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**Effect of SiO<sub>2</sub> nanoparticles in the physicochemical, mechanical, and structural properties of sweet potato starch edible films**

**Efecto de las nanopartículas de SiO<sub>2</sub> en las propiedades fisicoquímicas, mecánicas y estructurales de las películas comestibles de almidón de camote**

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**Abstract**

This study aimed to evaluate the effect of SiO<sub>2</sub> nanoparticles on the mechanical, structural, and physicochemical properties of sweet potato (*Ipomoea batatas*) starch edible films. Two edible films were made, films without SiO<sub>2</sub> nanoparticles (EF), and films with SiO<sub>2</sub> nanoparticles (DEF). The tensile strength for EF and DEF was 23.79 MPa and 24.65 MPa, respectively, and the elasticity for EF and DEF was 1.53% and 1.13%, respectively. The water vapor permeability, solubility, and water absorption for EF were  $2.2 \times 10^{-9}$  g/m·s·Pa, 19.52% and 4.51%, while for DEF they were  $1.7 \times 10^{-9}$  g/m·s·Pa, 18.19%, and 3.03%, respectively. According to FTIR results, the SiO<sub>2</sub> nanoparticles increased the short-range crystallinity of starch and reduced water vapor permeability without affect its mechanical properties.

*Keywords:* solubility, permeability, elasticity, tensile strength, crystallinity.

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**Resumen**

Este estudio tuvo como objetivo evaluar el efecto de nanopartículas de SiO<sub>2</sub> en las propiedades mecánicas, estructurales y fisicoquímicas de películas comestibles de almidón de camote. Dos formulaciones de películas fueron preparadas, películas sin nanopartículas de SiO<sub>2</sub> (FE) y películas con nanopartículas de SiO<sub>2</sub> (DEF). La resistencia a la tracción para EF y DEF fue 23.79 MPa y 24.65 MPa, respectivamente y la elasticidad para EF y DEF fue 1.53% y 1.13%, respectivamente. La permeabilidad al vapor de agua, solubilidad y absorción de agua para EF fueron  $2.2 \times 10^{-9}$  g/m·s·Pa, 19.52% y 4.51%, en cambio para DEF fueron  $1.7 \times 10^{-9}$  g/m·s·Pa, 18.19% y 3.03%, respectivamente. De acuerdo con los resultados de FTIR, las nanopartículas de SiO<sub>2</sub> incrementaron la cristalinidad a corto alcance del almidón sin afectar las propiedades mecánicas y reduciendo la permeabilidad al vapor de agua.

*Palabras clave:* solubilidad, permeabilidad, elasticidad, resistencia a la tracción, cristalinidad.

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## 1 Introduction

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Currently, it is possible to find many studies on the preparation of nanocomposite biodegradable and edible films, which can be used for food packages. Edible films are thin layers that provide an impediment to mass transfer (oxygen, moisture, and solutes movement) between the environment and the food (Gutiérrez & Álvarez, 2017). So, the application of coatings and edible films to food products packing can prevent aroma loss, moisture loss, solute transport, oxygen penetration, and water absorption (Cazón *et al.*, 2017). Starch is a popular biopolymer used as a material to produce edible films due to its abundance in nature, low cost, and biodegradability (Gadhav *et al.* 2018).

Sweet potato (*Ipomoea batatas*) is an important economic crop in Asia, Africa, and Latin America. It is rich in starch, dietary fiber, provitamin A, iron, and minerals, and is used as an energy source in the human diet. Sweet potato cultivars exhibit differences in size and skin color. This variety in color is due to the difference in the content of phenolic compounds and pigments in root tuber (Guo *et al.*, 2019; Wang *et al.*, 2018). Sweet potato is a respiration climacteric tuber crops and approximately 10-15% of all harvested sweet potato cannot be used because they are perishable during storage (Wang *et al.*, 2020).

Sweet potatoes contain large amounts of starch (over 60% of dry weight) (Wang *et al.* 2020). However, it is known that starch edible films have higher permeability to water vapor, poor mechanical and thermal properties, which reduces their application in food packaging (Zhao *et al.*, 2020). So, it is necessary to search for new materials that may be added to starch films for improving their structural, mechanical, and physicochemical properties. In this way, the inorganic nanoparticles are an option to keep exceptional interfacial interactions in edible films, and significantly improve their properties, since nanometric dimension of nanoparticles may improve the distribution and compactness of polymeric structure (Da Silva *et al.*, 2018).

