



FOOD ECO-COVERING: CHARACTERIZATION OF BIODEGRADABLE FILMS MADE WITH STARCH FROM ANDEAN *Oxalis tuberosa*

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ABSTRACT

Objective: This research focused on the characterization of biodegradable films made from nopal (*Opuntia ficus-indica*) mucilage and oca (*Oxalis tuberosa*) starch to cover food.

Theoretical Framework: This study is grounded in the growing environmental concerns surrounding conventional plastics and the urgent need for sustainable alternatives in the food industry. The research builds upon the emerging concept of circular economy, focusing on biodegradable materials derived from underutilized agricultural resources.

Method: Nine biodegradable films were prepared by thermoforming and dried at 40, 50, and 60 °C. Their physical properties and degradation versus temperature were evaluated by thermogravimetry, and structural identification was carried out by infrared (IR) spectroscopy.

Results and Discussion: The films exhibited a water solubility more significant than 91%. Water activity ranged between 0.402 and 0.573, correlating with the percentage of humidity (<25.3%). The starch and pectin content influenced these properties, as well as transparency ($T < 2.08$). Thermogravimetric analysis revealed that the films reached the highest mass loss at around 300 °C. Finally, the IR analysis demonstrated the relationship between the components and the new molecular arrangement of the films, influenced by the concentrations of oca starch.

Research Implications: It provides significant implications for sustainable food packaging and agricultural waste utilization, offering a potential solution to reduce plastic waste in the food industry. The findings suggest that local agricultural products can be transformed into value-added materials with environmental benefits.

Originality/Value: The study offers unique value through its comprehensive characterization and its potential for promoting local agricultural resources in sustainable packaging solutions.

Keywords: Biodegradable Films, *Oxalis Tuberosa* Starch, FTIR, TGA.

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ECOCOBERTURAS ALIMENTARES: CARACTERIZAÇÃO DE FILMES BIODEGRADÁVEIS FEITOS COM AMIDO ANDINO *Oxalis tuberosa*

RESUMO

Objetivo: Esta pesquisa focou na caracterização de filmes biodegradáveis para cobrir alimentos, feitos de mucilagem de palma (*Opuntia ficus-indica*) e amido de oca (*Oxalis tuberosa*).

Referencial Teórico: Este estudo é fundamentado nas crescentes preocupações ambientais em torno dos plásticos convencionais e a urgente necessidade de alternativas sustentáveis na indústria alimentícia. A pesquisa se baseia no conceito emergente de economia circular, concentrando-se em materiais biodegradáveis derivados de recursos agrícolas subutilizados.

Método: Nove filmes biodegradáveis foram preparados por termoformagem, secos a 40, 50 e 60 °C. Suas propriedades físicas, bem como a degradação dos filmes versus temperatura, foram avaliadas por termogravimetria e a identificação estrutural foi realizada por espectroscopia no infravermelho (IV).

Resultados e Discussão: Os filmes apresentaram solubilidade em água superior a 91%. A atividade de água variou entre 0,402 e 0,573, correlacionando-se com a porcentagem de umidade (<25,3%). O conteúdo de amido e pectina influenciou essas propriedades, bem como a transparência ($T < 2,08$). A análise termogravimétrica revelou que os filmes atingiram a maior perda de massa em torno de 300 °C. Por fim, a análise de IV demonstrou a relação entre os componentes e o novo arranjo molecular dos filmes, influenciado pelas concentrações de amido de oca.

Implicações da Pesquisa: fornece implicações significativas para embalagens alimentícias sustentáveis e utilização de resíduos agrícolas, oferece uma potencial solução para reduzir o desperdício de plástico na indústria alimentícia. Os resultados sugerem que produtos agrícolas locais podem ser transformados em materiais de valor agregado com benefícios ambientais.

Originalidade/Valor: O estudo oferece valor único através de sua caracterização abrangente e seu potencial para promover recursos agrícolas locais em soluções de embalagem sustentáveis.

Palavras-chave: Filmes Biodegradáveis, Amido de *Oxalis tuberosa*, FTIR, TGA.

