

Effect of Glycerin and Urea on the Synthesis of Potato and Cassava Starch-Based Biopolymers: Hardness, Micrography, and Thermogravimetric Analyses

Efecto de la glicerina y la urea en la síntesis de biopolímeros a base de almidón de papa y yuca: análisis de dureza, micrografía y termogravimetría

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ABSTRACT

Petroleum-derived polymers used in everyday products generate large amounts of waste and negative environmental impacts due to their slow decomposition. To address this issue, other options have been studied, such as biopolymers based on starch, a polysaccharide formed by chains of amylose and amylopectin that can be linked together by heat and water to form a polymeric matrix similar to petroleum-derived plastics. In this study, glycerin and urea were used as plasticizing additives to improve the flexibility of biopolymers. The objective was to expand knowledge on biopolymers and their potential applications as an alternative to petroleum-derived plastics. A quantitative and qualitative factorial experimental design was implemented which consisted of three factors: the percentage of starch type, the percentage of plasticizer type, and the total percentage of plasticizers. Once the biopolymers were synthesized, they were characterized through thermal stability tests using thermogravimetric, hardness, and micrography analysis. The results indicated that glycerin increases the flexibility of the biopolymer, while urea increases its hardness. The working temperature of the biopolymer is below 130 °C, causing no disintegration of the material. In addition, it was identified that the presence of gas inclusions, both internal and superficial, can significantly affect the mechanical properties of the biopolymers. In conclusion, it was demonstrated that starch-based biopolymers with plasticizing additives have the potential to become a viable and sustainable alternative to petroleum-derived plastics in everyday products.

Keywords: biopolymers, glycerin, urea, starch, TGA, hardness, micrograph

RESUMEN

Los polímeros derivados del petróleo que se utilizan en productos de uso cotidiano generan grandes cantidades de residuos y un impacto ambiental negativo debido a su lenta descomposición. Para combatir este problema, se han estudiado otras opciones como los biopolímeros a base de almidón, un polisacárido formado por cadenas de amilosa y amilopectina que pueden enlazarse mediante calor y agua para formar una matriz polimérica similar a los plásticos derivados del petróleo. En este estudio se utilizó glicerina y urea como aditivos plastificantes para mejorar la flexibilidad de los biopolímeros. El objetivo fue ampliar el conocimiento sobre los biopolímeros y sus posibles aplicaciones como alternativa a los plásticos derivados del petróleo. Se implementó un diseño experimental factorial cuantitativo y cualitativo que constaba de tres factores: el porcentaje del tipo de almidón, el porcentaje del tipo de plastificantes y el porcentaje total de plastificantes. Una vez sintetizados los biopolímeros, se caracterizaron mediante ensayos de estabilidad térmica con TGA, dureza y micrografía. Los resultados indicaron que la glicerina aumenta la flexibilidad del biopolímero, mientras que la urea aumenta su dureza. La temperatura de trabajo del biopolímero se encuentra por debajo de los 130 °C, sin generar desintegración en el material. Además, se identificó que la presencia de inclusiones gaseosas, tanto internas como superficiales, puede incidir significativamente en las propiedades mecánicas de los biopolímeros. En conclusión, se demostró que los biopolímeros a base de almidón con aditivos plastificantes tienen el potencial de convertirse en una alternativa viable y sostenible a los plásticos derivados del petróleo en productos de uso cotidiano.

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Palabras clave: biopolímeros, glicerina, urea, almidón, TGA, dureza, micrografía

Received: January __th 20xx

Accepted: January __th 20xx

Introduction

Prior to the 19th century, plastic was not widely used [1], [2]. It was during the 20th century when interest in its use increased and its demand grew, given the need to develop advanced materials [3]–[5]. The production and manufacture of plastic products entails lower costs compared to other materials, which has significantly influenced its production and applicability in various industrial sectors [6], [7].