Silicon dioxide nanoparticles (Nano-SiO<sub>2</sub>) is an amorphous powder which owns a three-dimensional net molecular structure that can be used to modify properties of different polymers such as starch, because it has small size, high energy, hydroxyl groups on the surface, unsaturated chemical bonds and large specific surface area (Tang *et al.*, 2009). These

nanoparticles have been synthesized by different techniques such as sol-gel, Stöber, modified Stöber and microemulsion, with spherical morphologies, hollow spheres, tubes and fibers of structurally amorphous nano-SiO<sub>2</sub> (Wu *et al.*, 2013). Nano-SiO<sub>2</sub> can form hydrogen bonds that bind to the polymeric matrix due to the silicon dioxide nanoparticles have a large specific surface area and numerous substituted hydroxyl groups on the surface. Between the uses, nano-SiO<sub>2</sub> are added to organic polymers like starch to improve their mechanical properties, water resistance, chemical, and thermal stability (Yang *et al.*, 2016). These were employed into chitosan-based coatings due to their low price, non-toxicity, and potential enhancement of microbial abilities. Is also known, that nano-SiO<sub>2</sub> are widely used to extend shelf life and inhibit fruit decay or employed as safe additives in medical devices and biomaterials (Tian *et al.*, 2019).

On the other hand, nano-SiO<sub>2</sub> is stable, compared with other types of nanoparticles, furthermore, non-toxic and it is an additive that have been widely used in food processing and preservation (Yu *et al.*, 2012), and the consumption of silicon dioxide nanoparticles ranging between 30 and 200 nm present in various food products was estimated to be 1.8 mg/kg per day (Dekkers *et al.*, 2011). The addition of nanoparticles enhances the mechanical strength, gas barrier, heat resistance, and antimicrobial properties of edible films. However, changes in short-range crystallinity when SiO<sub>2</sub> nanoparticles are added to edible films have not previously been determined. So, in this work the objective was to evaluate changes in short-range crystallinity when SiO<sub>2</sub> nanoparticles are added to the edible films and its influence in the mechanical and physicochemical properties of these.

## 2 Materials and methods

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SiO<sub>2</sub> nano-powder with a specific surface area 175-225 m<sup>2</sup>/g was purchased from Sigma Aldrich, Mexico. Sweet potatoes were purchased from market of Xalapa, Veracruz, Mexico.

### 2.1 Extraction of sweet potato starch

Sweet potato starch was obtained according to Akintayo *et al.* (2019). First, the sweet potatoes were washed and peeled, then they were ground with distilled water in a conventional blender for 5 min and filtered, after that, the liquid mixture was placed in the

refrigerator overnight, then, the sediment was frozen and dried using a lyophilizer (Labconco FreeZone 1, USA) for two days at a pressure of 0.05 mbar. Finally, the dried sediment was crushed in a mortar and sieved through a 80  $\mu\text{m}$  mesh.

## 2.2 Preparation of edible films

Edible films of sweet potato starch (EF) and edible film of sweet potato starch with  $\text{SiO}_2$  nanoparticles (DEF) were elaborated using the same methodology described by Ballesteros-Mártinez (2020) with some modifications, but in the case of DEF was added  $\text{SiO}_2$  nanoparticles (1% with respect to the dry base of starch) to the sweet potato starch solution. First, a 3% solution of sweet potato starch were prepared and stirred at 85°C for 20 min. After, glycerol was added (15% with respect to the dry base of starch) and stirred for 10 more min. Finally, 10 ml of this suspension was dried in Petri dishes (internal diameter: 8.3 cm) at 28°C for 28 h. Moisture content was determined by a thermobalance (OHAUS MB200, Germany) and films were conditioned at 65% relative humidity before to carry out the analytical determinations.

## 2.3 Physicochemical analysis

### 2.3.1 Thickness and transparency

Thickness was determined using a Yosoo digital micrometer  $\pm 0.001$  mm. Transparency (T) was determined according to Bao *et al.* (2009), which consisted of measuring the absorbance at 550 nm ( $A_{550\text{nm}}$ ) using a Spectrophotometer (Thermo Scientific UV-Vis GENESYS 10S, Uruguay) and then transparency (T) was obtained using equation 1.

$$T = \frac{A_{550\text{nm}}}{x} \quad (1)$$

Where  $x$  is thickness.

### 2.3.2 Water solubility

The Bertuzzi (2007) method was used to determine water solubility (S). Film sample was immersed in 40 ml of distilled water for 24 h at room temperature and maintaining gentle agitation. Water was drained and film sample was dried at 105°C to constant weight, the water solubility (S) was evaluated using the equation 2.

$$S = \frac{W_i - W_f}{W_i} \times 100 \quad (2)$$

Where  $W_i$  is the initial weight of sample and  $W_f$  is the final weight of sample after dried.