ECOCUBIERTAS DE ALIMENTOS: CARACTERIZACIÓN DE PELÍCULAS BIODEGRADABLES ELABORADAS CON ALMIDÓN DE *Oxalis tuberosa* ANDINA

RESUMEN

Objetivo: Esta investigación se centró en la caracterización de películas biodegradables para cubrir alimentos, elaboradas con mucílago de nopal (*Opuntia ficus-indica*) y almidón de oca (*Oxalis tuberosa*).

Marco Teórico: Este estudio se fundamenta en las crecientes preocupaciones ambientales en torno a los plásticos convencionales y la urgente necesidad de alternativas sostenibles en la industria alimentaria. La investigación se basa en el concepto emergente de economía circular, enfocándose en materiales biodegradables derivados de recursos agrícolas subutilizados.

Método: Nueve películas biodegradables fueron preparadas por termoformación, secadas a 40, 50 y 60 °C. Se evaluaron sus propiedades físicas, así como también la degradación de las películas frente a la temperatura por termogravimetría y la identificación estructural se realizó mediante espectroscopía de infrarrojo (IR).

Resultados y Discusión: Las películas exhibieron una solubilidad en agua superior al 91%. La actividad del agua osciló entre 0,402 y 0,573, correlacionándose con el porcentaje de humedad (<25,3%). El contenido de almidón y pectina influyó en estas propiedades, así como en la transparencia ($T < 2,08$). El análisis termogravimétrico reveló que las películas alcanzaron la mayor pérdida de masa alrededor de 300 °C. Finalmente, el análisis de IR demostró la relación entre los componentes y el nuevo arreglo molecular de las películas, influenciado por las concentraciones de almidón de oca.

Implicaciones de la Investigación: son significativas para empaques alimentarios sostenibles y utilización de residuos agrícolas ofreciendo una solución potencial para reducir los desechos plásticos en la industria alimentaria.



Los hallazgos sugieren que los productos agrícolas locales pueden transformarse en materiales de valor agregado con beneficios ambientales.

Originalidad/Valor: El estudio ofrece un valor único a través de su caracterización integral y su potencial para promover recursos agrícolas locales en soluciones de empaque sostenibles.

Palabras clave: Películas Biodegradables, Almidón de *Oxalis tuberosa*, FTIR, TGA.

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1 INTRODUCTION

Growing concern about the environmental impact of conventional plastics has driven the search for sustainable alternatives in the food industry (Cazón et al., 2017; González-Seligra et al., 2017). Global plastic production reached approximately 368 million metric tons in 2019, with packaging accounting for 40 % of plastic demand, highlighting the urgent need for ecological solutions (PlasticsEurope, 2020; Geissdoerfer et al., 2017). Single-use plastics pose significant environmental challenges, contributing to pollution, marine ecosystem degradation, and long-term ecological damage (United Nations Environment Programme [UNEP], 2018; Yokota et al., 2017).

In this context, biodegradable films made from usable resources with very little economic value, such as starch, have gained considerable attention due to their potential to reduce pollution caused by the use of plastics and promote the circular economy (García-Guzmán et al., 2022; Thakur et al., 2019). Biodegradable materials offer promising alternatives by providing environmentally friendly solutions that can decompose naturally, reducing carbon footprint and minimizing waste accumulation (Piao et al., 2024; Yu & Flury, 2024; Omer & Hassan, 2024; Song et al., 2009). These innovative materials not only address environmental concerns but also contribute to sustainable development goals by transforming agricultural by-products into value-added materials (Singh et al., 2024; Samir et al., 2022; Cristofoli et al., 2023; Karan et al., 2019).

Oxalis tuberosa, commonly known as oca in the Peruvian Andes region and other regions of South America, is an Andean tuber with a high starch content that has unique properties for making films (Fonseca-Santanilla & Betancourt-López, 2022; Chirinos et al., 2009; Estrada-Fernández et al., 2021). Research has demonstrated that Andean tubers possess complex molecular structures and unique chemical compositions making them excellent candidates for biodegradable film production (Pico et al., 2022). Likewise, it is important to



value the use of nopal, a food widely consumed in various regions of America (Sáenz et al., 2004); however, in Peru, its consumption is limited to producing its fruit. This underutilization represents a significant opportunity for innovative material development and optimizing sustainable agricultural resources (González-Sandoval et al., 2019; Fortunati et al., 2019; Wikström et al., 2019).

