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Development of expanded matrix elaborated from starch and cassava flour by extrusion*

Desarrollo de una matriz expandida elaborada a partir de almidón y harina de yuca por extrusión

Desenvolvimento de uma matriz expandida feita por farinha e amido de mandioca por extrusão

LUCIO-IDROBO, YERALDIN[1]; ARBOLEDA-MUÑOZ, GERMAN-ANTONIO[2];
DELGADO-MUÑOZ, KAREN-LORENA[3]; VILLADA-CASTILLO, HÉCTOR-SAMUEL[4]

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1 Universidad del Cauca, Research Group Science and Technology of Biomolecules of Agroindustrial Interest, CYTBIA. Agroindustrial Engineer. Popayán, Colombia. https://orcid.org/0000-0003-1283-3125

2 Universidad del Cauca, Research Group Science and Technology of Biomolecules of Agroindustrial Interest, CYTBIA. Master in Organization and Project Management. Popayán, Colombia. http://orcid.org/0000-0003-2900-880X

3 Universidad del Cauca, Research Group Science and Technology of Biomolecules of Agroindustrial Interest, CYTBIA. Specialist in Technological Innovation Management. Student of Doctorate in Engineering in Universidad del Valle. Popayán, Colombia. https://orcid.org/0000-0002-5251-5984

4 Universidad del Cauca, Agroindustry Department, ViceChancellor of Research, Research Group Science and Technology of Biomolecules of Agroindustrial Interest, CYTBIA. PhD in Engineering. Popayán, Colombia. http://orcid.org/0000-0002-5557-3215

Correspondence: garboleda@unicauca.edu.co

ABSTRACT

In the search for alternatives to materials such as expanded polystyrene, interest in the development of packaging derived from starch has grown, which represent challenges from a technological point of view due to the weaknesses they may present. Against this, the inclusion of raw materials such as flours can contribute to increasing its properties. Due to this, the aim of this study was to obtain an expanded material from starch and cassava flour by single screw extrusion. Two experimental designs were made: the first included two factors, cassava flour content and moisture percentage and the second design used two factors, temperature profile and screw speed. According to the main results, the combinations that presented outstanding mechanical properties were mixing with 15% flour and 22% moisture, under process conditions of 100 rpm and 125°C. From this, it was concluded that it was possible to obtain an expanded matrix from a mixture of starch and cassava flour with characteristics of high density, low expansion index and low compressive strength.

KEYWORDS:
Biodegradable; Packaging; Foam; Starch; Flour; Matrix;

RESUMEN

En marco de la búsqueda de alternativas a materiales como el poliestireno expandido, ha crecido el interés por el desarrollo de empaques derivados de almidón, los cuales representan retos desde el punto de vista tecnológico por las debilidades que pueden presentar. Frente a esto, la inclusión de materias primas como harinas puede contribuir a incrementar sus propiedades. Debido a esto, se planteó como objetivo la obtención de un material expandido a partir de almidón y harina de yuca por extrusión de tornillo simple. Para esto, se realizaron dos diseños experimentales: el primero incluyó dos factores, el contenido de harina de yuca y el porcentaje de humedad y el segundo diseño utilizó dos factores, el perfil de temperatura y la velocidad del tornillo. De acuerdo con los principales resultados, las combinaciones que presentaron propiedades mecánicas sobresalientes fueron la mezcla con 15% de harina y 22% de humedad, en condiciones de proceso de 100 rpm y 125°C. A partir de esto, se concluyó que fue posible obtener una matriz expandida a partir de mezcla de almidón y harina de yuca con características de alta densidad, bajo índice de expansión y baja resistencia a la compresión.

PALABRAS CLAVE:
Biodegradable; Empaque; Espuma, Almidón; Harina; matriz.

PALAVRAS-CHAVE:
Biodegradável; Embalagem; Espuma; Amido; Farinha; Matriz.

RESUMO

Na busca de alternativas a materiais como o poliestireno expandido, aumentou o interesse no desenvolvimento de embalagens derivadas de amido, o que representa desafios do ponto de vista tecnológico devido às fragilidades que podem apresentar. Contra isso, a inclusão de matérias-primas como farinhas pode con-

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tribuir para o aumento de suas propriedades. Devido a isso, o objetivo era obter um material expandido a partir de amido e farinha de mandioca por extrusão com um único parafuso. Foram realizados dois delineamentos experimentais: o primeiro, com dois fatores, o teor de farinha de mandioca e a porcentagem de umidade, eo segundo, dois fatores, perfil de temperatura e velocidade do parafuso. De acordo com os principais resultados, as combinações que apresentaram excelentes propriedades mecânicas foram misturadas com 15% de farinha e 22% de umidade, sob condições de processo de 100 rpm e 125°C. A partir disso, concluiu-se que era possível obter uma matriz expandida a partir de uma mistura de amido e farinha de mandioca com características de alta densidade, baixo índice de expansão e baixa resistência à compressão.

INTRODUCTION

In recent years, development of foamed materials has focused on the production of green polymer foams from renewable raw materials [1]. The development of starch-based foams is emerging as an important substitute for traditional foams derived from petroleum [2], in particular application as loss fill, because starch is non-toxic, inexpensive, abundant, biodegradable, versatile and a renewable agricultural resource [3].