Petrochemical polymers are materials characterized by their easily moldable molecular structure, flexibility, and corrosion resistance. Single-use petrochemicals, also known as *disposable products*, constitute a significant portion of everyday products and are manufactured in large quantities due to their high demand [3], [8]. However, their final disposal in landfills and ecosystems generates a substantial environmental impact, as they can take decades or even hundreds of years to degrade [9]–[11].

Various research approaches are currently being explored to address the issue of plastics and their replacement. One widely studied option is biopolymers, which include starch, a polysaccharide or *glucose polymer* that occurs in granular form and serves as an energy source for plants and animals. The polymeric chains of amylose and amylopectin that make up starch vary according to their source [12]–[14]. Under controlled humidity and temperature conditions, these chains can cross-link to form a polymer matrix suitable for the production of bioplastic films [15]–[19].

In this context, the use of industrial waste from processing companies handling potato (*Solanum tuberosum*) and cassava (*Manihot esculenta*) has been proposed. These carbohydrate-rich foods are widely cultivated and processed worldwide [20]. According to the Food and Agriculture Organization (FAO), the annual potato production exceeded 300 million tons in 2016 [21]. For cassava and other tubers, global processing is estimated to exceed 320 million tons, generating 5–30% waste, such as peels and pulp, at each production stage. This approach reduces the impact of using food crops in biopolymer production, thereby promoting a more sustainable and ethical resource utilization [20].

Peels, common byproducts in the processing of these tubers, represent an affordable raw material that is available in large volumes and has potential applications in biopolymer production. In the potato industry, processing generates between 70 000 and 140 000 tons of peels per year, which are generally discarded or used in low-value products such as animal feed [21]. Similarly, cassava processing yields up to 1.6 tons of peels and 280 tons of bagasse with high moisture content, which are often wasted without adequate treatment, ending up in landfills and watercourses [22].

The starch composition of these crops varies: potatoes contain 17–24% amylose and 76–83% amylopectin, whereas cassava contains 16–22% amylose and 81–83% amylopectin [23]. These differences are crucial in the gelatinization process, which determines the formation and properties of the polymer matrix in bioplastics [24]. Leveraging these industrial sources of starch not only contributes to sustainability but also offers specific properties in the resulting bioplastic films.

Thus, this study aims to create biopolymers from potato and cassava starch, with the addition of plasticizers to enhance their flexibility and physical properties. According to the free volume theory [18], the addition of plasticizers with flexible chains increases the free volume of molecules, resulting in more flexible biopolymers [25], [26]. Additionally, it has been found that biopolymers synthesized solely with water and starch tend to be brittle, making the addition of plasticizers crucial to enhance their flexibility [27], [26].

Plasticizers are widely used to enhance the flexibility and processability of plastic materials, with their selection being a crucial aspect that depends on chemical and physical compatibility with the polymer matrix [28]. Among the most commonly used plasticizers are urea, sorbitol, and glycerol, given their effectiveness in modifying the mechanical properties of biopolymers [29], [30]. Other compounds, such as ethylene glycol, diethylene glycol, and triethylene glycol, are recognized for their ability to reduce the rigidity of the polymer matrix [31].

Additionally, more specialized plasticizers like triethyl citrate have demonstrated significant potential in applications requiring greater thermal and chemical stability [32], and biodegradable materials such as chitosan and polylactic acid have been investigated as alternative plasticizers for sustainable biopolymer production, standing out for their renewable origin and reduced environmental impact [33].

For this research, we used glycerol and urea. To date, the extent to which plasticizers affect material properties when added to a combined potato and cassava thermoplastic starch polymeric matrix for the production of biodegradable and compostable films has not been investigated [34].

Methodology

The methodology used in this study was divided into four phases, namely extraction, polymerization, fabrication, and characterization.

