# Crosslinking of glucomannan-based biofilms by tartaric acid: Rheology and barrier properties of the biofilms for food packaging

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**Abstract**

The demand for food-wrapping films is increasing worldwide; however, the need for environmentally friendly films draws attention to plants and agricultural wastes. Hemicelluloses were studied to produce wrapping biofilms. Glucomannan from konjac roots and Xylan from corn cob wastes were used as a blend (mass ratio of 70:30) g/g to make food wrapping biofilms. Here, we studied the effect of cross-linking with tartaric acid on the blend's rheology and moisture barrier properties. The mass fraction of tartaric acid is based on the % mass fraction of the mixture (100%, 50% and 25% in 100 ml of the polymers' suspension in deionized water). The filmogenic solution is made as a suspension before drying, which has the potential to make a film. All the cross-linked solutions, TA100, TA50, TA25, and TA00, proved to be shear-thinning polymers, and their viscosity decreased as the mass of tartaric acid increased. After casting, the dried films were excellent moisture barriers.

## 1. Introduction

As climate change impacts people, animals, and plants, protecting the environment and seeking eco-friendly alternatives is a general research concern. Developing green technologies that are friendly to the environment reduces everything that generates global warming and, as a result, climate change. Examples are recycling of wastes, elimination of emissions, electric vehicles, green packaging and so on [1] [2]. Food packaging includes metals, glass, plastic, cardboard boxes and containers. In flexible food packaging, the sources of stretch food wrapping and cling films vary based on their origin. Plastic wraps are made of petroleum [3], and usually, they are transparent, have strong tear, and keep the food fresh and cheap. Nevertheless, they are hard to recycle or biodegrade and pile up in landfills. The other type of wrapping film is produced from renewable re-

sources like plants or agricultural wastes, called biofilms. For instance, polylactic acid (PLA), cellulose and some types of hemicelluloses [4], [5] [6]. The cellulose-based films carry suitable mechanical properties, biodegradability and excellent physical properties. On the other side, cellulose films are highly absorbent to water [7], [8], [9]. Hemicellulose-based films are still in the trial processes [10] [11] [12]. In this research, a mixture of two hemicelluloses was used, glucomannan from the roots of the Konjac plant and Xylan from the wastes of corn cobs. Sorbitol was also added, a polysaccharide used as a plasticizer to make the biofilm flexible and elastic. Figures (1) and (2) show the chemical structure of all ingredients [13] [14]. Tartaric acid is a natural, white, crystalline, cross-linking agent. Its salt is known as cream of tartar and is used in baking. To our knowledge, it is the first time

it has been used to enhance the water vapour resistance of glucomannan-based biofilms.
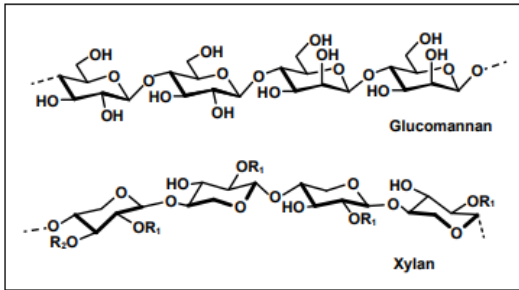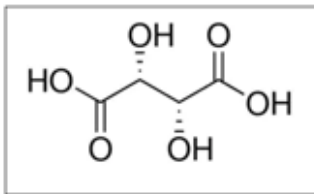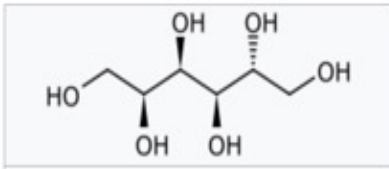


Fig. 1: Glucomannan and Xylan [14].



(a): Tartaric acid



b): Sorbitol

Fig. 2: Tartaric acid and sorbitol [13].

