. Moench) by-products potentially represent suitable materials for the development of biofoams as they have high cellulose content, which is needed for biofoams to exert strong mechanical properties and water resistance. Previous research has investigated pure cellulose and xylan as raw materials by using extrusion or hot mould baking as methods to produce biodegradable packaging [1]. Biodegradable foams can be used in combination with biodegradable fillers, such as lignin, for the development of biocomposites, which have applications in a variety of industries, including food, agricultural, automotive and chemical.

In biodegradable foam production, starch is the main material due to its swelling properties. The types of starch may influence the characteristics of biofoam due to the ratio of amylose to amylopectin. The foam developing process includes the gelatinisation of starch, evaporation of water and finally
molding [2]–[4]. However, biodegradable foams are susceptible to water absorption and relative humidity which affect the mechanical properties [2], [3], [5], [6]. Therefore, several studies have focused on the improvement of water resistance properties of foams by adding fibre [2], [3], [5], oil [2], protein [7], chemical compounds such as PVA [4], chitosan [2], kaolin, and beeswax [5].

In this study, the focus was on the development of packaging materials, in particular food trays. Starch-based foams were developed in order to replace the expanded polystyrene foam packaging currently in use. Key aspects that were investigated include the compatibility between sorghum extracted cellulose and xylan used for biocomposites and the production method on three dimensional structures of biofoams, as well as their physical properties. A filler such lignin which has hydrophobic compounds could also potentially added into formulation to increase the water resistant and shelf life of the biofoam.

2. Materials and Methods

2.1. Materials

Commercial cassava and corn starches were used in this study, and had a moisture content of 12% (dry basis). The amylose content of cassava and corn starch was 28% (w/w) and 17% (w/w), respectively. Cellulose, xylan and lignin fractions of sorghum by-products were also used in this study. Commercial polyvinyl alcohol and magnesium stearate were used as additives. 2-Octenyl succinic anhydride (OSA) was obtained from Sigma Aldrich.

2.2. Methods

2.2.1. Tray processing by thermopressing

The trays were prepared with two types of starch (cassava and corn starch), testing several formulations. Table 1 is list of formulation using corn and cross-link corn starch, whereas formulation using cassava starch is depicted in Table 2. The additives (PVA, magnesium stearate, xylan, and lignin) were mixed with starch (60-80% w/w). PVA (Polyvinyl alcohol) and magnesium stearate (MgS) were added to the batter.

**Table 1.** Food tray formulation from corn-starch, cross-linked corn starch, cellulose, polyvinyl alcohol (PVA), hemicellulose of bran, lignin and water (w/v of solid proportion)

| Sample | Batter solid mass (60 g)** | S (%) | PVA | MgS | C | H | L |
|--------|-----------------------------|-------|-----|-----|---|---|---|
| A      | 80%                         | 10    | 5   | 5   | - | - | - |
| B      | 80%                         | 10    | 5   | 5*  | - | - | - |
| C      | 50%                         | 15    | 5   | 2   | - | - | - |
| D      | CL 80%                      | 10    | 5   | 2   | 1 | - | - |
| D-1    | CL 80%                      | 10    | 5   | 3   | 1 | - | - |
| D-2    | CL 80%                      | 10    | 5   | 4   | 1 | - | - |
| D-3    | CL 80%                      | 10    | 5   | 2   | 5 | - | - |
| D-4    | CL 80%                      | 10    | 5   | 2   | 10| - | - |
| E      | 80%                         | 10    | 5   | 2   | 1 | - | - |
| F      | 60%                         | 20    | 5   | 2   | 1 | - | - |
| F-1    | CL 60%                      | 20    | 5   | 2   | 1 | - | - |
| G      | 60%                         | 20    | 5   | 2   | 1 | 10| - |

*cellulose was soaked in water for 30 min to increase the moisture content. S: starch, PVA: polyvinyl alcohol, MgS: magnesium stearate, C: cellulose, H: hemicellulose, and L: lignin. **40-50 mL water was added to the batter and baking time was 240 s.
The method in this study was based on previous study [8]. Cellulose (2, 3, 4, 5% w/w) and water (45-50 g) were also added into the dough and mixed using a mixer. 70-80 g dough of each formulation were layered into a thermopressing plate. A Teflon lid was placed over the plate and the system was set at 170 °C for 3-4 min. Finally, trays were removed from the equipment and were cooled in room temperature and cut into a square mold. The trays were stored at room temperature and RH 50% before further characterisation analysis.