However, these are generally fragile with low mechanical properties and high hydrophilicity [4]. Thus, cassava flour consists mainly of starch (76,4%), moisture (11,9%), protein (2,8%), fiber (2,0%), ash (2,0%) and fat (0,6%) and sticking temperature around 74°C [5, 6]. Cassava flour has 72,61% to 87,71% amylopectin and 12,28% to 27,38% amylose [7]. Elements like fraction of lignocellulosic fibers, protein, and lipids, can improve starch-based foams properties [4] and this can help to bioplastic industry because they present advantages compared to the synthetic packaging like use of renewable sources, potential and edibility biodegradability [7].

Others elements can be added to the formulation to improve starch foam properties, like biopolymers and natural fibers [8]. Different studies have used starch by production of foam using other components like polylactic acid (PLA) [9], sugarcane bagasse [10] or polyvinyl alcohol [11]. The aim of this research was to obtain an expanded matrix using starch and cassava flour mixtures using a single-screw extrusion process.

METHOD

Materials

Cassava starch (*Manihot esculenta Crantz*) variety *Verónica* (Almidones de Sucre, Córdoba-Colombia), refined cassava flour (Clayuca S.A.S, Cali-Colombia) and distilled water (Biotechnology Laboratory, Universidad del Cauca, Popayan - Cauca) were used. Glycerol was used as an additive (99,5% purity, Disan S.A, Cali- Colombia).

Extrusion conditions

Mixes of cassava flour, starch, water and glycerol were prepared and mixed. Using a compositional water balance was adjusted to the required moisture and mixed for 10 minutes, until a homogeneous mixture was achieved. Glycerol content (2%) was constant by every sample. The mixtures were stored in an airtight container and left to stand for 48 hours. After they were processed in a ThermoScientific single screw extruder, model Haake Polylab OS, Germany, provided with a 19 mm diameter barrel, a screw with a 5:1 compression ratio and L/D ratio of 25 with a die of 3 mm diameter nozzle.

Experimental design

Two consecutive experimental designs were made.

Experimental design 1. Determination of the mixture composition. An experimental factorial design 3^2 was carried out, with three replicates per treatment, where two factors were evaluated (flour content and moisture in the mixture) with three levels each one. Table 1 shows first experimental design. The treatments were obtained using a temperature profile of 125°C with a screw speed of 100 rpm. The conditions were determined based on preliminary essays.

Experimental design 2. Determination of extrusion conditions in the mixture. In this factorial experimental design 3^2 the factors were evaluated: temperature profile (°C) and screw rotation speed (rpm), both with three levels, with three replicas per treatment. The mixture to be processed was the one chosen in the experimental design for the mixing conditions. Table 2 shows second experimental design.

The tests were carried out in the Laboratory of Rheology and Packaging of the Universidad del Cauca, in Popayán, Cauca, Colombia (1760 m MSL, 19°C average temperature and 77% average humidity).

The obtained materials were conditioned to a constant relative humidity of 50 ± 1% and temperature of 23 ± 2°C for a period of 8 days in a climatic chamber according ASTM D618-13 [12].

Determination of physical properties

Apparent density. It was defined according to the parameters established in ASTM D3748 [13], which sets forth the standard practice for the evaluation of high density rigid cellular plastics, in addition to making reference to test me-

Table 1. Experimental design for the mixture composition.

Treatment	Composition		Response Variables
	Cassava flour (%)	Humidity of the mixture (%)	
TM1	15	18	1
TM2		20	
TM3		22	
TM4	30	18	
TM5		20	
TM6		22	
TM7	45	18	
TM8		20	
TM9		22	

[1]Compressive strength (MPa), Modulus of elasticity (MPa); Apparent density (kg/m^3); Radial Expansion Ratio

Table 2. Experimental design for process conditions.

Treatment	Process conditions		Response variables
	Screw rotation speed (rpm)	Average temperature profile (°C)	
TP1	100	125,00	1
TP2	120		
TP3	140		
TP4	100	127,25	
TP5	120		
TP6	140		
TP7	100	129,50	
TP8	120		
TP9	140		

[1]Compressive strength (MPa), Modulus of elasticity (MPa); Apparent density (kg/m^3); Radial Expansion Ratio

thods such as ASTM 1622 [14]. The diameter of the extruded materials was measured along a 10 cm long section. Averages of ten diameter measurements were used. A circular cross-sectional area of the extruded part, calculated from the average diameter, was assumed. All measurements were made in triplicate. The volume was determined by taking its dimensions (diameter and height) with a caliper foot and then each of the specimens was weighed in an analytical balance to finally calculate the density through the relationship between weight and volume.

Radial expansion index. It was determined using the relationship between the cross-sectional area of the expanded extrudate and the cross-sectional area of the die socket [15]. The diameter of the extruded materials was measured along a 10 cm long section with the aid of a caliper foot. Averages of ten diameter measurements were used. A circular cross-section area of the extruded part, calculated from the average diameter, was assumed, for which three replicates per treatment were made.

Mechanical properties of compression. The guidelines contemplated in ASTM D3748 [13], which sets forth the standard practice for the evaluation of high density rigid cellular plastics, were followed, in addition to making reference to test methods such as ASTM D-695 [16], which establishes the procedure for executing the compression test on rigid plastic materials, where it is described that the test sample must have dimensions such that the ratio of slenderness (Length/Radius) is of an interval of 11 to 16:1. Compressive strength and modulus of elasticity were measured on a universal machine under the following operating conditions: 500 N cell, 1,3 mm/min head speed and 500 points/s data collection speed.

