Synthesis of starch powder from different organic wastes: A green approach to a valuable material

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Abstract. Plastic pollution has become one of the main causes of irreversible damages to the environment. Despite the well-known adverse effects, the lack of culture in recycling is causing overcrowded landfills with tons of this disposable material. Developing new products is necessary to replace plastics with eco-friendly materials, in this way, biopolymers are a suitable alternative. Biopolymer synthesis requires a natural polymer known as starch, which is a common compound in roots, tubers, fruits, cereals and aquatic plants. In the present work, we study different solid organic wastes such as cassava, potatoes, plantain, corn and two types of algae Elodea Canadensis and Myriophyllum Quitense, in order to discover its feasibility as starch sources. For this purpose, we established an extraction method taking into account the main structure of each organic waste. By means of Fourier-transform infrared spectroscopy (FTIR) and iodine test, we obtained the chemical structures and principal characteristics of each starch. Synthesized powders exhibited characteristics of thermoplastic starches making them available for its potential use in biopolymers.

1. Introduction

Plastics derived from petroleum have become necessary in several activities of human population thanks to its benefits; however, its massive use is causing negative impacts to the earth. This environmental problem has stimulated research to discover and propose new materials that can replace plastics [1]. Accumulation of plastics rises up to 4900 millions of tons (almost 60% of total production) in the atmosphere, according to current trends and dynamics of global waste management, by 2050 almost 9000 millions of tons will be recycled, 12000 millions of tons incinerated and 12000 millions of tons left in landfills or in natural environments [2].

To develop eco-friendly alternatives to replace plastics is necessary to find a cheap, abundant and renewable raw material; in this way, starch currently represents a widely used source for plastic production with biodegradable properties. One natural source of starch is food; its wide consumption is constantly creating high amounts of organic wastes that can potentially cause an environmental impact. These organic wastes has an important content of starch that can be used [3]. Taking into account that in Colombia, roots, fruits and tubers are involved within the food basket, a high consumption of these could generate high amounts of organic husks. Nowadays the lack of recycling culture involve an inadequate management of the organic wastes becoming sources of contamination [4].
Another source of available starch that is causing significant environmental impacts is aquatic plants; the principal cause is due to its endemic growth, which reduces the extension of lakes and lagoons. This non-controlled growth cause decomposition, water quality alterations, poisoning of species and ecological imbalance. Due to fertilizers used in the peripheral of the lacustrine body, algae modified its chemical composition causing a negative impact on other species [5].

One of the alternatives to mitigate these environmental problems are biopolymers, a class of polymers made from renewable raw materials or derived from environment. These natural sources makes them a sustainable option in terms of production. To develop natural polymers or biopolymers is necessary a starch source. This polysaccharide is stored as energy reserve in plants; common starch sources are roots (cassava), tubers (potatoes), aquatic plants (Elodea Canadensis and Myriophyllum Quitense), fruits (plantain) and cereals (corn) [6].

Several works previously reported different extraction methods of starch, which include the wet or traditional method. This method consisted of a solution of lactic acid and sulfur dioxide, which would generate a change in the starch due to nixtamalization; a variation of the wet method is carried out with the use of distilled water in other cases 25% sodium hydroxide [8] or buffer solutions [9] are used as solvents. To accelerate the drying process and make an efficient separation, the centrifuge is usually used [10], another procedure consist of decant for a long time suspending the material in water until the sediment is obtained. Another existing method is through the dry route, in this procedure is necessary to carry out repeated grinding, thus modifying the sugar content of the starch [11]. Currently, there are methods whose purpose is to increase efficiency of extraction process using ultrasound-assisted equipment. The method uses frozen roots, which has shown that 10 minutes of sonication promotes an increase yield of starch. Additionally, it was shown that does not modify the structure of the starch granule [12] making this method viable for the extraction of starch without changing the structure and optimizing the yield.

In the present work, we investigate five different wastes as potential starch sources these include: potato peels, cassava, plantain, corn (leaves and teas) and aquatic plants. To our knowledge, there are reduced studies linked to starch extraction from the proposed wastes; therefore, findings of this work are necessary and opening for further quantitative studies. Synthesis of starch carried out three phases; in the first stage, we designed the extraction process, in the second we performed an iodine test for each starch and last, we studied by means of Fourier-transform infrared spectroscopy (FTIR) each sample in order to discover principal bands of chemical bonds formed. Finally, according to the extraction efficiency we propose which organic wastes are potential starch sources to obtain biopolymers with molecular balanced structures.