Table 2. Food tray formulation from cassava-starch, cellulose, polyvinyl alcohol (PVA), hemicellulose of bran, lignin and water (w/v of solid proportion)

| Sample | Batter solid mass (60 g)* | S  | PVA | MgS | C | H | L | OSA |
|--------|---------------------------|----|-----|-----|---|---|---|-----|
| H      | 83%                       | 10 | 5   | 2  |   |   |   |     |
| I      | 73%                       | -  | 5   | 4  | 8 | 10|   |     |
| J      | 71%                       | -  | 5   | 4  | 10| 10| 5% (w/v of water) |     |

S: starch, PVA: polyvinyl alcohol, MgS: magnesium stearate, C: cellulose, H: hemicellulose, and L: lignin. *40-50 mL water was added to the batter and baking time was 240 s.

Density was calculated as the relationship between weight and volume [9]. Reported values are the mean of five independent measurements for each formulation.

2.2.2. Mechanical properties of food trays

Before testing, the thickness was measured by a digital micrometer. The measurement was conducted in triplicate. A texture analyser model Texturepro CT V1.2 Build 9 (Brookfield Engineers Lab. Inc) using probe TA 18 was used to measure tensile strength. The condition set up was trigger load 50 g, test speed 1 mm/s, load cell 4.5 g and return speed 1 mm/s. It was used to determine compression of samples according to ASTM Standard Method D882-88. The trays were compressed (each measuring 3 cm x 3 cm) at load cells 4500 g. Each tray formulation was tested 3 times, and the reported values are the averages of each of the 3 samples tested. Tensile strength was calculated by dividing the load at break by the original width. The results were expressed in megapascals (MPa).

tensile strength = (load at break)/(original width) (original thickness)

3. Result and Discussion

3.1. Pasting behavior

Commercial cassava, corn, and modified corn starches were used in this study as the main component of the developed food trays. These two starches have their own pasting behaviour and their effect on the biofoam properties was investigated. The starches’ pasting behavior is presented in Table 3. The most important qualities determining the starch functionality are amylose to amylopectin (AM/AP) ratios, viscosity development characteristics, and some other minor constituents of starch content such as protein, phosphorus, and lipid.

Table 3. Parameters of pasting characteristics of native corn, cross-linked corn, and native cassava starches.

| Samples      | AM/AP  | PT (°C)     | PV (cP)     | HPV (cP)    | BD (cP)     | FV (cP)    | SB (cP)    |
|--------------|--------|-------------|-------------|-------------|-------------|------------|------------|
| Native corn  | 0.66±0.01a | 76.7±0.01a | 3838±76a   | 2684±62a   | 1154±14a   | 2776±71a  | 417±14a   |
| CL corn      | 0.65±0.02b | 75.8±0.21b | 2584±22b   | 1034±37b   | 1125±38b   | 1150±16b  | 1855±69b  |
| Native cassava | 0.53±0.03c | 71.1±0.14c | 6509±37c   | 2383±4.59c | 2522±2.12c | 4126±16c  | -3240±69c |

Values followed by the same letters in columns are not statistically different at α= 0.05; n=2. AM/AP-ratio amylose to amylopectin, PT-pasting temperature, PV-peak viscosity, HPV-hot paste viscosity, BD-breakdown (PV-HPV), FV-final viscosity, SB-setback (FV-HPV).
It can be observed that the ratios of AM/AP and all pasting behavior among all starches were significantly different \((p<0.05)\). The modification of corn starch using 2-OSA gave significant effect on all pasting behaviors of the samples. The pasting properties of starch can be affected by amylose, lipid content and branch chain length distribution of amylopectin [10]. The pasting temperature or gelatinisation of native corn \((76.7 \, ^\circ C)\) and CL-corn starches \((75.8 \, ^\circ C)\) in this study were higher than the cassava starch \((71.1 \, ^\circ C)\) (Table 3). This result is in agreement with a previous study conducted by Jane et al. [11], in which the cassava starch had lower pasting temperature than normal and waxy corn starches. This difference could be attributed to differences in starch granule sizes and the starch component. It has been reported that starches with large granules undergo gelatinisation relatively faster than smaller fractions because of the less molecular bonding in which they swell faster as well as break down faster [12]. The structure of starch consists of \(\alpha\)-(1,4)-D-glucose units (amylose) and \(\alpha\)-(1,6)-D-glucose units (amylopectin). Amylose and amylopectin link through hydrogen bonding and arrange themselves radially in layers to form granules. Larger starch granules generates higher viscosity than smaller size of granules which the size of the granule makes it more sensitive to shear [13].

