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Rice flour-based films: effects of type and solvent and plasticizer concentration

Películas a base de harina de arroz: efectos del tipo y concentración de disolvente y plastificante

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Abstract

The use of rice flour as a polymeric matrix for the development of biodegradable films is an economical alternative to the use of rice starch. In the present work, the comparison of the type of solvent in films made with rice flour (3.15 g), was evaluated using glycerol as plasticizer (0.75, 1, 1.25, 1.5, 1.75, and 2 ml) and two solvents: water and 0.5M acetic acid. A final volume of 70 ml was reached in all formulations. The films obtained were characterized by thickness, solubility, permeability to water vapor, FTIR, Scanning Electron Microscopy (SEM), and mechanical tests. It was found that when using acetic acid as a solvent, it favorably affects all the characteristics of the films obtained, however, with increases in glycerol (> 1.25 ml) it was observed that the films showed minimal differences due to solvent effects; since glycerol is the one who determines the properties of the films.

Keywords: Flour, rice, films, glycerol, water, and acetic acid.

Resumen

La utilización de harina de arroz como matriz polimérica para el desarrollo de películas biodegradables es una alternativa económica respecto al uso de almidón de arroz. En el presente trabajo se evaluó la comparación del tipo de solvente en películas elaboradas con harina de arroz (3.15 g), utilizando glicerol como plastificante (0.75, 1, 1.25, 1.5, 1.75 y 2 ml) y dos solventes: agua y ácido acético 0.5M. En todas las formulaciones se alcanzó un volumen final de 70 ml. Las películas obtenidas fueron caracterizadas mediante: espesor, solubilidad, permeabilidad al vapor de agua, FTIR, Microscopía Electrónica de Barrido (SEM) y pruebas mecánicas. Se encontró que al utilizar ácido acético como solvente, éste afecta favorablemente todas las características de las películas obtenidas, sin embargo, con incrementos de glicerol (> 1.25 ml) se observó que las películas mostraron mínimas diferencias por efectos del solvente; pues el glicerol es quien determina las propiedades de las películas.

Palabras clave: Harina, arroz, películas, glicerol, agua y ácido acético.

1 Introduction

According to MINAM (2018), the presence of petroleum-derived plastics represents 10% of the total volume of all waste generated in Peru. The accumulation and persistence of synthetic plastics endanger the stability of the ecosystem, as well as basic services in public health, generating the need to develop new materials that partially replace some type of use of these synthetic materials.

Increasing consumer demand for safe, healthy, high quality, convenient foods with long shelf life has driven interests and advanced research activities in edible packaging in the food and packaging industries. (Janjarasskul *et al*., 2014). Fitch-Vargas *et al*., (2019) also mentions that the use of this type of coatings is a promising technology to maintain quality of fruits and vegetables.

Núñez-Gastelum *et al*., (2019) and López-Díaz *et al*., (2018) mentioned that the materials commonly used as polymeric matrix can be lipids, resins, proteins, and polysaccharides.

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Rice flour is a mixture of polysaccharides, proteins, and lipids, the presence of these would improve the functional characteristics of the films developed without the need to seek synergy using other polymers. Dias *et al*., (2011) states that making films with rice flour is cheaper than with commercial starches.

Starch is the main chemical component of a grain of rice, with an approximate 90% presence in the grain, as well as proteins and lipids are significant; having about 6.5% and 0.8% respectively. (Zhou *et al*., 2002). In addition, starch is the most important polysaccharide used as a polymeric matrix for the biodegradable packaging of food (López-García *et al*., 2017).

The addition of organic acids in films containing starch, generate a substitution of hydroxyl groups (hydrophilic) by ester groups (hydrophobic), modifying the behavior of the films, compared to those that use only water as a solvent (Ghanbarzadeh *et al*., 2011).

The high affinity of glycerol for water promotes the diffusion of molecules because glycerol is a small molecule that can easily be inserted between starch chains to form a hydrogen bond between amylose and amylopectin by acting as a plasticizer (Laohakunjit & Noomhorm, 2004).

The objective of this work is the study of the influence of two different solvents in the production of biodegradable plastics based on rice flour and the incorporation of glycerol in relation to their properties.

2 Materials and methods

2.1 Materials

Commercial rice flour (Tres Estrellas©) was purchased; acetic acid, glycerol, and Sodium Chloride from Sigma-Aldrich.

