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BIOPLASTIC MADE OF ORANGE PEELS

BIOPLÁSTICO ELABORADO DE CÁSCARAS DE NARANJA

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ABSTRACT:


Plastics have prohibited its use in certain applications because of environmental issues. Therefore, a more eco-friendly option is needed for food packaging and containers. This study uses the waste of orange peel to create a biopolymer that can be used for these purposes. Orange peel of 100 and 250 micrometers, starch, from two sources: corn and potato (7.5%), glycerol (6.0%) and water (71.5%) are the main materials used in these formulations. In addition, two technological procedures were evaluated: the cooling gel method (room temperatures, refrigeration (4 °C) freezing (-18 °C) and the way in which water was eliminated: solar, oven (120 °C) and dehydration (42°C). In order to obtain the best results, 54 experiments were carried out in triplicate and the parameters were evaluated: flexibility, porosity, water absorption, fracture force and biodegradability. Best results were obtained when utilizing orange powder of 250 micrometers, the bioplastic showed a better texture than the one utilizing powder of 100 micrometers. Different ratios of corn and potato starch hardly reveal any differences in the final biopolymer properties, however, from an economical point of view; corn starch is the best option. The best cooling methods of the gel are both refrigeration and room temperature; whereas the latter implies less energetic consumption and therefore it is suggested. The biopolymer made with 100% corn starch and using solar drying showed to be the most flexible one, as well presented less porosity, which is translated into less water absorption; exhibiting a biodegradability of 63% in 21 days. In future studies.

Keywords: bioplastic, biopolymer, orange peels, circular economy, economic prosperity

RESUMEN:

Los plásticos han sido prohibidos en ciertos países para ciertas aplicaciones ya que existe una preocupación ambiental por su uso. Por lo que, este estudio usa los desperdicios de las cáscaras de naranja para crear biopolímeros, utilizando como ingredientes principales: cáscara de naranja de 100 y 250 micrómetros (15%); almidón de maíz y papa (7.5%), glicerol (6.0%) y agua (71.5%). Además, se evaluaron dos procedimientos tecnológicos para su elaboración: forma de enfriar el gel ya sea a temperatura ambiente, refrigeración (4°C) y congelación (-18 °C). Así como la forma de eliminar el agua de la mezcla: al sol, en horno y mediante deshidratación. Para evaluar las mejores condiciones de obtención, se llevaron a cabo 54 experimentos por triplicado, donde se evaluaron: flexibilidad, porosidad, absorción de agua, fuerza de fractura y biodegradabilidad. Los mejores resultados se obtuvieron al utilizar polvo de cáscara de naranja con un tamaño de 250 micrómetros. El almidón de papa y de maíz, fue analizado en distintas proporciones, identificando que no existieron diferencias entre proporciones, sin embargo, se sugiere utilizar el almidón de maíz únicamente por que es un componente con un costo menor. Se estudiaron dos procedimientos tecnológicos: el enfriamiento de la masa de gel la cual presenta mejores resultados tanto a refrigeración como a temperatura ambiente; se sugiere utilizar el último ya que implica menor consumo energético. El biopolímero formado con 100% de almidón de maíz y secado al sol presenta los mejores valores de flexibilidad, así como mínima porosidad y por ende menor absorción de agua; obteniendo una biodegradabilidad de un 63% en 21 días.

Palabras clave: bioplástico, biopolímero, cáscaras de naranja, economía circular, prosperidad económica

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1. INTRODUCTION


There is an increasing interest to develop biopolymers, which can become an alternative to existing materials in particular to conventional plastics. Biopolymers can be produced by natural materials or be synthesized by microorganisms (1). Nowadays, society wants to shift towards more sustainable approaches, and using agrifood residues and transform them into value added products has become a challenge (2).

Conventional plastics offer useful properties and have therefore become an indispensable material in our daily life that is widely used such as in food packaging. However, plastics turn into difficult to manage waste when no longer required (3,4). Additionally, landfilling plastics are difficult to degrade because of their long-lasting properties (5). This resistance causes an alarming accumulation of plastics in the soil, rivers and oceans and even in living organisms.