High amylopectin starches have been found to re-associate more hardly than high amylose starches. This is because the branch chains cannot align parallel to each other and limited to move. On the other hand, the higher amylose starches generate lower swelling power since starch swelling is mainly characteristic of amylopectin which in turn decrease the crystallinity [14]. The granules less absorb water, resist to swell and cannot freely leach amylose. It is therefore modification is essentially needed to improve the functionality of starches such as crosslinking starches. In this study, the starch modification improved the property of native corn starch to be less hygroscopic. It suggested that the hydroxyl groups in the starch molecules which are capable of forming hydrogen bonds with surrounding moisture, leading to increase the strength of foams [15].

During cooling in pasting process, the viscosity decreased due to the disintegration of starch granules and polymer realignment. It can be observed that CL-corn starch could maintain the paste viscosity as opposed to other starches. The 2-OSA as a crosslink agent of corn starch generates linear or branched chains which interconnected covalently to starch chains. Therefore, this modified starch was more stable, clearer, and retard retrogradation or recrystallization of starch molecules. The short-term and long-term developments of crystallinity in starch have been attributed to the gelation and retrogradation of the amylose and amylopectin fractions, respectively. This processes take place within the temperature range of \(T_g < T < T_m\) [15], [16]. This pasting behavior is essential to determine the suitability of starch in biodegradable foams and it can affect the tray’s characteristics as well as the interaction of starch with other components in trays’ batter.

### 3.2. Elaboration of biodegradable foams

Small concentrations of each component (starch, cellulose, hemicellulose, PVA, and lignin) (Table 1 and 2) were chosen for the preparation of the tray batters, based on preliminary tests and previous studies. PVA was added to the batter to increase the foam strength and water resistance [17]. Magnesium stearate has a role as a mold release agent and preventing sticking of starch to the mold [4]. The minimal water volume was 45-50 mL which improved the addition of cellulose to the batters and allowed to obtain homogeneous dispersions. To improve the mechanical properties of trays, not more than 5% (w/w) of cellulose was added.

Biodegradable foams with 5% (w/w) cellulose led to agglomeration of cellulose in the biofoams, as seen in Fig. 1A and 1B. This finding suggested that cellulose cannot disperse well in the matrix of batter. However, Fig 1C exhibited agglomeration of cellulose and led into not fully well-shaped trays due to lack of starch. The rest of formulations (60-80% (w/w) of starch and 2-4% (w/w) of cellulose) used in this study successfully formed well-shaped trays lacking of evident cracks (Fig 1).
3.3. Physical properties of the trays

The thickness of the foams ranged from 3.00 to 4.43 mm (Table 4). The addition lignin to the batter was correlated with a higher thickness value (G and J). Furthermore, lignin resulted in on trays darker in color and very susceptible in high relative humidity (RH). In this study, the thickness values of starch biofoams with additional cellulose fibre are comparable to previous study by Polat et al. [18], in which the thickness of corn starch with additional corn husk fibre, kaolin and beeswax were approximately 3.46 to 4.11 mm.

In terms of moisture content, it can be observed that trays based on cassava starch tended to have higher moisture content (16.54-22% dry basis) compared to corn starch and CL-corn starches’ trays (Table 4). The volume of water added to each formulation was related to the fibre content based on previous study by [7], [8]. Water was added to improve the homogenous dispersion of batters. The moisture content values of corn and CL-corn starch biofoams were comparable to what has been done in a previous study, with resulting values ranging approximately from 8.65 to 11.37% [18]. It is likely that different types of starches, fibres and additives give significant effect to the water content of the final trays.

