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Development of corn starch biodegradable films reinforced by nanocellulose for food packaging

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ABSTRACT: The present paper evaluates the mechanical and physical characteristics of edible films made from corn starch (*Zea mays* L.) reinforced with nanocellulose. To do this, films were made with 0.4 and 0.08% cotton nanocellulose (NC) with plasticizer (0.4 and 1%). The mixture was emulsified between 1 and 3 min and dried for 20 h (36 and 50 °C). NC was obtained by acid hydrolysis for 6 days at 25 °C with 50% sulfuric acid and dialyzed to neutral pH. Cotton NC and NC-included film were analyzed using Fourier transform infrared spectroscopy. About the physical characteristics, it will be observed that there was no significant difference between the inclusion percentages. Accordingly, a 5% inclusion would be sufficient for smooth, transparent, flexible, non-cracked and homogeneous films. It is determined that the higher the percentage of NC, the lower the elasticity values, improving the films' hardness and transparency. Given that the percentage present in the NC test solution is 1.6%, its inclusion would be 0.4% (25 ml NC) and 0.08% (5 ml NC), indicating its great influence on films.
Keywords: biodegradable films; starch; mechanical properties.

Desenvolvimento de filmes biodegradáveis de amido de milho reforçados com nanocelulose para embalagens de alimentos

RESUMO: No presente trabalho são avaliadas as características mecânicas e físicas de filmes comestíveis elaborados a partir de amido de milho (*Zea mays* L.) reforçados com nanocelulose. Para isso foram confeccionados filmes com 0,4 e 0,08% de nanocelulose de algodão (NC) com plastificante (0,4 e 1%): A mistura foi emulsionada entre 1 e 3 min e seca por 20 h (36 e 50 °C). A NC foi obtida por hidrólise ácida durante 6 dias a 25 °C com ácido sulfúrico a 50% e dialisada até pH neutro. Algodão NC e filmes incluídos em NC foram analisados por espectroscopia de infravermelho com transformada de Fourier. Em relação às características físicas, observar-se-á que não houve diferença significativa entre os percentuais de inclusão. Assim, uma inclusão de 5% seria suficiente para obter filmes lisos, transparentes, flexíveis, não fissurados e homogêneos. Determina-se que quanto maior o percentual de NC, os valores de elasticidade diminuem, melhorando a dureza e a transparência dos filmes. Dado que a percentagem presente na solução teste NC é de 1,6%, a sua inclusão seria de 0,4% (25 ml NC) e 0,08% (5 ml NC), o que indica a grande influência que tem nos filmes.
Palavras-chave: filmes biodegradáveis; amido; propriedades mecânicas.

1. INTRODUCTION

Due to its low cost and wide availability, starch, a biopolymer used in packaging, allows us to obtain biodegradable materials (SCHAEFER et al., 2020). Starch-based edible films have high water vapor permeability and poor mechanical properties, as noted by FANG et al. (2019). Also, JOSHI et al. (2018) stated that forming these films with poor barrier and mechanical properties has restricted their development and practical application. Likewise, CHEN et al. (2019) and FARAJPOUR et al. (2020) reported that starch films mostly show high sensitivity to humidity and low mechanical strength.

However, compared to synthetic materials, starch films, due to their hydrophilic nature and poor mechanical characteristics, can be improved by incorporating other materials in the polymer matrix (MÜLLER et al., 2011). The material added as reinforcement must interact with the polymer matrix to have a union between the material's properties. If this does not occur, the composite material shows poor mechanical properties compared to pure polymers (TEODORO et al., 2015). Still, improving mechanical properties and reducing moisture sensitivity are two continuous challenges for starch films (CHEN et al., 2019).

Nanocellulose-based polymers can modify surface chemistry and mechanical properties, making them an ideal role for reinforcement (ZINGE; KANDASUBRAMANIAN, 2020). When nano-sized aggregates are dispersed in the polymer matrix, nanocomposite films show great mechanical performance and adequate barrier properties (Sharma et al., 2017), as also detailed by Ghanbari et al. (2018) for corn starch films. Da Silva et al. (2012) stated that with the addition of 0.2% by weight of nanocellulose, the starch-based films had significantly improved in mechanical properties by 92% concerning surface tension and 400% in Young's modulus, compared to the control films.

