Surface properties, rheology and thermorheology of octenyl succinic anhydride (OSA) waxy maize starch

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ABSTRACT

The present work is aimed to realize a complementary characterization of octenyl succinate anhydride-modified waxy maize starch (OSA WM starch) in terms of surface properties rheology, and thermorheology. Conductimetry analysis and surface tension measurements confirmed the amphiphilc character of the modified polysaccharide. The critical concentration of aggregation was found to be 0.1% and the corresponding measured surface tension was 54.5 mN/m. In parallel, the rheological and thermorheological behaviors of OSA starch were determined. The model of Herschel-Bulkley was utilized for the evaluation of the rheological parameters. Furthermore, the viscoelasticity study showed that the elastic modulus (G') and the loss modulus (G") were sensitive to the polymer concentration and are dependent of frequency and strain. The modified polysaccharide exhibited seemingly a solid-like behavior (G'>G"). Starting at critical temperature, measurements in oscillation of the effect of temperature showed that system becomes much more elastic. The great increase in elasticity confirms the thermoreversible behavior.

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1. INTRODUCTION

Starch is, after cellulose, the most common reserve substance in higher plants; it is present in a large number of agricultural raw materials such as cereals, tubers, legumes, and certain fruits [1]. It is a semi-crystalline natural polysaccharide that is attracting growing interest in several fields; however, starch is mainly used in food because of its nutritional value [2,3]. Apart from these properties specific to carbohydrates, starches are added to food preparations because they exert a great influence on the viscosity, texture, and taste of the products in which they are incorporated [4]. So, starch can be found in the presence of various ingredients that can influence its structural properties [5].

Native starches are suitable for most industrial applications. However, it may be necessary, in some cases, to improve their performance through a set of physical, chemical, and enzymatic modifications, to meet specific technological and nutritional needs [6]. The chemical modifications applied to native starches lead to more or less important variations of their structures, depending on the composition of the starch, water content, conditions applied, and organization of the amylose and amylopectin in grains of native starch [7]. Thus, modification by octenyl succinic anhydride (OSA) leads to the formation of functional starches that can be utilized as additives, thickeners, emulsifiers, or gelling agents [8,9]. Because of the potential use of OSA starch, several studies were devoted to investigating its properties. Ezzeroug et al. [10] evaluated the physicochemical and size distribution of OSA starch using dynamic light scattering. Zhang et al. [11] demonstrated the relationships between the emulsification properties of OSA starch and its molecular structure. Moreover, the rheological and pasting behavior of the modified polysaccharides were investigated by Punia et al. [12], where they were proposed in cake formulation as fat replacers.

Therefore, the rheological characteristics of starch pastes are controlled by various factors; the most important of them are the type and concentration of starch, the temperature of measurement and the method by which the paste is prepared *ie* the rate, time, and temperature of heating, the way of stirring [13]. Thus, a relationship between the granule size distribution and the rheological properties of different forms of starch was revealed [14].

Recently, we investigated the chemical structure, morphology, and thermal properties of a commercial OSA waxy maize starch [15]. In order to complete its characterization, rheological and thermo-rheological analyses were performed, with the aim of relating the behavior differences under the thermal effect, flow curves, and viscoelasticity. In parallel, the amphiphilic character was evaluated by measuring the surface properties of the modified biopolymer in an aqueous solution.

2. MATERIAL AND METHODS

2.1. Material and sample preparation

Octenyl succinate anhydride-modified waxy maize starch (OSA WM starch), marketed as Gel′N′Melt, was purchased from Ingredion (Manchester, UK). The grade of the commercial modified starch is for food and pharmaceutical uses. Stock solutions were prepared by dissolving 10 g of OSA WM starch in 100 mL of ultra-pure water at 65 °C and left at room temperature for one day, and then diluted to obtain desired concentrations.

2.2. Determination of surface properties

Several methods have been used to determine the surface tension (ST), the critical aggregation concentration (CAC), and the Krafft point temperature (T_K). The most important are tensiometry and conductimetry [16]. The conductivity of polymer solutions was measured using an Orion 3-Star conductimeter (Thermo Fisher Scientific, MA, USA). The surface tension was determined by a TSD digital tensiometer (Gibertini, Italy).