Inspired by those increasing environmental problems and health concerns, biodegradable food packaging with environmentally friendly properties has received great interest in the food industry. Therefore, more research is focused on new methods for developing safe, non-toxic and biodegradable packaging rich in bio-sources, that stabilizes the carbon dioxide cycle, enhances composting and avoids toxic emissions to the environment (6, 7,8). Biodegradable plastics have similar properties to common plastics, the difference becomes apparent, when they are no longer needed. Naturally occurring microorganisms such as bacteria, fungi and algae can decompose biodegradable plastics without causing harmful substances (9,10). Therefore, biodegradable polymers based on natural organic components represent the smart alternative to synthetic petroleum-based plastic packaging materials because they are, sustainable, biocompatible, non-toxic and therefore safe for the environment and human health (11,12,13).

Research on biopolymers is growing very fast, a trend, which is noticeable in the rising number of articles and patents, related to our research (14). Universidad Panamericana performed a study in December 2020, and found 40 patents related to biopolymers, using citric peel as an ingredient. Four of these patents were detected as closely related to this study: KR100879528B1, CN105766840A, KR101816786B1, CN107446361A (5,15,16,17) (Proper elaboration of Universidad Panamericana, Mexico, using data from Orbit, V.1.9.8; Orbit Intelligence by Questle, December 2020). Countries with the most patents in that area are the United States and China followed by the United Kingdom, Germany, France and India. More than 400 related scientific articles were published in the time between 1979 to 2019, with exponentially rising publications in the last years.

The urgency for smartly designed non-toxic products is as important as the integration of waste management, where waste is recognized and used as a secondary raw material, a strategy known as circular economy (18,19,20,21). Bioplastics that use bio-waste, like orange peels, as an ingredient and renewable energies in their production process are economically interesting because of their reduced production costs (22, 23, 24, 25, 26). The aim of this study is therefore to find a new formulation for a novel biodegradable polymer based on orange peels.

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2. – MATERIAL AND METHODS


The methodology used to elaborate the bioplastic consisted in evaluating the formulation of the biopolymer and the technological parameters associated with the process.

2.1. General Orange Biopolymer Composition

The elaboration of the formulation is based on four main ingredients that are orange peel of 100 and 250 micrometers, starch from two sources: corn and potato (7.5%), glycerol (6.0%) and water (71.5%). The composition of this formulation was based on previous studies performed by our research group (27), where the ingredients were mixed according to the methodology explained generating an emulsion o/w. It is important to note that all ingredients used in the formulation are GRAS (Generally Recognized as Safe) to ensure that the formulation meets the requirements for human consumption as this bioplastic could be used for the production of disposable plates or in food packaging.

2.2. Orange Biopolymer Procedure

Figure 1 shows the methodology for obtaining the biopolymer, including the variables that were studied to obtain the best biopolymer that will be explained in detail.