This study focuses on characterizing biodegradable films made with *Oxalis tuberosa* starch, exploring their potential as food eco-covering. The research addresses the analysis of physical and thermogravimetric properties and the identification of functional groups, contributing to the growing body of knowledge in sustainable packaging materials (Choque-Quispe et al., 2021; Fakhouri et al., 2012; Torrenegra et al., 2018).

2 THEORETICAL FRAMEWORK

2.1 OXALIS TUBEROSA

It is known as oca, and it is cultivated from Argentina to Venezuela (Ugent & Ochoa, 2006). It is grown at altitudes between 2,000 and 4,000 meters (Fernandez & Rodríguez, 2007). It is notable for its hardiness, tolerance to adverse climates, low incidence of pests and diseases, and ease of agronomic management, with no chemicals used during production (National Research Council, 1989). Additionally, it is considered economically important due to its carbohydrates, minerals, and oxalate content (Talledo & Escobar, 1995).

2.2 STARCH

It is a complex carbohydrate consisting of two similar polysaccharides: amylose and amylopectin. It exhibits a sophisticated granular organization that varies significantly depending on its botanical origin. This natural polymer's structure and properties are fundamental to its functionality in various applications, including film formation and food preservation (Cowieson, 2005).

2.3 NOPAL MUCILAGE (*Opuntia ficus-indica*)

A highly branched fibrous polysaccharide with the capacity to form molecular networks and retain large quantities of water while modifying the physicochemical properties of



substances it interacts. Its complex structure enables it to function as a natural hydrocolloid with significant applications in food science and technology (Gibson & Nobel, 1990).

2.4 FOOD ECO-COVERING

These materials, derived from renewable natural resources such as starches, proteins, and polysaccharides, naturally decompose in the environment, significantly reducing ecological impact (García-Guzmán et al., 2022; Thakur et al., 2019). Their development addresses critical environmental challenges and promotes the valorization of local agricultural resources and the advancement of circular economies (Karan et al., 2019). Biodegradable films represent a sustainable innovation in the packaging industry (Cazón et al., 2017).

3 METHODOLOGY

3.1 RAW MATERIAL

The *Oxalis tuberosa* was cultivated in Sucaraylla at 3641 meters of altitude located in José María Arguedas district at coordinates 13° 44' 02'' S 73° 21' 01'' W. For starch extraction, the decantation methodology described by (Melian, 2010) was used with some modifications and adapted for the oca samples. In the case of the extraction of the nopal mucilage, the proposal made by (Orozco, 2017) was used. The material was obtained in Talavera district (13° 39' 23'' S 73° 25' 29'' W).