2. Materials and methods

2.1. Extraction process from organic wastes

Collected organic wastes used in this study such as corn husks, potato, plantain and cassava peels were from different food companies located in Sogamoso, Colombia. The extraction process of starch was designed based on the wet method [7] and is depicted in Figure 1. The process consisted of seven phases, each one completed as follows:

- Selection: In this phase, we removed damaged and irregular peels from collected organic wastes.
- Washing: Organic wastes were subjected to three washes to remove impurities; first, raw materials were cleaned carefully in a container with deionized water. Then, peels were disinfected using 0.5% of hypochlorite mixed with 1000 mL of distilled water; solution was used per 1000 g of peels.
- Grinding: Raw materials were grinded using a disc mill to obtain a homogenous mass with less polysaccharides.
- Filtration: 1000 g of mass was mixed with 1000 mL of deionized water was added and flocculated for 2 hours to allow separation of starch granules from mass. Then, the mixture was passed through a gauze filter to separate raw fiber from liquid. This process is repeated twice with the dry matter remaining.
- Sedimentation: Filtered liquid was left at room temperature for 12 hours to allow accumulation of starch particles. The sediment was washed using 200 mL of water per gram of starch, until a clean material was obtained and finally decanted.
- Drying: The starch was dried at 40°C for 24 hours in an electric stove.
- Milling and sieving: Dry starch was passed through No. 40 sieve (400 microns).

Figure 1. Scheme of extraction process from wastes: corn husks, potato, plantain and cassava peels.

2.2. Extraction process from algaes

Two types of algaes were used *Elodea Canadensis* and *Myriophyllum Quitense*, both collected from Lake Tota located in Boyacá, Colombia. As is showed in Figure 2 treatment of algaes and extraction process of starch involved eight phases, described as follows:

- Washing: Aquatic plants were carefully cleaned with distilled water and disinfected with 0.5% hypochlorite. Then, algaes were washed using 1000 mL of water per 1000 g of aquatic plants.
- Drying: Cleaned algaes were left on a surface and dried for 1 week at room temperature, avoiding sun exposure.
- Crushing: Using an electric disk mill algaes were reduced to 400 microns.
- Filtration: 50 g of grinded aquatic plants were mixed with 700 mL of distilled water; the mixture was flocculated for 2 hours to allow the release of starch particles. Then, the fiber was filtered from liquid. This process was repeated twice with the remaining dry matter.
- Agitation: The mixture was stirred on a hotplate at 30 °C for 30 minutes and then filtered once.
- Sedimentation: The mixture was left for 24 hours to allow decantation of starch particles.
- Drying: Sediment was dried using a stove at 40 °C for 24 hours.
- Milling and sieving: Dried mass was milled to 400 microns using a mesh No. 40.
The starch obtained of each sample was characterized through Fourier-transform infrared spectroscopy (FTIR), for which the dry sample was mixed with KBr and pressed to obtain a tablet, which was introduced in a Nicolet™ iS50 FTIR spectrometer. For each of the samples, 32 scans were recorded in the region of 400 to 4000 cm⁻¹ [16].

3. Results & Discussion

3.1. Extraction efficiency

The amount of starch obtained in weight is shown in table 1 extraction efficiency obtained from potato was 7.3%. Compared with an industrialized one with a yield of 15.56 to 17.76% [5] of starch from potato residues, in this case, efficiency variation was affected by the extraction method used. The method was carried out based on Gonzales L, Gómez S and Abad works, which were proposed as an effective industrialized development. Based on works of Villalobos M, López P, Rodriguez and Prado M, the extraction process is similar to the proposed in the present work and presented a starch content of approximately 7 to 8 % [13]. The increase of efficiency in extraction of starch from potato peels, an industrialized process is more convenient. In comparison with Bilgin work, starch extraction of plantain peels has a higher starch efficiency reaching 11.3%. Works from Giraldo J, Cuarán J, García L, and Pardo L, showed an efficiency of 12.3% [14]. The differences could be associated with quality of plantain peels or differences in extraction processes. It should be noted that grain size could affected the extraction efficiency of starch, since two solid phases remained once suspended water was removed. Characteristic gray colour was observed as well, which could be characteristic of traces of proteins, lipids and small starch granules. The last due to its low density which could not separate from water remaining in white colour on surface [15]. The efficiency calculation was determined by equation (1), determining the percentage efficiency (E) of each starch extracted from organic wastes and algaes:

\[ E = \frac{\text{weight from starch extracted}}{\text{weight sample processed}} \times 100 \]  

(1)

Table 1. Extraction efficiencies of potential starches as a percentage.