Cotton nanofibers, composed mainly of cellulose, can be a promising alternative for obtaining this type of nanostructure (GÓMEZ et al., 2016). Furthermore, cotton nanofibers present a strong interaction with the polymer matrix, which leads to an improvement in the properties of the film, as it represents an alternative to the use of synthetic products (WANG et al., 2019). According to Savadekar; Mhaske (2012), the addition of nanocellulose in thermoplastic starch films increased the surface tension and the mechanical properties improved (WANG et al., 2019).

Among the most important cereals for human consumption are corn (*Zea mays* L.), wheat, and rice, essential raw materials for the processing industry. These cereals produce starch, oils, proteins, alcoholic beverages, food sweeteners, and more (YU; MOON, 2022). Corn grains contain approximately 90% starch and 9% protein, and small amounts of oils, minerals and trace elements (Ikem et al., 2023), with starch being the main constituent of corn (TETLOW et al., 2004).

The global production of corn starch is approximately 60 million tons, of which 60% is used for food applications and the rest for non-edible and pharmaceutical applications (COPELAND et al., 2009). According to figures from the III National Agricultural Census, Ecuador has approximately 248982 hectares and 82000 production units destined for the cultivation and production of corn. According to MAGAP statistics, in the last decade, around 270 thousand to 360 thousand hectares of corn have been planted annually in Ecuador, with a current production that exceeds one million tons (TRIVIÑO; VILLENA, 2019).

As mentioned, starch-based films have limitations such as high water vapor permeability and poor mechanical properties, which restrict their industrial application. Several studies have reported that adding reinforcing materials like nanocellulose can improve these films' mechanical and barrier properties. Despite this, further research on integrating nanocellulose into the starch matrix is still needed to improve its moisture barrier characteristics and mechanical strength. In this context, the present work evaluated the effect of cotton nanocellulose on the mechanical and physical characteristics of edible corn starch (*Zea mays* L.) films.

2. MATERIAL AND METHODS

2.1. Preparation of nanocellulose

Cellulose nanofibers were obtained by cutting commercial cotton fibers to a size of approximately 2 to 4 mm. They were treated by basic hydrolysis with 5% (m/v) sodium hydroxide for 3 h at 10^3 Pa. The fibers were washed and subjected to hydrolysis (Habibi et al., 2010) with 50% (v/v) sulfuric acid for 6 days at 25 °C. The hydrolyzed acid fibers were dialyzed for 7 days with daily exchanges of deionized water until a pH between 6 and 7 was achieved (DONG et al., 1996). Subsequently, they were passed through a membrane filter (Polyethersulfone, pore size 0.22 µm, JiAn City Qingfeng Filter Equipment Material Co., Ltd., Jiangxi, China). The solution was adjusted to a concentration of 1.6% (m/v) of cellulose nanofibers (DONG et al., 1996), because the nanofibers tend to polymerize at a higher concentration. This solution was stored between 8 and 15 ºC.

2.2. Preparation of films with reinforcement

Based on our previous unpublished experimental results, corn starch films with nanocellulose were prepared from a 1.8% (m/v) starch solution (Royal, Mondelez Ecuador Cía. Ltda., Quito), at which was added glycerin at 0.4 and 1% (Merck KGa, Darmstadt) as a plasticizer and cotton nanocellulose at 5 and 25% (m/v). It was heated on a magnetic stirrer (Velp Scientifica Srl, Italy) until reaching 95 °C for 3 min. Subsequently, the solution was mixed with a mechanical stirrer (Imusa click & mix) for 1 and 3 min, respectively, at around 50 °C. Depending on the treatment, the solution was poured into plastic Petri dishes and dried at 36 and 50 °C for 20 h in an oven with air circulation. They were then demoulded and stored at 25 °C and 50% relative humidity. Table 1 shows the variables and levels for the experiments' development and the response variables.