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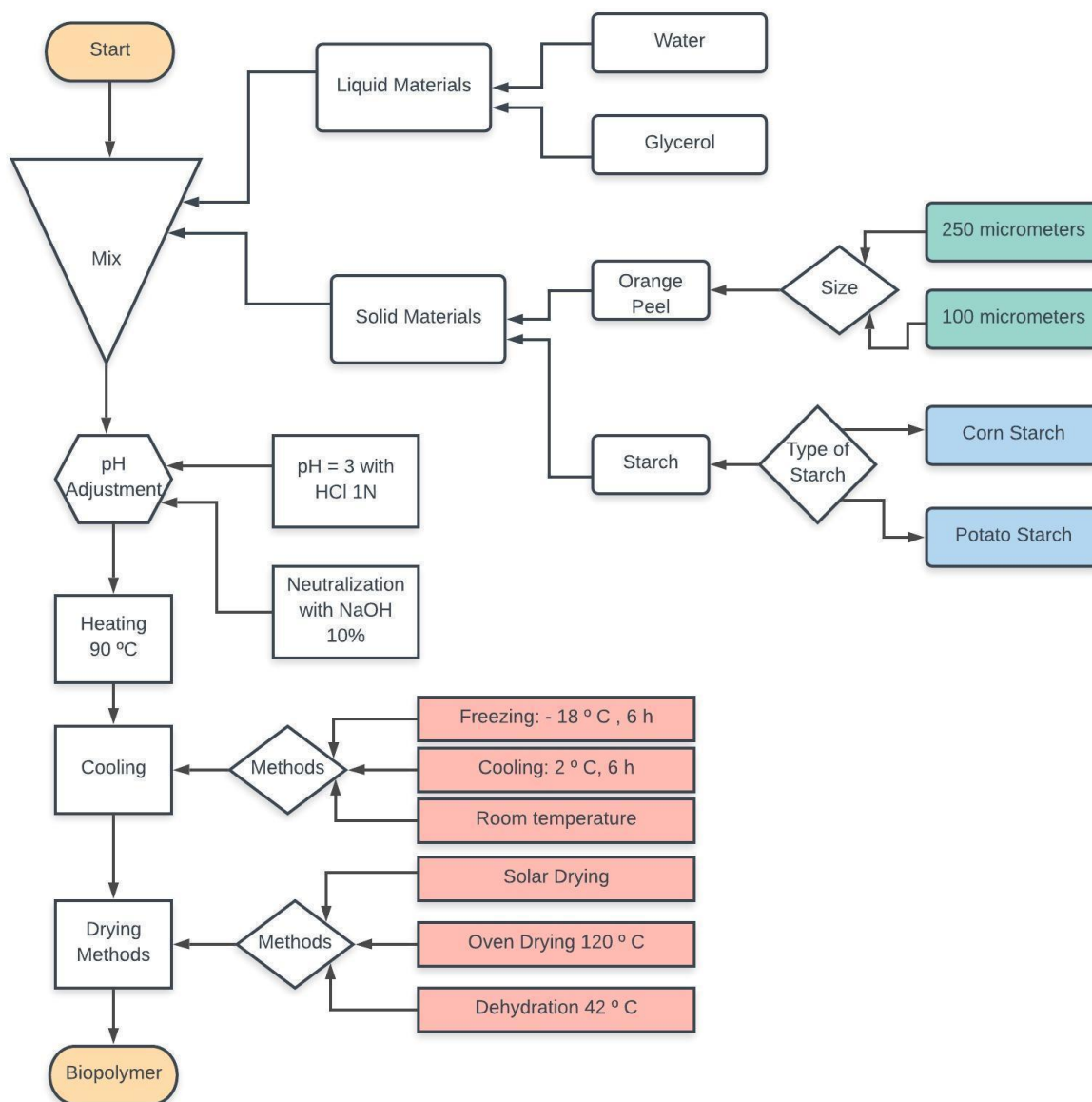



Fig. 1. Methodology followed for obtaining orange biopolymer

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2.2.1. Solid Materials

The influence of the solid ingredients was studied. The orange powder was obtained through four processes (Figure 2). First, the peel of oranges was grinded (Thermomix TM31, Germany), followed by a dehydration process (Ecalibur 3526T EUA) at 60 °C for 24 h. Afterwards, the material was grinded again (Thermomix TM31, Germany). Last, the powder was sieved, so that the fractions of 250 nm y 100 nm were obtained (Figure 2). The first variable studied in this paper is the influence of the particle size in the final properties of the bioplastic. It has been established that there are different ways to make a measurement and to classify particles according to the size. For this study we used ASTM E-11-61 that uses different sieves from the Tylor series in order to separate different extracts according to their size (28).