2.3. Morphology of granules

The morphology of granules was examined by a scanning electron microscope (Jeol JSM-6400, Japan). The electron acceleration voltage used was 5 kV, and the detector-sample distance was between 9 and 14 mm. The samples were fixed onto metallic sample holders with conducting silver glue and sputtered with a layer of gold.

2.4. Rheological analysis

For the rheological study, a plate-plate (25 cm diameter, 1 mm gap) rheometer (Anton Paar, Germany) was utilized. Aqueous solution containing OSA WM starch at different concentrations (0.7% to 10%, w/v) were prepared and subjected to rheological measurements at a temperature of 25 °C. Flow curves were first determined by the variation in the apparent viscosity as a function of shear rate ranging from 0.001 to 1000 (1/s). Linear and dynamic viscoelastic properties were then determined by strain sweep tests at a frequency of 1 Hz in a strain range of 0.001-0.1% and a frequency sweep test in the range of 0.01-10 rad/s at a constant strain value (deducted from the strain sweep test). For the determination of the thermo-rheological properties, the samples were subjected to heating and cooling from 2 to 85 °C and from 85 to 2 °C at a rate of 5 °C/min. The viscoelastic parameters, such as the storage modulus (G') and loss modulus (G") were measured as a function of temperature.

3. RESULTS AND DISCUSSION

3.1. Surface properties of OSA WM starch

Fig.1 shows the conductivity profile of OSA WM starch as a function of concentration at 25 °C. The experimental curve reveals that the conductivity is proportional to the polymer concentration. By definition of the solution conductivity, the increase in conductivity as a function of polymer concentration can only be explained by an increase in the number of free counterions in the solution. It was also observed that the curve has changed the slope at two points. This is due to the ionic character of OSA WM starch, which behaves like a surfactant in solution [17].

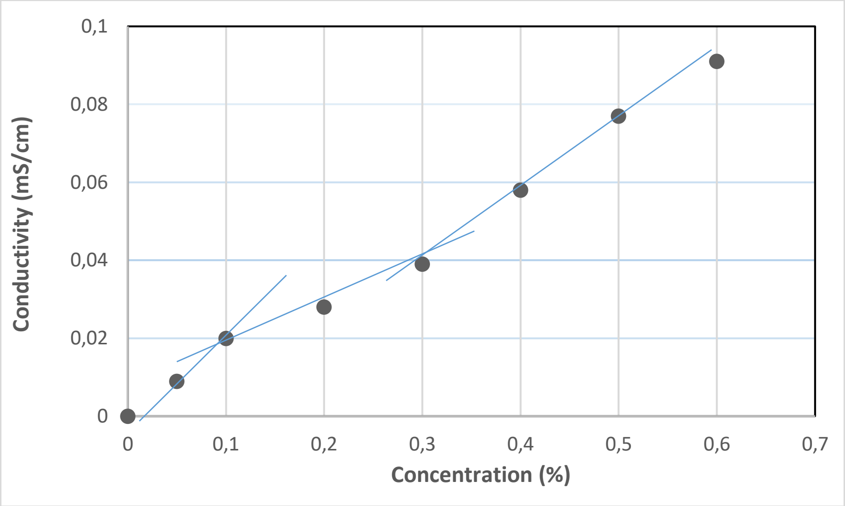


Figure 1. Variation in conductivity as a function of biopolymer concentration (25°C)

Two linear regions were observed before and after the CAC which was around 0.1%. Before the CAC, where the concentration of macromolecules is low, the amount of polymer is not sufficient enough to form micelles. OSA starch dissolves in water to give simple macromolecules. After the CAC, the formation of micelles continues and the presence of macromolecules also increases, leading to an increase of the conductivity but to a lesser extent than in the absence of micelles. At the CAC, the ST was found equal to 54.5 mN/m. This value confirms the results found by several authors who have worked on the surface properties of several modified starches [8,9]. The second change in slope can be interpreted by a polymer saturation point (PSP) indicating an oversaturation in OSA starch polymer. Beyond this point, which is about 0.33%, the conductivity continues to increase.

