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BIO-BASED ELECTROACTIVE COMPOSITE BIOPOLYMER FILMS FROM CASSAVA STARCH / ANACARDIC ACID

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ABSTRACT

The United Nations have established Responsible Consumption and Production as one Sustainable Development Goal. This goal demands strategies for the efficient utilization of natural resources, agro-industrial waste, and the development of eco-friendly materials. Thus, the objective of this work was to develop bio-based films synthesized with anacardic acid (AA) extracted from the Cashew Nut Shell Liquid (CNSL) and starch extracted from cassava. Biopolymer films were synthesized at different pH values with various concentrations of AA. The biopolymer films prepared at basic pH presented higher tensile strength and the addition of AA also contributed to increasing the tensile strength. Infrared spectroscopy allowed the establishment of possible hydrogen bond interaction between AA and the polymeric chain of cassava starch, which could contribute to its higher tensile strength. The degradation rate was not affected by the synthesis pH, but by the presence of AA. Cyclic voltammetry allowed evidence of the electroactivity of glucose and AA in the composite biopolymer films. The results allowed it to conclude that it was possible to obtain a composite biopolymer from thermochemical synthesis with AA and cassava starch. The presence of AA can improve tensile strength, decrease the rate of biodegradability, and improve the electroactivity of the films.

Keywords: Anacardic Acid, Cassava, Starch, Biopolymer, Electroactivity.

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This manuscript is focusing **SDG-12:** Responsible Consumption and Production

INTRODUCTION

Synthetic plastics are used in a large number of applications, especially within single-use products such as packaging and disposable goods. The contamination of synthetic plastics is not only limited to their low biodegradability but from the extraction of oil as a raw material, a contaminating footprint is generated, going through the production process and final disposal.[1,2] In addition, during the degradation process, the so-called micro-pollution or microplastic pollution is generated because the molecules are not fully degraded and are integrated into the ecosystems.[3,4] Pollution is an international interest problem, of such magnitude that the United Nations has included it in the Sustainable Development Goals (SDGs) and has called for consumption and responsible production.[5] Therefore, due to the pollution problems generated by plastics of petrochemical origin, an interest in the development of alternatives that can substitute plastics or synthetic polymers is growing. In this sense, there are several strategies used for the development of bio-based materials, as well as the sources of origin. According to the IUPAC, biopolymers are polymers formed by living organisms from biomass.[6] Numerous studies have been reported in the literature on biopolymers production from different sources such as cellulose, chitosan, alginate, and starch, among others.[7-10] Starch is found mainly in seeds and tubers of various plant species as a reserve carbohydrate and is considered one of the most plentiful biopolymers in nature. In addition to its wide use in the food industry, it also has numerous non-food applications as a binder and stabilizer.[11] Recently, starch has become an object of interest for the development of biopolymers mainly due to its abundance, low cost, biodegradability, and biocompatibility, in addition to its easy process of obtaining. Starch is a polysaccharide made up of two classes of glucose macromolecules (amylopectin and amylose). Amylose is a glucose linear biopolymer linked with α-1,4 bonds, consisting of 250 to 2500 units. Amylopectin is a branched glucose biopolymer, with α-1,4 bonds in the linear part and α-1,6 bonds in the branches, which occur every 20 to 30 glucose units.[12-14] The proportions of amylose and amylopectin are approximately 20