Biopolymers are considered viscoelastic materials because they possess viscous and elastic properties, such as paints, gels or films [15]. Cross-linking is a chemical process in which the polymer chains combine by creating covalent bonds between their functional groups. They improve the barrier and mechanical properties of films and coatings [16] [17]. Figure (3) demonstrates a general cross-linking process. Some examples are tartaric acid, cross-linked starch films [18], and citric acid cross-linked glucomannan [19] and starch films [20].
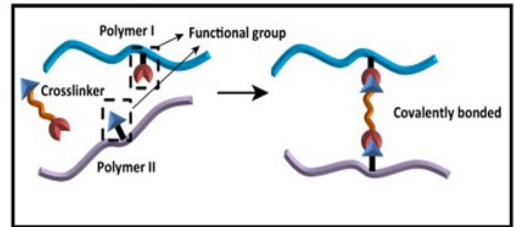


Fig. 3: Schematic of the crosslinking process [16].

The cross-linking between the biopolymer chains leads to a rheology change in these suspensions. The rheology can be studied using rotational rheometers for starch-glucomannan blends [21] [22]. Viscosity is the internal resistance to flow and describes the performance of the fluids during industrial processing. The dynamic viscosity of the fluids plotted against the shear rate shows Newtonian and non-Newtonian behaviour of materials, as illustrated in Fig. 4.
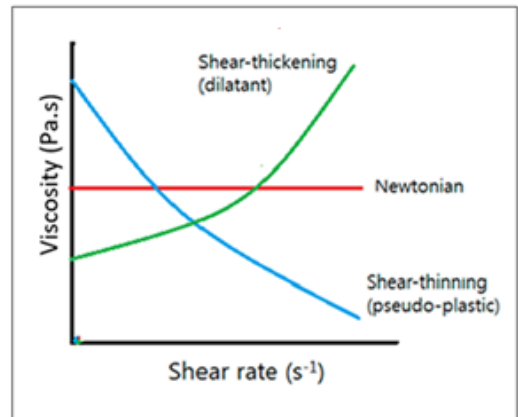


Fig. 4: Viscosity behaviour of fluids at different shear rates

Non-Newtonian materials flow in laminar flow, which hinders mixing and dispersion in tanks, consumes extreme power for pumping in pipes and fittings and affects the selection of pumps [23]. Viscoelasticity means that the material is not purely elastic, solid, viscous, or liquid, and time determines which behaviour prevails. During oscillation deformation, some polymers experience a viscosity decrease with angular shear rate. Therefore, they are called shear-thinning materials. The viscosity-shear rate relation can be modelled using simple equations, such as the Power law, Cross and Sisko [21] models. The proper model to apply depends on the region of the viscosity-shear rate curve and the available data. Newton's law relates stress τ and strain γ of solids and liquids:

$$\tau = \mu\gamma \qquad \text{where μ is the viscosity} \qquad 1$$

and Hooke's law:

$$\tau = G\gamma, \text{ where G is the shear modulus [24]} \qquad 2$$

The permeability of the biofilms to moisture is measured based on ASTM E96 desiccant test methods. The moisture vapour transmission rate (MVTR) of a sheet is defined as:

$$MVTR = \frac{m}{Axt} = \frac{\left(\frac{m}{t}\right)}{A} \qquad 3$$

Where m: is the mass change of water vapour or moisture transmitted through the film in g, A: area in cm$^2$, t: time in a day, and m/t is the slope of the straight line in g/day.

Sometimes, the value of MVTR is reported as g/m$^2$/day. The mass of a cup assembly sealed by the biofilm is monitored for several days and recorded. The mass is then plotted against time, the slope is calculated, and the MVTR is estimated [25]. According to TAPPI 488, the sample should be monitored for three days, and then the MVTR is calculated from the previous equation. However, if the weight changes by ~5%, it is assumed to be constant, and the film represents a good barrier. If there is no gain in weight, keeping the sample for extra four days and recording the weight is necessary. If it is kept constant, the film is considered a practically impressive barrier, which means an excellent moisture barrier.