Table 1. Test variables and response variables
Tabela 1. Variáveis de teste e variáveis de resposta

X1	X2	X3	X4	Response variables
Nanocellulose	Plasticizer	Temperature	Mixing time	Elasticity
25	0.4	36	3	Maximum strength,
5	1	50	1	moisture, thickness

2.3. Analysis of nanocellulose

For microscopy, a drop of the cellulose nanofiber solution was taken after coating with gold-palladium to improve the conductivity of the material to achieve higher magnifications in the scanning electron microscope (FEI Inspect S50) with a voltage of 10.5 kV at High vacuum mode and 4.5 Spot pressure at 12-15 Pa). An ETD detector (Everhart-Thornley Detector) was also used at a working distance of 10 mm for the sample holder. In addition, the purity of cellulose was determined using the Kurshner and Hoffer method through alcoholic digestion in nitric acid (HESSLER; MEROLA, 1949).

2.4. Analysis of the biodegradable films

The thickness of the films was measured with a digital micrometer (0-12.7 mm with a sensitivity of 0.001 mm), with 10 random repetitions at different points of each film. Additionally, the moisture content was determined using the gravimetric method (AOAC, 2012), water solubility according to the methodology described by Chiumarelli;

Hubinger (2014) with some modifications, and tensile strength and elasticity (ANCHUNDIA et al., 2016).

In addition, the ease of mold separation, texture, adhesion capacity, flexibility, homogeneity, transparency and weakness of the edges were subjectively assessed. The separation property of the mold refers to not leaving part of the film in the Petri dish when it is removed with the tweezers, texture indicates that the film does not have lumps, adhesion capacity means completely detaching from the surface, flexibility refers to bending no cracking and edge weakness indicates sticky edges or Petri dishes with film residue on edge. These properties were measured from 1 to 10, with 10 being the highest and 1 being the lowest characteristic.

Attenuation modulus Fourier transform infrared spectroscopy (ATR-FT-IR) was used to identify the main functional groups and determine the interactions of nanocellulose in the film. Before measurement, the nanocellulose-reinforced starch membranes were dried at 40 °C for 2 h to remove moisture. ATR-FT-IR spectra were obtained with a Nicolet 6700 spectrophotometer (Thermo, USA) from 4000 to 400 cm^{-1} with an ATR diamond crystal. The measurements were made with a resolution of 4 cm^{-1} and an accumulation of 64 measurements.

2.5. Statistical analysis

The analysis of the results is based on a factorial statistical design with the Statgraphics program (StatPoint Technologies Inc., 2010). In addition, the Statistical Package for Social Sciences (SPSS, v. 25.0, IBM Corp., 2017) was used. The Kolmogorov-Smirnov test was carried out to check the normal distribution of the results, and, if it did not exist, the non-parametric Mann-Whitney U test was applied since the random variables (ease of mold separation, texture, capacity of adhesion, flexibility, homogeneity, transparency, weakness of the edges when removing the film from the Petri dish) were considered as ordinal and were contrasted with two groups defined in each grouping variable (NC concentration, plasticizer, temperature and mixing time). The confidence interval was 95% ($p \leq 0.05$) for all cases.

Considering the categories of each grouping variable, two groups were formed, in which different random variables were contrasted. For this, hypotheses were formulated in each contrast (H_0: there are no differences between the groups in terms of the random variables and H_1: there are differences between the groups in terms of the random variables), working with a significance level of 5%, of such that with $p \leq 0.05$, H_0 would be rejected (it would be accepted that there are differences) and with $p > 0.05$, H_0 would not be rejected (homogeneity would be accepted).

3. RESULTS

3.1. Scanning electron microscopy

The solution containing 1.6% (± 0.2% standard deviation) of nanocellulose was milky white (Figure 1A). After drying, the fibers were obtained at a nanometric scale, resulting in a transparent material (Figure 1C).

Figure 2 shows the SEM images of the nanocellulose obtained by acid hydrolysis. The diameter of the cellulose nanofibers varied between 180 and 200 nm. Figure 2 shows the SEM images of the nanocellulose obtained by acid hydrolysis. These images reveal a characteristic morphology of cellulose nanofibers, with diameters ranging between 180 and 200 nm. Acid hydrolysis is a commonly used method to decompose cellulose into nanofibers, resulting in high purity and size uniformity in the obtained products. This process involves breaking chemical bonds in the cellulose structure through the action of acids, which leads to the formation of nanofibers with unique properties and a wide range of applications in fields such as the materials industry and biotechnology. The controlled dimensions of these nanofibers are of particular interest to numerous applications, such as reinforcements in composite materials, filter membranes, and cell culture substrates.