All statistical comparison tests were performed with the SPSS statistical package (SPSS, Inc., Version 21). The results obtained in each experimental design were subjected to a normality test using the Shapiro-Wilk test. Followed by a variance analysis (Anova) to evaluate the influence of the factors studied on the properties of the expanded one, followed by the Levene test and finally the test of multiple comparisons according to the homogeneity of their variances (T3 of Dunnett or Tukey) to establish the differences between the treatments contemplated in the experimental designs All the analyses were performed with a significance level of 0,05.

RESULTS

Experimental design I

All data were found to conform to a normal distribution, based on the results of the Shapiro-Wilk test. According to the variance analysis test (Anova) there was an important effect on all the response variables, with the exception of the flour content on the expansion index. The effect of the inclusion of the flour on the apparent density and of the humidity on the compressive strength was evident.

The interaction between the factors generated a significant effect on the totality of the response variables, indicating that the factors did not produce their effect independently, but on the contrary the effect produced was linked to a joint action of both the flour and water content. When this situation occurred, it became necessary to carry out the analysis by treatments, given that these are the expression of the interaction between the two factors. Subsequently, it was verified by means of the Levene test that the response variables fulfilled the assumption of homogeneity of variances, with the exception of the modulus of elasticity. It was therefore necessary to carry out a multiple comparison test by Tukey and T3 by Dunnett, according to the homogeneity of the variances.

Dunnett's T3 test for the modulus of elasticity yielded five subgroups, resulting in different TM7 and TM3 treatments, with values of 102,44 MPa and 305,41 MPa respectively. As it was shown in the Anova, the flour content was the factor that caused the greatest effect on this response variable, as it presented a higher F statistic. Table 3 shows Anova results.

Table 3. Variance analysis for mixture composition.

Factor	Dependent variable	F	Sig.
Cassava flour	1	12,891	,000
	2	33,016	,000
	3	72,575	,000
	4	3,421	,055
Humidity of the mixture	1	172,457	,000
	2	16,158	,000
	3	102,866	,000
	4	42,267	,000
Interaction	1	26,903	,000
	2	31,010	,000
	3	12,395	,000
	4	13,329	,000

[1]Compressive strength (MPa), [2]Modulus of elasticity (MPa); [3]Apparent density (kg/m^3); [4]Radial Expansion Ratio

Regarding the influence of the moisture content on these results, although in the TM3 treatment it contained higher humidity (22%) than the TM7 treatment (18%), where according to what was previously established, higher moisture content would mean a higher expansion rate, however this was not effectively presented for in this study.

For apparent density and radial expansion ratio Tukey's test yielded four subgroups for each response variable. Regarding apparent density, TM7 treatment had the lowest value (561,67 kg/m^3) and TM3 treatment had the highest value (876,33 kg/m^3). Contrary behavior was observed with the expansion index, where the TM7 treatment reported a higher index (6,89) than the TM3 treatment (4,73), thus proving an inversely proportional relationship between these response variables.

When observing the behavior of the apparent density with the interaction of the percentage of flour and humidity in the mixture, a directly proportional relationship is found between the content of humidity and the apparent density at 15% flour content and inversely proportional between the content of flour and the apparent density at 22% moisture content. That is to say that the lowest values in density were obtained by the treatments with the lowest percentage of humidity (18%) but depending on the percentage of flour. Therefore, the TM7 treatment with the highest flour content (45%) and lowest percentage of moisture (18%) was the one that presented the lowest value of apparent density with 561,67 kg/m^3. As presented in Table 4.

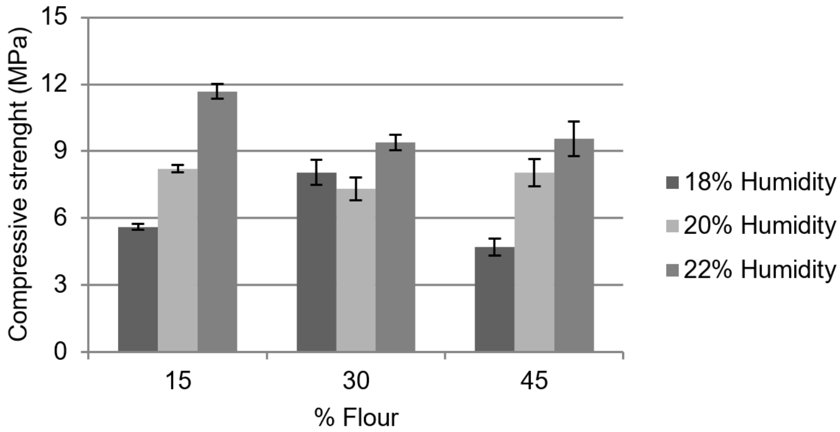
Table 4. Results of aparent density and radial expansion ratio for experimental design 1.

TM	1	Aparent density (kg/m^3)	Radial expansion ratio
TM1	15:18	687,67±0,03^{b}	6,40±0,20^{c,d}
TM2	15:20	825,33±0,02^{c,d}	5,71±0,10^{b,c}
TM3	15:22	876,33±0,01^{d}	4,73±0,15^{a}
TM4	30:18	718,00±0,03^{b}	5,75±0,45^{b,c}
TM5	30:20	725,00±0,02^{b}	6,00±0,27^{b,c}
TM6	30:22	792,67±0,01^{c}	5,50±0,23^{b}
TM7	45:18	561,67±0,03^{a}	6,89±0,29^{d}
TM8	45:20	726,33±0,01^{b}	5,34±0,08^{a,b}
TM9	45:22	729,67±0,02^{b}	5,54±0,31^{b}

[1]Cassava flour:Humidity

In Figure 1, shows the compressive strength values for each of the combinations studied. The different flour concentrations at 22% moisture content showed higher values of compressive strength than at 18 and 20% moisture content. It is thus that this percentage level of humidity presented increases of 29,9% and 66,7% of compressive strength, as opposed to the percentages of 20 and 18% of humidity respectively.