Extraction

Starch from potato and cassava was extracted according to the method described by [35], which involved crushing the raw materials in an aqueous medium. The resulting wet mass with lumps was diluted in water and agitated to separate the starch granules from unwanted materials [35]. The mixture was then filtered, and the resulting liquid was decanted to obtain the starch together with other impurities in the form of sediment. Three washes were performed with distilled water to remove these impurities, and the resulting wet starch was dried in an electric oven at 60 °C for 24 h [36].

Polymerization

The entire polymerization process was conducted while following the proportions outlined in the experimental design. This design, summarized in Table I, was developed to assess the impact of three primary factors on biopolymer synthesis: % starch type, % plasticizer type, and % total plasticizer, all on a basis of 10 g total starch per sample. The factor levels considered potato/cassava starch combinations (100/0, 60/40, 40/60, and 0/100), urea/glycerin ratios (100/0, 60/40, 40/60, and 0/100), and total plasticizer percentages (5, 10, and 20%). For instance, for the 40/60

potato/cassava combination, 4 g of potato starch and 6 g of cassava starch were used. With a 5% total plasticizer level, corresponding to 0.5 g, and a 40/60 urea/glycerin ratio, 0.2 g of urea and 0.3 g of glycerin were incorporated. This approach was replicated for all combinations, enabling a comprehensive evaluation of the resulting properties.

Table I. Experimental design factors

| Factors | | % Total Plasticizer | | | | |
|---------|---------------------|---------------------|-----|-----|-----|--|
| | | | 5% | 10% | 20% | |
| 100/0 | % Glycerin/ Urea | 100/0 | P1 | P2 | P3 | |
| | | 60/40 | P4 | P5 | P6 | |
| | | 40/60 | P7 | P8 | P9 | |
| | | 0/100 | P10 | P11 | P12 | |
| 60/40 | % Glycerin/ Urea | 100/0 | P13 | P14 | P15 | |
| | | 60/40 | P16 | P17 | P18 | |
| | | 40/60 | P19 | P20 | P21 | |
| | | 0/100 | P22 | P23 | P24 | |
| 40/60 | % Glycerin/ Urea | 100/0 | P25 | P26 | P27 | |
| | | 60/40 | P28 | P29 | P30 | |
| | | 40/60 | P31 | P32 | P33 | |
| | | 0/100 | P34 | P35 | P36 | |
| 0/100 | % Glycerin/ Urea | 100/0 | P37 | P38 | P39 | |
| | | 60/40 | P40 | P41 | P42 | |
| | | 40/60 | P43 | P44 | P45 | |
| | | 0/100 | P46 | P47 | P48 | |

Source: Authors

For each factor combination, four specimens were synthesized per sample in order to ensure the repeatability and reliability of the data obtained. The physical-mechanical properties of the biopolymer were measured from these specimens. The results of these measurements were averaged and used for statistical and graphical analysis, allowing for the identification of trends and relationships between the evaluated factors.

A full factorial design was employed in the statistical analysis, enabling the evaluation of interactions between factors and their combined effect on biopolymer properties. This robust approach facilitated the interpretation of the results and allowed for precise comparisons between different experimental combinations.

This methodology enabled a comprehensive analysis of the interactions between factors and their impact on biopolymer properties, providing reliable data for interpreting the results and drawing conclusions.

The polymerization method used in this study involved the addition of plasticizing agents in an aqueous medium, following the methodology of [37], with modifications to suit the additives and equipment used in the process. On a hot plate at 100 °C, a homogeneous mixture of water, starch, and urea was prepared in predetermined proportions. Continuous stirring was maintained to prevent lump formation and ensure uniformity. Heating caused the starch granules to swell, allowing water to penetrate the amorphous regions of the starch, thus fragmenting the granular structure [38].

In the formulations containing glycerin, this component was added when the mixture reached 40 °C, allowing polymer chains to form before the gelatinization process began. This approach was based

on the findings of [39], who reported that starch gelatinization starts between 40 and 45 °C. Throughout the procedure, constant stirring was also maintained to prevent lump formation and ensure a homogeneous mixture.