This work aimed to determine what concentration of the crosslinker TA will lead to proper rheology and the best water vapour barrier of the biofilms.

## 2. Experimental work

### 2.1 Materials
Xylan (Biosynth Carbosynth Company); Glucomannan (NOW Food Company); Tartaric acid, TA (Nature Vibe Botanicals); Mono Sodium Tartrate, MST (Sigma-Aldrich); D-Sorbitol (Alfa Aesar); Citric Acid, CA (Sigma Aldrich); Sodium Hypophosphate, SHP (Sigma Aldric) and Deionizedater, DI (Western Michigan University).

### 2.2 Film formation
The biofilms were formed by the casting method at different tartaric acid concentrations. All chemicals are water-soluble. Table 1 shows the formulation of the biofilms. A cross-linked biofilm with citric acid as a crosslinker was used as a control biofilm to compare the properties with the tartaric acid cross-linked ones. The biofilms were named based on the amount of tartaric acid in the formula. For example, TA100

means that the formula contains 1 g of TA and 0.5 g MST in 100 ml water suspension, while CA25 has the composition of 0.25 g of citric acid and 0.125 g of sodium hypophosphate (SHP) in 100 ml of DI.

Table 1: Formulation of glucomannan-xylan biofilms (In 100 ml of DI water)

| Biofilm | Gluco-manan [g] | Xylan [g] | Sorbitol [g] | Tartaric acid, TA [g] | Mono-sodium tratrate ,MST [g] |
|---------|-----------------|-----------|--------------|-----------------------|-------------------------------|
| TA100 | 0.7 | 0.3 | 0.2 | 1 | 0.5 |
| TA50 | 0.7 | 0.3 | 0.2 | 0.5 | 0.25 |
| TA25 | 0.7 | 0.3 | 0.2 | 0.25 | 0.125 |
| TA00 | 0.7 | 0.3 | 0.2 | 0 | 0 |
| CA25 | 0.7 | 0.3 | 0.2 | Citric acid: 0.25 | SHP: 0.125 |

The procedure to synthesize the biofilms, following our previous work [14], starts with dissolving the glucomannan in 100 ml of DI water, transferring the beaker into a water bath at 45° C and mixing the solution using a rotary mixer at 6000 rpm for 15 minutes. Xylan was added and mixed for 5 minutes, followed by sorbitol and mixed for 5 minutes, and then tartaric acid and its catalyst MST were added and mixed for 30 minutes to allow for cross-linking between the polymers. The produced suspension (filmogenic solution) was tested for density and pH. We used an Anton Paar rheometer with a plate geometry for rheology tests (viscosity, storage and loss moduli). The desiccant method tested the water vapour barrier property after drying the casted biofilms, and a GLAD cling wrap was also used as a control.

## 2.3 Rheology tests

During an oscillation test of viscoelastic materials, first, the filmogenic solution of each film had its viscosity and frequency sweep test of the shear stresses at different shear strains are measured by the Anton-Paar Rheometer at 25℃. Viscosity is the internal resistance to flow and is defined as:

$$\text{Viscosity (Pas)} = \frac{\text{Shear stress}}{\text{Shear rate}} \text{ or } \mu = \frac{\tau}{\dot{\gamma}} \qquad 4$$

Viscosity is plotted at different shear rates. During the sweep test, Stress τ* and strain γ sinusoidal signals have a phase shift called δ and usually have the value 0°-90°. The shear modulus of non-Newtonian fluids has two parts, loss modulus G" which represents the viscous behaviour or tendency to flow of the viscoelastic material and storage modulus, G"which is the elastic behaviour or tendency to retain shape. The complex number G* is composed of G" and G' such that:

$$G^{*} = \sqrt{G^{"2} + G^{'2}} \text{ and } \tan\delta = \frac{G^{"}}{G^{'}} \qquad 5$$

