

Rheological behavior and texture of corn starch gels (*Zea mays*), arrowroot (*Maranta arundinaceaea* L.) and cassava (*Manihote sculenta* Crantz)

Comportamento reológico e textura de géis de amido de milho (*Zea mays*) e de féculas de araruta (*Maranta arundinaceaea* L.) e de mandioca (*Manihote sculenta* Crantz)

Comportamiento reológico y textura de geles de almidón de maíz (*Zea mays*) y féculas de árbol (*Maranta arundinaceaea* L.) y yuca (*Manihote sculenta* Crantz)

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Abstract

The main energy reserve in plants is starch. It is unique among polysaccharides for being in the form of granules. Starch granules are heterogeneous mixtures of amylose and amylopectin. Gelatinization and retrogradation of starch depend on the ratio of amylose to amylopectin, type of crystallinity, along with the sizes and structure of starch granules. The present work determined the rheological behavior in a dynamic state, in terms of sweeping frequency, time and temperature, in addition to the extrusion of corn starch gels and arrowroot and cassava starches. The rheological study demonstrated that in all samples analyzed, whether in a non-gelatinized liquid mixture or in the form of gels, behaved as non-Newtonian fluids independent of time. Non-gelatinized mixtures showed properties of dilating non-Newtonian fluids and the gels of pseudoplastic non-Newtonian fluids. Gels were classified as elastic, as it was found that the storage module is larger than the dissipation module with the storage module decreasing with increasing temperature, and thus temperature dependent. With increased temperature, the gels showed low stability which is characteristic of weak gels. The more elastic the gel, the greater its resistance and the corn starch gel was the most resistant when compared to arrowroot and cassava starch.

Keywords: Gelatinization; Retrogradation; Fluids; Elasticity.

Resumo

A principal reserva de energia nas plantas é o amido, sendo abundante em sementes, raízes e tubérculos. De todos os polissacarídeos, o amido é o único produzido em pequenos agregados individuais, denominados grânulos. Os grânulos de amido são misturas heterogêneas de duas macromoléculas, amilose e amilopectina, que diferem no tamanho molecular e grau de ramificação. As propriedades de gelatinização e retrogradação do amido estão relacionadas a vários fatores, incluindo proporção de amilose e amilopectina, tipo de cristalinidade, tamanho e estrutura do grânulo de amido. Desta forma, o objetivo deste trabalho foi determinar o comportamento reológico em estado dinâmico, quanto à varredura de frequência, tempo e temperatura, além da extrusão dos géis de amido de milho e féculas de araruta e de mandioca. Demonstrou-se através do estudo reológico que todas as amostras analisadas, sendo em mistura líquida não gelatinizada ou em forma de géis, apresentaram características de fluidos Não-Newtonianos independentes do tempo. As misturas não gelatinizadas apresentaram

propriedades de fluídos Não-Newtonianos dilatantes e os géis de fluidos Não-Newtonianos pseudoplásticos. Os géis foram classificados como elásticos, pois verificou-se que o módulo de armazenamento é maior que o módulo de dissipação e dependentemente da temperatura, decresce com o seu aumento. Concluiu-se que com o aumento progressivo da temperatura, os géis apresentaram baixa estabilidade, característica de géis fracos. Quanto mais elástico é o gel, maior será a sua resistência, sendo que o gel de amido de milho foi mais resistente quando comparado às féculas de araruta e de mandioca.

Palavras-chave: Gelatinização; Retrogradação; Fluidos; Elasticidade.