Acetic acid was subsequently added when the mixture reached 60 °C, in order to completely disrupt the starch granules and thus optimize the efficiency of polymer chain formation. The temperature was continuously monitored to ensure it did not exceed 80 °C, with heat adjustments made on the hot plate as needed. The mixture was stirred until a fully homogeneous sol-gel was achieved and removed from the hot plate after 40 min [40].

The pH of the mixture was carefully controlled throughout the process to prevent variations that could compromise the integrity of the starch granules. According to [41], pH changes can affect the starch gelatinization process, potentially influencing granule stability. No significant pH alterations were observed during the tests. However, in the event of an unexpected decrease, the procedure would need to be restarted and the amount of acetic acid adjusted in order to restore optimal conditions.

Molding

The bioplastic specimens were produced using the casting method, following the methodology proposed by [42]. In this technique, the biopolymer is poured in a sol-gel state onto a mold coated with a release agent. The resulting sheet is then dried at 50 °C for 24 h in an electric oven before being demolded and cut into the desired specimen shape.

Material characterization

Thermogravimetric analysis (TGA) is a quantitative analytical technique that monitors the mass of a sample as the temperature increases in an oven under a stable or changing gas flow [43]. This technique allows detecting weight changes that occur in a sample subjected to heat treatment, providing valuable information about its thermal stability, maximum working temperature, moisture loss temperature, disintegration temperature, and ash generation temperature [44].

Optical microscopy was used to study the surface morphology of starch-based biopolymer sheets. The samples were analyzed using a Nikon SMZ800 stereo microscope equipped with a SONY video camera, with magnifications of 10x, 20x, and 60x. The inspection of the micrographs focused on the conformation and appearance of the films, providing information on their structural characteristics and potential performance.

Hardness is defined as a material's resistance to plastic deformation from penetration or scratching by another solid body. In this project, the Shore scale was used, as it is widely employed to evaluate the hardness of soft and semi-rigid materials like plastics and elastomers [45]. Measurements were conducted using a PC-511/A digital durometer (type A) equipped with an indenter, following the parameters specified by the Shore A scale.

Results and discussion

TGA

Fig. 1 illustrates the weight loss as a function of time and temperature for biopolymer samples containing different plasticizer types and concentrations (glycerin and urea). Overall, the thermogravimetric behavior was similar among samples with equivalent plasticizer content, although specific differences were observed at each stage of the thermal analysis.

During the initial phase, spanning temperatures between 0 and 100 °C, weight loss corresponds to the evaporation of water absorbed or physically bound to the biopolymer. Samples with higher plasticizer content exhibited faster weight loss, indicating that these additives increase moisture retention by interacting with the starch chains. This trend reflects the hygroscopic nature of the plasticizers used in the polymer matrix.

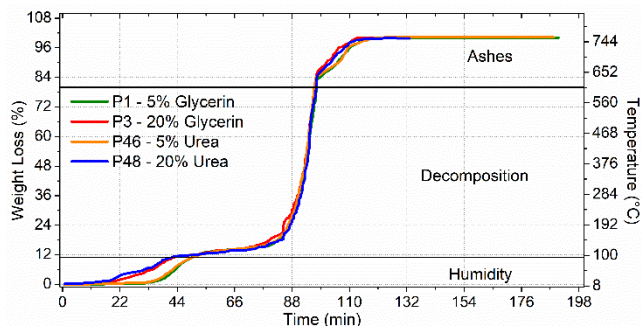


Figure 1. TGA of the biopolymers
Source: Authors

In the second phase, at temperatures ranging from 150 to 500 °C, the thermal degradation of polymer chains and plasticizing additives takes place. The onset temperature of decomposition varies depending on the plasticizer type and concentration. Despite these variations, the samples followed a uniform degradation pattern within the specified range. This behavior suggests that, while plasticizers influence the thermal stability of the material, they do not significantly alter the decomposition mechanism.