### 2.3.3 Absorption capacity of water

Absorption capacity of water was determined according to Tang *et al.* (2009). A 30 mm  $\times$  50 mm film sample was weighed and immersed in water at 25°C during 10 min. After, drained and weighted. The water absorption capacity ( $W_a$ ) was calculated as:

$$W_a = \frac{W_1 - W_0}{W_0} \times 100 \quad (3)$$

Where  $W_0$  is the initial weight of film sample and  $W_1$  is the final weight of film sample after drained.

### 2.3.4 Water vapor permeability

Water vapor permeability was determined in three relative humidity gradients ( $\Delta\text{RH}$ : 67, 47 y 25%). In this work, the ASTM E96 (ASTM, 1995) method reported by Bertuzzi *et al.*, (2007) was used. WVP was determined as shown below:

$$\text{WVP} = \frac{G \cdot x}{A \cdot \Delta p} \quad (4)$$

In this case, G is the slope estimated from the linear regression plot of weight variation over time (kg/s),  $\Delta p$  is the vapor pressure gradient between the sides of the film (Pa), A represent the area ( $\text{m}^2$ ), and x is the thickness (m).

## 2.4 Determination of mechanical properties

A tensile test at 0.5 mm/s was performed using a Texturometer (Stable Micro Systems, TA.XTplusC, UK), where the films were placed in 30 mm  $\times$  60 mm strips. Tensile strength (ST) and percentage elongation (EAB) were calculated using the following formulas given by Dong *et al.* (2022):

$$\text{ST}(\text{MPa}) = \frac{F}{h \times W} \quad (5)$$

Where F is the stress at break of the film sample (N); h is the thickness (mm) and W represent the width (mm).

$$\text{EAB}(\%) = \frac{l_b - l_0}{l_0} \times 100 \quad (6)$$

Where  $l_b$  is the elongation measured at break (mm);  $l_0$  represent the initial length of the sample (mm).

## 2.5 Determination of structural properties

### 2.5.1 Environmental Scanning Electron Microscopy (ESEM)

The surface morphology of the edible films was analyzed by an environmental scanning electron microscope (Elecmi, SEM- Quanta FEG 250, The Netherlands), operated at 5.0 kV voltage and 1000× magnification.

### 2.5.2 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared spectra of edible films were obtained using an infrared spectrometer (IlliminatIR II JY Smiths Detection coupled to an optical microscope OLYMPUS BX41, Japan). Samples were analyzed using the total reflection (ATR) method. All samples were analyzed at a resolution of  $4\text{ cm}^{-1}$  for 32 consecutive scans. Short range crystallinity was determined following the method reported by Van Soest *et al.* (1995). To extract information on the molecular organization of the edible films components, the spectrum was deconvoluted using Lorentzian and Gaussian functions.

## 2.6 Statistical analysis

Statistical analysis of data was made by the Student's t test to evaluate the means between the two groups with a 0.05 significance level, using the SigmaStat 3.5 software.

## 3 Results and discussion

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### 3.1 Thickness and transparency

The thickness of EF and DEF were  $0.045 \pm 0.012$  mm ( $45 \pm 0.12\mu\text{m}$ ) and  $0.046 \pm 0.016$  mm ( $46 \pm 0.16\mu\text{m}$ ), respectively. The transparency was  $3.4 \pm 0.08$  for EF and  $2.89 \pm 0.04$  for DEF. There was not significant difference between results of thickness, while the transparency was different in both films ( $p < 0.05$ ). The measure of thickness and transparency is important because they can influence in the determination of their functionality and acceptability by consumers. Thickness of EF and DEF was lower than others starch films reported by different authors; for example, López- García *et al.* (2017) made starch films with nopal mucilage obtaining a thickness range

of 176 to 368  $\mu\text{m}$ , while López-Chavez *et al.* (2017) made nanocomposites films of starch with organoclay obtaining  $91 \pm 2\mu\text{m}$  thickness, also they reported a small increase in the thickness of starch films when the organoclay increase. In this work, the addition of  $\text{SiO}_2$  nanoparticles increased the thickness, too, but this difference was not statistically significant. Regarding transparency, De Azevedo *et al.* (2021) reported that the transparency values of all corn starch-based films with the addition of silica powder were lower than those observed in the control bioplastic. Similar results were obtained in this work, because of DEF presented less transparency than EF.