| Type of starch           | % efficiency of obtaining |
|--------------------------|---------------------------|
| Potato                   | 7.3                       |
| Plantain                 | 6.7                       |
| Elodea Canadensis        | 4.15                      |
| Myriophyllum Quitense    | 3.82                      |
3.2. Analysis of Fourier-transform infrared spectroscopy (FTIR)

The spectrum depicted in Figure 3 shows C-C elongation signals at 764 cm\(^{-1}\), the signal 860 cm\(^{-1}\) allows displays a deformation of CH\(_2\); the range of 900 to 1250 cm\(^{-1}\) corresponds to the mostly asymmetric stretching of the C-O bond vibrations, which are associated with the elongations of the polysaccharide particles [17]. Besides, a decrease in the signals attributed to bending vibrations is observed in 1243 cm\(^{-1}\) y 1650 cm\(^{-1}\). Also by elongation (3250-3900 cm\(^{-1}\)) of the O-H groups; this implies that the samples examined are hydrophilic. It is also noted that at 1344 cm\(^{-1}\) there is a deformation of CH\(_2\).

Concerning the peak of the 1462 cm\(^{-1}\) which is why its deformation is evident. The signal at 2920 cm\(^{-1}\) corresponds to the C-H stretches of the glucose unit of the starch molecule; additionally, it was observed that the 1740 cm\(^{-1}\) in each of the samples corresponds to the voltage vibration of C=O. The results obtained showed considerable similarities with the typical band of a thermoplastic starch [18]. The links identified are shown in Table 2.

**Table 2.** Bonds resulting from Fourier-transform infrared spectroscopy.

| Wavenumber (cm\(^{-1}\)) | Type of bond | Description |
|---------------------------|--------------|-------------|
| 3250-3900                 | O-H elongation | Stretches related to intra and intermolecular hydrogen bonds. |
| 2920                      | Elongation CH |             |
| 1740                      | Elongation C=O |             |
| 1650                      | Bending (O-H)(Water) | Hydroxyl groups confer hydrophilic properties. |
| 1462                      | CH deformation or CH\(_2\) vibration |            |
| 1344                      | C-O-H bending, CH\(_2\) vibration | Characteristic bonds of crystalline starch/ amorphous starch region |
| 1243                      | O-H bending |             |
| 1010, 1080, 1150          | C-O and C-C Stretch |            |
| 900                       | Elongation C-O | Anhydroglucose units that are stretched in the starch. |
| 860                       | Deformation CH\(_2\) |            |
| 764                       | C-C elongation |             |

According to the infrared transmission spectroscopy (Figure 4), the combinations of starches that could be compatible for the manufacture of bioplastics are those that have flexions, elongations and deformations (near wave numbers) in similar fragments, that is, they present chemical bonds related; those mixtures were the potato-plantain, and corn-cassava [20].
3.3. Iodine test

To verify starch structures in the organic material and algae, the iodine test was performed [21]. The procedure consist of adding 0.25 mL of iodine to a solution of 10 mL of distilled water with 1 g of material to be tested. The reaction occurs when amylose is present, which has a non-branched helical structure that allows the iodine to penetrate the helix of the starch, thus modifying the properties of light absorption until obtain a blue color, although high concentrations displayed black color. As shown in figure 4, after test was performed black color was displayed showing the presence of starch.
4. Conclusions

Extraction efficiencies obtained showed that algae used presented approximately 50% less starch than organic peels, although the extraction process was carried out by the same method, and algae were subjected to an additional flocculation procedure using temperature.

The behavior found in starches extracted from potato, cassava and corn residues presented IR bands that corresponded to typical structures of starch granules. Also, aquatic plants Elodea Canadensis and Myriophyllum Quitense, possess the characteristics of the starch molecule, for this reason, it is possible, in a next phase to give it a sustainable use as a biopolymer.

According to the FTIR analysis, starches have chemical functions similar to each other, because they present the deformations, flexions and peaks on the same wavenumbers, therefore, the carbohydrates as more similar bases for the elaboration of the bioplastics, were potato-plantain, and corn-cassava, varying the quantities of the aquatic plants for the biopolymer.

The results of the iodine test applied to the material obtained from the aquatic plants showed a positive result for the presence of starch, the most consistent coloration was in the Myriophyllum Quitense concerning the Elodea Canadensis; which corroborates the existence of bonds associated with the polysaccharide molecules detected by means of the analysis indicated by the infrared transmission spectroscopy.

5. Future work

Based on the results obtained we recommend to characterized the fiber obtained as co-products from the starch extraction process in order to take advantage of them for further applications.

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