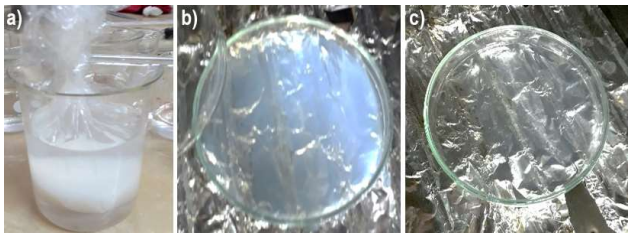

Figure 1. Nanocellulose. a) Dialysis; b) Nanocellulose sample during drying; c) Nanocellulose sample after drying.
Figura 1. Nanocelulose. a) Diálise; b) Amostra de nanocelulose durante a secagem; c) Amostra de nanocelulose após secagem.

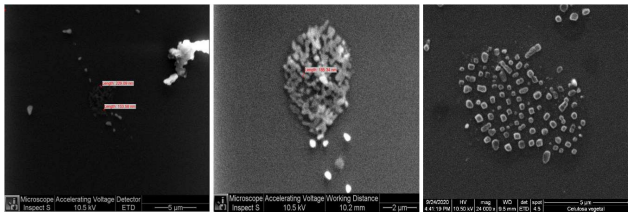

Figure 2. Scanning electron micrographs of cotton nanocellulose obtained by acid hydrolysis.
Figura 2. Micrografias eletrônicas de varredura de nanocelulose de algodão obtidas por hidrólise ácida.

3.2. FTIR

Nanocellulose fibers comprise cellulose, whose elements are similar to the constituents of starch and glycerin. Figure 3 shows the spectra of the cotton nanocellulose and the film with nanocellulose. The latter spectrum includes the band signals of nanocellulose with a less pronounced peak difference at 1626.1 cm^{-1}. In the film, more peaks were observed between 847.9 and 1626.1 cm^{-1}, which could be due to the inclusion of starch and glycerin.

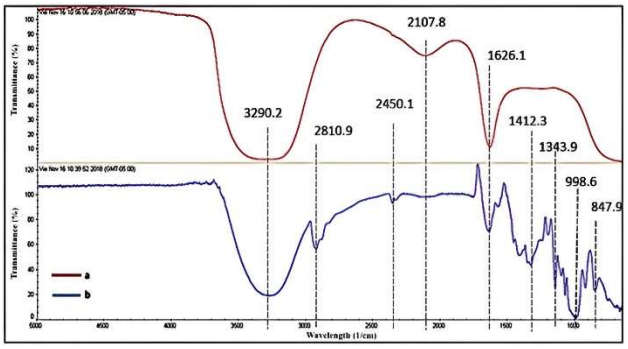

Figure 3. FTIR spectrum: a) Cotton nanocellulose and b) Film with nanocellulose.
Figura 3. Espectro FTIR: a) Nanocelulose de algodão e b) Filme com nanocelulose.

3.3. Film properties

According to Table 2, the elasticity with the highest value was 1.88 MPa, this is due to the inclusion of 25 ml of NC, 1 ml of plasticizer, 50 °C drying and 3 minutes of mixing. The highest value for tensile strength was 42.10 N, which corresponds to the inclusion of 25 mL of NC, 0.4 mL plasticizer, 50 °C drying temperature and 1 min of mixing.

The result of the greatest thickness was 0.1473 mm, which corresponds to the inclusion of 25 mL of NC, 0.4 mL plasticizer and 36 °C drying in 1 min of mixing. The highest solubility value was 22.71%, including 5 mL of NC, 0.4 mL plasticizer and 36 °C drying in 1 min of mixing. Regarding the solubility, the lowest value was 9.60%, corresponding to the inclusion of 5 mL of NC, 0.4 mL plasticizer and 36 °C temperature in 3 min of mixing.

In Figure 4A-C, the inclusion of NC influences the properties of elasticity, tensile strength and thickness, while it had no major effect on solubility. However, factors such as the combination of nitrocellulose, plasticizer and temperature influence the final humidity of the film. Figure 5 shows the relationships between the plasticizer and the response variables. It shows a proportional relationship between plasticizer and solubility (Figure 5D); while elasticity, maximum strength, thickness and moisture increase, the plasticizer decreases (Figures 5A, 5B, 5C and 5E).