### 3.2 Solubility in water and water absorption

Water solubility is an essential property in starch-based packaging, this indicates the film's integrity in an aqueous medium, where higher solubility represents lower water resistance (Calderón-Castro *et al.*, 2022). Solubility in water and water absorption showed significant differences ( $p < 0.05$ ) between both films. Solubility in water of films were 19.52 % and 18.19% for EF and DEF, respectively. In this work,  $\text{SiO}_2$  nanoparticles in the sweet potato starch films caused a reduction in their solubility (approximately 1.33%) and in their water absorption. There are many studies that use different inorganic material to decrease the interaction of hydrophilic groups of starch with the water, such as Rammak *et al.* (2021), who had high solubility values in their cassava starch films (44.87%) due to the hydrophilic property of starch, so they added kaolin and observed a decrease in solubility about 8% (33.20%). Piñeros-Guerrero *et al.* (2021) analyse the incorporation of montmorillonite clays (MM), and polycaprolactone (PCL) to a matrix of thermoplastic cassava starch (TPS). The addition of polycaprolactone and montmorillonite reduced the water solubility of the films by up to 24.2%. Torabi & Mohammadi (2013) obtained similar values to this work, who obtained 20% solubility in their potato starch films and 18% solubility in the films with silicon dioxide nanoparticles. Water absorption of EF and DEF was 4.51% and 3.03%, respectively. So, the incorporation of  $\text{SiO}_2$  nanoparticles in the film matrix decreased the water absorption capacity, too. This is due to the interactions between  $\text{SiO}_2$  and other compounds in its structure showing a greater resistance to water, this due to the obstruction of the microparticles to free water diffusion in the film matrix. Salazar *et al.* (2019) mentioned that the

incorporation of silicon dioxide nanoparticles (SiO<sub>2</sub> NPs) in pectin-based edible films improved their moisture barrier compared to their control films, giving the possibility that the hydroxyl groups in the surface of the SiO<sub>2</sub> nanoparticles can form hydrogen bonds with the hydrophilic groups of the pectin chains, reducing the amount of water sorption sites. So, in this work, the SiO<sub>2</sub> nanoparticles caused an increase in electrostatic interactions between these and the polymeric matrix that decreased the interaction between free water molecules and the starch; and the moisture content of the DEF was higher than in the EF because silicon dioxide particles have a nanoporous surface that promoted better adsorption of water vapor from the environment and therefore a higher moisture content.

### 3.3 Water vapor permeability

It is widely known that polysaccharides and proteins have ability to form films, but although they can act as effective barriers to gas transport (O<sub>2</sub> and CO<sub>2</sub>), they have a high water vapor permeability (Dehghani *et al.*, 2018). The results obtained for EF and DEF of water vapor permeability are shown in Figure 1. The water vapor permeability of DEF was lower than that of EF in the three  $\Delta RH$  studied. Rammak *et al.* (2021) evaluated the water vapor permeability (WVP) applying the ASTM method E96/E96M-10 in their cassava starch films with kaolin microparticles and obtained statistically significant differences up to the addition of 10% in weight of kaolin microparticles. They concluded that the improvement was derived from the tortuous pathway caused for the microparticles that inhibited the transport of free water through the starch structure. WVP value reported by these authors was  $3.5 \times 10^{-5}$  g/m·day·Pa ( $9 \times 10^{-9}$  g/m·s·Pa), which is a higher value obtained for the DEF films in this work. Similarly, Torabi & Mohammadi (2013) reported a higher WVP value ( $5 \times 10^{-7}$  g/m·s·Pa) than those obtained in this work, they made potato starch films with SiO<sub>2</sub> nanoparticles, in which they observed that the nanoparticles decreased WVP due to the fact that nanoparticles fill the pores in the structure of the macromolecules in the matrix. Spatafora *et al.* (2019) made edible films with SiO<sub>2</sub> nanoparticles, using orange and mango pectin, they evaluated the water vapor permeability by ASTM E96-00 method and observed in their results that the incorporation of nanoparticles improved this barrier property, giving the possibility that the hydroxyl groups on the

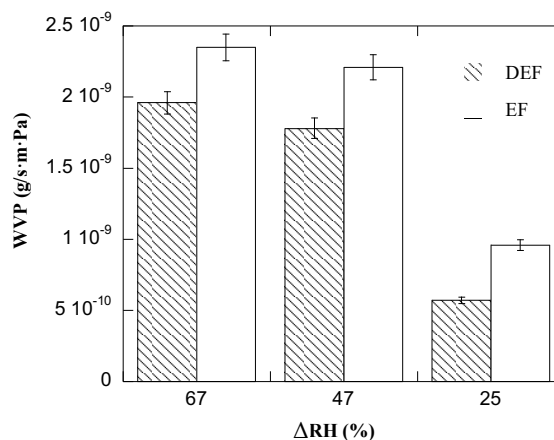


Figure 1. Water vapor permeability of sweet potato starch film (EF) and sweet potato starch film with SiO<sub>2</sub> nanoparticles (DEF) at 25°C.

