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Research Article

Electrochemical Study of Cassava Starch Conductive Biopolymers Synthesized at Different pH

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Abstract: The aim of the study was to determine the effect of pH on the electrochemical properties of chemically-synthesized conductive polymers from alkoxylation of cassava starch at different pH (3, 4, 5, 8, 10, 11, 12) and with addition of different plasticizers (glycerol, glutaraldehyde, polyethylene glycol) and lithium perchlorate. The better integrity in their electrochemical structure was observed at pH 4, 5 and 8, due to the overlapping of the voltammograms of each one of the assays, indicating the stability of each material. Finally, it was observed that properties of the materials were unstable at extreme pH conditions (very low or very high pH), corroborating the progressive rearrangement of the structure thereof.

Keywords: Biopolymers, cassava starch, conductive polymer, cyclic voltammetry, *Manihot esculenta* crantz

INTRODUCTION

Hydrocarbons have been the central axis of energy utilization for technological and industrial development and have been protagonists until nowadays of the well-being of modern society, but its use has brought with it a sharp environmental impact as a result of combustion subproducts and because they are forms of non-renewable energies (Singh and Gu, 2010). So, it has been emphasized the need to advance in the development of alternative sources of non-conventional and renewable energies, also known as clean energies, as an alternative of contingency to the fuels used today (Tsai *et al*., 2016).

In the field of material science and technology, there is an active search of new materials that meet with the requirements of functionality of the demand imposed by the market and at the same time, that mitigate the environmental impact resultingfrom society lifestyle (Arrieta *et al*., 2017). The most widely used polymers cannot be recyclable without the inevitable loss of their properties, generating a constant increase on their consumption and a significant negative impact on the environment; therefore, the development of so-called biodegradable polymers from mainly from renewable resources have emerged as a promising alternative to hydrocarbon derived materials (De Aquino *et al*., 2015). Thus, biodegradable polymers have been studied for agricultural applications, biomedical and industrial applications among others (Garcés and Palencia, 2017; Pandit, 2017; Palencia *et al*., 2017). A specific working line in the study of new materials is the development of conductive biopolymers, which have showed satisfactory results in terms of electrical and mechanical properties (Arrieta *et al*., 2010; Deep *et al*., 2012), being their applications focused in their use as conductive materials and semiconductors (Venugopal *et al*., 2014).

The aim of this work is to study the electrochemical behavior and chemical stability of cassava starch conductive biopolymer films. This study analyses the influence of the different pH conditions used in the synthesis process on the electrochemical properties of films made from cassava starch.

MATERIALS AND METHODS

For the extraction of the cassava starch from amilolytic tissue, which is provided with amylose and amylopectin, is an essential stage for the synthesis of the conductive biopolymer. For extraction, the traditional method was used, consisting of washing, peeling, grating, decanting, filtering, drying and maceration processes. The resulting starch was sieved

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Fig. 1: Solid sample cell used for electrochemical characterization

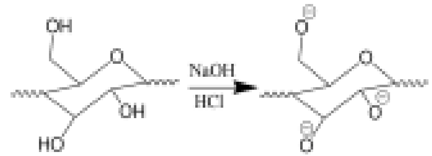

Fig. 2: Schematic representation of the alkoxylation reaction of starch

to obtain a particle size of 60 µm and ensure a homogeneous mixture of pre-adjusted pH aqueous solution. Glycerol (GLY), glutaraldehyde (GLU), polyethylene glycol (PEG) and lithium perchlorate (LP) were purchased from Sigma-Aldrich. For the chemical synthesis of the starch-conductive biopolymer at different pH, it was necessary to adjust the pH (3, 4, 5, 8, 10, 11, 12) according to the assay in a volume of 100 mL of ultra-pure water (Milli-Q). GLY (1.99 g), GLU (5.31 g), PEG (0.99 g) and LP (1.5 g) were added to cassava starch solution at room temperature and constant stirring. The pH was adjusted by addition of NaOH (0.1 mol/L) and HCl (0.1 mol/L), according to mixture requirements (Arrieta *et al*., 2017).

The solutions were heated at constant temperature (75±1.0°C) for 15 min and then poured into Petri dishes, finally brought to an oven at a temperature of 70°C for 48 h.

For the electrochemical characterization of the conductive polymer, a solid sample cell was made up of two 2 cm×2 cm stainless steel sheets, fitted into an acrylic support, which were closed with a press type nut system as shown in the Fig. 1. This assembly was required for the application of the Cyclic Voltammetry (CV) technique at the working conditions with a potential range of 2.0 V to -2.0 V, a scanning rate of 100 mV/s and 30 voltammetric cycles. The measurements were made using the Open Circuit Potential (OPC), which corresponded to 0.1 V (Arrieta *et al*., 2017).

RESULTS AND DISCUSSION

The cyclic voltammetry was carried out in freshly prepared films. The electrochemical analysis of the starch films was performed for each of the pH conditions in order to determine the changes in the electrochemical behavior of the material, representing the potentials in volts in the ordinate and the current in amps on the abscissa.

The starch dissolved by disrupting of the starch granules breaks free the polymeric molecules into the solution. The addition of NaOH and HCl breaks the intra- and intermolecular hydrogen bonds. The starch hydroxyl groups (-OH) on starch molecules are transformed to alkoxy group (starch-O^-). This reaction is showed in the Fig. 2. The charges on polymer chain allows to starch films to conduct the electric current and exhibit the electrochemical activity.