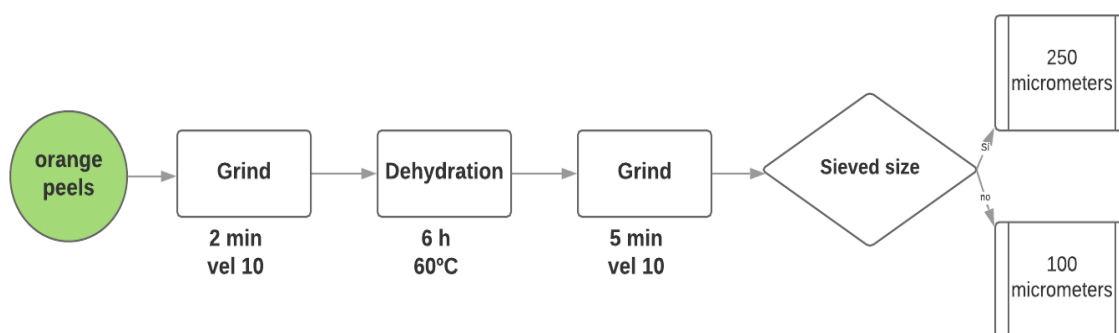


Fig. 2. Steps for obtaining orange peel powder


Two types of starch were evaluated in this study corn starch (CS) and potato starch (PS) (Figure 3). They were added in different ratios and their influence on the final properties of the bioplastic obtained was investigated. Different mixtures were studied: 100 % corn starch; 70% corn starch with 30% potato starch and 50% corn starch with 50% potato starch.

2.2.2. Polymer formation

The polymer formed when pH of the mixture was reduced. First, pH was measured and reduced to a range of 2 - 3 where hydrochloric acid 1N (Karat, Mexico) was used. In the heating process described in 2.2.3, the pH was returned to neutral pH 7.0 with a 10% NaOH solution (J.T.Baker, USA).

2.2.3. Heating and gel formation

The gel formation initiated at 65°C, but the heating process continued until 90 °C in order to eliminate water and to obtain a moldable paste.

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2.2.4. Cooling

The third variable studied (Figure 3) was the influence of the cooling process. We selected three methods: refrigeration (True T49 USA) for 6 h, freezing -18 °C for 6 h in a freezer (True T49F, USA) and cooling at room temperature. We wanted to study the influence of heated starch when being exposed to hydration and gelatinization and later when going on to cooling with the objective of obtaining a high quality gel.


2.2.5. Drying methods

Three drying methods were tested for water elimination: convection oven with a temperature of 120 °C (Rational 61, Germany), dehydration at 42 °C (Ecalibur 3526T, USA) and solar drying.

Using the established methodology, 54 different treatments were tested, as shown in Figure 4.

Treatment	Particle Size		Concentration			Type of cooling method			Drying Method		
	100 micrometers	250 micrometers	Corn starch 100%	Corn starch 70% Potato starch 30%	Corn Starch 50% Potato Starch 50%	Refrigeration	Freezing	Room Temperature	Convection Oven	Dehydration	Solar Drying
1											
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Fig. 3. Different treatments studied

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2.3. EVALUATIONS

2.3.1. Flexibility and Porosity

Flexibility refers to the capacity of the biomaterial to bend easily without any breaking risks. The assessment scale was performed according to the scale presented in Figure 4a, whereas porosity refers to the existence of roundish pores in the biomaterial that may affect the desirable softness appearance. Figure 4b shows the assessment scale (29).

a			b		
Flexibility Measurement	Scale presented	Description	Porous Measurement	Scale presented	Description
Highly flexible	3	The material shows a great flexibility and material toughness	Non-porous	3	Any type of liquid can penetrate the polymer
Moderately flexible	2	The material shows an average flexibility and material toughness	Moderately porous	2	The liquid can slightly penetrate the polymer
Not flexible	1	The material breaks easily and shows no flexibility to bending or torsion	Highly porous	1	When the liquid penetrates the polymer

Fig. 4a. Flexibility Scale (right); Fig. 4b. Porosity scale (left).