Resumen

La principal reserva energética de las plantas es el almidón, siendo abundante en semillas, raíces y tubérculos. De todos los polisacáridos, el almidón es el único que se produce en pequeños agregados individuales, llamados gránulos. Los gránulos de almidón son mezclas heterogéneas de dos macromoléculas, amilosa y amilopectina, que difieren en tamaño molecular y grado de ramificación. Las propiedades de gelatinización y retrogradación del almidón están relacionadas con varios factores, incluida la proporción de amilosa y amilopectina, el tipo de cristalinidad, el tamaño y la estructura del gránulo de almidón. Así, el objetivo de este trabajo fue determinar el comportamiento reológico en estado dinámico, en términos de frecuencia de barrido, tiempo y temperatura, además de la extrusión de geles de almidón de maíz y almidones de arrurruz y yuca. Se demostró mediante el estudio reológico que todas las muestras analizadas, al estar en una mezcla líquida no gelatinizada o en forma de geles, presentaban características de fluidos no newtonianos independientes del tiempo. Las mezclas no gelatinizadas mostraron propiedades de dilatación de fluidos no newtonianos y los geles de fluidos pseudoplásticos no newtonianos. Los geles se clasificaron como elásticos, ya que se encontró que el módulo de almacenamiento es más grande que el módulo de disipación y, dependiendo de la temperatura, disminuye con su aumento. Se concluyó que con el aumento progresivo de temperatura, los geles mostraron baja estabilidad, característica de los geles débiles. Cuanto más elástico es el gel, mayor es su resistencia, y el gel de almidón de maíz es más resistente en comparación con el almidón de arrurruz y yuca.

Palabras clave: Gelatinización; Retrogradación; Fluidos; Elasticidad.

1. Introduction

Starch is an important energy reserve in plants, making it an important source of food for humans and important to the industrial sector. Its physical-chemical characteristics has applications are not limited to the food, pharmaceutical, textile, paper, and metallurgical industries (Vamadevan & Bertoft, 2015). Because of its diverse properties, food starch is widely used to improve the texture of products, to increase water retention, as well as a thickener, stabilizer and gelling agent (Silva, 2019). Its many uses require specific structural and functional properties (Zhang et al., 2018).

The shape and size of starch granules depend on its botanical source (Franco et al., 2001). Starch granules are heterogeneous mixtures of two macromolecules, amylose and amylopectin, made up of glucose units, which differ in molecular size and degree of branching (Mizukami; Takeda & Hizukuri, 1999). However, starch is not composed solely of carbohydrates, it also contains small amounts of proteins, lipids and minerals (Oyeyinka & Oyeyinka, 2017).

The gelatinization and retrogradation of starches are a function of several factors. These include the relative proportion of amylose and amylopectin, type of crystallinity, size and structure of starch granules (Lindeboom; Chang & Tyler, 2004). Gelatinization generally occurs over a wide temperature range, characteristic for each source of starch, affected primarily by the presence of water. Retrogradation happens when the starch molecules lose energy and the hydrogen bonds become stronger, with chains beginning to re-associate into a more orderly state (Eliasson, 1996; Singh et al., 2006). Starch is not found alone in food products. This makes the study of the interactions of starch with other components such as proteins, sucrose, salts, among others important, because many common products such as breads, pasta, sauces and other industrially processed products have their own characteristic textural and sensory attributes, which are a function of these complex interactions.

The rheological and textural properties are affected during the gelatinization and retrogradation process, which makes it important to evaluate and study each type separately (Roberts & Cameron, 2002). The basic feature of starch rheology is its viscosity (Alcázar-Alay & Meireles, 2015).

The objective of the present work was to determine the rheological behavior of a dynamic state, in terms of sweeping frequency, time and temperature, in addition to the extrusion of corn, arrowroot, and cassava starches.

2. Methodology

The raw materials, corn, arrowroot, and cassava starches, were purchased in the city of Goiânia-GO, at the Central Market. Analyses were performed at the School of Agronomy (EA) of the Federal University of Goiás (UFG), in the Multi-User Laboratory for Texture Analysis, Rheology and HPLC (LabMulti).

Rheology was studied, in terms of scanning the frequency, time and temperature of corn, arrowroot, and cassava starches, in liquid mixtures with non-gelatinized water, in liquid mixtures at the same gelatinized concentrations, and by extruded mixtures of gelatinized solids at a higher concentration.