## 2.4 Barrier properties tests

For the biofilm barrier property test for the moisture vapour transmission rate (MVTR), 50 ml of the filmogenic solution was cast into a Petri dish and dried in Caron 6010 Environmental chamber for 24 hours at 60℃ and 35% Relative Humidity, RH, then transferred to the testing laboratory at 23℃ and 50% RH for conditioning. After 24 hours, the biofilm was manually peeled off the Petri dish and tested. According to the ASTM E96 test-desiccant method, a circle of 2.5 inches of biofilm was cut and sealed on the top of the cup with enough mass of anhydrous $CaCl_2$ to keep 0% RH inside. The Thwing-Albert EZ-Cup was weighed to measure the MVTR for several days and recorded. Some films needed a

week to prove that it is a good barrier.

## 3. Results and Discussion
The rheological characterization of the filmogenic solution was done as viscosity and shear measurements of the cross-linked biofilms at different shear rates.

### 3.1 Filmogenic solutions
The density ($\rho$) measurement of the suspensions was carried out using the pycnometer, The average density of all filmogenic solutions was 1.0 ±0.05 g/cm³, and they had an average pH= 4.80±0.05. Both measurements showed that the cross-linking did not affect the density or the pH. The reason could be explained by the fact that the change of the masses of TA and its catalyst added was small and didn't affect the density value rounded to 2 digits. The same can be said about the pH.

### 3.1.1 Viscosity μ
Biopolymers are viscoelastic materials, and their response to deformation of shear stress τ defines their characteristics by their viscosity μ and its shear modulus G. The viscosity of the filmogenic solutions of TA100, TA50, TA25 and TA00 was plotted against shear rate, by Origin Pro 8.5 software, as in Figure 5. All the solutions exhibited a shear-thinning behaviour, where the viscosity decreased as the shear rate increased. The least viscous gel was the filmogenic solution of the uncrosslinked sample TA00. At the same time, TA50 was the most viscous gel, which can be explained as follows: the carboxylic groups of TA were reacting with -OH groups of the polymers (Xylan and glucomannan) in the esterification reaction. Since citric acid has three -COOH groups and tartaric acid two -COOH groups, it is likely that the reaction can involve different polysaccharidic chains, resulting in cross-linking of those other chains,

making different layers and networks and thus increase the internal resistance to flow under shear stress, i.e., increase in viscosity. Since all the suspensions have shear-thinning behaviour, their bonds are broken isothermally and reversibly under shear stress. The aggregates of the chains of polymers in the suspension are destroyed due to the angular shear, and their dissociation rate is bigger than that of association [23]. Pumping or injecting these solutions during the biofilm processing is the same as honey at room temperature of 10,000 mPa.s. This can be explained by the strong intermolecular forces between the polymer's chains in the matrix.

The dynamic viscosity μ, or η in some references, was modelled using the Cross model for TA100, TA50, TA25 and TA00. The cross-model equation of non-Newtonian fluids [26] is:

$$\mu = \frac{\mu_0 - \mu_\infty}{1 + \left(k\,\dot{y}\right)^n} + \mu_\infty \qquad 6$$

Where $\mu_\infty$: infinite shear viscosity (Pas), $\mu_0$: zero shear viscosity (Pas), $\gamma$ : shear rate (s$^{-1}$) and k and n are specific parameters of each fluid. The cross-model fit was in excellent agreement with the experimental values of all solutions, as shown in Figure 6. The observed viscosity (scattered) and its modelled value by the cross model (line) were plotted in the same colour to compare the two values.
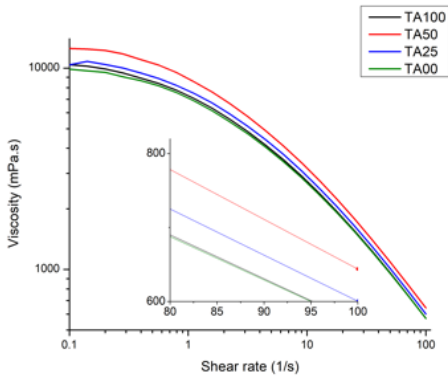
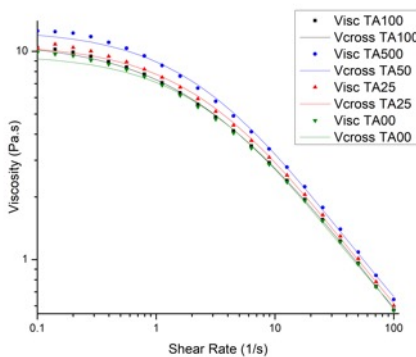Fig. 5: Viscosity of cross-linked filmogenic solutions.