2.2 Film preparation

Rice flour films were prepared via casting, following the methodology described by Medrano de Jara *et al*., (2020), with some modifications, 3.15 g of rice flour was dissolved in 65 ml of the solvent (water or acetic acid 0.5M), with magnetic stirring (IKA® C-MAG HS7) for 20 min at 80 °C, Glycerol was added (0.75, 1, 1.25, 1.5, 1.75 or 2 ml for each treatment) and complete with solvent up to 70 ml, with magnetic stirring for 10 min more. After that, solutions were placed into an ultrasonic bath (Branson® 2510) for 30 min in order to homogenize the solutions and eliminate air bubbles. The prepared solutions were transferred to glass molds of 11x16 cm. The solutions were dried into the oven (Memmert SN55) for 15 h at 40 °C and finally, the films were stored in plastic bags at 28 °C. Each film was prepared in triplicate. All formulations are shown in Table 1.

2.3 Film Thickness

Film thickness measurements were carried out at 10 different positions of each film selected randomly, using a Digimatic micrometer IP65 MITUTOYO, model MDC-1 MJ, with an error range of ± 0.00005.

Table 1. Formulations used with 3.15 g of rice flour in 70 ml of filmogenic solution.

Formulation	Label	Glycerol	Water	Acetic Acid 0.5N
1	T-0.75G- AcAc	0.75 ml	0 ml	69.25 ml
2	T-1.00G- AcAc	1.00 ml	0 ml	69.00 ml
3	T-1.25G- AcAc	1.25 ml	0 ml	68.75 ml
4	T-1.50G- AcAc	1.50 ml	0 ml	68.50 ml
5	T-1.75G- AcAc	1.75 ml	0 ml	68.25 ml
6	T-2.00G- AcAc	2.00 ml	0 ml	68.00 ml
7	T-0.75G-W	0.75 ml	69.25 ml	0 ml
8	T-1.00G-W	1.00 ml	69.00 ml	0 ml
9	T-1.25G-W	1.25 ml	68.75 ml	0 ml
10	T-1.50G-W	1.50 ml	68.50 ml	0 ml
11	T-1.75G-W	1.75 ml	68.25 ml	0 ml
12	T-2.00G-W	2.00 ml	68.00 ml	0 ml

T= Treatment, G=glycerol, W=water & AcAc= Acetic Acid

2.4 Water solubility

Water solubility was determined according to ASTM standard method D570-98. Sample films with dimensions of 2 × 2 cm were weighed and placed into plastic vials, then 30 ml of distilled water was added and samples were kept at rest for 24 hours at room temperature. After the time, undissolved samples were removed and dried in an oven for 24 hours at 100 °C. Measurements were replicated three times and results were reported as the average. Water solubility was calculated with Eq. 1.

$$\%Solubility = \frac{W_I - W_F}{W_I} * 100 \quad (1)$$

where W_I was the initial weight of the films (g) and W_F was the final weight after drying.

2.5 Fourier-transform infrared spectroscopy (FTIR) analysis

Fourier transform infrared spectroscopy (FTIR) analysis was performed to analyze the chemical interaction of acetate, glycerol, and rice flour. Film samples were analyzed with an FTIR instrument (Perkin Elmer Spectrum Two with Spectrum® Software) with ATR accessory; 16 scans with 4 cm^{-1} resolution were used in the wavenumber range of 650 to 4000 cm^{-1}. The measurements were replicated twice.

2.6 Morphological structure

A scanning electron microscope (SEM) (JEOL, JSM-6010 model) with an accelerating voltage of 2 kV was used to investigate the microstructure and the surface morphology of the films using magnification 150 X.

2.7 Mechanical properties

The tensile strength and percentage of deformation at break were determined according to ASTM standard method D882-02. Sample films, with dimensions of 1 x 10 cm, were loaded in a universal testing machine (BlueHill Lite INSTRON, model 2519-107). The crosshead speed and initial distance were set to 0.5 mm/s and 5 cm respectively. Measurements were replicated three times and results were reported as the average.