surface of the SiO<sub>2</sub> nanoparticles could form hydrogen bonds with the hydrophilic groups of pectin chains, reducing the number of water sorption sites. Similar results reported Acosta-Domínguez *et al.*, (2021), who made sweet potato starch films using alginate microparticles, they attributed the decreased of WVP to effect of micropores present in the surface of alginate microparticles. When the molecules permeate through the matrix, a part of these water molecules are diffused through the particles and are adsorbed into the micropores, causing that less water transfer through the film. On the other hand, Santana & Kieckbusch (2013), who made alginate films with Ca<sup>2+</sup> particles, explained that the lower permeability values are due to greater, stronger and better intramolecular structuring or organization caused by the crosslinking of the polymeric structure with the Ca<sup>2+</sup>. Dong *et al.*, (2022) described that the increase of SiO<sub>2</sub> nanoparticles concentration in biodegradable chitosan film caused a decrease in its permeability, and they explained that the nanoparticles could occupy the spaces of the film matrix promoting a dense tridimensional network and improving diffusion of water molecules on the surface. In the present work, the effect of the addition of SiO<sub>2</sub> nanoparticles to the films on the intermolecular structure was verified with the FTIR study on short-range crystallinity (Fig. 5), where it is clearly observed that the crystallinity of DEF was higher than that of EF, which implies a better intramolecular organization with stronger bonds between starch and nanoparticles, this coupled with the porous nature of SiO<sub>2</sub> nanoparticles caused a decrease in the permeability of water.

Table 1. Mechanical properties of EF and DEF.

Sample	Tensile strength (MPa)	F Max (N)	E (%)	Young modulus (kg/mm <sup>2</sup> )
EF	23.79±0.55 <sup>a</sup>	18.89±1.25 <sup>b</sup>	1.53±0.10 <sup>c</sup>	0.0469±0.003 <sup>d</sup>
DEF	24.65±0.70 <sup>a</sup>	20.20±0.97 <sup>b</sup>	1.13±0.32 <sup>c</sup>	0.0484±0.002 <sup>d</sup>

Values are expressed as means ± standard deviation ( $n = 3$ ). Values in the same column followed by different superscript letter are significantly different according to T student test at  $p < 0.05$ .

### 3.4 Mechanical properties

The addition of SiO<sub>2</sub> nanoparticles did not change the mechanical properties of both edible films (Table 1), which is considered a desirable result, since generally the addition of inorganic particles causes an increase in their hardness and rigidity, making them brittle and with a lower percentage of elongation. Similar results presented De Azevedo *et al.* (2021), who showed that the addition of silica powder in corn and potato starch bioplastics does not contribute to the increase in tensile strength. However, the reinforcing effect of the addition of inorganic microparticles in films has been reported by several authors, where the tensile strength increases, but the percentage of elongation decreases. Acosta-Domínguez *et al.* (2021) made edible films of sweet potato starch with calcium alginate microparticles and determined the maximum breaking strength, observing that the incorporation of the microparticles resulted in a decrease in tensile strength, since less force was needed to break them ( $F_{max}=4.5N$ ). Rammak *et al.* (2021) elaborated edible films of cassava starch with microparticles of kaolin 10% and determined the elongation at break and tensile strength, obtaining results of 2.99 MPa and 96.57%, respectively. In films without microparticles obtained 0.64 MPa and 146.63%, respectively. Torabi & Mohammadi (2013) performed a tensile test on their potato starch films with SiO<sub>2</sub> nanoparticles, in their results observed that the addition of nanoparticles of up to 5% by weight increased Young's modulus and tensile strength, whereas elongation at break decreased. So, the reinforcing effect of SiO<sub>2</sub> nanoparticles caused the reduction of mobility of the starch chains, which increased rigidity and fragility. Sornsumdaeng *et al.*, (2021), evaluated the mechanical properties of rice starch films reinforced with CaO particles, attributed this effect to due to esterification reactions and crosslinking in the polymer matrix. In this work, the mechanical properties of ED and DEF did not present significant differences due to the possible formation of intermolecular interactions such

as hydrogen bonds and Van der Waals forces. These intermolecular interactions among the nanoparticles and the polymer matrix kept the structure together and prevented that the polymer matrix lose mechanical strength, as shown in the Table 1 and FTIR results. So, the addition of SiO<sub>2</sub> to the sweet potato edible films is recommended because the WVP can be reduced without affecting its mechanical properties.