The viscoelastic behavior of starch gels was studied through dynamic tests. The recorded rheological parameters were the storage (G') and dissipation (G'') modules.

2.1 Rheological study

For the preparation of the mixtures, a concentration of 3% of the raw material (w/v) was used (6 g per 200 ml of water), for each treatment. After homogenization, 50 ml of each mixture was removed, and kept in falcon tubes. The remainder was put into beakers for heating using a Bunsen burner for gelatinization. This made for a total of three ungelled and three gelled samples (one each of corn, arrowroot and manioc starches). Flow curves were made with the mixtures before and after gelatinization.

The parameters configured in the bench rheometer (Anton Paar Physica MCR 101, Ostfildern, Germany) were: deformation rate with rising, falling and rising ramps (1 to 500 s^{-1} ; 500 to 1 s^{-1} ; 1 to 500 s^{-1}), CP50-1 probe (cone-plate with 50 mm diameter and cone angle of 1°), a gap between the geometry and the rheometer plate 0.101 mm, and scanning at a linear rate at a temperature of 25° C .

2.2 Extrusion of corn, arrowroot, and cassava starches

The analysis was performed according to Ferreira (2014), with some modifications. For the preparation of the gels, a concentration of 20% of the raw material (w / v) was used, with 100g per 500 ml of water, for each treatment. After homogenization, in a volumetric flask, they were placed in 1000 ml beakers and heated until gelatinization with a Bunsen

burner, heating one treatment at a time. Then, they were molded into cylindrical shapes 36 mm in diameter and 26 mm high, with a base area of 936 mm².

The texture analysis was performed after 24 h of the samples preparation, in duplicate, in a texturometer (Texture Analyzer TA-XT Plus, Surrey, England), in compression force mode and operation to evaluate the maximum breaking force, obtained through the registration of the force x time curve. The texturometer was configured with the following settings: compression rate of 30% deformation; pre-test speed of 2 mm/s; test speed of 1 mm/s; post-test speed of 10 mm/s. A 36 mm diameter probe (P36-R), lubricated with mineral oil, was used. The analysis was performed at an ambient temperature of 25°C. This was followed by the AACCI 74-09.01 (2010) method adapted for gel texture.

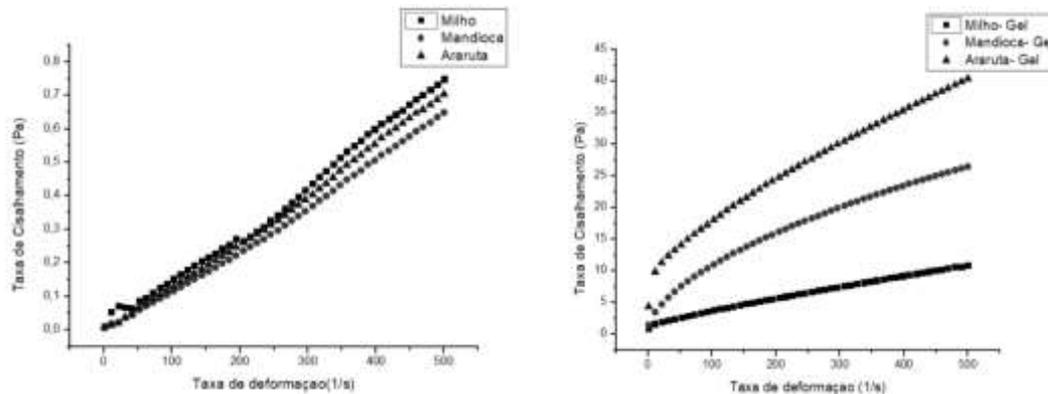
3. Results and Discussion

3.1 Rheological study

The description of the rheological behavior of the materials is made using models that varied the shear stress and the strain rate. When Newtonian fluids are deformed, the shear stress generated is directly proportional to the deformation rate. non-Newtonian fluids can be classified according to how apparent viscosity varies with strain rate, specifically whether it increases or decreases with the increase in the strain rate (Steffe, 1996).