However, the density of trays from cassava starch were lower (0.15-0.19 mm) compared to corn and CL-corn starch trays (Table 4). The addition of 4% of cellulose in cassava starch trays reduced the foam density as reported by [7], in which a reduction of foam density was promoted by the addition of corn fibre. All density values in the present study (approximately 0.05-0.09 g/cm$^3$) were higher than those of expanded polystyrene (EPS) (0.05–0.09 g/cm$^3$) [19]. Low density is preferable in this product [2]. It is due to the practical use in everyday life (light and easy to store).

In terms of color parameters of the generated bio-foams, the additives influence the color of starch foams (Table 4). Cellulose had natural yellowish color and hemicellulose had white color. The corn and cassava starch trays had yellowish color as depicted in Fig 1. It can be seen that CL-corn starch
trays had higher value (L) compared to the other trays. The color difference (∆E) of CL-corn starch showed lower values than other starch trays.

Starch foams are very susceptible to moisture particularly in high relative humidity. Water molecules attack the hydrogen bonds of starch, weakening them and decreasing the functional properties of the foams [20]. WAC was determined to investigate the resistance of biofoams to water. The highest WAC was observed in cassava starch biofoams (H, I, and J), approximately 16-22%. This result is in agreement with previous study by Kaisangri [2], in which cassava starch foams had high water absorption index (WAI) since the dissociation of water molecules to H-bonds with OH groups of glucose units along the polymer chains could have easily occurred. In addition, the water holding capacity of cellulose rich-fraction from sorghum stalk could hold 22.76 to 35.27 times water per dry weight at room temperature [21]. The WAC of trays depends on the porous and surface area of trays. It suggested that the water resided inside the pores and was bound to the cellulose fibrils through hydrogen bonding as those reported by Qiu et al. [21].

Table 4. Physical properties of biodegradable foams based on corn, crosslink corn, and cassava starch

| Formulations | Thickness (mm) | Moisture content (%) | Density (g cm⁻³) |
|--------------|----------------|----------------------|-----------------|
| A            | 3.03±0.11      | 16.54±4.49           | 0.64±0.02       |
| B            | 3.00±0.08      | 9.06±01.25           | 0.60±0.03       |
| C            | 3.01±0.04      | 14.63±1.56           | 0.56±0.01       |
| D            | 3.66±0.30      | 17.91±4.1            | 0.25±0.02       |
| D-1          | 4.08±0.06      | 10.2±0.07            | 0.25±0.02       |
| D-2          | 4.43±0.13      | 10.67±0.01           | 0.29±0.01       |
| D-3          | 4.09±0.01      | 12.21±0.11           | 0.16±0.01       |
| D-4          | 4.14±0.03      | 10.2±0.07            | 0.17±0.00       |
| E            | 3.61±0.13      | 10.44±2.7            | 0.25±0.01       |
| F            | 3.46±0.01      | 8.94±0.02            | 0.26±0.00       |
| F-1          | 3.16±0.01      | 11.41±0.06           | 0.17±0.01       |
| G            | 4.17±0.19      | 11.21±3.79           | 0.31±0.00       |
| H            | 3.66±0.27      | 16.54±0.87           | 0.15±0.00       |
| I            | 3.72±0.09      | 22.54±2.51           | 0.17±0.00       |
| J            | 4.31±0.28      | 19.47±0.89           | 0.19±0.00       |

Taking all these parameters into account, the CL-corn starch (formulation D) was selected to be further studied due to its better WAC, density, moisture content, thickness and color properties compared to other foams. The effect of cellulose and hemicellulose addition to the batter foams was observed. The addition of cellulose (D-1 and D-2) and hemicellulose (D-3 and D-4) to the CL-corn starch formulation (D) increased the thickness of trays, indicating that the filler addition changed the ability of the starch paste to foam. The lowest foam density (0.25 g/cm³) was from CL-corn starch mixed with 1% (w/w) of hemicellulose and 2% (w/w) of cellulose.