### 3.5 Environmental scanning electron microscopy (ESEM)

Surface structure and nanoparticles distribution in the edible films was analyzed by ESEM, the micrographs are shown in the Figure 2. The adhesive face of EF and DEF (letter A and B, respectively) shows a homogeneous and smooth surface, but the image of DEF shows a random distribution of SiO<sub>2</sub> in the matrix. In the Figure 2, letter C and D (EF and DEF, respectively) show the adhesive surface opposite, where it is possible to see roughness in some areas and certain points with agglomerations of nanoparticles because they occupied the pores found on the surface. Also, DEF shows less empty starch granules (commonly called "ghosts") than EF in its structure. The results obtained are similar to those found by Garcia-Diaz *et al.* (2016), who added different concentrations of CaCO<sub>3</sub> to samples of gelatinized corn starch, observing a disruption of these ghosts as the concentration of CaCO<sub>3</sub> particles increased. Dong *et al.* (2022), analyzed the morphology of chitosan-based biodegradable films with and without nanoparticles of silicon dioxide-gallic acid, in the scans they can observe a smooth and homogeneous surface in the films without nanoparticles. Concerning the films with nanoparticles, it can be observed rough patches with more densely shape on the surface of the composite films, this is indicative of the formation of a cross-linking network structure between the chitosan polymer matrix and the nanoparticles. Rammak *et al.* (2021), analyzed the adhesive face of the cassava starch film without kaolin microparticles, which showed homogeneity and smoothness, while the films

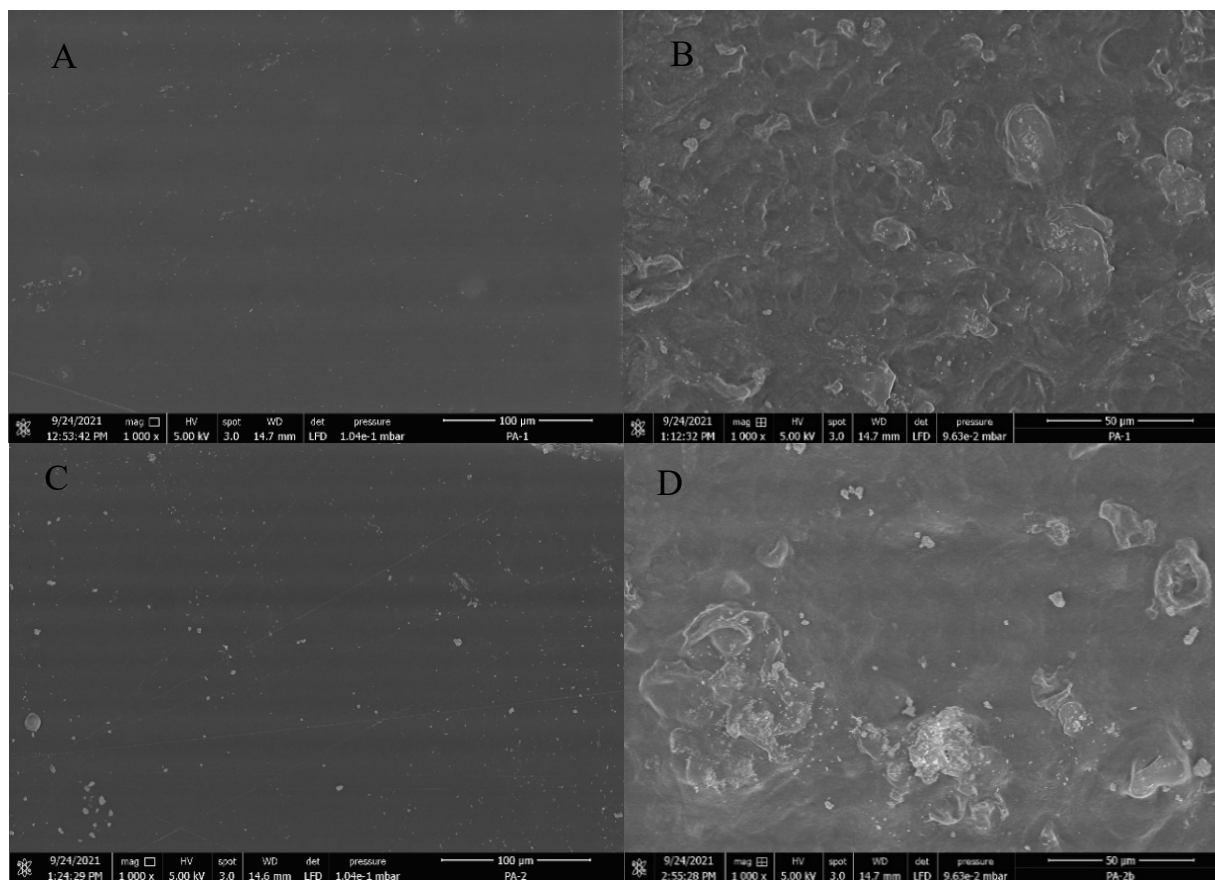


Figure 2. ESEM images of the adhesive and anti-adhesive surface. A-B: sweet potato starch film (EF) and C-D: sweet potato starch film with SiO<sub>2</sub> nanoparticles (DEF).

with kaolin microparticles showed an absence of holes or cracks due to the good dispersion and embedding in the starch structure.