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2.3.2. Water absorption

Weight samples of 3 x 1 cm were immersed for 30 s in distilled water (Figure 5a). Samples were let to dry and weighted at constant weight; the process was performed twice, so that the first point lasted 1 minute. This test was performed 4 times, which allowed samples to be immersed for 4 minutes. This test was performed in accordance with the methodology established by (36). The percentage in mol of absorption (Q_t) was calculated according to Equation 1. Changes within time were plotted and the equation that characterized this behavior was established.

$$Q_t(\text{mol}\%) = \left[\left\{ \frac{\text{mass of solvent sorbed by biomaterial}}{\text{Molecular mass of the solvent}} \right\} \frac{\text{initial mass of biomaterial}}{\text{initial mass of biomaterial}} \right] \times 100 \quad (1)$$

2.3.3. Fracture Force

For the fracture force, the sample was cut into pieces of 3 x 1 cm, and were analyzed in TAXT2i Texture Analyzer (Stabler Microsystem Ltd. Haslemere, U.K) at 20 °C at a speed of 5mm/s performing three repetitions. Fracture force was carried out by puncture, using a cylindrical probe as shown in Fig. 5. Results were analyzed using ANOVA Minitab.

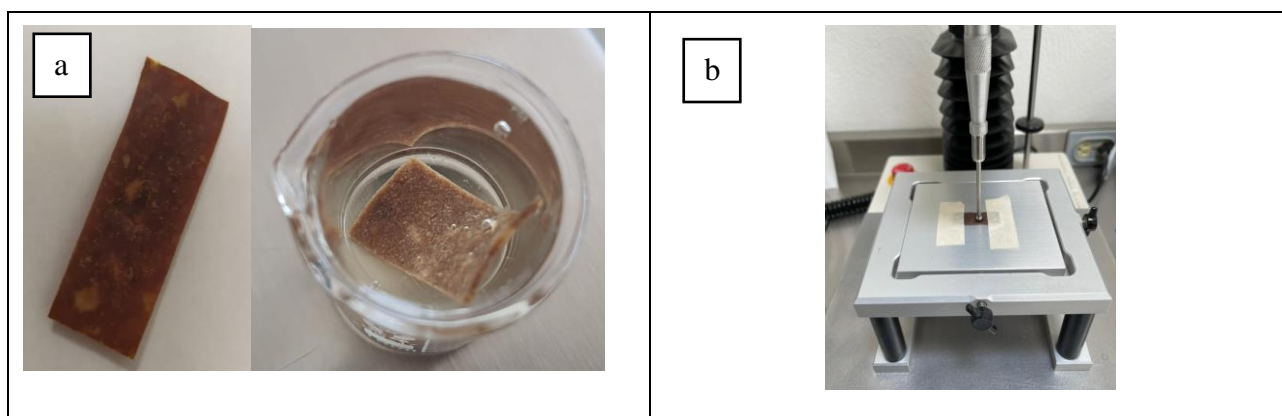



Fig 5a. Water absorption (left) and Fig. 5b. Fracture force of biopolymer (left).

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2.3.4. Biodegradability

The biodegradability test was performed unearthing the sample in soil. The biopolymer was previously weighted, and was placed in a 5 cm depth of dirt for 20 days. The material was collected and weighed again. The percentage of biodegradability was calculated according to Equation 2.

$$\text{Biodegradability}(\%) = \frac{\text{Initial weight}(g)}{\text{Final weight}(g)} \times 100 \quad (2)$$

3. RESULTS


It is important to establish that 54 different treatments were studied, according to Fig. 3. The procedure followed was to test each parameter, taking into consideration that if any variable was not viable the experiments were stopped, as established in (27).

We found that the interaction in the mixture that generates the polymer depends highly on the particle size of the orange peel powder, which was measured with a sieve according to ASTM E-11-61. Accordingly, the particle size was crucial for the resulting biomaterial and showed better results at a particle size of 250 micrometer, since this allowed homogeneous consistency to the final product compared to the one of 100 micrometer. When using the 100 micrometer orange peel powder the pH of the mixture could not be brought back to pH 7, therefore the value based on the flexibility scale is null, whereas when using orange powder of 250 micrometers it is considered to be very flexible.