The results obtained from conductivity and ST measurements agree with those reported by Krstonošić et al. [18] and, Varona et al. [19] who determined significant values for waxy maize OSA starch derivatives. In addition, Shorgen and Biresaw [20] studied the surface properties of water-soluble starches; they reported that the ST of OSA starch (DS: 0.02-0.04) was about 42-43 mN/m. This value is slightly lower than that found experimentally in this study.

3.2. Determination of the Krafft point temperature

The determination of the Krafft point temperature (T_K) was carried out by varying the conductivity of the polymer as a function of temperature between 10 and 100 °C. It can be seen from Fig.2 that the conductivity increases with temperature. Thus, the intersection of the two slopes corresponding to the conductivity curves, allows the determination of T_K.

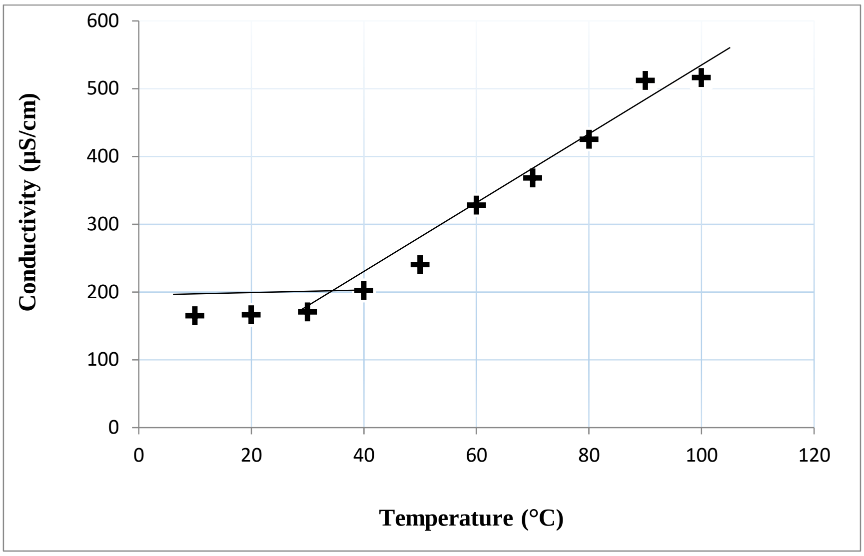


Figure 2. Conductivity of OSA starch as a function of temperature

In addition, the conductivity increases almost exponentially from T_K (critical micellization temperature); the micellization process is then initiated. Below T_K and at low amphiphilic polymer concentrations, monomer solutions were obtained. At higher concentrations, the macromolecules precipitate in the form of hydrated crystals and therefore, there is no formation of micelles. Above T_K and at low concentrations, solutions of monomers are concentrated which lead to the formation of micelles. For ionic polymer surfactants, the conductivity suddenly increases almost exponentially from a value of T_K around 31°C, which corresponds to the critical micellization temperature [21].

Hence, the formation of micelles enhances the dissolution of amphiphiles in water. The water solubility of ionic polymer surfactants increases sharply above T_K. Consequently, the knowledge of T_K is often necessary and, in most applications, it is essential to choose a polymer surfactant with a value of T_K lower than the temperature of use.

3.3. Rheological behavior

3.3.1. Flow curves

The flow curves of OSA starch dispersions are shown in Fig.3, which clearly showed that the studied biopolymer exhibits a shear-thinning behavior, as the viscosity decreases with the shear rate.