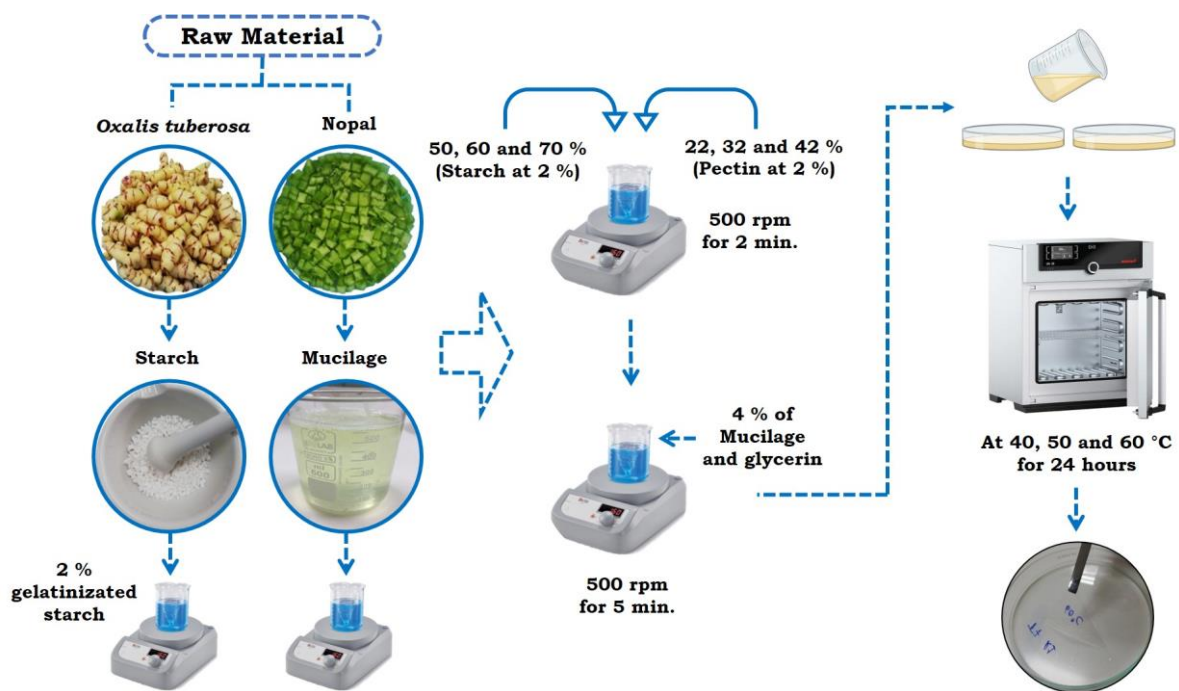
3.2 PREPARATION OF BIODEGRADABLE FILM

For the preparation of biodegradable films, nine treatments were formulated (T1-40, T2-40, T3-40, T4-50, T5-50, T6-50, T7-60, T8-60, and T9-60) at different starch concentrations (50, 60 and 70%) that were gelatinized at 2%. The pectin percentages were 22, 32, and 42%, prepared at 2%. They were thermoformed at temperatures 40, 50, and 60 °C for 24 hours. The mucilage concentration (4%) was the same for all treatments. Glycerin was used as a plasticizer (4%), as shown in Figure 1.



Figure 1

Sample Preparation Flowchart



3.3 MOISTURE

This test was performed using the methodology (AOAC International, 2005). The films were cut to approximately 2 cm² and placed in aluminum foil containers. A Binder brand forced convection oven model FED 115 was used at 105 °C for 24 h.

3.4 TRANSPARENCY

The methodology proposed by (Oregel-Zamudio et al., 2016) was used. The absorbance of a film strip (1 x 4 cm) was determined at a wavelength of 600 nm in a UV-Vis spectrophotometric equipment brand Thermo Scientific, model Genesys 150. The absorbance values obtained were divided by the thickness of the film, and the transparency was calculated (abs.mm⁻¹).

3.5 SOLUBILITY (%S)

The method (Bertuzzi et al., 2007) was used with some modifications. The film samples were placed in a desiccator for one week to obtain the initial dry weight. The previously



weighed films were placed in 40 mL of deionized water at 25 °C for 24 h at 100 rpm in a Velp Scientifica brand agitator model F10230270. The remains that were not solubilized were removed by centrifugation and dried to obtain the final dry weight.

$$\% S = \frac{W_{si} - W_{sf}}{W_{si}} \times 100 \quad (1)$$

where:

W_{si} , initial dry weight

W_{sf} , final dry weight.

3.6 WATER ACTIVITY (a_w)

The a_w of the biodegradable films was evaluated through a Rotronic brand water activity analyzer model HP23-AW.

3.7 THERMOGRAVIMETRIC ANALYSIS (TGA)

The thermal stability of the biodegradable films was analyzed in a LINSEIS brand equipment model STA PT1600. The ASTM E1131-08-2014 standard was considered (ASTM International, 2014).

3.8 IR ANALYSIS

Infrared analysis was performed on a Thermo Fisher brand Nicolet iS50 323/5000 spectrometer. Pressed tablets were prepared with 0.1 % of the film in KBr and taken to the transmission module of the FT-IR spectrometer, which has a resolution of 4 cm⁻¹ and a range of 400 to 4000 cm⁻¹.

3.9 STATISTICAL ANALYSIS

The physicochemical properties were analyzed using analysis of variance (ANOVA) and Tukey's multiple range test at 5% significance.



4 RESULTS AND DISCUSSIONS

4.1 PHYSICAL CHARACTERIZATION

The results in Table 1 show that the humidity of the treatments ranges from 22.3 to 25.3 %. The T2-40 treatment is the one that showed the highest humidity; this phenomenon is because pectin has open structures with at least two hydroxyl groups that facilitate the bond with water (Galindez et al., 2019). The T9-60 treatment is the one that showed the lowest humidity content; this could be due to the interaction between the polymer chains and the water molecules, which allow the evaporation of water during the drying process (Damodaran et al., 2007). Similar results were obtained by Galindez et al., (2019) and Susmitha et al., (2021) in edible biofilms made from ulluco starch and corn starch-gelatin, respectively.