Regarding the type of starch, three formulations were tested, including (CS) and potato starch (PS). Results show that the characteristics observed in the biopolymers with different concentrations of corn starch and potato starch (50:50, 70:30, and 0:100%, respectively) showed no differences in flexibility and porosity.

The cooling method that showed the best results was room temperature, which could mean that structure is formed slowly in this kind of biopolymers. Probably, when freezing, crystals are formed which makes the structure fragile, giving low results in flexibility and porosity.

Solar drying formed less fracturable materials and with less porosity. The biopolymer formed is smoother with more plasticizer characteristics when corn starch is used in combination with sun drying. When analyzing the fracture force (Fig. 6b) we can observe that there was a significant difference ($\alpha \geq 0.014$) in this parameter when taking into account the drying method, and the biopolymer who offered more resistance was the biopolymer that was sun dried. Moreover, no significant difference was observed when analyzing the interaction of both variables (technology of drying and sources of starches in different ratios).

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Therefore, the best biopolymers results were obtained in the following four treatments: a) 100% of corn starch biopolymer obtained with sun drying; b) 70% of corn starch combined with 30% of potato starch applying sun drying, c) 100% of corn starch biopolymer obtained with dehydration and d) 70% of corn starch combined with 30% of potato starch biopolymer obtained with sun drying (Figure 6). When analyzing the samples we can observe that the biopolymers with the highest porosity/flexibility scale occurs when the biopolymers were sundried as previously discussed (Fig. 6a).

When analyzing the water absorption, we can observe that the sun dried biopolymers absorb the least amount of water, as shown in Fig. 6c. This is an important feature of this type of material that could be used in the food industry, while the dehydrated biopolymers absorbed more water (Fig. 6c)