Figure 1. Compressive strength behaviour according to combinations % Flour-% Humidity.



In Tukey's test for compressive strength five subgroups were obtained, presenting significant differences in treatments TM7 and TM3, with 4,70 MPa and 11,68 MPa respectively, the latter being the one that presented significant differences with respect to the other treatments, so it did not share subgroup with any other combination.

When water is mixed at high pressure and temperature it ends up being released as steam just at the nozzle outlet at atmospheric pressure [17]. Water can act as blowing agent and plasticizer, where Meng found that water content above 18% the starch-based materials foamed very well, because they had a high melting strength and rich blowing agent and the pressure of the die decreased with increasing water content of the starch pellets [18].

According to the results, the treatment with a higher modulus of elasticity coincided with the higher density values of the treatments, as well as with the higher compressive strength. In front of this, [17] they affirm that to obtain expanded of low density it is required that these are stable and do not shrink beyond the matrix, that when contracted this way a foam of high density is produced, which can be related with the obtained results, since it was identified at the moment of the processing as the contraction generated at the exit of the die nozzle was evident, which could derive in those high levels of apparent density.

This coincides with what was found for the TM7 treatment under which the lower modulus values were found, as well as the lower apparent density results and the higher expansion rates, possibly associated with the combined effect of cell diameter and cell wall thickness and high correlation between the apparent density and mechanical properties [19], causing that in expanded with a greater expansion the size of the cellular walls and its solid phase is reduced, resulting in a lower rigidity, influencing the behavior of the foam modulus that depends mainly on the cellular structure and density [20].

A higher compressive strength was possibly generated in one of the treatments that contained the maximum percentage of humidity (22%) because the starch granules could be easily disintegrated in the presence of a greater amount of water, thus allowing the formation of a continuous and strong surface [15].

Although it was found that the highest rate of radial expansion was obtained in a treatment with the maximum percentage of flour (45%), it is known that starch is the main component of flours, but it also contains other minor components such as fibers, which plays an important role in physical and mechanical properties of the extruded foams [21], where the cellulosic or lignocellulosic fibers could increase the mechanical properties for their chemical compatibility with starch [22].

This can corresponds to an action of flour as a nucleating agent, which added to a greater generation of internal pressure associated with the presence of fiber [23]. Then, it has contributed to reducing the apparent density values of the material. Although fiber can cause different effects: on the one hand, natural fibre/filler can be a good nucleating agent, but also can cause non-uniform cell size, irregular cell nucleation or a low cell density foam [24].

In addition, as mentioned above, humidity was the most influential factor on apparent density, possibly due to the fact that pressure and temperature fall drastically when the extruded material leaves the extrusion nozzle [25], which could have generated cellular spaces that when occupied by air, influenced in obtaining a higher or lower density with a higher intensity than that reflected by the flour content, where water can act as a plasticizer and blowing agent in extrusion processes, some of the water in TPS becomes steam at high temperature and acts as blowing agent, while the other part of the water bonds with starch chain and acts as plasticizer to enable starch to obtain the melting strength needed for foaming [18], where humidity content of the mixtures have an important impact on the extrusion-cooking process and on the quality of the foamed extrudates [26].

Other studies, like [27] used potato flour by obtaining of loose-fill foams, using water like blowing agent in a single screw extrusion process, where increasing the moisture contents tended to increase rigidity and irregular surfaces of foams, but, the expansion ratio and cell size of foams decreased as the moisture contents of potato flour increased.

With this first experimental design we found the mixture composition to obtain an expanded matrix by extrusion, which presented outstanding physical and mechanical properties. It was chosen that the mechanical properties of compression were the variable of decision, because these characteristics can be a significant factor in the application of the obtained material. Once analyzed the behavior of the different treatments in front of the diverse variables of response, it can be affirmed that the treatment that offered a greater compressive strength was the treatment TM3 with a value of 11,68 MPa (15% Flour and 22% Humidity), which was selected as the appropriate one for the pursuit of the obtaining of the expanded material in the following stage, in which the conditions of processing were determined.

Experimental design II

The parameters of the extrusion process, such as the temperature and speed of the screw, have a direct influence on the density, expansion ratio and other physical properties of the expanded materials [15]. Due to this, it was considered important to review under which conditions of temperature and screw speed the most favorable combination for the development of the expanded matrix of starch and cassava flour could be found.

Initially, the Shapiro-Wilk normality test was carried out, from which it was possible to prove that the data obtained from the valued response variables followed a normal distribution. Subsequently it was established with the Anova variance analysis test that in none of the screw rotation speed levels generated a significant effect on the four evaluated response variables. A similar situation occurred with the temperature profile, with the exception of the modulus of elasticity and apparent density, on which a significant effect was caused in at least one of their levels.

However, the interaction of screw speed with temperature did showed an effect on the response variables as obtained from the Anova analysis showed in Table 5. Due to this, we proceeded to analyze the treatments carrying out multiple comparisons of Tukey. Previously the Levene test was carried out where the homogeneity of variances of the samples was verified.

