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# Caracterización térmica y estructural del bagazo de Oca

## Structural and thermal characterization of the esterified oca bagasse

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**Abstract:**

The structural and thermal characterization of the esterified Oca bagasse was carried out, in which, through the scanning electron microscopy images it was shown that the starch granules that are in the bagasse, apparently didn't suffer any structural damage, however, concavities on the granules' surface were observed. The bagasse sample, as well as the homegrown and altered one, revealed a type B diffraction pattern that is distinctive of the tubers. In the altered bagasse, a 1727 $cm^{-1}$ new peak was observed. While the calorimetry results decreased in the endothermic peak. Nevertheless, an additional investigation is required to explore the bagasse's applications and functions, in order to enhance its use in food industry.

***Keywords:***

*Esterification, Oca and Bagase*

**Resumen:**

Se realizó la caracterización estructural y térmica del bagazo de Oca, donde a través de las imágenes de microscopía electrónica de barrido sé muestra que los gránulos de almidón presentes en el bagazo, aparentemente no sufrieron un daño estructural, sin embargo, si se observaron concavidades en la superficie de los gránulos. La muestra de bagazo, tanto nativo como modificado, mostró un patrón de difracción tipo B, que es característico de los tubérculos. En el bagazo modificado, se observó un nuevo pico a 1727 $cm^{-1}$. Mientras los resultados de calorimetría mostraron una disminución del pico endotérmico. Sin embargo, se requiere una investigación adicional para explorar las aplicaciones y la funcionalidad de los bagazos, para potencializar su uso en la industria alimentaria.

***Palabras Clave**:*

*Esterificacion, Oca y Bagazo*

## Introduction

The starch is an important ingredient in a wide range of food products, which can be extracted from different botanical sources such as cereals, legumes and tubers. In Hidalgo tubers like Oca (*Oxalis tuberosa molina*) are cultivated exclusively for self-consumption. On the other hand, it is known that the starch isolation process produces a residue or bagasse that contains diverse components like proteins, lipids, minerals fiber and starch, the last one being the major component with a 30 to 60% starch percentage, (Versino *et al*., 2015; Hernández *et al*., 2014) consequently this bagasse, should be proposed as an alternative flour instead of other tubers or cereals. However, to diversify its use, it is necessary to typify it and modify its main component. Whereby the starches' science and technology investigations have proposed modification methods. The esterification is one of them, due to the starch's chemical substitutions could obstruct sterically the enzyme attack and the altered starch couldn't decompose completely. In comparison with many other substances used for the starch's chemical modification, the citric acid is of low toxicity and can improve the metabolism in our body (Anastassiadis *et al*., 2008). Nevertheless, there is very little work on the influence of the esterified bagasse components that could substitute a conventional flour. For which this investigation has as an objective to determine the Oca

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bagasse structural and thermal properties for its possible use in food industry.

**MATERIALS AND METHODS**

***Material****:* was used as raw material for bagasse extraction, Oca tubers (*Oxalis Tuberosa*) which were bought in Tulancingo de Bravo's commercial swap market in Hidalgo State.

***Bagasse Extraction*****:** To obtain the bagasse, the used method was the same followed by Jiménez-Hernández *et al*., (2007). The tubers were chopped up and grinded in an industrial blender in a 1:2 proportion (tuber/water), the solution was sifted successively in meshes of 100, 200 y 325 U.S. the retained residue in each mesh was washed until the supernatant became transparent. Subsequently, it was dried in a conventional oven at 40 °C for 24 hours, to sieve it afterwards in a 100 U.S. mesh.

***Bagasse Esterification:*** The bagasse esterification was made with citric acid, to form the ester bond, applying the method used by Xia *et al*., (2016).

**Scanning Electron Microscopy:** The homegrown and altered bagasse was observed using a Hitachi S4700 microscope (Hitachi HTA, Japan), following the Xia *et al*., 2015 method.

***Differential scanning calorimetry:*** The gelatinization process of homegrown and altered bagasse was evaluated in a differential scanning calorimetry equipment (DSC 200 TA series 900796.901), according to the method followed by Chen *et al*., 2015.

***Fourier Transformed Infrared Spectroscopy (FT-IR):*** The spectroscopy analysis of the bagasse samples, was held following the Hernández *et al*., 2017 method, made by a Perkin Elmer Series 100 spectroscopy equipment featured with an attenuated total reflectance (ATR-FTIR).