Figure 1 shows the shear stress behavior in relation to the strain rate applied to corn, arrowroot, and cassava starches. All samples, in liquid mixture and in the form of gels, showed non-Newtonian fluid behavior independent of time, that is, its behavior cannot be described linearly, with variations in trajectory, with apparent viscosity dependent either on the deformation rate or shear stress.

Figure 1. Rheological behavior of mixtures (A) and gels (B) of corn, arrowroot, and cassava starches.



Source: Authors.

Starch gels are non-Newtonian fluids that can exhibit an initial shear stress at low strain rates (Giboreau, et. al., 1994; Rao, et al., 2005). The gels did not undergo a sudden shear, which suggests that they kept their structures with minimal changes. Apparent viscosity decreases as the deformation rate and shear time increase, because of the orientation of the molecules in the flow direction and the breakdown of aggregates, which reduce the resistance to movement (Barnes et al., 1989).

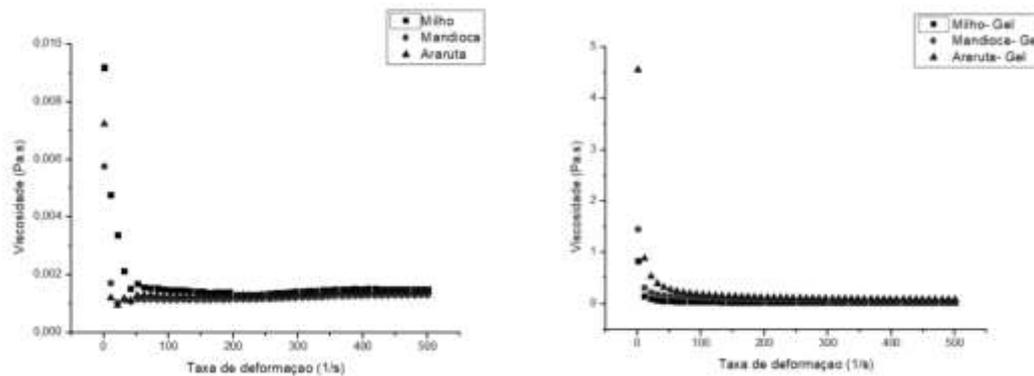
3.2 Fluid characteristics

The flow behavior either depends only on the deformation rate and not on the shear duration (independent of time), or it may also depend on the shear duration. Various types of behavior of food fluids independent of time can be classified (Rao, 2014). Figure 2 shows the fluid characteristics of each mixture and of each gel. Corn, arrowroot, and cassava starches displayed similar characteristics, being both non-Newtonian dilating fluids, when not gelatinized, and pseudoplastic non-Newtonian fluids, when gelatinized.

Expansion fluids show an increase in viscosity when the deformation rate increases (Scharamm, 2006; Araujo, 2003), causing the separation of the particles and, consequently, an increase in the overall volume of the mixture. As the tensions increase, the particles start to interact with each other, increasing the viscosity of the system (Holdsworth, 1971). Pseudoplastic fluids, on the other hand, suffer a decrease in viscosity when the deformation rate increases. Modification of the structure of long-chain molecules occurs with the increase

of the speed gradient. These chains tend to line up parallel to the current lines, decreasing the flow resistance (Thomaz, 2002).

Figure 2. Characteristics of mixtures (A) and gels (B) of corn, arrowroot, and cassava starches.



Source: Authors.

Gelatinized starches exhibit either pseudoplastic or dilating behavior depending on the degree of gelatinization (Bagley & Christianson, 1982; Okechukwu & Rao, 1995).