### 3.6 Fourier Transform Infrared Spectroscopy

Figure 3 shows FTIR spectra of EF and DEF, where starch films presented IR main signals in the range of 1300-1000, 1475-1450, 3000-2800, and 3500-3250 cm<sup>-1</sup>, associated with C-O-C stretching, O-H bending, stretching of -CH<sub>2</sub>-, C-H asymmetric and O-H stretching, respectively (Bower & Maddams., 1996). The formation of new absorption peaks was not observed, which indicates that the addition of the nanoparticles did not involve the formation of new chemical bonds, only changed some electrostatic interactions confirmed by the shifts of the absorption bands observed in the spectra. Dong *et al.* (2022) did not find new peaks by the adding of SiO<sub>2</sub> nanoparticles in the spectra of their alginate-based

films, but the N-H/O-H stretching bands shifting

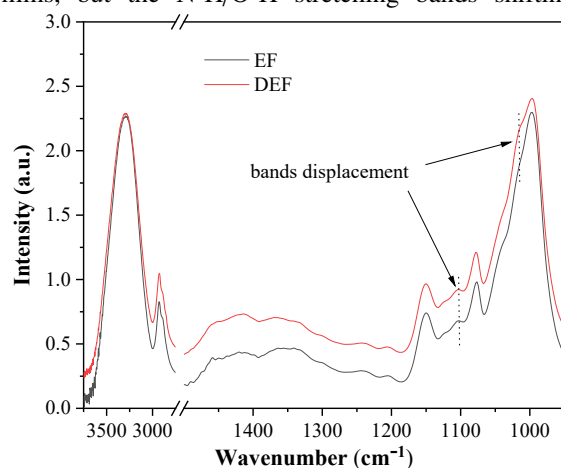


Figure 3. FTIR spectra of sweet potato starch film (EF) and sweet potato starch film with SiO<sub>2</sub> nanoparticles (DEF).

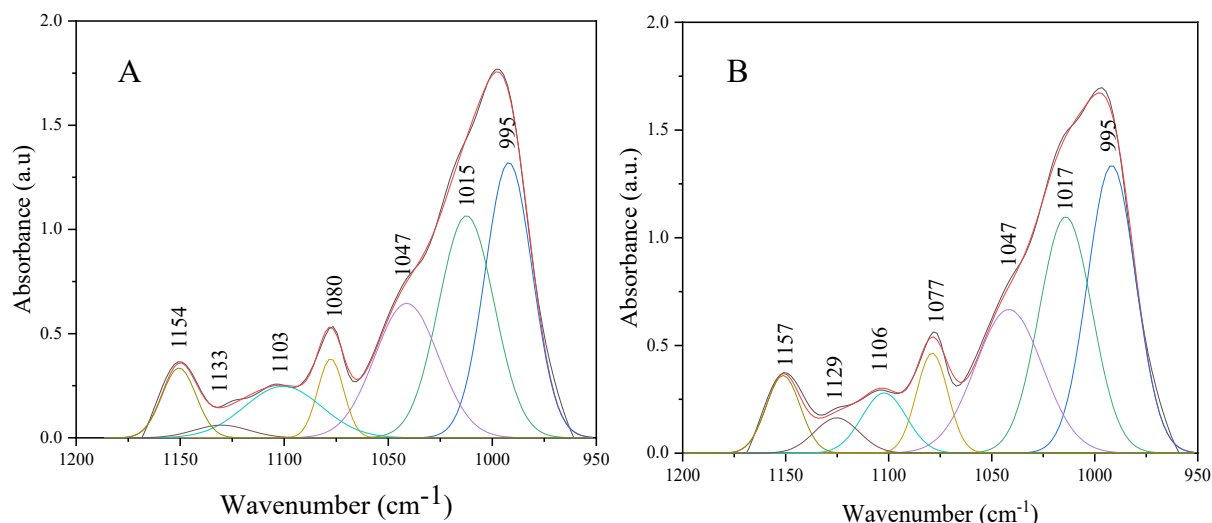


Figure 4. Deconvoluted FTIR. A: sweet potato starch film (EF) and B: sweet potato starch film with SiO<sub>2</sub> nanoparticles (DEF).