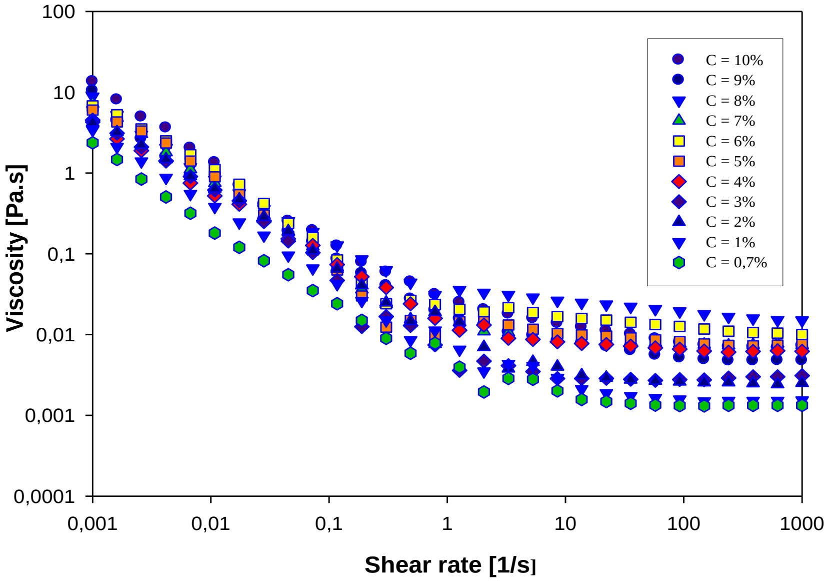


Figure 3. Profiles of the apparent viscosity as a function of the shear rate of OSA starch at different concentrations

These results agree with those found by Ezzeroug et al. [10], and Park et al. [22]. It was also observed that the viscosity of the polymer is low especially at high shear rates. During shearing, particles are deformed and eventually disrupted, resulting in reduction in the viscosity at high shear rates. In addition, the viscosity increases with the increase of the polymer concentration (C). However, the appearance of the flow curves for different concentrations have the same behavior.

For all concentration profiles, three regions are observed. In the first region, at low shear rate, the shear-thinning behavior is observed with the absence of a Newtonian plateau; such profiles are characteristic of fluids with yield stress [23]. So, the second region is found at intermediate shear rates, where we observe a short pseudo-Newtonian plateau that is followed by the third region corresponding to the Newtonian plateau at high shear rates. These findings may be explained at first by the presence of two populations of OSA starch particles. The first one is composed of particles with smaller average sizes that they are less aggregated compared to the second population formed by a residual fraction of unmodified starch particles.

On the other hand, it was observed that the viscosity of the different dispersions increases significantly when the shear rate applied to the flow tends to be cancelled. This implies the need for a minimal stress. The knowledge of the yield stress is very important at the practical level to facilitate the conditioning of products [24]. Its presence reflects the state of packing of the starch grains; it would be due to the existence of interactions between the swollen grains or between the swollen grains and the macromolecular network [25].

For the modeling of the rheological curves, the model of Herschel-Bulkley was selected (Eq.1). This model integrates several characteristic parameters. In addition, it is assumed that the structure of the fluid is stable, so the time of application of the stress or the shear does not influence the model.

$$\tau = \tau_0 + K_* \gamma^n \qquad (1)$$

where τ is the shear stress, γ is the shear rate, τ_0 is the yield stress, K is the consistency index and *n* is the flow index (n is between 0 and 1 for shear-thinning fluids). The rheological parameters obtained for OSA WM starches using the model of Herschel-Bulkley are summarized in Table 1.

The results confirmed the mechanical behavior of the modified polysaccharide, where it was clearly shown that the rheological curves obey to the proposed model. The parameter values also confirmed the shear-thinning behavior ($0<n<1$). In addition, there are highly significant correlations where the values of the coefficients of determination are around 0.99 for all studied concentrations. In addition, the obtained values indicated that the shear-thinning behavior is accentuated, where high consistency and low *n* values are obtained.

Furthermore, it was observed that some rheological parameters depend on the biopolymer concentration. Thus, the consistency index does not vary much with concentration. However, the yield stress is strongly dependent on the concentration; it increases sharply when it varies from 0.7 to 2% and, thereafter it increases in a linear fashion. It should be emphasized that the rheological parameters-concentration relationships can provide a very simple means for predicting the behavior of the test fluids without going through viscometric tests.