Table 3 summarizes the influence of NC inclusion on the physical characteristics by analyzing the subjectively measured properties, showing the p values of the Mann-Whitney U test. There is no significant difference between the inclusion percentages. Five percentage points would be enough for smooth, transparent, flexible, non-cracked and homogeneous films. The plasticizer, drying temperature and mixing time influenced the physical characteristics and presented significant differences between treatments, except inhomogeneity, where there was no difference.

Table 2. Data and response variables.
Tabela 2. Dados e variáveis de resposta.

Nanocellulose (mL)	Plasticizer (mL)	Temperature (ºC)	Mixing time (min)	Humidity (%)	Thickness (mm)	Solubility (%)	Elasticity (MPa)	Tensile strength (N)
5	0.4	36	1	39.87	0.0626	22.71	0.92	32.07
5	0.4	36	3	25.44	0.0556	9.60	0.98	36.10
25	0.4	36	1	17.09	0.1473	11.18	0.67	40.63
25	0.4	50	1	16.30	0.0606	20.10	1.24	42.10
25	0.4	36	3	24.01	0.0487	18.36	1.26	24.64
5	1.0	36	3	23.46	0.06012	22.51	0.10	10.54
25	1.0	50	3	19.35	0.0949	21.41	1.88	35.09
5	0.4	50	3	18.23	0.06234	20.34	0.03	17.15
5	0.4	50	1	17.98	0.06212	20.89	0.05	15.67
25	1.0	50	1	25.25	0.0929	20.21	0.45	13.13
25	1.0	36	3	23.06	0.0906	16.78	0.73	22.63
5	1.0	36	1	24.83	0.0649	20.69	0.10	20.88
25	0.4	50	3	30.70	0.1153	20.73	0.68	32.35
5	1.0	50	1	30.08	0.0897	21.21	0.56	13.85
5	1.0	50	3	27.10	0.06	15.00	0.61	10.97
25	1.0	36	1	22.95	0.0946	10.26	0.80	28.85

Table 3. Results of the non-parametric Mann-Whitney U test.
Tabela 3. Resultados do teste não paramétrico U de Mann-Whitney.

Grouping variables	Ease of mold separation	Structure	Texture	Adhesion capacity	Flexibility	Homogeneity	Transparency	Edge weakness	Integrity
Nanocellulose concentration	0.252	0.611	0.442	0.744	0.897	0.399	0.729	0.404	0.622
Plasticizer	0.547	0.021	0.111	0.191	0.000	0.747	0.945	0.027	0.000
Drying temperature	0.000	0.904	0.697	0.002	0.738	0.144	0.006	0.424	0.073
Mixing time	0.869	0.357	0.013	0.157	0.158	0.694	0.008	0.699	0.858

4. DISCUSSION

In the edible film, the interactions between the NC and the identification of the functional groups were possible by using FTIR spectroscopy. Using this technique, PULIDO et al. (2016) propose an increase in intensity in most bands due to the increase in crystallinity. Furthermore, for 3290.2 $cm^{-1,}$ the spectral bands corresponding to the intramolecular O-H stretching of cellulose, the peak at 2810 cm^{-1} due to the stretching of the C-H bond, the bands at 1626 cm^{-1} correspond to C=O vibrations, while the bands 1412.3 and 1343.9 cm^{-1} correspond to CH_3 bonds and the peaks at 998.6 and 847.9 cm^{-1} are related with the vibrations and stretching of the C-O-C group, which coincides with the literature (ZARA et al., 2017). According to YU et al. (2017), when incorporating NC into the starch films, the transmittance relationship was found between the peaks at 3290.2 and 1626.1 cm^{-1}, considered an important parameter to show the intensity of the hydrogen bond of cellulose.

Increasing the amount of NC (from 5 to 25 mL) generally decreased the solubility and elasticity of the films. This suggests that higher NC concentrations can increase the material's stiffness. Using more plasticizers (from 0.4 to 1.0

ml) increased the solubility and elasticity but decreased the tensile strength of the films. An increase in temperature (from 36 to 50 ºC) generally favored greater tensile strength. A longer mixing time (3 min compared to 1 min) often resulted in thinner, less elastic films.