Fig. 6: Cross-model fit of the viscosity of all samples.

### 3.1.2: Storage and loss moduli

Polymers show a strong temperature and time-dependent behaviour, which can be modelled by the Maxwell Model [24]; however, in Anton Paar, the tests were conducted isothermally at 25° C. As mentioned earlier, viscoelastic materials possess pure viscous fluids, modelled as dashpot, and ideal elastic solid, modelled as spring [24]. Therefore, if the material deforms under shear stress, it will be described by Newton's law for viscosity μ and Hooke law for modulus G. The Maxwell model combines the dashpot and spring in series. The applied shear stress is the same, but the strain will be additive, as explained in Figure 7. When the sinusoidal stress is applied, the spring expands, and the dashpot slows down the deformation of the whole system. The loss modulus G" represents the amount of energy dissipated during the deformation, while the energy stored and retained is expressed as storage of modulus G". If GG'G", then the material is considered mainly elastic, else it is viscous.
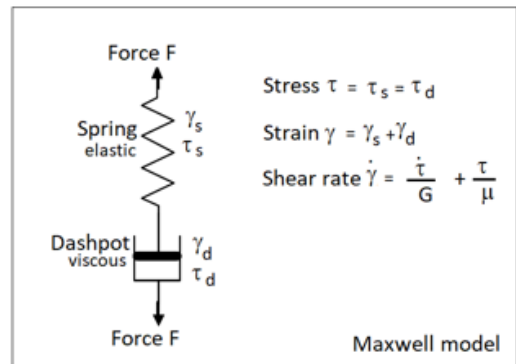


Fig. 7: Maxwell model.

The storage and loss moduli were measured through a sweep test with an angular frequency of 0.1 to 100 rad/s and a strain of 5% and plotted for each filmogenic solution. The graphs showed that all solutions were liquid-like at low shear rates where GG' <G" and turned into soft solids or gels at ~ 10 rad/s where G'>G". The turning-over point is called the gelation point. At angular frequency $\omega$ =10 rad/s, the values of G' and G" of the samples were very close to each other but not equal 100%, TA100 had an absolute difference of 1.0 rad/s, TA50 of 0.5 rad/s, TA25 of 0.8 rad/s and TA00 had 1.4 rad/s; this shows that cross-linking with TA delayed the gel formation since it has the highest G' and G" values.