Table 1. Characteristics parameters of Herschel-Bulkley model

Samples	Herschel-Bulkley factors			
C (%)	τ_0 (Pa)	K (Pa.s),	*n*	R^2
0.7	0.0039 ± 0.0004	0.0014 ± 0.0002	0.9912 ± 0.0018	0.990
1	0.0059 ± 0.0003	0.0015 ± 0.0001	0.9849 ± 0.0123	0.993
2	0.0065 ± 0.0001	0.0025 ± 0.0004	0.9795 ± 0.0010	0.999
3	0.0055 ± 0.0003	0.0032 ± 0.0002	1.1085 ± 0.0025	0.997
4	0.0066 ± 0.0001	0.0065 ± 0.0004	0.9745 ± 0.0016	0.998
5	0.0062 ± 0.0002	0.0081 ± 0.0007	1.0000 ± 0.0027	0.997
6	0.0091 ± 0.0001	0.0123 ± 0.0009	0.9728 ± 0.0021	0.996
7	0.0084 ± 0.0002	0.0076 ± 0.0005	1.0503 ± 0.0024	0.998
8	0.0098 ± 0.0001	0.0200 ± 0.0010	0.9119 ± 0.0019	0.998
9	0.0098 ± 0.0002	0.0051 ± 0.0003	0.9133 ± 0.0028	0.998
10	0.0122 ± 0.0003	0.0078 ± 0.0005	0.8463 ± 0.0003	0.999

Values are mean ± SD of three replications for each sample

3.3.2. Viscoelastic properties

The viscoelastic character is a very important and very frequent non-Newtonian behavior in polymer solutions. The fluid response to a strain has both elastic and viscous aspects. In the first case, the characteristic time of the stress is less than the characteristic time of the material; the elementary components do not have time to deform significantly and an elastic response is observed. When the stress time is greater than the characteristic time of the material, the response is viscous.

Thus, at low frequencies, it was noticed for the present case that the loss modulus (G") is much higher than the elastic modulus (G') as shown in Fig.4. This indicates that the viscous behavior is predominant. As the strain increases, G' tends to a plateau and G" decreases rapidly. Consequently, the elastic behavior becomes predominant.

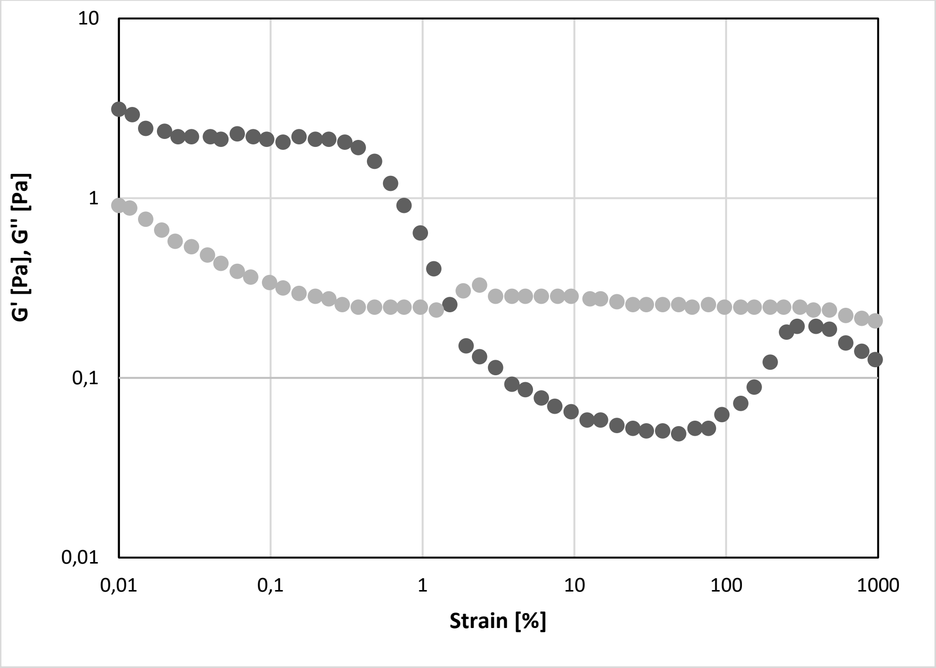
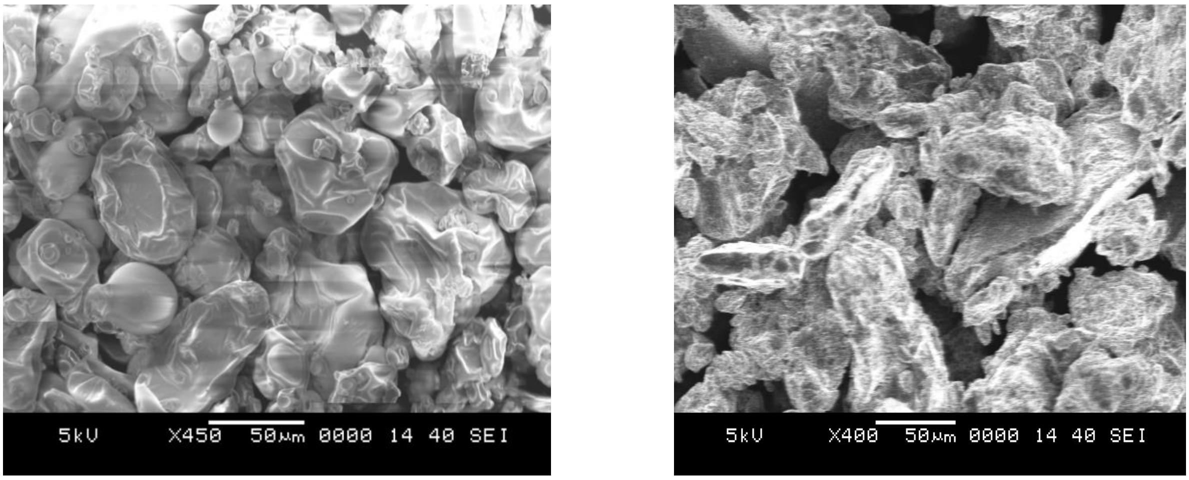