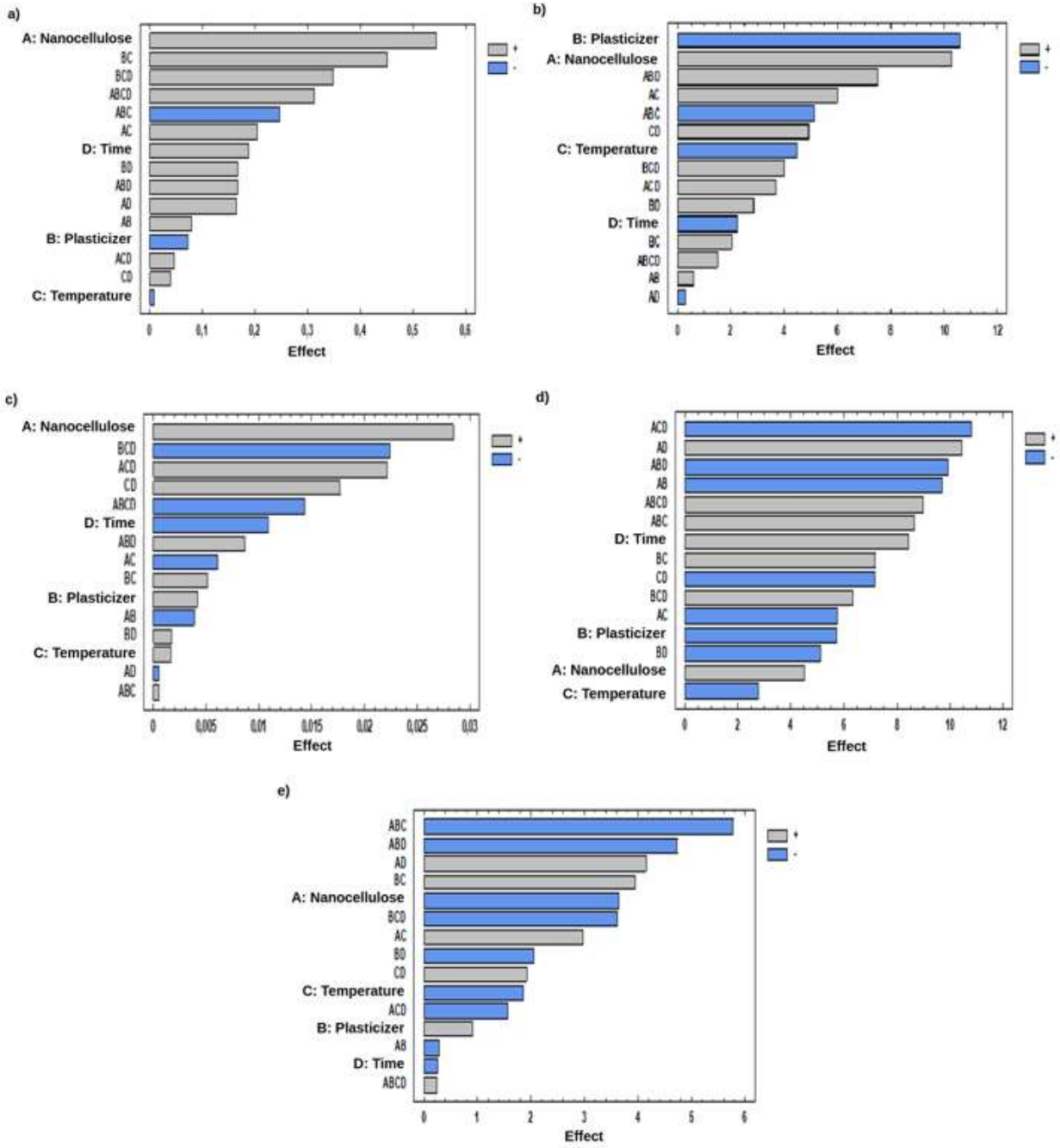

Figure 4. Pareto diagrams: a) Elasticity, b) Maximum strength, c) Thickness, d) Solubility and e) Humidity.
Figura 4. Diagramas de Pareto: a) Elasticidade, b) Resistência máxima, c) Espessura, d) Solubilidade e e) Umidade.