Finally, at temperatures exceeding 600 °C, the samples reach a residual state corresponding to ash formation. At this stage, the decomposition of organic components is complete, and differences in residual weight are associated with the initial composition of the biopolymer and the type of plasticizer used.

On a broader scale, it is noted that the onset temperatures of degradation in this study (approximately 130 °C) are lower than those reported in previous research, such as [46] and [47], which indicate onset temperatures between 150 and 250 °C. However, the weight loss patterns are consistent with the characteristic values for starch-based biopolymers. This suggests that the studied samples can be safely processed at temperatures below 130 °C, while ash formation consistently occurs above 600 °C, in line with prior studies. The graph clearly illustrates these phases and highlights the influence of plasticizer concentration and type at each stage of the TGA.



Figure 2. Biopolymer micrographs 10x, 20x, and 60x
Source: Authors

Optical microscopy

Micrographs were obtained from various tests performed on biopolymer samples, which, as previously noted, were formulated with different proportions of the evaluated variables: starch type (% potato/cassava), plasticizer type (% glycerin/urea), and total plasticizer concentration. The micrographs, captured at magnifications of 10x, 20x, and 60x, are presented in Fig. 2. The analyzed samples included P1 (% potato/cassava: 100/0, % glycerin/urea: 100/0, total plasticizer: 5%), P14 (% potato/cassava: 60/40, % glycerin/urea: 100/0, total plasticizer: 10%), P28 (% potato/cassava: 40/60, % glycerin/urea: 60/40, total plasticizer 5%), P31 (% potato/cassava: 40/60, % glycerin/urea: 40/60, total plasticizer: 5%), and P46 (% potato/cassava: 0/100, % glycerin/urea: 0/100, total plasticizer: 5%).

At 10x and 20x, a non-uniform surface conformation was observed in the biopolymer samples, with noticeable bubbles on the surface and within the formed materials. This phenomenon suggests that, during the synthesis process, the agitation used to homogenize the material introduced air bubbles which could not fully escape the matrix before solidification due to the high viscosity of the system.

At 60x, an irregular surface texture was observed, associated not only with the presence of bubbles, but also with other structural imperfections related to the manufacturing technique employed, *i.e.*, casting. While effective for producing biopolymers, this method is influenced by multiple variables that are challenging to control, such as solidification speed, plasticizer distribution within the matrix, and environmental conditions, which hinder the attainment of a fully homogeneous surface.

These imperfections could potentially be minimized by manufacturing the biopolymers via alternative processing techniques such

as injection molding or thermoforming. These methodologies allow for greater precision in controlling material distribution and processing conditions, thereby reducing bubble formation and improving surface uniformity. Injection molding, for instance, ensures consistent pressure and homogeneous components distribution, while thermoforming facilitates more uniform solidification through controlled molds and temperatures. This suggests that optimizing the manufacturing technique is essential to enhancing the final quality of biopolymers and their suitability for end-use applications.

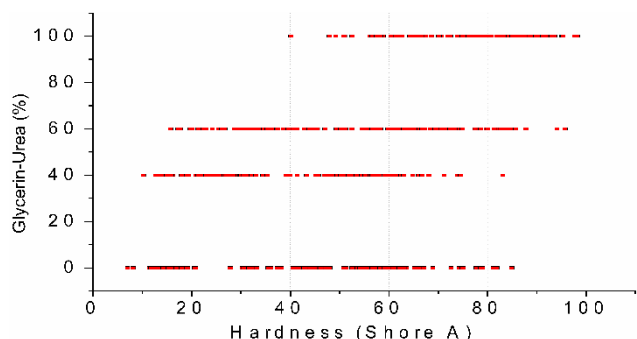


Figure 3. Scatter plot: hardness vs. glycerin-urea (%)
Source: Authors

Hardness

The data presented in Figs. 3 and 4 demonstrate an inverse relationship between the glycerin content and the hardness of the biopolymer. An increase in the percentage of glycerin incorporated into the polymer matrix leads to a reduction in material hardness, accompanied by greater variability in the data for different biopolymer samples. Additionally, Fig. 4 highlights the significant impact of increasing the overall percentage of plasticizers on data dispersion across various biopolymer samples. This behavior can be attributed to the effect of plasticizers on material hardness, wherein glycerin imparts greater flexibility. Moreover, biopolymer compositions with higher urea concentrations exhibit reduced data variability and increased hardness.