Table 5. Variance analysis for process conditions.

Factor	Dependent variable	F	Sig.
Screw rotation speed	1	0,887	,429
	2	0,721	,500
	3	2,206	,139
	4	0,418	,665
Temperature profile	1	2,359	,123
	2	38,139	,000
	3	11,917	,001
	4	1,982	,167
Interaction	1	5,310	,005
	2	5,567	,004
	3	3,441	,030
	4	4,614	,010

[1]Compressive strength (MPa), [2]Modulus of elasticity (MPa); [3]Apparent density (kg/m^3); [4]Radial Expansion Ratio

Tukey's multiple comparison test grouped the modulus of elasticity results into two subsets, presenting significant differences between TP3 (138,31 MPa) and TP1 (175,09 MPa) treatments, being the combinations with the lowest and highest modulus of elasticity values respectively. Apart from these, there were no significant differences between the other treatments. Given that the interaction of the factors evaluated generated significant changes in this response variable, it can be stated that temperature was the factor with the greatest impact, according to what was obtained in the analysis of Anova variance and also by the highest statistic F. Experimental results for modulus of elasticity are presented in Table 6.

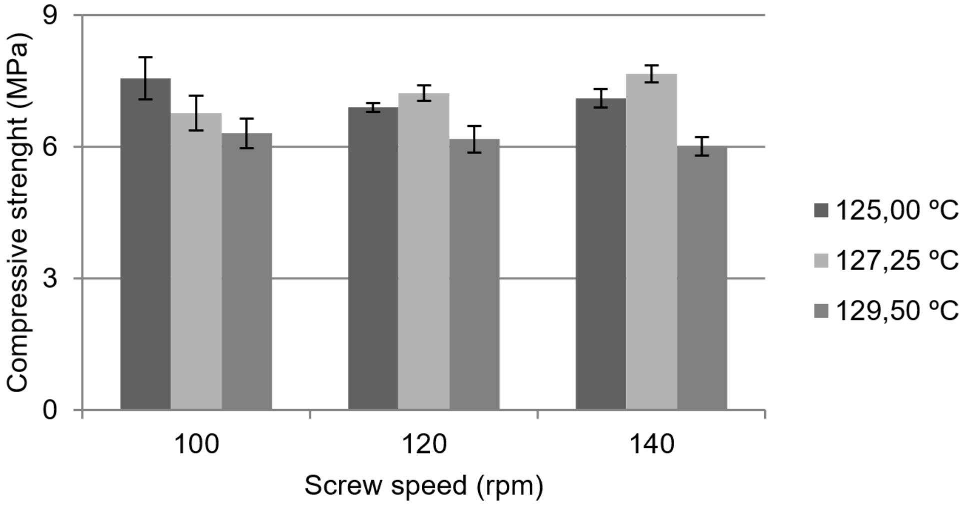
Table 6. Results of modulus of elasticity in experimental design 2.

TP	Screw speed: Profile temperature	Modulus of elasticity (MPa)
TP1	100:125,00	175,09±18,73^{b}
TP2	120:125,00	142,73±8,13^{a,b}
TP3	140:125,00	138,31±5,45^{a}
TP4	100:127,25	151,20±14,72^{a,b}
TP5	120:127,25	165,71±11,38^{a,b}
TP6	140:127,25	168,68±21,00^{a,b}
TP7	100:129,50	148,42±10,47^{a,b}
TP8	120:129,50	158,37±14,12^{a,b}
TP9	140:129,50	145,76±4,82^{a,b}

The results of compressive strength, for the Tukey test, were distributed in five subsets, presenting a significant difference between TP9 and TP2 treatment, TP3, TP5, TP1 and TP6. The lowest value (6,01 MPa) corresponding to the TP9 treatment and the highest value (7,67 MPa) of the TP6 treatment. According to Anova, the interaction speed and temperature profile was the one that most impacted the behavior of this variable. In Figure 2, it can be observed the change in the behavior of the compressive strength against the different combinations of screw speed and temperature.

In terms of apparent density, two subsets were found, in the first subset the lowest value of this response variable corresponding to the TP7 treatment was presented (675 kg/m^3), however this treatment only presented signifi-

Figure 2. Compressive strength behavior according to velocity-temperature profile combinations.



cant differences compared to the TP2, TP5 and TP6 treatments, the latter being the one in which the density was higher (781 kg/m^3). In order to determine some type of trend with temperature profile 2 and a higher speed level, the density of the material became higher, so much so that the TP6 treatment had a higher density than the TP7 treatment. Experimental results for apparent density and radial expansion ratio are presented in Table 7.

In this sense, higher levels of screw speed represented higher values of compressive strength for the intermediate temperature profile (127,25°C), being even higher than those found for processed samples with higher temperature profile. Accordingly, a higher temperature resulted in a lower compression strength value. This can be related to what was stated by [25] when identifying that at high temperatures they also favor coalescence and decrease the density of the number of cells; which allows inducing a reduction in the mechanical properties, since when there are higher densities they imply higher resistances to compression [20].

With respect to the results obtained from the expansion index, it was found that only between TP6 and TP3 treatments there were significant differences, reporting respectively the lowest (5,53) and highest (6,35) value, both processed at a speed of 140 rpm. According to the analysis of Anova variance, the interaction of the factors had greater significance against this response variable, hence, the temperature profile with its individual action could have influenced to a greater extent the action of this interaction.

Table 7. Results of apparent density and radial expansion ratio in experimental design 2.