NC influenced the elasticity, and according to Gray et al. (2018), adding 2% of NC gave 10% in the elastic modulus compared to the control sample. However, with 1% NC, they gave optimal values to the elastic modulus with a value of 8.5 MPa, compared to that of the present work at 5% NC was 0.98 MPa. This means that the higher the percentage of NC, the lower the elasticity values, improving the films' tensile strength. This could be due to the presence of hydroxyl groups and the formation of hydrogen bonds for better reinforcement of the final compounds (MEESORN et al., 2017; LOMELÍ-RAMÍREZ et al., 2018). According to De Andrade et al. (2019) and Bagde; Nadanathangam (2019), incorporating NC can have a negative influence by making the material more rigid. However, according to De Andrade et al. (2019), it increases the mechanical resistance of the materials, and Bagde; Nadanathangam (2019) show a further increase in fragility in the film. With higher NC, the fragility of the biocomposite increases, and its flexibility decreases (TIMHADJELT et al., 2015; YU et al., 2017). Variability in elasticity and tensile strength was observed between different experimental conditions, highlighting the importance of optimizing process parameters to obtain desired properties.

Humidity significantly impacted the thickness of the films and, therefore, their mechanical properties, with higher levels of humidity associated with thinner films. The thickness of the films presented a value of 0.1473 mm, lower than those obtained by Bagde; Nadanathangam (2019) (0.184 mm) and higher than those reported by Jiménez et al. (2019) (~0.040 mm) for films without NC, which demonstrates the influence of the inclusion of NC on the increase in film thickness.