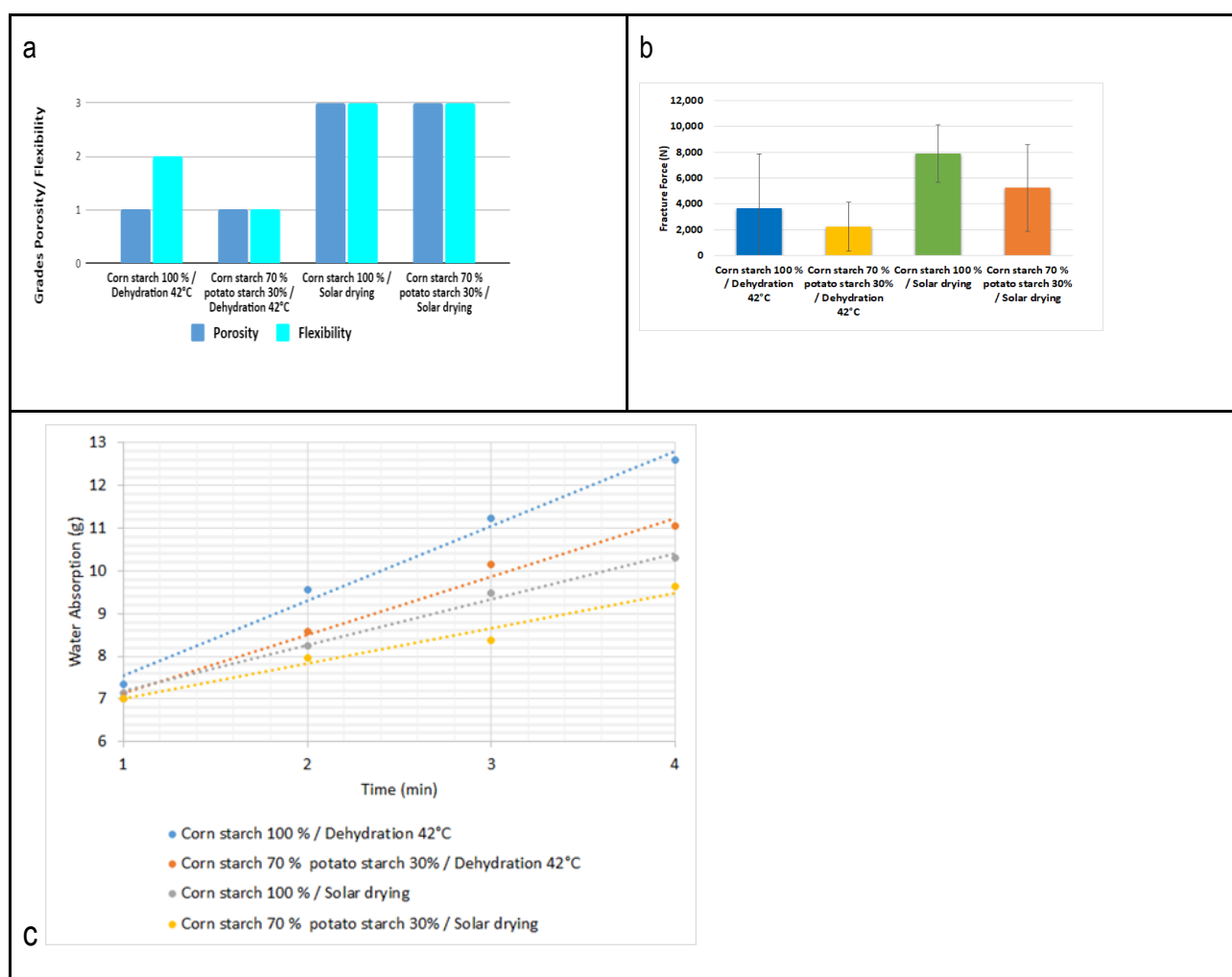


Fig.6. Bioplastic formulations analysis based on porosity and flexibility (a: upper left) and fracture force (b: upper right) and water absorption capacity (c: down)


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Figure 7 shows that after 21 days the biodegradability of the biopolymer that was made of 100% corn starch and subjected to solar drying, was 67.3 ± 0.1607 , which refers to the percentage of weight loss as established in Equation 2.

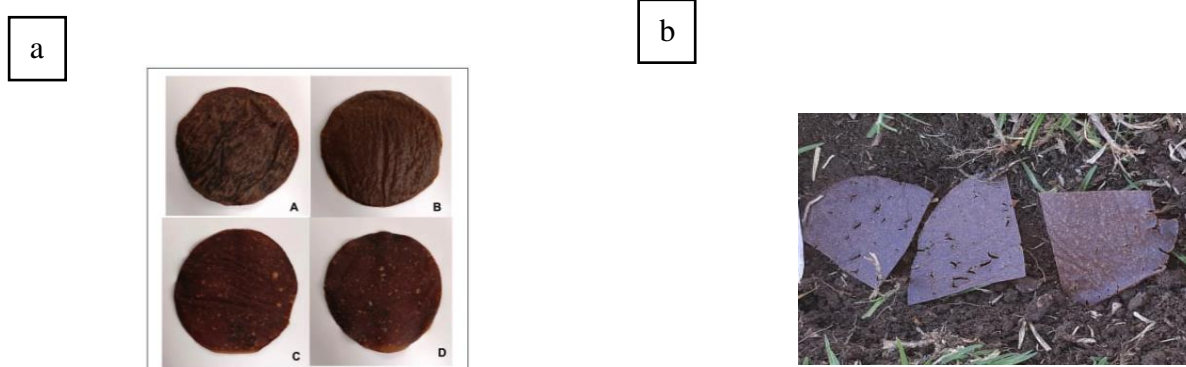



Fig. 7 a. Bioplastic formulations based on composition and drying method (left) **A:** Corn starch 100 % / Dehydration 42°C; **B:** Corn starch 70 %; potato starch 30% / Dehydration 42°C; **C:** Corn starch 100 % / Solar drying; **D:** Corn starch 70 % ; potato starch 30% / Solar drying; Figure 8 b. Biodegradability of 100% of corn starch biopolymer obtained with solar dryin

4.- DISCUSSION

When generating biopolymers it is important to analyze the different materials that can be used, as well as the ratio in which they have to be put up together. The biopolymer formulated in this study consisted of four major materials: water (71.5%), glycerol (6%), orange peel powder (15%) and starch (7.5%). Which means that the correct formulation can generate adequate biopolymer properties, being flexible and having a high fracture force; in consequence they should absorb less water.