2.8 Water vapor permeability (WVP)

The water vapor permeability (WVP) was determined according to ASTM method E96-00, with a few modifications. A circular section of each 8 mm diameter film was cut. The thickness of each sample was recorded and placed on the top of a 2 ml vial containing 1 ml of supersaturated potassium nitrate solution to generate a constant relative humidity. The vials were placed in a desiccator containing a humidity indicator and Silicagel as a drying agent. The test was performed in triplicate for each sample. Every hour, the mass of the cells was recorded using an analytical balance (± 0.00001 g) during 8 h. The slope of the straight lines, G/t, was calculated by linear regression and the water vapor permeability (WVP) was calculated according to equation 2.

$$WVP = \frac{\left(\frac{G}{t}\right)}{P_{sat}(RH_1 - RH_2)A} * z \quad (2)$$

where: G/t is the slope of the line of weight changes as a function of time, z is the thickness of the biofilm, A is the exposed area of the biofilm and P_{sat} is the saturation pressure at the working temperature. RH_1 and RH_2 represent the relative humidity of the supersaturated KNO_3 solution and the relative humidity of the environment inside the desiccator respectively.

2.9 Statistical analysis

All the results obtained in the tests were statistically treated using the software MiniTab 18 Statistical Software (Minitab Inc, State College, PA, USA); performing Analysis of Variance (ANOVA) to compare the solubility, water vapor permeability, and mechanical properties of the films, at a significance level of 0.05 (α = 0.05). The Tukey method was used to determine the significant differences in the composition of the films in relation to the parameters evaluated, except for the thickness.

3 Results and discussion

3.1 Film thickness

The thickness is subject to different factors: quantity and type of plasticizer, and the type of solvent. The thickness increases according to the increase of the amount of plasticizer within the mixture (Table 2), is

due to the amount of glycerol that increases the size of the starch molecule reflected in a new redistribution of the free volume of starch. However, this effect is a consequence of the high bond between plasticizer and polymer. Otherwise, when water is replaced by acetic acid as a solvent, this effect was inverse. According to the ANOVA test, there is a significant difference between the thicknesses of water or acid-based films for 95% confidence (p-value = 0.000).

3.2 *Water solubility*

The water solubility of the films is an important property to evaluate their probable application in food packaging. Figure 1 shows an increase proportional to the volume of plasticizer used for films developed in water and acetic acid. Due to the presence of more ester groups (acetate) in the films with acetic acid as a solvent, the solubility decreases. These substitutions made the films more hydrophobic, according to Ortega-Toro *et al*., 2014. The ANOVA performed, indicated there is a significant difference for all films at a 95% confidence level (p-value $<\alpha$).

3.3 *Water vapor permeability (WVP)*

It can be seen that the permeability of the films increases as the amount of glycerol increases (Figure 2), as a consequence of the hydrophilic nature of glycerol, easily forms hydrogen bonds with water molecules (Alves *et al*., 2007). Likewise, it can also be seen that the greatest increases in water permeability were obtained when the film contained water instead of acetic acid. Otherwise, the high affinity of glycerol for water promotes the diffusion of molecules (Laohakunjit & Noomhorm, 2004), the increase in glycerol concentration increases the WVP of the films, because, the presence of hydroxyl groups in each of the carbons of the glycerol molecule increases its hydrophilic character, favoring the adsorption of water vapor molecules (Galdeano *et al*., 2009).

Table 2. Film thicknesses based on 0.5N acetic acid and water.

Film	Thickness (mm)	Film	Thickness (mm)
T-0.75G-AcAc	0.1917 ± 0.0138^{d}	T-0.75G-W	0.2133 ± 0.0202^{d}
T-1G-AcAc	0.1910 ± 0.0122^{d}	T-1G-W	0.2183 ± 0.0181^{d}
T-1.25G-AcAc	0.2160 ± 0.0157^{c}	T-1.25G-W	0.2300 ± 0.0219^{d}
T-1.5G-AcAc	0.2373 ± 0.0282^{b}	T-1.5G-W	0.2483 ± 0.0189^{c}
T-1.75G-AcAc	0.2347 ± 0.0141^{b}	T-1.75G-W	0.2483 ± 0.0262^{b}
T-2G-AcAc	0.2807 ± 0.0242^{a}	T-2G-W	0.2963 ± 0.0275^{a}

*Different letters indicate significant differences ($p < 0.05$) for each kind of film.