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and 80% respectively, their proportionality depending on the plant species of origin.[12,13,15] The most used plant species for starch extraction are corn, cassava, potatoes, rice, yams, and bananas. Unmodified and modified starch has been used to develop a variety of biopolymers for food packaging, artificial muscles, medicine, and water treatment.[16-19] Another of the strategies used to elaborate starch biopolymers has been their combination with other types of materials, this allows improve and modulate their properties and expand their technological potential. The starch has been combined with graphene, copper nanoparticles, polyvinyl alcohol, chitosan, and polyacrylonitrile, among others.[20-23] One of the most interesting strategies in recent years is the use of by-products and waste from agroindustry to generate compounds and materials of technological and industrial interest. In this sense, in the production process of the Anacardium occidentale (Cashew) nut, the shell becomes a waste from which an oil known as CNSL (Cashew Nut Shell Liquid) can be extracted. CNSL represents 30% of the discarded walnut shell and is predominantly confirmed by cardanol (23 to 11%), AA (70 to 80%), and cardol (7 to 9%). 24 AA is a phenolic compound, with a carboxylic group and a chain of 15 carbon atoms, which gives it insecticidal, anti-inflammatory, fungicidal, and antioxidant properties, among others. In addition, it has the potential to develop a wide variety of bioactive products and biomaterials.[24-26] AA has been used to make polymers, combined and incorporated with other materials to make bioactive materials. Its combination with chitosan has been reported to produce films for bioactive packaging, cellulose membranes for extraction in the water of 4-amino-2-chloropyridine, and films with antimicrobial properties, among others.[26-28] Although works on the application of AA and its combination with different biopolymers have been reported, no references were found on the combination of AA with starch biopolymers. In this work, the synthesis of bio-based composite biopolymer films from cassava starch / AA and their characterization by infrared spectroscopy, cyclic voltammetry, tensile strength, and biodegradability test is presented.

EXPERIMENTAL

Material and Reagents

Reagents were purchased from Merck and Sigma-Aldrich. The preparation of the solutions and synthesis reactions was carried out with ultrapure water. The reagents used were: glycerin (Gly), Glutaraldehyde (Glu), polyethylene glycol (PEG), methanol, calcium hydroxide, chlorohydric acid, ethyl acetate, and anhydrous sodium sulfate. Starch extraction was carried out from tubers of Manihot esculenta (Crantz variety) and AA was extracted from CNSL obtained from cashew shells of *Anacardium occidentale* Yucao Ao3 variety. Starch was extracted using the previously described wet extraction process.[29] The tubers (500 g) were washed, peeled, and disintegrated by blending at 25 000 rpm with 2.5 L of water. The material obtained was filtered (200 μm) and the liquid obtained was decanted and dried for 24 h at 50 °C. The extracted starch was washed (three times) and dried for 24 h at 50°C. The dry cassava starch was sieved (50 μm) and a fine bright white powder was obtained. The AA extraction was carried out from CNSL following the previously reported chemical method.[30] CNSL was extracted from fresh and dried shells by mechanical extraction at room temperature using an oil press machine (US Solid). 100 g of CNSL was dissolved in 400 mL of 5% methanol and 50 g of calcium hydroxide was added. The formed calcium anacardate precipitate was filtered and washed with water. Calcium anacardate was heated at 45 °C for 3 h, suspended in HCl 11.0 M, and AA was extracted with ethyl acetate and dried with anhydrous sodium sulfate. Starch and AA were identified by infrared spectroscopy. The purity of the cassava starch was determined by the Association of Official Analytical Chemists (AOAC) method and AA by HPLC.[29,30]

Synthesis of Bio-Based Composite Biopolymer Films

Composite biopolymer films were elaborated by thermochemical synthesis. 3 g of starch was dispersed by stirring at 1500 rpm in 100 ml of water with pH balanced to the addition of NaOH or HCl (0.1 M) as required, and heated at 70 (±5) °C until complete dissolution (approx. 15 min). The pH values used in the synthesis solutions were 3, 7, and 11. The solution was cooled to room temperature and the plasticizers (Gly 2g; Glu 1.5g; and PEG 2g) were added with constant stirring until dissolved. AA was added to the synthesis solutions; mixtures with AA contents of 0.0%, 1.0%, and 2.0% were prepared. They were then heated at 70 (±5) °C and constantly stirred for 15 min. The synthesis solutions were placed in a Teflon petri dish to be dried in an oven for 48 hours at 70 °C. The films were manually removed and left to rest for 24 hours. Each film was prepared in triplicate.

