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A NEW METHOD TO OBTAIN THE CRYSTALLINITY OF CORN PERICARP

Felipe Caballero Briones, * Augusto A. Iribarren Alfonso, [1] Juan L. Peña Chapa, and Álvaro Zapata-Navarro.

CICATA-IPN, Unidad Altamira, Km. 14.5 Carretera Tampico-Puerto Industrial Altamira, AP 165 89600 Altamira Tamaulipas, México. Email: langosta@hotmail.com
[1] Instituto de Materiales y Reactivos, Universidad de La Habana, Vedado 10400 La Habana, Cuba.

Abstract

In this work we propose a new method to obtain the crystallinity of corn pericarp by x-ray diffraction, separating the amorphous components by extraction and using these date to processes the spectra, and compare our result with the traditional method. We found that both method give very different values showing that our method allows a more accurate determination of the structure of the pericarp.

The degree of crystallinity of bio-polymeric materials is related with many of their properties. Determination this very important quantity from x-ray diffractograms, is often inaccurate due to the difficulty to find a proper demarcation line between the amorphous and crystalline contributions.[1] In this work we present a method to calculate the crystallinity of corn pericarp separating some amorphous components by extraction and processing their x-ray difractograms to deconvoluting these into the crystalline and amorphous components signals, with the future scope of study the corn pericarp during *nixtamalization*.

Corn pericarp was sequentially extracted with DI H_2O, NaOH 1%, $NaClO_2$ 2%, and KOH 18% as reported previously.[2] The extracted materials in each step were properly collected and analyzed by X-ray diffraction in a Siemens D5000 diffractometer using a Bragg-Brentano geometry. The normalized diffractograms of the H_2O, NaOH and $NaClO_2$ extracted samples, and commercial microcrystalline cellulose are shown in the Fig. 1. The arrow-marked zones are related with the amorphous hemicelluloses, and diminish after the NaOH extraction, that is expected by hemicelluloses solubilization.[3] The treatment with $NaClO_2$ does not affect significatively. The Figure 2 shows the NaOH solubilized sample diffractogram. From its width and shape it corresponds to an amorphous material, so a lorentzian function was fitted to this diffractogram. This lorentzian was used to subtract a primary amorphous contribution to the unprocessed pericarp

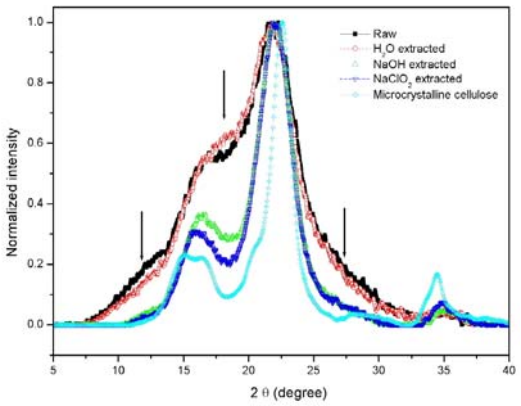

Figure 1. Normalized X-ray diffractograms of Extracted materials and microcrystalline cellulose.

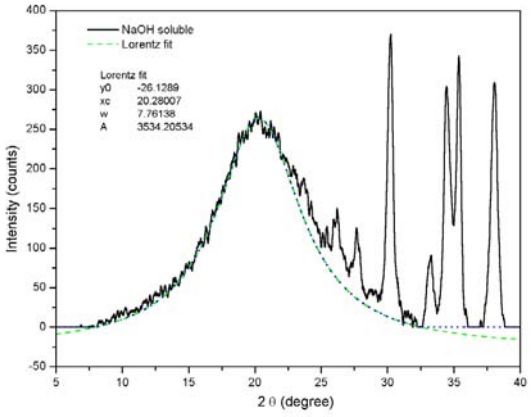

Figure 2. NaOH solubilized amorphous sample diffractogram and a fitted lorentzian.

diffractogram. In this way we got a reduced spectrum showing another amorphous region around 12 degrees which was fitted an other lorentzian, getting in this way the diffraction signal of the two main amorphous components of the unprocessed pericarp. Figure 3 shows the diffractograms of all samples after subtracting amorphous fitted signal, getting in this way the crystalline part of the samples. It can be noted that they all consist of cellulose with slight changes in crystalline orientation.

Having the amorphous and crystalline components we calculate the crystallinities of the obtained samples and the results are shown in Table I, also we calculate the crystallinities of the samples with the traditional method that uses parabolic functions to subtract the amorphous component to compare both methods. As can be seen in the Table I there is a huge difference in the values obtained with each method, but we consider that our method must be more precise because we use real amorphous signal. Also from Table I and Fig. 3 can be noted that changes in the preferential orientation of the material induce a greater error in the crystallinity calculated using the traditional method, because the parabolic adjustment for the amorphous component takes away some crystalline part.

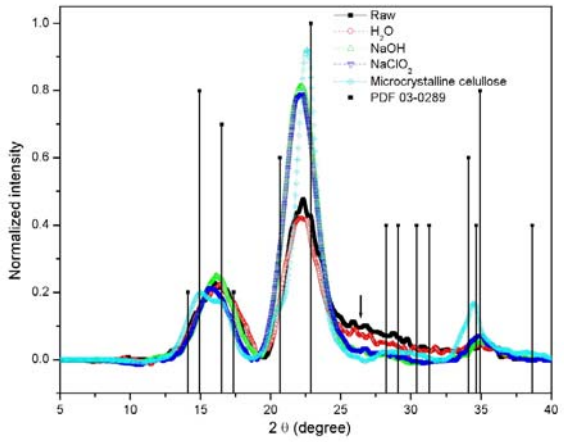

Figure 3. Ddiffractograms of samples after subtracting amorphous fitted signal.

Table I. Crystallinity index of the samples calculated by both methods.

Sample	Crystallinity index[a]		Difference (percent)
	Our Method	Traditional	
Unprocessed	31.6	15.4	105
H_2O extracted	29.7	13.9	186
NaOH extracted	58.3	42.3	38
$NaClO_2$ extracted	59.4	52.4	13
Microcrystalline cellulose	75.5	68.3	11

[a] *X* = Crystalline area/ Total area x 100 (the areas are under the diffractograms)

This new method to calculate the crystallinity of corn pericarp by x-ray diffraction separating the amorphous and crystalline components considers the real amorphous material present in the sample, so it is more accurate than the traditional one.

References

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