TP	1	Aparent density (kg/m^3)	Radial expansion ratio
TP1	100:125,00	703,67±0,03^a	5,74±0,36^{a,b}
TP2	120:125,00	727,00±0,02^{a,b}	5,96±0,25^{a,b}
TP3	140:125,00	682,00±0,03^a	6,35±0,41^b
TP4	100:127,25	707,33±0,01^a	5,97±0,18^{a,b}
TP5	120:127,25	731,00±0,01^{a,b}	5,86±0,10^{a,b}
TP6	140:127,25	781,00±0,03^b	5,53±0,33^a
TP7	100:129,50	675,00±0,03^a	6,25±0,13^{a,b}
TP8	120:129,50	678,30±0,02^a	5,93±0,32^{a,b}
TP9	140:129,50	692,67±0,04^a	5,74±0,18^{a,b}

[1]Screw speed (rpm): Profile temperature (°C)

A general analysis of the treatment with respect to the response variables, it was evidenced that the behavior of the material with respect to the compressive strength was noted as the TP6 treatment offered 7,67 MPa, being 0,11 MPa above the TP1 treatment, so much so that according to Tukey's test, these two treatments were part of the same subgroup.

With respect to density, it was the TP6 treatment that yielded a material with higher density, with significant differences compared to that shown by the TP1 treatment, which although it did not represent the combination that offered the lowest density, if it was within the same subgroup of the treatment as if it were, such as the TP7 treatment. As regards the elasticity modulus and expansion index, there were no significant differences between the two, being these ones that showed the highest modulus of elasticity, the opposite case with the expansion index, where they showed lower levels.

It is possible that the lower results in the upper temperature profile are due to the fact that, if temperature is higher excessively, then melt strength of the polymer can be low-inducing cell rupture [28], which could have generated a significant reduction in the values of compressive strength. In addition, as a higher statistical value F was found, it showed that the effects on compressive strength were more due to the action of temperature.

The upper density value was found for the combination of 140 rpm and 127;25°C, i.e. for the upper screw speed level, possibly due to the interaction with the intermediate temperature profile, under which the viscosity may not have been significantly reduced thus preventing further expansion and therefore lower apparent density values [29]. At higher temperature profiles, the apparent density decreased, which coincides with what was reported by [15] who reported that the extrusion temperature could generate changes in cell wall thickness and viscosity, causing a reduction in the solid phase of the polymer, thus decreasing its density.

From the previous, it was determined that the TP1 treatment, that is, the combination of temperature profile 1 (125,00°C) at a screw speed of 100 rpm, corresponded to the relevant extrusion conditions to obtain the expanded one. As it offered high values of compressive strength without representing a material with the highest densities.

In foams made from thermoplastic cassava starch, obtained by single screw extrusion, apparent density values between 473 and 673 kg/m^3 and radial expansion ratios between 4,5 and 7,8 were reported [30]. Other authors reported values from 6,10 to 6,14 for radial expansion and densities from 110 to 430 kg/m^3 for cassava starch foams with cellulose fibers [21]. Also [15] reported that the foam density of starch-based products ranged from 18,7 to 30,5 kg/m^3, 2,5 times denser than foam based on expanded polystyrene. Although commercial products like Ethafoam220 is a closed cell polyethylene foam of medium density 35 kg/m^3 [31].

Mixtures of potato starch with poly(vinyl alcohol) presented apparent densities from 175 to 295 kg/m^3 and modulus of elasticity from 206 to 414 MPa [19]. Other studies report results from 133 to 935 MPa for compressive strength in potato starch and poly(vinyl alcohol) foams using the extrusion-cooking technique [11]. Other results of foams made from potato flour showed expansion ratios from 1,61 to 3,49 with compressive strength from 0.29 to 1,35 MPa [27]. Compared to these studies, the results obtained with foams made from starch and cassava flour showed high density (675-781 kg/m3), similar expansion rates (5,53-6,35), low compressive strength rates (6,01–7,67 MPa) and low modulus of elasticity rates (138-175 MPa).

However, future work around starch-based materials can have different perspectives: decreasing cost and improving performance [32], where application of raw materials like cassava flour can help to increase applications and impact for use of this kind of materials. Further efforts will be required to enhance the functional properties of materials developed with this type of raw material, as mixtures with other polymers such as polylactic acid, poly(vinyl alcohol) and use of twin-screw extrusion.

CONCLUSIONS

It was possible to obtain an expanded matrix using starch and cassava flour as raw materials using the simple screw extrusion process. The interaction of the factors flour content and moisture generated significant effect on the mechanical and physical properties of the material, finding the highest values of compressive strength with a combination of 15% flour content, 22% moisture. The temperature profile and screw speed factors interacted significantly on the mechanical and physical properties of the extruded material. The development of an expanded material obtained by extruding a single screw of starch, cassava flour and other additives resulted in a rigid matrix, with high density and low compressive strength, compared to other investigations of biodegradable foams for loose filling.