Characterization of Bio-Based Composite Biopolymer Films

Characterization measurements were carried out on each of the synthesized cassava starch/AA films. The films were characterized by FTIR-ATR spectroscopy performed with a Spectrum-two from Perkin-Elmer spectrometer, with an ATR (Attenuated Total Reflectance) accessory. To record the spectra, a resolution of 4 cm^{-1}, a wavenumber range of 550 cm^{-1} to 4000 cm^{-1}, and 100 scans were used. Tensile strength measurements were carried out with an Instron universal testing machine 5882 at room temperature. The dimensions and geometry of the test samples were defined agreeing to the ASTM-D638 standard. Measurements of the test samples were carried out after calibration of the 50 N load cell (calibrated by a National Certification Institute). Electrochemical properties were carried out by cyclic voltammetry with a Gamry 1010E potentiostat/galvanostat using a dry measurement cell, formed from two stainless steel sheets and sandwiching the sample (1 cm x 1 cm).[29] Cyclic voltammetry was recorded at 0.1 V s^{-1} scan rate, scan range from +1.5 V to -1.5 V starting at 0 V. The reference potential used was the Open Circuit Potential (0.15 V). The biodegradability of the films was measured by soil burial test, the soil mixture was prepared using silty silt (23%), organic matter/cow manure (23%), and (23%), and distilled water (23%). The test samples were weighed before being buried 10 cm deep at 25 (± 5) °C, previously packed in perforated bags.

RESULTS AND DISCUSSION

The bio-based composite biopolymer films synthesized at distinct concentrations of AA and with distinct pH exhibited a stable consistency to manual traction and could be easily detached from Petri dishes without fractures or breakage. Composite biopolymer films presented a dark amber coloration, due to the presence of AA, while biopolymer films without AA (0%) showed a transparent yellow color. Figure-1 shows an image of the different films synthesized at distinct concentrations of AA and with distinct pH. It has been previously reported that plasticizers confer mechanical stability to starch films owing to the generation of hydrogen bonds with OH (hydroxyl groups) in the polymeric starch molecules. Therefore, hydrogen bonds can also be established between the hydroxyl groups present in the polymeric chain of starch and the hydroxyl and carboxyl groups present in the molecular structure of AA.

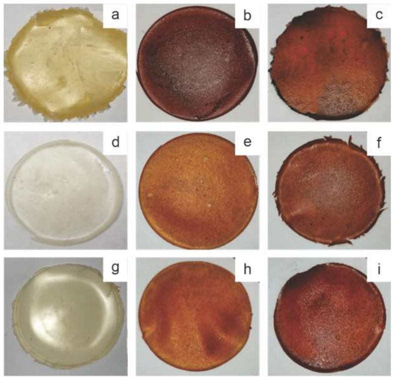

Fig.-1: Image of Biopolymeric Films Elaborated to (a) pH 3 and 0.0% AA; (b) pH 3 and 1.0% AA; (c) pH 3 and 2.0% AA; (d) pH 7 and 0.0% AA; (e) pH 7 and 1.0% AA; (f) pH 7 and 2.0% AA; (g) pH 11 and 0.0% AA; (h) pH 11 and 1.0% AA; (i) pH 11 and 2.0% AA

Figure-2 shows a scheme of the possible interactions established between the molecules of AA and the polymeric chains of starch. To evidence, this possible interaction, the infrared spectra of AA, cassava starch biopolymer, and composite biopolymer were studied (Fig.-3). Figure-3a shows that the spectrum of AA and the characteristic bands of this compound can be observed; the bands present at 3425 and 1260 cm^{-1} are associated with the OH group, 3004 and 1535 cm^{-1} due to the aromatic ring, 2930 and 910 cm^{-1} corresponding to the carboxylic group, the C-C stretch bands assigned to carboxyl group at 1585 cm^{-1}, and at 1450, 1445 and 1230 cm^{-1} the bands associated with C-H bending, aromatic C=C, an aliphatic chain, respectively. The cassava starch biopolymer presented the typical bands; the vibration assigned to the OH group was registered at 3440 cm^{-1}, the stretching of CH_2 at 2800 and 3000 cm^{-1}, the bands between 1500-800 cm^{-1} is the fingerprint region, the bands at 1153 and 1080 cm^{-1} attributed to C-O. In the spectrum of the composite biopolymer (Fig.-3c), the bands corresponding to its components (AA and cassava starch) were evidenced. In the region from 800 to 1400 cm^{-1} (blue zone), the characteristic bands of starch predominated. In the region from 1500 to 1800 cm^{-1} (red zone), the bands associated with the aromatic ring (1535 cm^{-1}) were evidenced, and the segment between 2800 and 3700 cm^{-1} (purple zone) where the band associated with OH stretching predominated. This band is of special interest because, as has been established in previously published works, its widening and weakening can show the formation of hydrogen bonds.[31] In this sense, it was observed that the band associated with OH is wider in the spectrum of the composite biopolymer, showing the hydrogen bond formation between polymeric molecules of cassava starch and AA.