As for solubility, values were found in a range of 91.7 to 99.2 %. T4-50 treatment is the one that showed the highest solubility; this phenomenon is attributed to the amylose content in the starch because it does not retrograde (Vargas et al., 2016). In addition, it can be attributed to a higher content of hydrophilic groups (Fakhouri et al., 2012). Solubilities of 91.04% in cactus mucilage biofilms were reported by González Sandoval et al., (2019); however, Choque-Quispe et al., (2021) obtained values of up to 54.8% in edible biofilms based on native potato starch and nopal mucilage.

The transparency of the films presented values from 0.71 to 2.08 Abs.mm⁻¹. The T1-40 treatment presented the highest transparency with a 0.71 Abs.mm⁻¹ value. On the other hand, the T9-60 treatment showed the lowest transparency value, which was 2.08 Abs.mm⁻¹. It can be attributed to the fact that starch in higher concentrations is responsible for the increase in opacity due to its gelatinization process (Fakhouri et al., 2012); values from 1.06 to 1.55 in edible biofilms based on native cassava starch were reported by Galindez et al., (2019).

The a_w was found in a range of 0.401 to 0.573. T1-40 treatment presented the highest a_w with a value of 0.573. This fact is attributed to the availability of carbonyl and hydroxyl groups, as well as to the sugar residues of starch and mucilage that increase the available active hygroscopic sites (Choque-Quispe et al., 2021); Similar results were reported by Choque-Quispe et al., (2021) with values of 0.562 to 0.680 in edible biofilms.

**Table 1***Physical analysis*

Treatment	Moisture (%)		Solubility (%)		Transparency (Abs.mm ⁻¹)		Water activity (<i>a_w</i>)	
	$\bar{x}$	<i>SD</i>	$\bar{x}$	<i>SD</i>	$\bar{x}$	<i>SD</i>	$\bar{x}$	<i>SD</i>
T1-40	25.1 ^a ± 0.1		97.2 ^{ab} ± 1.6		0.71 ^f ± 0.03		0.573 ^a ± 0.005	
T2-40	25.3 ^a ± 0.4		93.9 ^{bc} ± 1.9		0.76 ^{ef} ± 0.06		0.478 ^c ± 0.003	
T3-40	24.0 ^{abc} ± 0.3		94.7 ^{bc} ± 2.3		1.05 ^d ± 0.09		0.443 ^d ± 0.006	
T4-50	24.6 ^{ab} ± 0.5		99.2 ^a ± 1.1		1.96 ^{ab} ± 0.12		0.531 ^b ± 0.007	
T5-50	24.4 ^{abc} ± 0.1		91.7 ^c ± 1.4		1.44 ^c ± 0.11		0.420 ^e ± 0.007	
T6-50	23.5 ^{bc} ± 0.3		93.4 ^{bc} ± 1.2		1.90 ^{ab} ± 0.05		0.418 ^e ± 0.006	
T7-60	23.5 ^{bc} ± 0.2		96.4 ^{ab} ± 1.1		0.98 ^{de} ± 0.01		0.449 ^d ± 0.008	
T8-60	23.1 ^{bc} ± 0.2		94.2 ^{bc} ± 0.7		1.73 ^b ± 0.15		0.408 ^{ef} ± 0.004	
T9-60	22.3 ^c ± 0.2		94.6 ^{bc} ± 0.3		2.08 ^a ± 0.08		0.402 ^f ± 0.003	