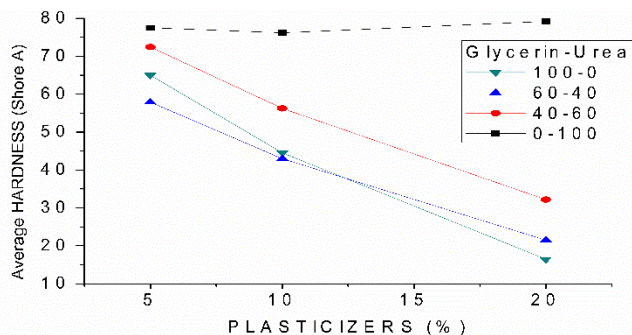


Figure 4. Linear graph: plasticizer hardness vs. glycerin-urea (%)
Source: Authors

Fig. 5 illustrates a clear decrease in material hardness as the ratio of total plasticizer to total starch increases, indicating that incorporating plasticizers enhances material flexibility and reduces rigidity, significantly impacting biopolymer hardness. In other words, the material's hardness is affected by the proportion of plasticizers used during production.

In the study titled *Physical characterization of biopolymers with starch from potato and cassava waste polymerized in water* [48], biopolymers synthesized from starch without the addition of plasticizers

reached a maximum hardness of 78,9 on the Shore A scale. Comparing these results to our findings reveals that an increase in the glycerin content within the polymer matrix reduces hardness, with values as low as 33.2 on the Shore A scale.

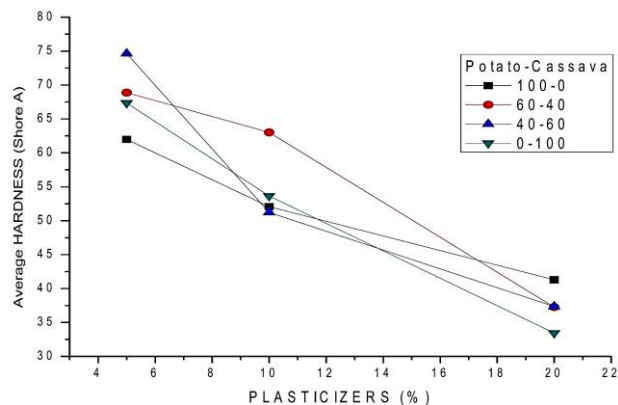


Figure 5. Linear graph: plasticizer hardness vs. potato-cassava (%)
Source: Authors

Fig. 6 explores the interaction between plasticizers and the potato and cassava starch ratios, showing that biopolymer hardness depends on the combination of these factors. Biopolymers with higher glycerin proportions (100/0) exhibit significantly lower hardness values across all starch combinations, confirming glycerin's predominantly plasticizing effect. Conversely, systems with higher urea content (0/100) tend to exhibit greater hardness, particularly in compositions with cassava starch (0/100), suggesting that urea promotes the formation of a stiffer matrix when interacting with cassava starch.