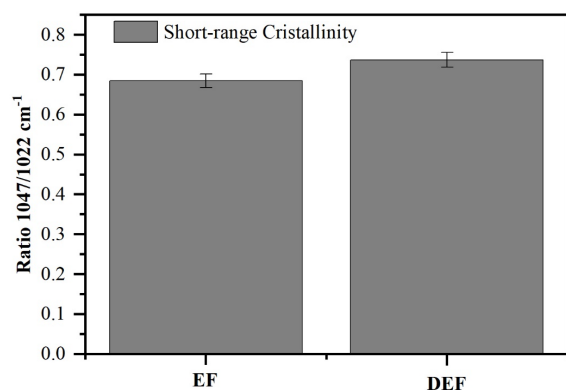


Figure 5. Short range crystallinity of sweet potato starch film (EF) and sweet potato starch film with SiO<sub>2</sub> nanoparticles (DEF). Statistical analysis was performed using paired Student t-test. P values 0.05 were considered significant.

at lower wavenumbers, as results of intermolecular hydrogen interactions between the nanoparticles and the alginate. Instead, Rammak *et al.*, (2021) analyzed the FTIR spectra of cassava starch edible films with and without kaolin microparticles. FTIR spectra presented a broad peak around 3280 cm<sup>-1</sup>, this signal was a characteristic peak related to the stretching of hydroxyl groups in the spectra of films without kaolin, which was shifted to higher wavenumbers in the spectra of the film composites. Deconvoluted FTIR spectra of EF and DEF are shown in Figure 4. Figure 4 B present the band shifts of the absorption peaks for DEF, slight displacements of absorption peaks

were observed at positions 1015, 1080, 1133, and 1154 cm<sup>-1</sup>, Stuart (2004), cited that band broadening and wavenumber shifts are indicative of chemical interactions among other components in a blend which implies changes in intermolecular interactions. In the case of the 1080 cm<sup>-1</sup> frequency, this absorption band corresponding to the C-O vibration (Jiménez *et al.*, 2014) and it was found in EF (Fig. 4 A), but in DEF it was displaced to 1077 cm<sup>-1</sup> (Fig. 4 B), which indicates higher stretching vibration between the C-O bonds according to the harmonic oscillator model. This theory mention that the molecular interaction formed by the bonds of diatomic molecules becomes stronger by changing the position of the peak to a lower wave number (Ahmet & Ilberg, 2016). However, the higher stretching vibration between the C-O bonds could occur in the starch polymer or between nanoparticles and the starch polymer. On the other hand, in this work the electrostatic interaction could be present between the positive charges of SiO<sub>2</sub> and the negative dipole of hydroxyl groups in starch film because the incorporation of the particles caused a change in the C-H asymmetric stretching of -CH<sub>2</sub>- and C-O-C stretching peak in the ranges 3000-2800 and 1300-1000 cm<sup>-1</sup>, respectively. Similar results reported Sornsumdaeng *et al.* 2021 in rice starch films with CaO. Figure 5 shows the 1047/1022 ratio of EF and DEF, where it is clearly observed that the edible films with SiO<sub>2</sub> had a higher value. Generally, the increase of 1047/1022 ratio determined from different starch sources is related to



the short-range order of double helices and relative crystallinity of starch microstructure (Van Soest *et al.*, 1995; Wang *et al.*, 2015; Garcia-Diaz *et al.*, 2016; Li *et al.*, 2018; González *et al.*, 2021). In this way, the results shows that the nanoparticles of SiO<sub>2</sub> produces starch fractions with a more ordered structure (González *et al.*, 2021). Alvarez-Ramirez (2019) studied the addition of Ca<sup>2+</sup> ions to improve the mechanical performance of starch films, the 1047/1022 ratio increased as a result of molecular interactions and structural modifications in starch chains caused through cross-linking with bivalent ions of Ca<sup>2+</sup>, authors mentioned that these interactions improved the short-range ordered starch structures and mechanical properties of starch films. In the present work, the increase of 1047/1022 ratio could be on line with it reported (Garcia-Diaz *et al.*, 2016; Alvarez-Ramirez *et al.*, 2019) where starch chains and bivalent ions, in this case Si<sup>2</sup>, promote an enhanced ordering of the starch microstructure in the edible films, with evident changes in the mechanical and physicochemical properties.

## Conclusion

The addition of SiO<sub>2</sub> nanoparticles to the sweet potato edible films increased the crystallinity indicating an improvement in intramolecular organization and changes in the electrostatic interactions between starch and SiO<sub>2</sub>, which decreased the water vapor permeability, solubility in water, and water absorption capacity and maintained the mechanical properties.

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