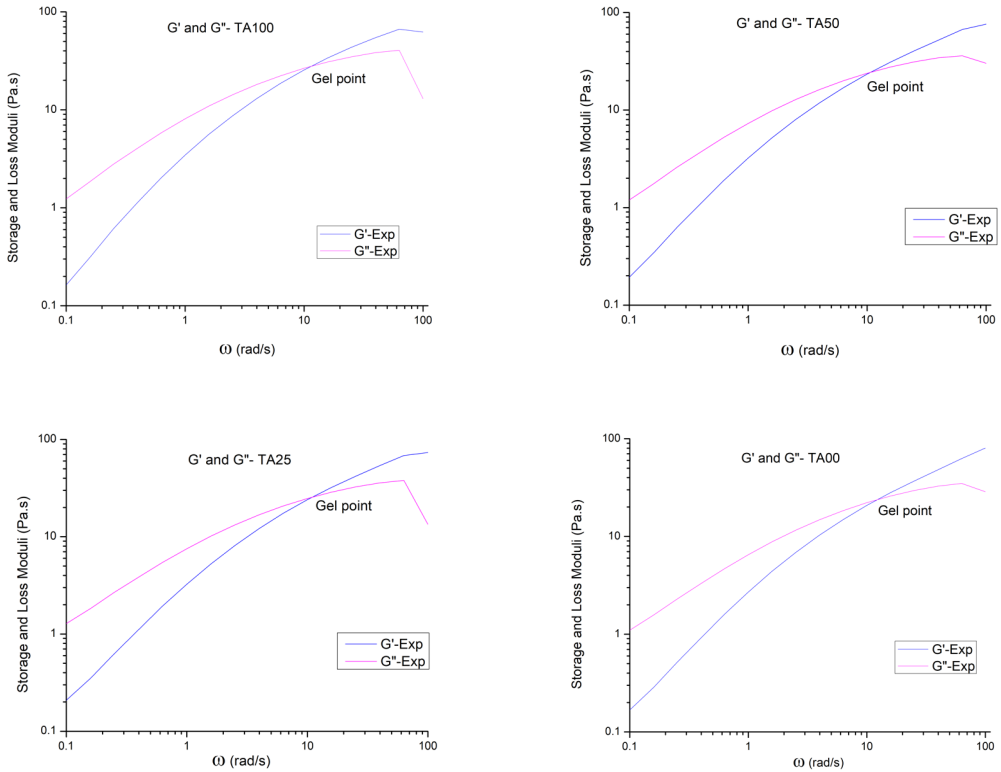However, TA00 became gel at 15 rad/s. Fig. 8 demonstrates these relations in all suspensions.

Fig. 8: A sweep test shows G' and G" as a function of the angular frequency.

The generalized Maxwell model represents more than spring-dashpot elements but will have more parameters of G and μ. In this model, three elements were used for the Maxwell model. Note that the zero-shear viscosity for the Cross-model fits and the zero-frequency viscosity for the Maxwell model fits are the same for each level of cross-linking agent (Figures 6 and 9), as required for consistency.
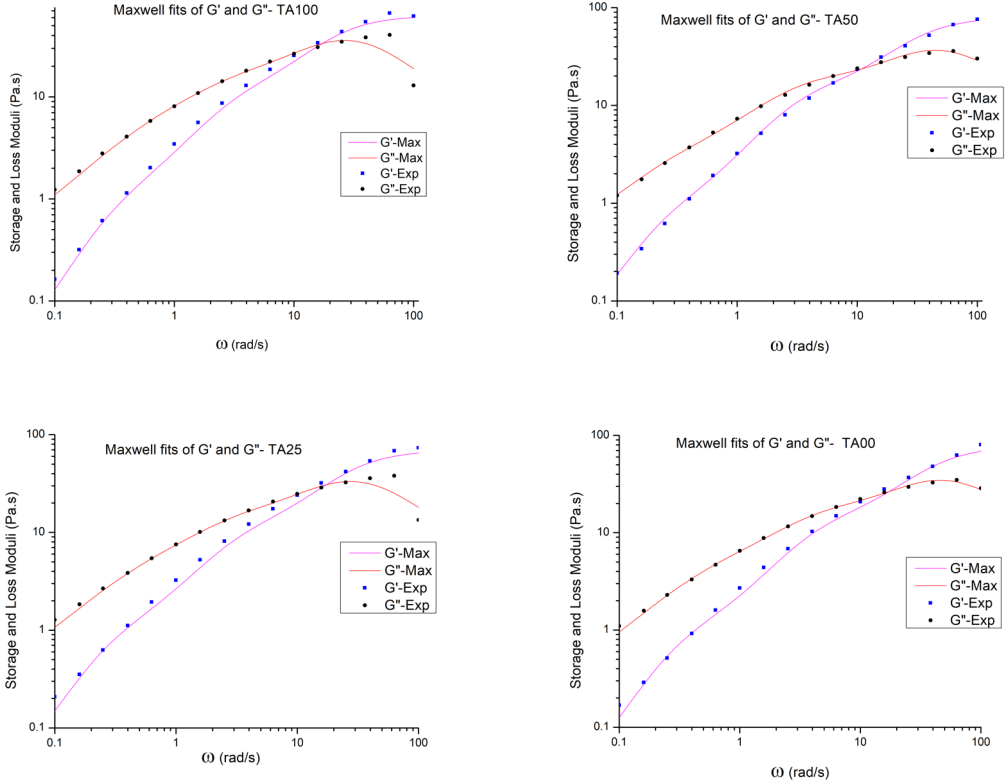
Fig. 9: Maxwell-fit of G' and G" compared to the experimental values.