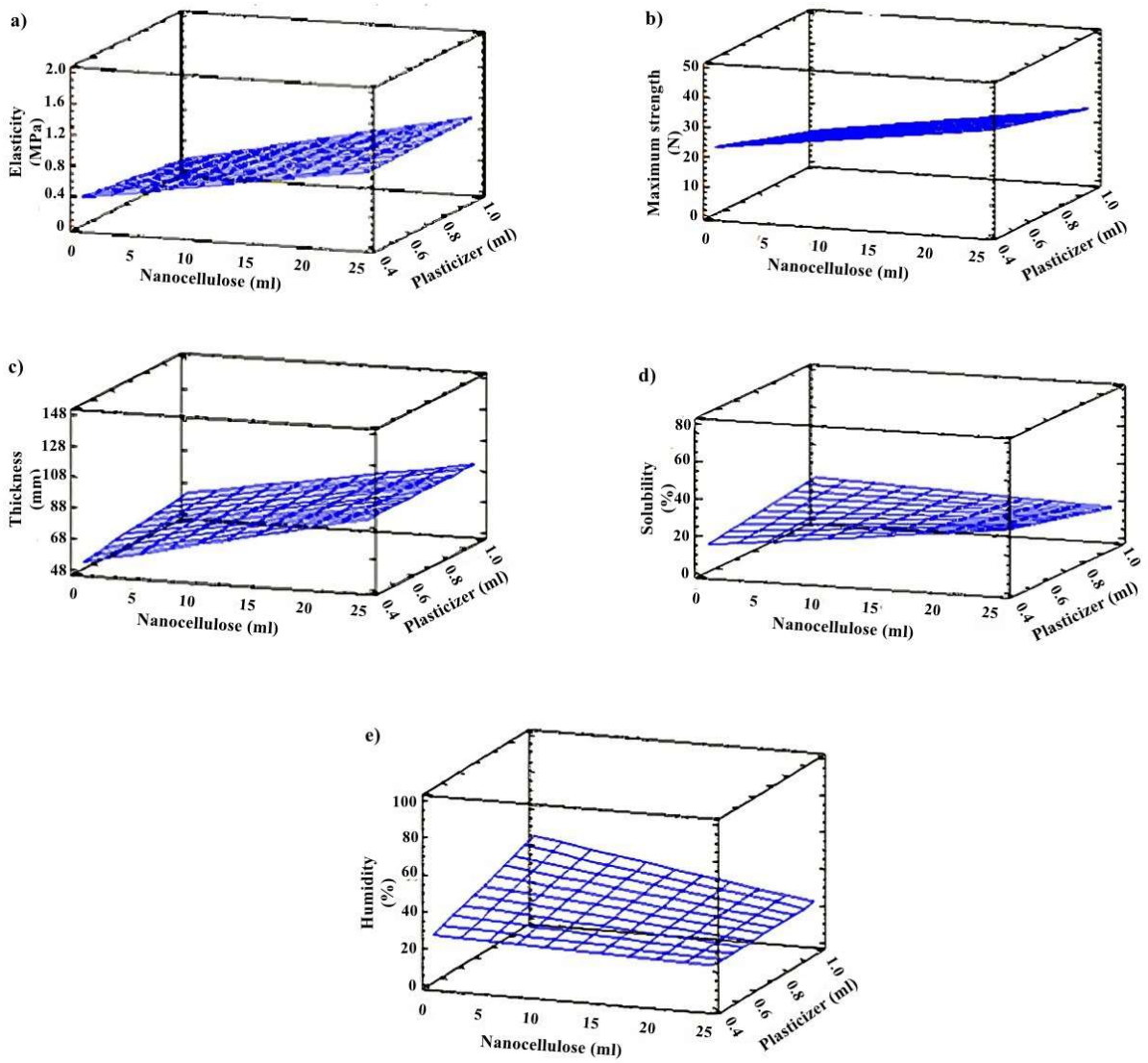

Figure 5. Relationship of the plasticizer with the different variables: a) Elasticity, b) Maximum strength, c) Thickness, d) Solubility, and e) Humidity.
Figura 5. Relação do plastificante com as diferentes variáveis: a) Elasticidade, b) Resistência máxima, c) Espessura, d) Solubilidade e e) Umidade.

In general, lower solubility indicates greater resistance of the material to dissolution in water, which may be desirable for applications where dimensional stability and resistance to moisture are sought (ALMEIDA et al., 2023). The optimal value of solubility of the film was 22.71%, referring to those proposed by Slavutsky; Bertuzzi (2014), which were between 18.5 and 26.6%, due to the addition of NC, which reduces the solubility of the film as it is indicative of strong interactions between the starch chains and the NC through hydrogen bonds with the hydroxyl groups of the NC (RHIM et al., 2007). For Bagde; Nadanathangam (2019), the solubility of the films was 36% after the incorporation of NC in corn starch films. However, in starch-chitosan films, there was no change in solubility with the addition of wood pulp NC at 2.8% by weight (YU et al., 2017).

Some studies have evaluated the solubility in starch films with nanocellulose. Sousa et al. (2023) found that adding nanocellulose and buriti oil decreased the solubility of cassava films compared to the control. The water solubility of corn starch films decreased with the addition of NC. The lowest values were for films containing 1% NC compared to films without NC. Some works have reported that adding nanocomposites can reduce the water solubility of starch-based films (PELISSARI et al., 2017; SOLTES DE ALMEIDA et al., 2020).

According to Mahardika et al. (2019), increasing the NC loading in the film with jicama (*Pachyrhizus erosus*), starch significantly reduced the moisture absorption value by approximately equal to 20%, and for Syafri et al. (2019), the humidity value was 22% in biocomposites with water hyacinth (*Eichhornia crassipes*) starch with the inclusion of NC

jicama (*Pachyrhizus erosus*), values lower than those indicated in this work. Including NC improved the films' transparency (SCHMIDT; MALWITZ, 2003; NOBBMANN; MORFESIS, 2009).

Given that the percentage in the NC test solution is 1.6%, its inclusion would be 0.4% (25 mL NC) and 0.08% (5 mL NC), indicating its great influence.

5. CONCLUSIONS

Using nanocellulose as a reinforcement in biodegradable corn starch films intended for food packaging has positively impacted their properties. Incorporating nanocellulose has significantly improved the films' elasticity, tensile strength and other physical characteristics. These changes translate into an increase in the maximum resistance and a reduction in the solubility of the films, suggesting their greater durability and functionality as packaging. These results support the viability and promise of biodegradable nanocellulose-reinforced corn starch films as a sustainable and effective alternative for food packaging.

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