Figure 4. Strain-dependent viscoelastic parameters of OS modified WM starch (C = 10%, F = 1 Hz)

Thus, when G' = G", the Maxwell model involves two perfect components [26]. Hence, three specific regions are observed. In the first zone, the flow is viscous (G'<G"). The second zone corresponds to the flow transition where the two moduli are of the same order of magnitude. The point of intersection of the two moduli (G' = G") often appears and in the case of Maxwell's model, it corresponds to the critical frequency. The third zone corresponds to the plateau or the rubbery zone; in this region, the elastic behavior dominates. It is also marked by a plateau of G'.

3.3.3. Thermo-rheological behavior

During a classic thermal transformation, the native starch grains swell in the presence of heat and water. This phenomenon is accompanied by the fusion of the crystallites and a progressive release of macromolecules of small weight from amylose and amylopectin [27]. This process will continue until the bursting of the granular structure and the total release of starch macromolecules. Thus and from a temperature of 65°C, the shape of the grains appears irregular with clusters of stuck particles, confirming the presence of aggregates (Fig.5). It was also observed a dominance of agglomerated particles of different sizes, totally fragmented with smaller cavities. Indeed, from the results obtained, it appears that the thermal effect causes a morphological and structural modification of OSA WM starch. On the other hand, the coexistence of individual particles with aggregated particles was observed, favoured by the extent of the fusion of grains during heating. Nevertheless, in the case of extrusion, the breaking of the granular structure, the release of macromolecules and the fusion of crystallites can also occur with or without the partial swelling of the grains. This is due to the high mechanical stress to which the biopolymer is subjected.



(a) **(b)**

Figure 5. SEM micrographs of: (a) OSA WM starch (at 25°C) and (b) OSA WM gelatinised starch (Tg = 65°C)

Thus, the temperature ramp makes it possible to follow the behavior of OSA WM starch at different concentrations (1, 5, 10, 20, and 30%) during heating-cooling when subjected to a shearing force. The results were recorded in the form of curves of variation of G' and G" as a function of temperature (Fig.6). The curves were expressed for a frequency of 1 Hz and a heating or cooling rate of 5 °C/min for a temperature range between 2 and 80 °C.