3.4. Mechanical properties of food trays

The tensile, elongation at break, and total work were determined using the foam plate specimens, which had the same formulation as the trays. The mechanical properties of biofoams are presented in Table 5. It can be observed that the addition of cellulose to the tray batter formulations decreased the tensile properties of the CL-corn starch biofoams. The increment of cellulose from 2% to 3% into the batter formulations caused tensile properties and elongation at break decreased; meanwhile, the total work increased. However, the increment of cellulose to 3% to 4% into the batter formulations increased the tensile strain and total work; on the other hand, decreased the tensile strength. The results demonstrated that the addition of cellulose did not affect the tensile properties which is in agreement with previous study which stated that the addition of corn fibre to tray formulations did not
significantly affect the tensile and flexural properties of the cross-linked starch foams [5].

The difference in the results is probably due to the size (molecular weight and degree of polymerisation), the concentration, and the type of fibre and synergistic of all components in the batter to the starch. The addition of hemicellulose 1% (D), 5% (D-3) and 10% (D-4) to batter formulations decreased the tensile properties and elongation at break, whereas it increased the total work. The addition of hemicellulose as hydrocolloid suggested to increase the viscosity of batter as those reported by Sullo & Foster (2010), leading to decrease in the density of trays. Therefore, the tensile properties and elongation declined. As previously mentioned, the mechanical properties depend on the synergetic effect and compatibility of all components in the batter (Shogren, Lawton, & Tiefenbacher (2002)).

Previous study reported that trays prepared with different proportions of additives had varying homogeneity and density and that trays having the smallest cell sizes were the most homogenous and exhibited the best mechanical properties (lower water absorption, lower thickness and a higher density). We found that the addition of hemicellulose and PVA increased the cell size in the centre of the foams and decreased tensile and flexural strength; however, these additives also reduced the water absorption of the foam trays. Overall, the formulation D was the best formulation in terms of mechanical properties.

Table 5. Mechanical properties of CL-corn starch biodegradable foams

| Samples | $S_t^a$ (MPa) | $\varepsilon_t^b$ (%) | $L_m^c$ (mm) | $F_m^d$ (mJ) |
|---------|--------------|-----------------|-------------|------------|
| D       | 0.77±0.07    | 14.35±2.62      | 2.94±0.50   | 9.46±2.29  |
| D-1     | 0.61±0.14    | 8.55±0.35       | 1.71±0.09   | 18.55±4.82 |
| D-2     | 0.48±0.09    | 9.1±1.56        | 1.79±0.29   | 20.29±2.11 |
| D-3     | 0.45±0.22    | 7.85±0.21       | 1.57±0.01   | 14.15±2.05 |
| D-4     | 0.43±0.21    | 10.75±1.77      | 2.12±0.35   | 16.98±4.01 |

Values are mean±standard deviation.

$^a$ Tensile strength;

$^b$ Tensile strain at the breaking point;

$^c$ Elongation at break;

$^d$ Total work.

Treatment condition: D (80%CL-corn starch,10% PVA, 5% MgS, 2% extracted cellulose, 1% extracted hemicellulose); D-1 (80%CL-corn starch,10% PVA, 5% MgS, 3% extracted cellulose, 1% extracted hemicellulose); D-2 (80%CL-corn starch,10% PVA, 5% MgS, 4% extracted cellulose, 1% extracted hemicellulose); D-3 (80%CL-corn starch,10% PVA, 5% MgS, 2% extracted cellulose, 5% extracted hemicellulose); D-4 (80%CL-corn starch,10% PVA, 5% MgS, 2% extracted cellulose, 10% extracted hemicellulose).

4. Conclusion

Cellulose and hemicellulose fractions extracted from sorghum by-products are suitable additives in biofoam formulations. However, the lignin fraction was not a compatible component due to its hygroscopic nature and the generation of a darker color of the biofoam. The addition of hemicellulose decreased the tensile properties and elongation at break. It can be inferred that the properties of biofoams are depend on interactions between components. Despite the higher density and lower elasticity of biofoams in this study compared to EPS, the present findings demonstrate that biofoams can be used as an alternative to EPS trays in applications where contact with moist food occurs for shorter periods. The density, elasticity, thermal properties and water absorption properties of cross-linked starch-based trays may be further improved.

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