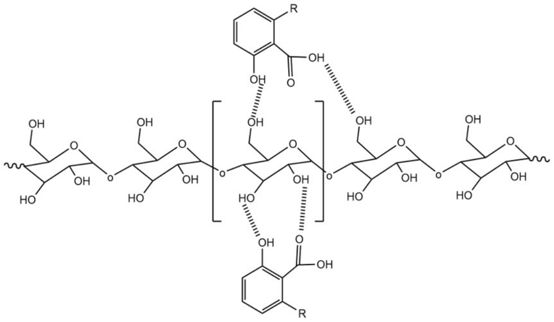

Fig.-2: Schematic Representation of Hydrogen Bonds Formed Between AA and Polymeric Molecule of Starch in the Composite Biopolymer

The interactions formed between the components of the composite biopolymer can influence its tensile strength. The results of the tensile strength tests are presented in Table-1. It could be observed that the concentration of AA proportionally affected the tensile strength; the higher the concentration, the higher the tensile strength was obtained. In addition, it was possible to establish that the films made in basic pH (pH 11), in general, presented higher tensile strength, this could be due to the differences in the crystallinity of the composite biopolymer, because, as has been previously established, starch films made at basic pH may exhibit higher crystallinity[7], which may increase tensile strength.

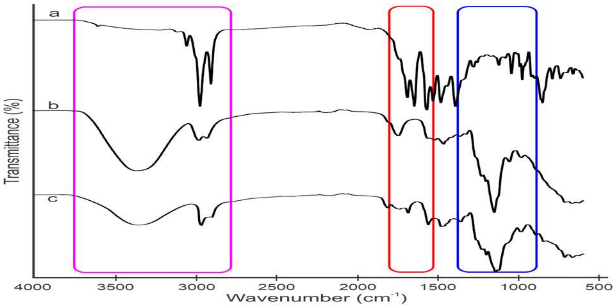

Fig.-3: FTIR-ATR Spectra of (a) AA; (b) Cassava Starch Biopolymer; (c) Composite Biopolymer Cassava Starch/AA

Figure-4 shows the biodegradability curves obtained with the composite biopolymer films. Only the weight loss percentage curves of the films synthesized with different concentrations of AA with pH 3.0 are presented because the films synthesized at different pH did not show any difference between them. Therefore, it was evidenced that the pH used in the synthesis process did not affect the biodegradation rate. On the other hand, AA content showed an effect on the rate of biodegradation in the first 13 days, slightly reducing weight loss. This behavior could be due to the presence of aromatic rings in the composite biopolymer films since this type of molecule can generate greater resistance to degradation.[32] The study of the electroactive behavior of the composite biopolymer films by cyclic voltammetry is presented in Fig.-5. Figure-5a shows the voltammetric responses of the biopolymer films without AA (0.0%). The films without AA showed voltammetric curves with a redox process when they were synthesized at pH 3 and 7, while the films synthesized at pH 11 did not show electroactivity. This behavior could be caused by the pH used in the synthesis of the films since basic pH can present more compact structures with greater crystallinity, which hinders the movement of charges in the polymeric matrix, decreasing the conductivity and electroactivity.