Figure 3 shows the voltammograms registered with the films prepared at pH 3 (Fig. 3a), pH 4 (Fig. 3b), pH 11 (Fig. 3c) and pH 12 (Fig. 3d). Oxidation and reduction peaks in each voltammogram are due to the electroactivity of the movement of charges of the inserted ions in the structure of the conductive starch biopolymer. The difference between the peaks in the voltammograms of each assay, at different values of pH, can be explained by different rearrangements of the polymer chains and possible structural changes with different electrochemical behavior.

Table 1 shows the different potential values of cathodic and anodic peaks for each biopolymer sample synthesized at different values of pH.

Different intensities of cathodic and anodic peaks in terms of milliamperes (mA) were evidenced, with appreciable changes in the peaks for the more acidic and basic environments (Table 1). These results possibly are associated with a greater opening of the polymer chains affecting the mobility of the electric charges that cross the material. The stability, determined by loss of signal intensity, showed that the films prepared at pH 8 and 9 were much more stable, with losses of intensity lower than 5% after 100 cycles. The less stable films were those prepared at pH 10, 11 and 12, with loss of intensity higher than 35% after 100 cycles.

As a result of this study, it was observed that when starch films were prepared with the components (glycerol, polyethylene glycol or lithium perchlorate) the recorded voltammetry showed a redox behavior in the films at the different acid environments; however, changes in the peak intensities and positions of some of the cassava starch conductive biofilms, as shown in biopolymers at pH 11 and pH 12 (Fig. 3c and 3d), may also be due to the basic environment at that the material was exposed. This behavior can be attributed to the fact that when higher amounts of NaOH are added in the synthesis of the starch films a rearrangement of the polymer chains is generated, producing possible changes in the structure and therefore in its electrochemical properties, a like to results observed when glycerol is added as plasticizer (Arrieta *et al*., 2014).

CONCLUSION

The voltammetric behavior of the different films synthesized at different pH shows that this synthesis

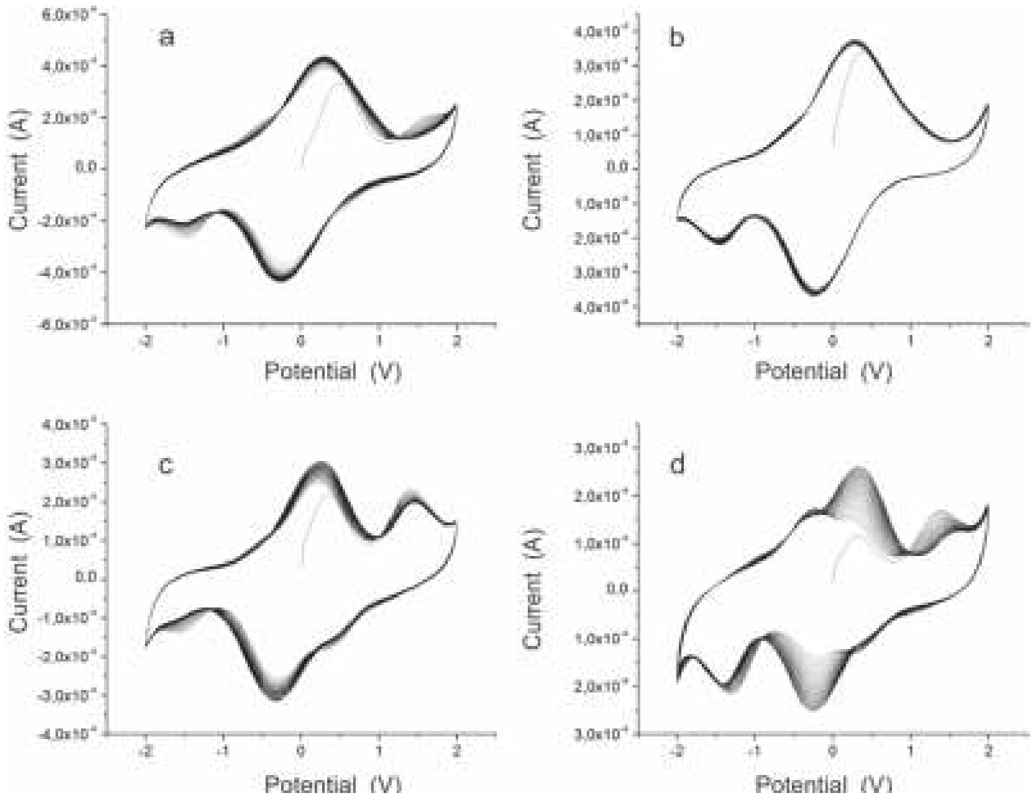

Fig. 3: Cyclic voltammetry of cassava starch biopolymers synthesized at; a): pH 3; b): pH 4; c): pH 11 and; d): pH 12

Table 1: Cathodic and anodic peaks of cassava starch biopolymers at different pH conditions

pH	Cathodic peak (V)	Anodic peak (V)
3	0.44	-0.44
4	0.38	-0.37
5	0.39	-0.39
8	0.37	-0.38
10	0.36	-0.38
11	0.30	-0.31
12	0.26	-0.25

factor affects the electrochemical behavior of the biopolymer films since in all the films it was observed electrochemical activity. However, the potentials were slightly different due to possible differences in their polymer structure. In addition, the stability of the films was affected by the pH of synthesis. At greater pH (10 to 12), the signals showed the greater loss of intensity (35%); whereas at medium and low values of pH, the loss of signal intensity was lower than 5%, which can be due to successive rearrangement of the structure of these materials.

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CONFLICT OF INTEREST

Authors have no conflict of interest

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