***X-ray diffraction:*** The samples were analyzed in a powder diffractometer equipped with a crystalline graphite monochromator under the following operating conditions: Radiation Cu Kα, Voltage 40 k V, chart speed 10 nm. Trawling speed, 2Ɵ/min, following the method of Moo *et al*., 2015.

***Statistical Analysis:*** The data's statistical analysis was carried out through a variance analysis, using the statistic software SPSS 22 (SPSS, Inc., U.S.A.), and for the measurement comparison Tukey's multiple test was applied ($\alpha < 0.05$).

**DISCUSSION OF RESULTS**

**Scanning Electron Microscopy**

In Fig. 1 the homegrown Oca Bagasse morphological characteristics are displayed, as shown the starch granules have an oval shape (Fig. 1 A). Furthermore, the bagasse modification didn't affect the shape or size of the granules. However, some concavities on the granules' surface were observed (Fig. 1 B). These results are comparable with Xia *et al*., 2015 where collapses and destructiveness of the potato starch granules esterified with citric acid were observed, in which ascribed results could be due to the destruction of the homegrown starch's granular structure by heating and recrystallization during storage.

**Fourier Transformed Infrared Spectroscopy (FT-IR)**

The FT-IR bagasse spectrums and citrate bagasse, are shown in the Fig. 2. An absorption peak of 3440 cm-1 and 2931 cm-1 should be the outcome of -OH y -CH of the glucose unit stretching vibration. The absorptions at 1644 cm-1 can be attributed due to the $H_2O$ bending vibration (Zhang *et al*., 2007). Unlike the homegrown bagasse (Fig. 2 A), the FT-IR Citrate bagasse spectrums (Fig. 2 B) displayed a strong absorption band at 1727 cm-1, due to the vibration caused by the disruption on the carbonyl symmetry of C-O, this new absorption band indicated that the starch present in the bagasse esterified in the process.

**X-Ray Diffraction**

The X-Ray diffraction patterns and crystallinity related to the homegrown and citrate bagasse samples are displayed in Fig. 3. The bagasse starch revealed a type B pattern with main peaks to 2θ = 15 °, 17 °, 18 ° y 23 ° (Zobel *et al*., 1988). As shown in the Fig. 3 C, the crystalline peaks of the citrate bagasse samples were smaller or even disappeared, in comparison with the ones in the homegrown bagasse. But the citric acid esterification didn't drastically alter the crystalline pattern of the bagasse's samples.

The starch's bagasse crystallinity was of 39.8% and for the citrate bagasse it was of 30%. This crystallinity reduction may well be due to the citric acid's insertion in the starch's chain, disrupts the crystalline structure. The substitution of the citric acid groups in the starch chain could form a highly-cross-linked bagasse and therefore limit the starch chain mobility in the bagasse's samples (Mei *et al*., 2015), nonetheless it is required to run starch digestion studies for confirmation.

**Differential scanning calorimetry**

In Fig. 4 the homegrown and citrate bagasse thermograms are displayed. The peak temperature for the homegrown Oca bagasse (60.70 °C). As shown in the Fig. 4 (A and B), the endothermic peak reduced gradually or even disappeared with the esterification, which indicates that the esterification reaction of the citric acid affected the starch's crystallinity present in the bagasse and eventually increased the starch's amorphous region (Mei *et al*., 2015). Regarding the enthalpy values also demonstrated significant statistic differences between the homegrown (4.54 J/g) and the citrate bagasse. The reduction of ΔH was due to the citrate substitution that altered the chain packing and generated more amorphous regions, which can be demonstrated with the content of the resistant starch and the relative crystallinity (Liu *et al*., 2014).

**CONCLUSIONS**

The results indicate that the esterification treatment held with citric acid doesn't affect drastically the starch's granules morphology that are present in the bagasse. On the other hand, changes were noted in the conducted thermograms and diffraction patterns, in addition to a new peak at 1727 cm-1. These results suggest that the citric acid esterification induced structural changes in the bagasse that affected significantly its digestibility and could become a potential method for starch preparation with thermal stabilization, apart from incorporating the use of a bagasse as raw material in the food industry.

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