When analyzing the formulation of our bioplastic, we can observe that the powder size can have an influence on their mechanical properties of the bioplastic. We have found that the powder size should not be too small (100 micrometers) because the biopolymer is not formed. Taking this into account it was decided to continue working with the powder size of 250 micrometers which is in accordance with studies published by (31) who reported to have developed bioplastics with a size of >300 micrometers.

It has been reported that when using different types of biodegradable materials that are mainly composed of polysaccharides (starch and cellulose) and proteins, biopolymers are formed by different molecule interactions (32). Starch is one of the main ingredients that has been used, it is formed by amylose and amylopectin, the ratio in which these two types of molecules are present will have a direct relation to the physicochemical characteristics. For example, if the quantity of amylose is high, the elongations of the bioplastic will be greater. Furthermore, the breaking force would be higher and this could lead to a material that could serve for a good gas permeation (33). Starch is commonly used in bioplastics because it is considered to have high moisture sensitivity (34). In this study, we wanted to evaluate if the type of starch had an influence on the bioplasticity, however the results show no evidence of difference among them. Nevertheless, we consider that the biopolymer formed with 100% corn starch should be chosen, regarding its lower cost when compared to potato starch which represents an economic advantage.

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Glycerol is the main by-product of the biodiesel industry (35), in particular glycerol is used as a plasticizer that prevents bacterial cells from adhering (32,26). In addition, glycerol, prevents the biopolymer from becoming brittle (37). Moreover, it is important to establish that glycerol promotes the formation of an emulsion, a characteristic that is desired to be obtained prior to the addition of solids (38,39).

In addition, we analyzed the technology used for our biopolymer. Our results reveal that drying had a significant influence on the biopolymer, when it was sun dried it was less porous and therefore presented less water absorption. Biomaterials dried at 120 °C easily broke; that temperature was chosen because we thought that a high temperature could promote a highly resistant biopolymer material, which would be less porous. However, this phenomenon could be explained because when high temperature is applied water evaporation occurs more rapidly affecting the internal structure of the biopolymers, which have effect mainly in starch (40).


Our results show that the process applied to the biopolymer has also an effect on the final properties of the bioplastic, showing that cooling must be performed slowly and drying should be with a solar process in order to obtain better results. However, this opens a gap for future work because probably a low controlled temperature could give better results. Moreover, the biopolymer was biodegradable.

In conclusions, based on the variables studied we were able to develop a biopolymer from orange peels that could be biodegradable, the best formulation parameters are as follow: orange peel with 250 micrometers, mixed with corn starch. The mix should be cooled slowly and dried at 42°C or less temperature in order to obtain a biopolymer with 14.28 ± 0.96 of humidity with good mechanical characteristics and that is less porous.

This study is an important basis for the development of future research where different ingredients could be analyzed for their application in the biopolymer industry. Considering that, most of them can be retrieved from subproducts of the agrifood industry, which could have a great impact in the circular economy (24,26,29,33).

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	<p>BIOPLASTIC MADE OF ORANGE PEELS</p>	<p>NEW MATERIALS AND NANOTECHNOLOGIES</p>
<p>RESEARCH ARTICLE</p>	<p>Julieta Domínguez-Soberanes, Pia Berger, Linda-Carolina Hernández-Lozano, Denise Ortega-Fraustro, María-Fernanda Macías-Ochoa and Crisdalith Cachutt-Alvarado</p>	<p>New materials</p>

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