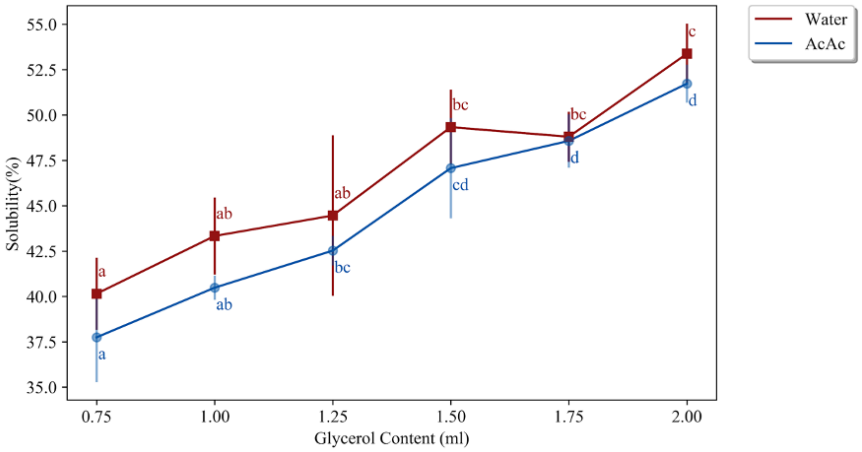

Fig. 1. Effect of solvent type on Solubility with respect to glycerol concentration. *Different letters indicate significant differences ($p < 0.05$) for each kind of film and solvent.

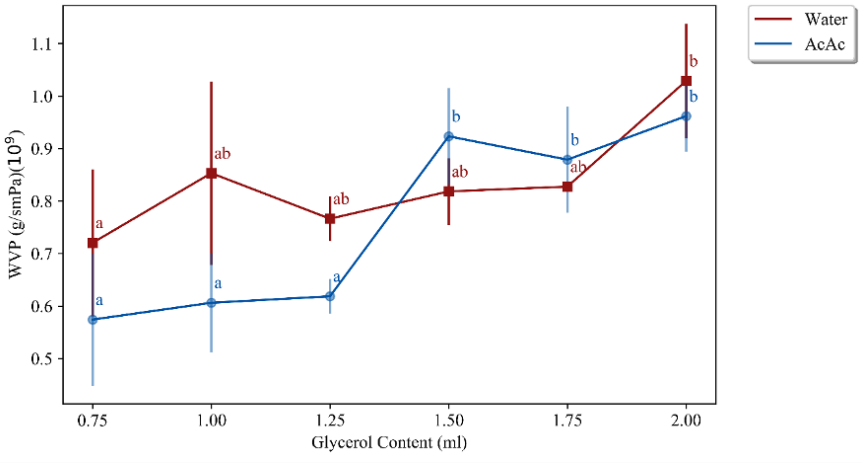

Fig. 2. Effect of solvent type on Water Vapor Permeability with respect to glycerol concentration. *Different letters indicate significant differences ($p < 0.05$) for each kind of film and solvent.

Table 3. Functional group and their respective wave numbers (cm^{-1}) for FTIR analysis (Silverstein *et al.*, 1981).

Functional group	Vibration type	Wavenumber (cm^{-1})
	Alcohol	
OH	Stretch-bonded H	3200-3600
OH	Stretch, free	3500-3700
CO	Stretching	1050-1150
	Alkane	
CH	Stretching	2850-3000
−CH	Flexion	1350-1480
	Alkene	
=CH	Stretching	3010-3100
=CH	Flexion	675-1000
C=C	Stretching	1620-1680
	Alkyne	
CH	Stretching	3300
C=C	Stretching	2100-2260
	Amino	
NH	Stretching	3300-3500
CN	Stretching	1080-1360
NH	Flexion	1600
C=O	Stretching	1670-1820
	Ester	
CO	Stretching	1000-1300
	Nitrile	
CN	Stretching	2210-2260
	Nitre	
N−O	Stretching	1515-1560

It is also important to note that the presence of acetic acid as a solvent in replacement of water presents a notable difference in the results of WVP as shown in Figure 2, this is because hydroxyl groups (hydrophilic) are substituted by acetate groups (hydrophobic), (Ghanbarzadeh *et al.*, 2011). However, it is observed that from concentrations greater than 1.25 ml of glycerol present in the film, it reduces the effect of the difference of solvents applied.