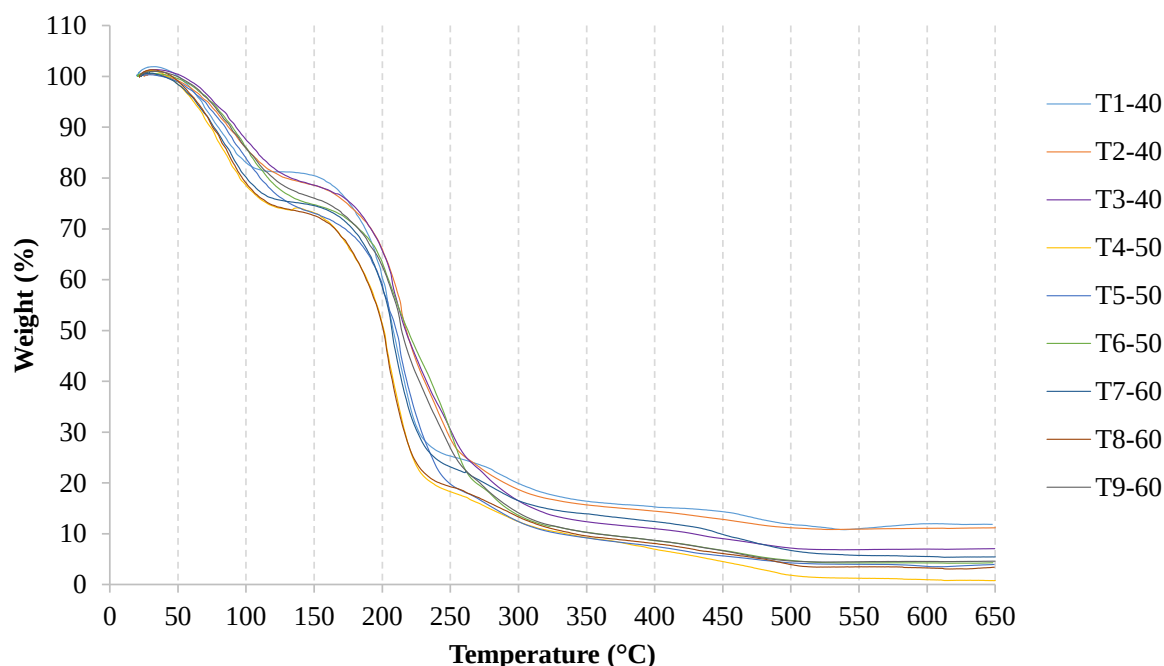
Note: $\bar{x}$, arithmetic mean; *SD*, standard deviation, and different letters indicate significant differences per row evaluated for triplicates at 5% significance.

4.2 THERMOGRAVIMETRIC ANALYSIS (TGA)

The thermograms are shown in Figure 1, and it can be seen that the formulations presented similar thermal behavior. From 50 °C onwards, mass degradation begins. It is mainly due to the evaporation of free and bound water (Badui Dergal, 2016). Likewise, it is observed that from 110 °C onwards. The mass remains slightly constant up to 165 °C, which implies that the polymeric structures of intermolecular bonds -C=O-, -OH, -COO-; of high molecular weight structures such as the polysaccharides present in the films have not yet degraded.

From 165 °C onwards, a greater mass degradation is observed up to approximately 300 °C. In this phase, organic compounds (Shafizadeh, 1971) such as simple sugars, including arabinose and galactose (Sáenz et al., 2004), are present in the chemical composition of the cactus; these sugars have melting points between 160 and 170 °C, and subsequently, their degradation begins. Similarly, carbohydrates present a mass decomposition in a temperature range of 200 to 400 °C.

In the third phase, it is observed that, from 300 °C, films suffer a slight and gradual loss of mass up to 500 °C. The loss of mass is attributed to the decomposition of high molecular weight components such as 1,4- α -D-galacturonic acid (GalpA) (Chasquibol-Silva et al., 2008) of the pectin, the IR spectrograms showed the presence of functional groups -COOH. Similar thermal degradation was observed by (Choque-Quispe et al., 2021) in biofilms made with potato starch.

**Figure 1***TGA Thermograms of the biodegradable films*

4.3 IR ANALYSIS

The IR spectra of the biodegradable films are shown in Figure 2, which shows that formulations presented intense bands of stretching vibration type at 3390 cm^{-1} , corresponding to the functional group -OH (de Jara et al., 2020). Similar spectra were reported by Choque-Quispe et al., (2021) in films made with potato starch. Methylene functional group $\text{-CH}_2\text{-}$ can be observed at 2930 cm^{-1} with an asymmetric stretching vibration type for all formulations.

Regarding the band located at 1650 cm^{-1} , it can be observed that the formulations T9-60, T7-60, and T4-50 presented a sharper peak, indicating a higher concentration of groups of the type -C=O- with a stretching vibration type. In addition, water H-OH is present with a bending vibration type.