REFERENCES

[1] FAN-LONG, JIN; ZHAO, MIAO; PARK, MIRA; SHOO-JIN,PARK . Recent trends of foaming in polymer processing: A Review. Polymers (Basel), v. 11, n. 6, 2019, p. 1-23.
https://doi.org/10.3390/polym11060953

[2] LIU, F.; LU, K.; SHAO, Z.; SHI, K.; LIN, X.; LIU, H.; YU, L. Thermal and rheological properties of starch-based materials. IOP Conference Series: Materials Science and Engineering, v. 479, n. 1, 2019, p. 1-4.
https://doi.org/10.1088/1757-899X/479/1/012006

[3] CHIARATHANAKRIT, CHALIGA; MAYAKUN, JARWAN; PRATHEP, ANCHANA; KAEWTATIP, KAEWTA. Comparison of the effects of calcified green macroalga (Halimeda macroloba Decaisne) and commercial $CaCO_3$ on the properties of composite starch foam trays. International Journal of Biological Macromolecules, v. 121, 2019, p. 71-76.
https://doi.org/10.1016/j.ijbiomac.2018.09.191

[4] MARTINS-MACHADO, CAROLINE; BENELLI, PATRICIA; TESSARO, ISABEL-CRISTINA. Study of interactions between cassava starch and peanut skin on biodegradable foams. International Journal of Biological Macromolecules, v. 147, n. 15, 2020, p. 1343-1353.
https://doi.org/10.1016/j.ijbiomac.2019.10.098

[5] LU, HAIQIN; GUO, LIYUN; ZHANG, LICHAO; XIE, CAIFENG; LI, WEN; GU, BI; LI, KAI. Study on quality characteristics of cassava flour and cassava flour short biscuits. Food Science and Nutrition, v. 8, n. 1, 2019, p. 521-533.
https://doi.org/10.1002/fsn3.1334

[6] ODEY-DORADA, NKA; YOUNG-LEE, WON. Evaluation of the quality characteristics of flour and pasta from fermented cassava roots. International Journal of Food Science and Technology, v. 55, n. 2, 2020, p. 813-822.
https://doi.org/10.1111/ijfs.14364

[7] SURYANTO, H.; RAHMAWAN, A.; SOLICHIN; SAHANA, R.; MUHAJIR, M.; YANUHAR U. Influence of carrageenan on the mechanical strength of starch bioplastic formed by extrusion process. International Conference on Mechanical Engineering Research and Application, v. 494, n. 1, 2019, p. 1-6.
https://doi.org/10.1088/1757-899X/494/1/012075

[8] GEORGES, ABINADER; LACOSTE, CATHERINE; DAMIEN, ERRE. Effect of formulation and process on the extrudability of starch-based foam cushions. Industrial Crops and Products, v. 115, 2018, p. 306-314.
https://doi.org/10.1016/j.indcrop.2018.02.001

[9] HASSAN, MOHAMMAD; LE GUEN, MARIE; TUCKER, NICK; PARKER, KATE. Thermo-mechanical, morphological and water absorption properties of thermoplastic starch/cellulose composite foams reinforced with PLA. Cellulose, v. 26, n. 7, 2019, p. 4463-4478.
https://doi.org/10.1007/s10570-019-02393-1

[10] CRUZ-TIRADO, JAM-PIER; VEJARANO, RICARDO; TAPIA-BLÁCIDO, DELIA; ANGELATS-SILVA, LUIS; SICHE, RAÚL. The addition of sugarcane bagasse and asparagus peel enhances the properties of sweet potato starch foams. Packaging Technology and Science, v. 32, n. 5, 2019, p. 227-237.
https://doi.org/10.1002/pts.2429

[11] COMBRZYŃSKI, MACIEJ; MATWIJCZUK, ARKADIUSZ; WÓJTOWICZ, AGNIESZKA; ONISZCZUK, TOMASZ; KARCZ, DARIUSZ; SZPONAR, JAROSLAW; NIEMCZYNOWICZ, AGNIESZKA; BOBER, DARIUSZ; MITRUS, MARCIN; KUPRYANIUK, KAROL; STASIAK, MATEUSZ; DOBRZAŃSKI, BOHDAN; ONISZCZUK, ANNA. Potato starch utilization in ecological loose-fill packaging materials—sustainability and characterization. Materials, v. 13, 2020, 1390.
https://doi.org/10.3390/ma13061390

[12] AMERICAN SOCIETY FOR TESTING AND MATERIALS (AOAC). ASTM D618-13: Standard Practice for Conditioning Plastics for Testing. Pennsylvania (USA): 2013, 4 p.

[13] AMERICAN SOCIETY FOR TESTING AND MATERIALS (AOAC). ASTM D3748-19: Standard Practice for Evaluating High-Density Rigid Cellular Plastics. Pennsylvania (USA): 2019, p. 2.

[14] AMERICAN SOCIETY FOR TESTING AND MATERIALS (AOAC). ASTM D1622/D1622M-14: Standard Test Method for Apparent Density of Rigid Cellular Plastics, Pennsylvania (USA): 2014, p. 4.

[15] MITRUS, MARCIN; MOSCICKI, LESZEK., Extrusion-cooking of starch protective loose-fill foams. Chemical Engineering Research and Design, v. 92, n. 4, 2014, p. 778–783.
https://doi.org/10.1016/j.cherd.2013.10.027

[16] AMERICAN SOCIETY FOR TESTING AND MATERIALS (AOAC). ASTM D695-15: Standard Test Method for Compressive Properties of Rigid Plastics. Pennsylvania (USA): 2015, p. 8.