3.4 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectra were contrasted with Table 3 (Silverstein *et al.*, 1981). In this sense, the spectrum shown in Figure 3 corresponding to pure rice flour shows that at 3714 cm^{-1} there is a peak that represents the stretching of the OH, and between 3625 and 3089 cm^{-1} are the stretches of the bound hydrogens of the oxidrile. Two peaks were also found in the spectrum between the band of 3089 and 2777 cm^{-1} that correspond to the aliphatic part (stretches CH_3, CH_2), the peak of 1643 cm^{-1} corresponds to the stretch of C=C (vinyl carbon) and flexion of the hydrogen of NH_2, in the band of 1464 and 1250 cm^{-1} represents the flexion of the alkyl group −CH, from 1250 to 1178 cm^{-1} corresponds to the stretching of CO, in the band of 1178 to 768 cm^{-1} are 5 peaks that are attributed to the COC bonds and vibrations of the OH group, this being characteristic of the polysaccharides (Aburto *et al.*, 1999) because the starch structure exhibits CO interactions with different chemical environments (COC, CH_2OH, and COH) (Ruiz, 2006).

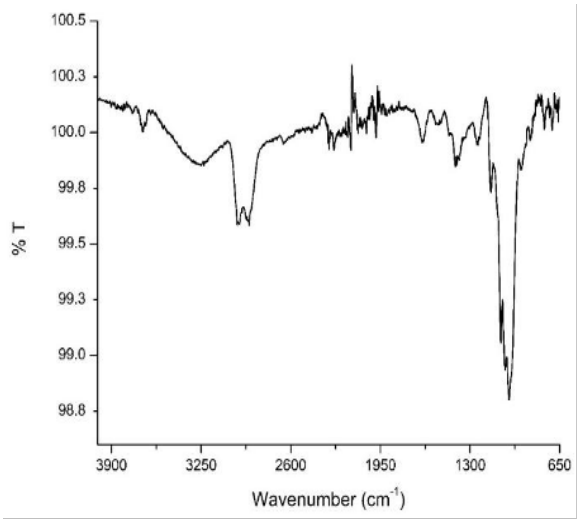

Fig. 3. FTIR spectrum of pure rice flour.

Figure 4 shows the spectra obtained from samples of films developed in water and acetic acid as well as the comparative spectra at different volumes of glycerol in water and acetic acid, respectively. Figure 4a shows that the corresponding band 3625 and 3089 cm^{-1} have a lower intensity in the peak corresponding to the film developed in acetic acid compared to that developed in water and this is due to the esterification of the groups OH of starch. (Ortega-Toro *et al*., 2014) (Cyras *et al*., 2006). A gradual increase in the intensity of the corresponding peak is observed in Figures 4b and 4c: to the oxidrile in the band from 3625 to 3089 cm^{-1}, to the aliphatic group in the band 2777 to 3089 cm^{-1}, and to the COC group in the band from 1178 to 768 cm^{-1}.

3.5 Scanning Electron Microscopy (SEM)

The morphological characterization of the films made with rice flour in water and acetic acid were observed by scanning electron microscopy. The films developed in acetic acid solution showed more homogeneous and continuous surfaces than those dissolved in water as shown in Figure 5. In both treatments, foreign particles were observed on the surface, probably were due to components of the rice flour, as indicated by Péroval *et al*. (2002). In all cases of evaluated films, showed irregularities at the microstructural level; could be attributed to the presence of lipids and mainly fiber, according to Dias et al (2010), which inhibits the formation and cohesiveness of a continuous matrix.