The results obtained clearly showed that the increase in temperature induces a transition from a fluid behavior to a viscoelastic one. For low temperatures, the aggregates are not yet formed. Beyond the critical granulation temperature, G' and G" increase with temperature. This increase is initially slow then sudden and depends on the polysaccharide concentration. This second mode identifies the fluid-crystal transition temperature which corresponds to the transition between a viscoelastic liquid mode (G'<G") and a viscoelastic solid mode (G'>G"). A translation towards higher temperatures between the particles leads to a dissociation of the aggregates, from which we obtain the crossing between the two moduli (G' = G"). In the intermediate zone, the elastic behavior prevails and G' presents a plateau, while G" presents a minimum. This can be explained by an increase in the mobility of the chains causing the opening of the aggregates to dissociate into smaller ones [28]. At a temperature above 60 °C, the modified starch grains undergo a complex process. First, there is a swelling of the grains due to the rupture of hydrogen bonds, solubilization of the granular contents, and a loss of the semi-crystalline structure of the biopolymer grains, which leads to a decrease in viscosity [29]. During the cooling, the presence of solubilized amylose makes it possible to form a gel, which embeds the dispersed grains. The gelation reinforces the elastic character; the moduli G' and G" evolve towards higher values after cooling. After that, a recovery in consistency is observed, due to a reassociation of the macromolecules, which will form a gel.

These observations made it possible to understand the influence of temperature on the rheological behavior of the modified polymer. Measurements in oscillation showed that from a critical temperature, the system becomes much more elastic. When the temperature increases, at first, the behavior of the polymer predominates. Then there is a great increase in the elasticity of the system. This is probably due to a change in the interactions between the particles leading to a dissociation of the aggregates. These findings confirmed the thermoreversible behavior of OSA WM starch.

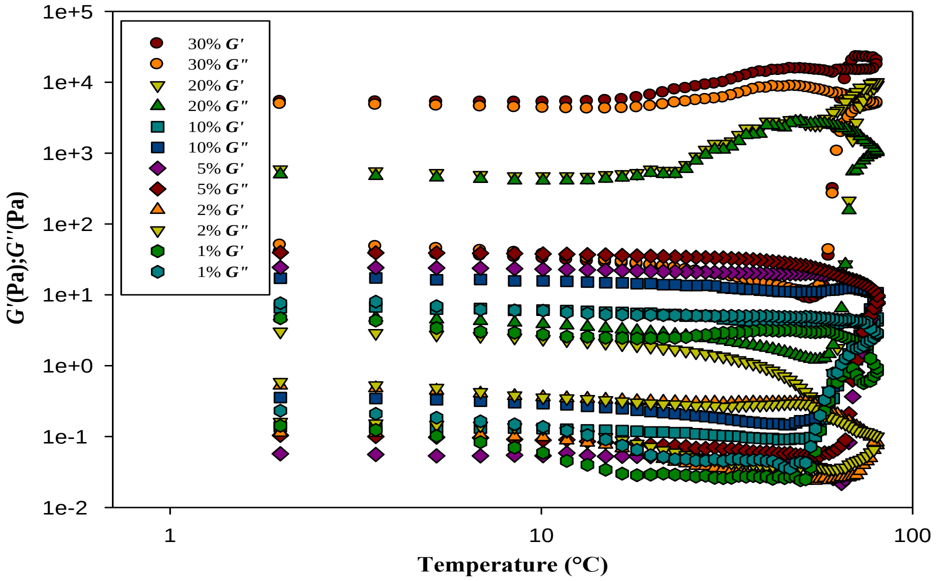


Figure 6. Effects of temperature and polymer concentration on the evolution of the elastic modules of OSA starch

4. CONCLUSION

The present investigation revealed that the commercial OSA WM starch (Gel N Melt) had amphiphilic properties which confirm its use in food formulations. On the other hand, the modified polysaccharide presented a shear-thinning behavior as the viscosity decreases with the shear rate. It was also observed the existence of two different populations of particles with a smaller average size and a second population formed by the residual fractions of unmodified particles. The thermorheological measurements showed clearly that the increase in the temperature induces a transition from a fluid behavior towards a viscoelastic behavior. In addition, the measurements in oscillation illustrated that from a critical temperature, the system becomes much more elastic which confirmed its thermoreversible behaviour. As a result, these findings can be considered as paramount elements to adapt in the biopolymer compositions for food applications.

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