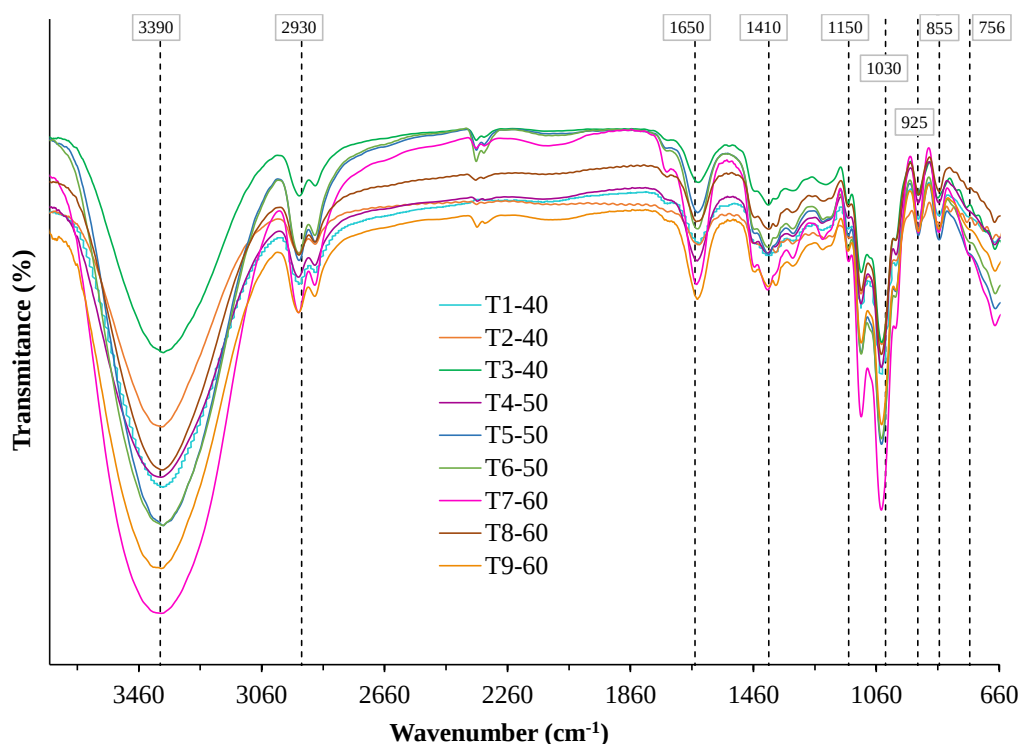
All treatments showed a slight peak at 1150 cm^{-1} , which is characteristic of the functional group C-O-C with a stretching type of vibration of the ether compound type. T7-60 treatment presented a peak of greater intensity corresponding to a structural vibration connected to an anomeric structure of D-glucose at 1030 cm^{-1} band. All treatments presented a low-intensity peak, which corresponds to the functional group -COOH in the band 925 cm^{-1} with a deformation type of vibration (Pérez-Vergara et al., 2020). Finally, in the 756 cm^{-1} band, all treatments present the C-O-C functional group with stretching vibration type, as reported by



(Choque-Quispe et al., 2021). Starch biofilms extracted from *Oxalis tuberosa* have similar functional groups (Pico et al., 2022), as well as spectra of films based on cassava starch (Torrenegra et al., 2018). The spectra of biofilms suggest the interactions between the different components.

Figure 2

IR spectrograms of the biodegradable films



5 CONCLUSIONS

Solubility is one of the most relevant physicochemical characteristics since they reached a solubility greater than 91%. They also had a water activity of less than 0.573 and a relatively low moisture content. The drying temperature and the amount of starch and pectin significantly influence transparency.

The treatments showed similar thermal behavior, with three main degradation phases: evaporation of water, decomposition of sugars and carbohydrates, and finally, decomposition of high-molecular-weight components such as pectin. The formulations containing 70% starch and 22% pectin showed the greatest mass loss.

The spectrograms showed a similar structural composition with a significant difference in the intensity of peaks in the different bands, which showed the relationship between the



components and the new molecular ordering of the films influenced by the oca starch concentrations.

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