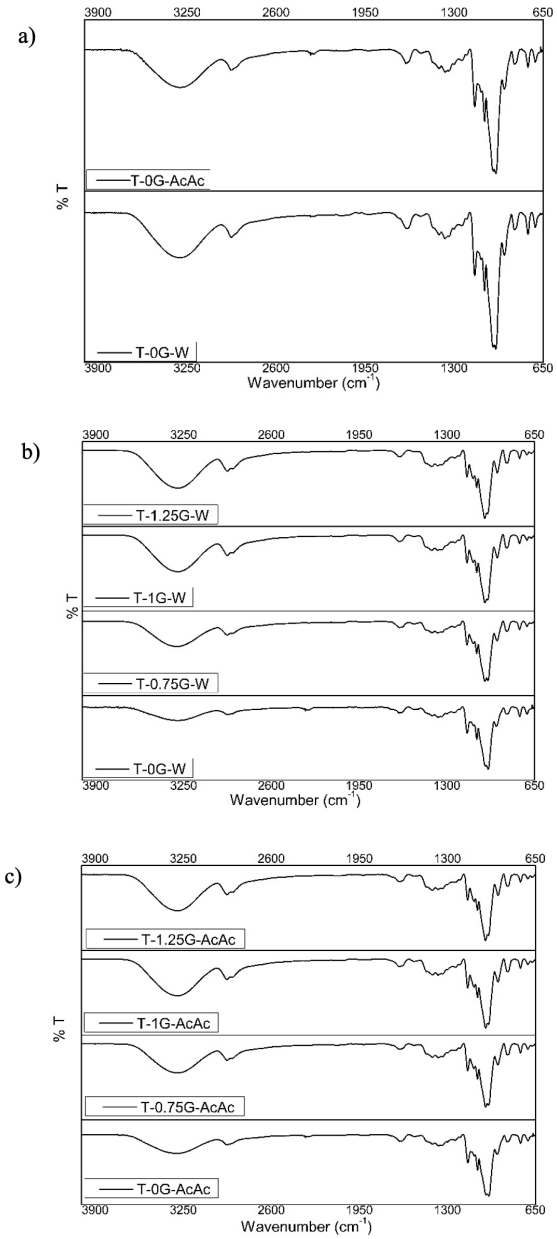

Fig. 4. a) FTIR spectrum of 3.15g rice flour in water 70 ml and 70 ml acid acetic 0.5M, b) FTIR spectrum comparison 0, 0.75, 1, 1.25 ml of glycerol in water c) FTIR spectrum comparison 0, 0.75, 1, 1.25 ml of glycerol in 0.5M acetic acid.

3.6 Mechanical properties

Figures 6 and 7, show that glycerol and the type of solvent (water and acetic acid) have significant differences in tensile strength and deformation among all the films developed ($p < 0.05$).

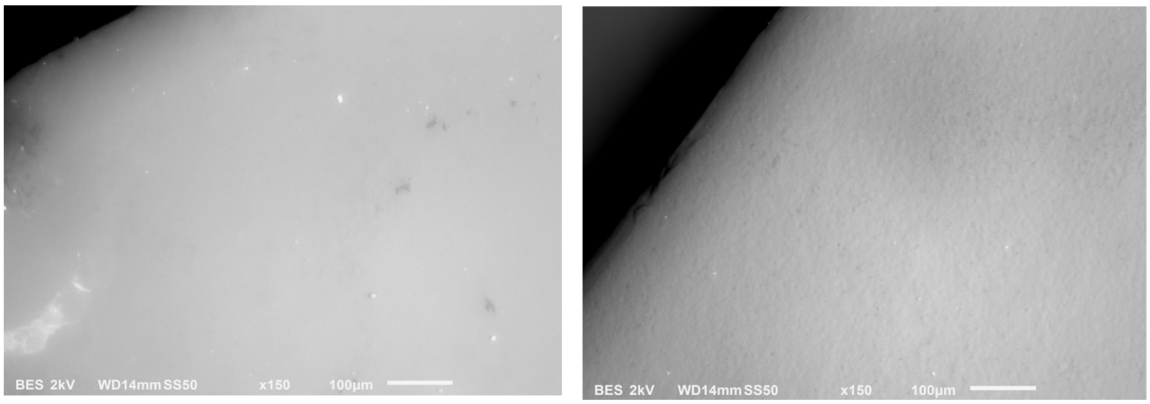

Fig. 5. Micrographs of rice flour films in water and acetic acid; (150 X, 2kV, 100 um). a) 1.25 ml Glycerol in water and b) 1.25 ml Glycerol in acetic acid.