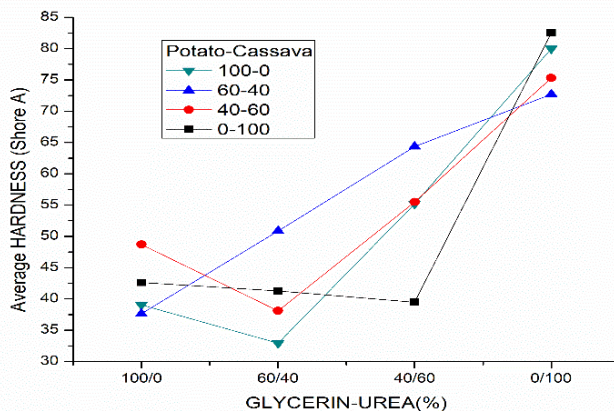


Figure 6. Linear graph: glycerin-urea hardness vs. potato-cassava (%)
Source: Authors

Furthermore, the lines corresponding to different starch combinations (100/0, 60/40, 40/60, and 0/100) indicate that the cassava content increases hardness in the presence of urea, while potato starch displays a more stable behavior under variations in plasticizer content. These differences suggest that the molecular structure and the interaction between polymers and plasticizers are key to tailoring the mechanical properties of the material.

The study titled *Analysis of factors affecting hardness behavior in biopolymers based on potato and banana peels: A factorial experimental evaluation* [49] also elucidates the relationship between increasing glycerin content and decreasing hardness, while noting that urea has a lower influence on material hardness.

Table II. Hardness data for petroleum-derived plastics

| Material | Hardness (Shore A) | |
|----------------------------------|--------------------|------|
| HDPE – High-density polyethylene | 98,5 | [50] |
| PE – Polyethylene | 95,13 | [51] |
| PET – Polyethylene terephthalate | 95,43 | [51] |
| PP – Polypropylene | 79,1 | [52] |
| PS – Polystyrene | 42,5 | [53] |
| PTFE – Polytetrafluoroethylene | 94,13 | [51] |

Source: Authors

The biopolymers synthesized in this study exhibit hardness values ranging from 30 to 85 on the Shore A scale depending on the type and percentage of the plasticizers incorporated into the polymer matrix. When compared to conventional plastics (Table II) such as HDPE (98,5), PE (95,13), PET (95,43), and PTFE (94,13), the biopolymers display lower hardness values. Still, they are competitive with PP (79,1), and, in certain combinations, they even surpass PS (42,5). This demonstrates that, by carefully adjusting the proportions and types of plasticizers, biopolymers with mechanical properties tailored for various applications can be achieved. In particular, this design flexibility suggests that these biopolymers could be synthesized to replace single-use plastics, offering a more sustainable and environmentally friendly alternative.

Conclusions

The addition of plasticizers to the polymeric matrix is known to provide materials with diverse physical-mechanical properties, making them potential substitutes for different petroleum-derived plastics. During the characterization of the biopolymers, the data revealed that the 60/40 potato/cassava starch ratio yielded superior biopolymer hardness. Regarding the plasticizer type, urea imparted significant hardness. However, in high proportions, it rendered the material fragile. In contrast, glycerin considerably reduced the hardness but provided flexibility to the biopolymer.

By combining urea and glycerin plasticizers in different proportions, hardness and plasticity properties can be tailored. Therefore, variations in the plasticizer type allow controlling the flexibility and hardness of the material, enabling the diversification of products. The TGA confirmed that the biopolymers can be processed at less than 130 °C for subsequent heat treatment to improve their properties while avoiding bioplastic disintegration.

The micrograph results showed that minimizing the bubbles in the biopolymers during the molding process can help to control the factors that affect mechanical properties, potentially yielding increased resistance to plastic deformation and a uniform material. Thus, optimizing the molding method is essential for achieving desired properties in biopolymers.

Author contributions

Daniel Nicolás Mariño Quintana, Diana Paola Sanabria, and Ricardo Alfonso Paredes conceived the project, defined its general structure, and designed the methodology. Daniel Nicolás Mariño Quintana and Diana Paola Sanabria were responsible for managing and conducting the research, including collecting the data, creating graphical visualizations, and drafting the initial manuscript. Ricardo Alfonso Paredes, Hugo Felipe Salazar, and Hugo Fernando Castro Silva supervised the project, validated the results, and performed a critical review of the draft. Finally, Daniel Nicolás Mariño Quintana and Diana Paola Sanabria edited the final manuscript.

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