Table-1: Tensile Strength of Composite Biopolymer Films Synthesized at Different pH Values and with Different AA Acid Concentrations

Assay	pH	Anacardic acid (%)	Tensile strength (MPa)
1	3.00	0.0	1.86 ± 0.28
2		1.0	2.31 ± 0.57
3		2.0	2.35 ± 0.32
4	7.00	0.0	2.09 ± 0.08
5		1.0	2.51 ± 0.44
6		2.0	2.89 ± 0.25
7	11.00	0.0	2.12 ± 0.09
8		1.0	3.25 ± 0.18
9		2.0	3.67 ± 0.07

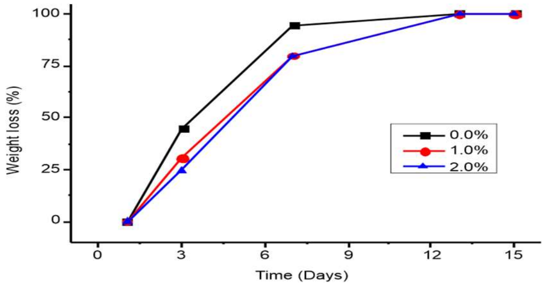

Fig.-4: Biodegradability Curves of Biopolymer Films by Soil Burial Test

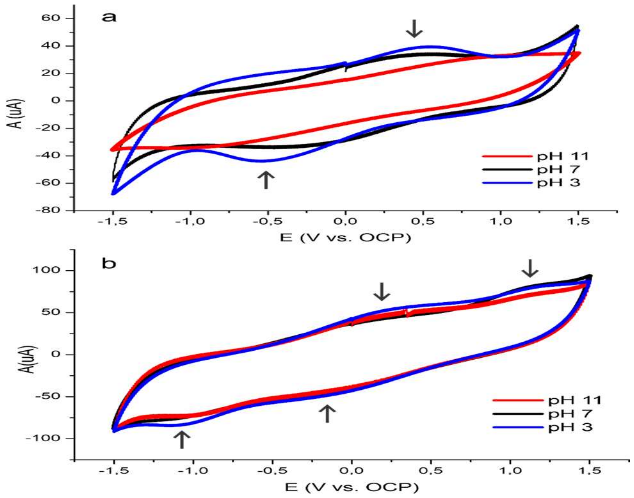

Fig.-5: Cyclic Voltammetry of Polymeric Films with a) 0.0% AA and b) 1.0% AA

Consequently, the curves presented lower current intensity and the absence of electroactivity (reduction and oxidation peaks). The redox process evidenced in synthesized films at pH 3 and 7, can be assigned to the oxidation and reduction of glucose units present in polymeric chain[33], and that due to the effect of pH presented more open and less compact structures that facilitated the movement of charges giving rise to oxidation and reduction processes when subjected to electrochemical stimuli. The responses of the films synthesized with 1.0% and 2.0% AA presented a similar behavior. Figure 5b shows the behavior of the films synthesized with 1.0% AA. The curves of these films showed electroactivity with all the synthesis pH values used, showing two redox processes, which were more defined in the biopolymeric films synthesized with pH 3 and 7. The oxidation-reduction processes peaks observed in the composite biopolymer films can be assigned to the redox processes of starch glucose units and AA present in the biopolymeric matrix.

CONCLUSION

The United Nations Sustainable Development Goals, demand strategies for the efficient use of resources and waste to reduce the environmental impact. As a contribution to these goals, a bio-based electroactive biopolymer composite was synthesized from cassava starch and AA extracted from CNSL. The effect of the amount of AA and the pH of synthesis were studied. In the study, it was concluded that the biopolymer was stable to manual traction and the presence of AA provided the highest tensile strength, as well as the use of basic pH for its synthesis. Infrared spectroscopy allowed the establishment of possible interactions by hydrogen bonds between the AA molecules and the polymeric chains of cassava starch in composite biopolymer films. The pH used in the synthesis process did not affect the biodegradability of the films, while AA decreased the rate of biodegradation of the composite biopolymer films. The basic synthesis pH decreased the electroactivity in the biopolymer films and the presence of AA allowed it to increase its electroactivity due to the presence in the voltammetry curves of the redox processes of glucose and AA.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing, and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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