### 3.1.3 Moisture vapour transmission rate (MVTR)

The mass of the Thawling Cups, including the biofilm and the desiccant, anhydride $CaCl_2$, was measured every 24 hours and recorded for three days and, in some cases to 7 days when the mass change of the system is constant or within 5% change. Table 2 illustrates the mass change of the cup assembly or moisture over time for the four crossed-linked biofilms and a Low-Density Polyethylene (LDPE) and the estimated MVTR values reported as (g/m²/day).

Table 2: Moisture Vapor Transmission Rate (MVTR) of the biofilms measured by EZ-cups.

| Sample | moisture, g | % moisture | MVTR (g/m²/day) |
|--------|-------------|------------|-----------------|
| TA100  | 0.01        | 0.01       | 0.45            |
| TA50   | 0.03        | 0.02       | 1.35            |
| TA25   | 0.03        | 0.02       | 1.35            |
| TA00   | 0.21        | 0.13       | 9.47            |
| CA25   | 0.10        | 0.06       | 4.51            |
| LDPE   | 0.01        | 0.01       | 0.45            |

The moisture entrapped inside the EZ-cups, or the change of the mass of the EZ-cup assembly, was little for all of the samples. A sample of commercial plastic wrap from GLAD Cling Wrap made of LDPE was also tested for three days as a control, and the value of the average MVTR in $g/m^2/day$ was reported. A reference gives MVTR of LDPE, but at 38°C and 90 % RH conditions, the value ranges from 16-20 $g/m^2/day$ [27]. However, the conditions are different, so that the comparison will be incorrect. The cross-linked biofilms showed less moisture in their cups than the uncrosslinked ones. TA00 allowed water vapour to go through and caused a change of 0.13% of its original mass, and therefore the value of its MVTR was the highest at 9.47 $g/m^2/day$. Biofilms cross-linked by TA showed resistance to water vapour transmission through them in the order of TA100>TA50>TA25. This is reasonable because the matrix had a higher amount of cross-linking agent, which made it denser. The value of MVTR of CA25 was less than TA25, even though CA provides higher chances of cross-linking. The GLAD plastic film had a 0.45 MVTR value as TA100. It is clear that cross-linking with TA or CA prevented the absorption of any moisture inside the cups and proved to be an excellent moisture barrier, and we can assume other vapours, too. This can be explained by the fact that the polymer network reduced the porosity of the films to the minimum and hindered water vapour transmission into the cups.

## 4. Conclusion

Glucomannan from Konjac roots and Xylan from corn cob wastes were used as a blend (70:30) g/g to produce food-wrapping biofilms. The cross-linking with tartaric acid at various concentrations affected the blend's rheology and moisture barrier properties. Cross-linking with tartaric acid enriched the polymer network, lowered the gel point of uncrossed filmogenic solutions, and reduced the water vapour permeability of the biofilms. Compared to citric acid, tartaric acid is stronger and cheaper; however, citric acid has the potential to create a more complex and linked network. The biofilms produced by cross-linking proved to have good resistance to allow water vapour molecules to pass through them. They are expected to prevent other flavours and odours of the same or larger size from penetrating them and spoiling the wrapped food.

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