[17] YANG, ZHIGUAN; GRAIVER, DANIEL; NARAYAN, RAMANI., Extrusion of humidity-resistant starch foam sheets. Polymer Engineering & Science, v. 53, n. 4, 2013, p. 857-867.
https://doi.org/10.1002/pen.23326

[18] LINGHAN, MENG; HONGSHENG, LIU; LONG, YU; QINGFEI, DUAN; LING, CHEN; FENGSONG, LIU; ZEZHENG, SHAO; KELEI, SHI; XIANYANG, LIN. How water acting as both blowing agent and plasticizer affect on starch based foam. Industrial Crops & Products, v. 134, 2019, p. 43-49.
https://doi.org/10.1016/j.indcrop.2019.03.056

[19] COMBRZYŃSKI, MACIEJ; MOŚCICKI, LESZEC; KWAŚNIEWSKA, ANITA; ONISZCZUK, TOMASZ; WÓJTOWICZ, AGNIESZKA; KRĘCISZ, MAGDALENA; SOŁOWIEJ, BARTOSZ; GŁADYSZEWSKA, BOZENA; MUSZYŃSKI, SIEMOWIT. Effect of PVA and PDE on selected structural characteristics of extrusion-cooked starch foams. Polímeros, v. 28,n. 1, 2018, p. 76-83.
https://doi.org/10.1590/0104-1428.02617

[20] LÓPEZ-GIL, A.; SILVA-BELLUCI, F.; VELASCO, D.; ARDANUY, N.; RODRIGUEZ-PEREZ, M., Cellular structure and mechanical properties of starch-based foamed blocks reinforced with natural fibers and produced by microwave heating. Industrial Crops and Products, v. 66, 2015, p. 194-205.
https://doi.org/10.1016/j.indcrop.2014.12.025

[21] KAISANGSRI, NATTAPON; KOWALSKI, RYAN; KERDCHOECHUEN, ORAPIN; LAOHAKUNJIT, NATTA; GANJYAL, GIRISH. Cellulose fiber enhances the physical characteristics of extruded biodegradable cassava starch foams. Industrial Crops and Products, v. 142, 2019, 111810.
https://doi.org/10.1016/j.indcrop.2019.111810

[22] MOO-TUN, NORA; IÑIGUEZ-COVARRUBIAS, IÑIGUEZ; VALADEZ-GONZALEZ, A. Assessing the effect of PLA, cellulose microfibers and $CaCO_3$ on the properties of starch-based foams using a factorial design. Polymer Testing, v. 86, 2020, 106482.
https://doi.org/10.1016/j.polymertesting.2020.106482

[23] LEUNG, SIU; WONG, ANSON; WANG, LILAC; PARK, CHUL. Mechanism of extensional stress-induced cell formation in polymeric foaming processes with the presence of nucleating agents. The Journal of Supercritical Fluids, v. 63, 2012, p. 187-198.
https://doi.org/10.1016/j.supflu.2011.12.018

[24] ABDUL-AZAM, FARAH-ATIQAT; NISHATA-ROYAN, RAJEDRAN-ROYAN; YUHANA, NI-YULIANA; MOHD-RADZUAN, NABILAH-AFIQAH, AHMAD, SAHRIM; ABU-BAKAR, SULONG. Fabrication of porous recycled HDPE biocomposites foam: effect of rice husk filler contents and surface treatments on the mechanical properties. Polymers (Basel), v. 12, n. 2, 2020, P. 475.
https://doi.org/10.3390/polym12020475

[25] BONIN, M. An investigation into the properties of starch-based foams [Ph.D. Thesis Doctor of Philosophy]. London (United Kingdom): Brunel University, School of Engineering and Design, 2010, 247 p.

[26] COMBRZYŃSKI, M.; MOŚCICKI, L.; MITRUS, M.; KUPRYANIUK, K.; ONISZCZUK, A. Application of extrusion-cooking technique for foamed starch-based materials. BIO Web of Conferences, 10, 2018, p. 1-6.
https://doi.org/10.1051/bioconf/20181001004

[27] BINTIIN, R.; PUNTAWONG, P.; THIRAPHATTARAPHUN, L. Properties of potato flour-based loose-fill foams. Materials Today: Proceedings, v. 17, 2019, p. 2078–2082.
https://doi.org/10.1016/j.matpr.2019.06.256

[28] ALTAN, M. In: Recent research in polymerization. London (United Kingdom): InTechOpen, 2018, p. 117-137.
http://dx.doi.org/10.5772/intechopen.71083

[29] FOLAYASO-FAYOSE, TITILOLA. Expansion characteristics of selected starchy crops during extrusion. The West Indian Journal of Engineering, v. 35, n. 2, 2013, p. 58-64.

[30] DELGADO, KAREN; ALBÁN-BOLAÑOS, PEDRO; MONTILLA-BUITRAGO, CAMILO-ELIAS; CERÓN-MOSQUERA, ALCY-RENE; VILLADA-CASTILLO, HÉCTOR SAMUEL. Evaluación de la densidad aparente e índice de expansión radial en espumas de almidón termoplástico. Agronomía Colombiana, v. 34, (Supl.1), 2016, p. S104-S106.

[31] CORDERO, MATEO; ROUILLARD, VINCENT; MILVERTON, JUSTIN. An evaluation of the mechanical performance of extruded wheat starch loose fill. Packaging Technology and Science, v. 32, n. 10, 2019, p. 511-521.
https://doi.org/10.1002/pts.2465

[32] JIANG, TIANYU; DUAN, QINGFEY; ZHU, JIAN; LIU, HONSGHENG; YU, LONG. Starch-based biodegradable materials: Challenges and opportunities. Advanced Industrial and Engineering Polymer Research, v. 3, n. 1, 2020, p. 8-18.
https://doi.org/10.1016/j.aiepr.2019.11.003