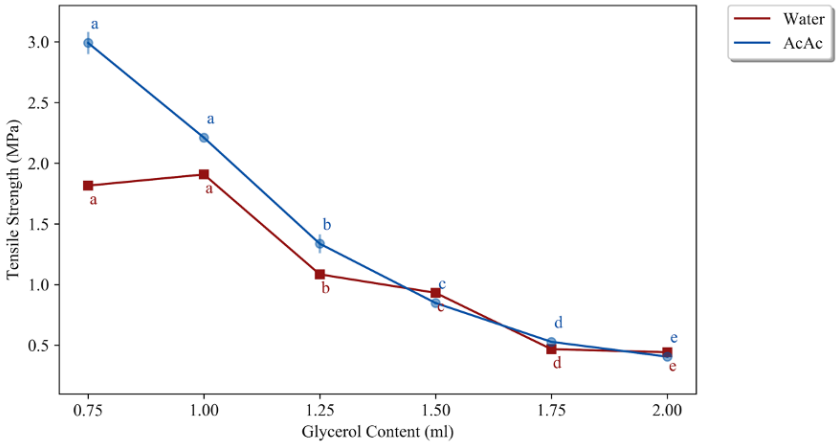

Fig. 6. Effect of solvent type on Tensile Strength with respect to glycerol concentration. *Different letters indicate significant differences ($p < 0.05$) for each kind of film and solvent.

Otherwise, figure 6 shows that films developed in acetic acid as a solvent have greater tensile stress compared to films developed with water. Oropeza *et al*., (2016) reported that the film in which acetic acid was used as a solvent showed greater mechanical strength (higher tensile strength value). This corroborates our results attributed to the replacement of the radicals −OH of the starch by the acetate radicals that lead to the decrease of bonds with glycerol, reducing the free volume of the main polymer matrix, reducing the plasticizing effect. However, as in the WVP, it is observed that from 1.25 ml of the plasticizer (glycerol) concentration generates saturation of this, showing the same behavior, regardless of the type of solvent used.

In Figure 7 it can be seen that films made with acetic acid show a tendency to increase tensile deformation in relation to the volume of plasticizer. This effect is not reflected in the films developed with water as a solvent and could be attributed to the amount of total starch, as well as the presence of other components in the rice flour affecting the interaction with glycerol and therefore the deformation of the films you develop (Mendoza Brito, 2012).

All this agrees with Oropeza *et al*. (2016), who show that, by using acids as a solvent within filmogenic solutions, a better response is obtained in all the evaluated characteristics, being able to obtain a material potential for commercial application.

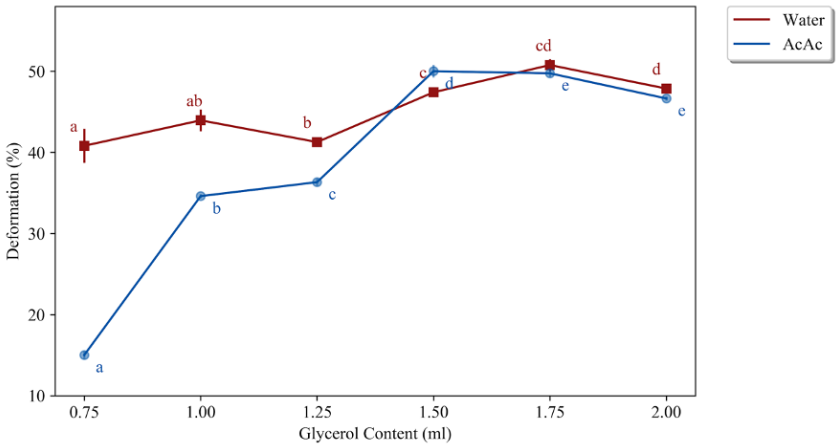

Fig. 7. Effect of solvent type on Deformation with respect to glycerol concentration. *Different letters indicate significant differences ($p < 0.05$) for each kind of film and solvent.

Conclusions

The rice flour films developed in acetic acid presented a lower solubility than those developed in water. Both the WVP and the mechanical properties showed a favorable behavior in acetic acid. The increase in plasticizer (greater than 1.25 ml) governs the system, leaving aside the effect of solvents on the film. The film developed with 3.15g rice flour, 1.25 ml of glycerol, 70 ml of 0.5M acetic acid (T-1.25 G-AcAc) presented the best performance for a possible application as food packaging.

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Nomenclature

ASTM	American Society for Testing and Materials
MPa	Mega-Pascals
μm	Micres
%	Percentage
°C	centigrades grades
WVP	Water Vapor Permeability
SEM	Scanning Electron Microscopy

Greek symbols

ϵ	Elongation
σ	stress

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