When the liquid mixtures were compared, similar characteristics were observed. When comparing the gelatinized material to the non-gelatinized material, there was a marked increase in viscosity. According to Coutinho and Cabello (2005), the cassava gel has non-Newtonian pseudoplastic fluid characteristics, giving an increase in the shear stress with the increase in the deformation rate. Rodrigues (2014) also detected in the arrowroot gel characteristics of a pseudoplastic fluid: both gels have behavior resulting from a fully organized network structure, formed by hydrogen interactions and polymer entanglement, which contributes for high viscosity at low shear rates.

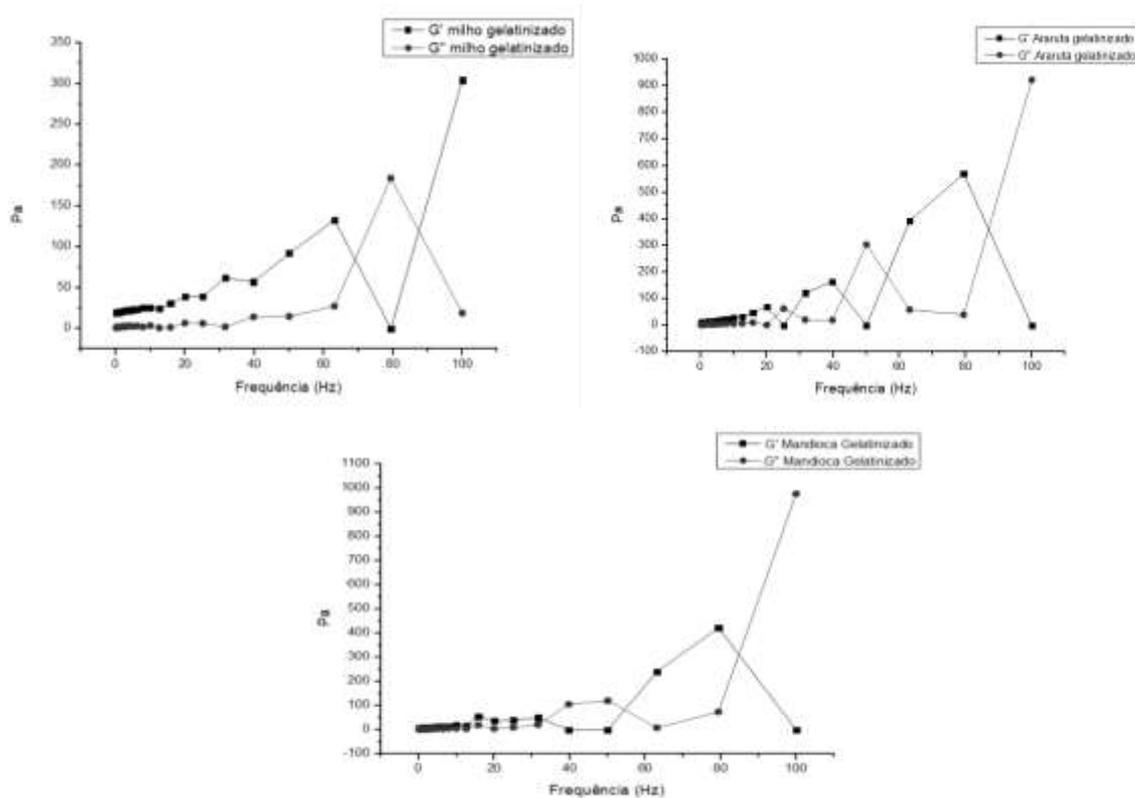
3.3 Dynamic state rheology

The variation of the storage (G') and dissipation (G'') modules with the oscillatory frequency provides information about the viscoelastic character of the system. Gels in which $G' > G''$ are characterized as elastic and the opposite behavior $G' < G''$ characterize them as viscous (Pereira et al., 2005).

For the behavior of corn starch and arrowroot and cassava starches (Figure 3), it was found that the storage module is larger than the dissipation module, $G' > G''$, that is, they are

characterized as elastic gels, where most of the energy is stored and the remainder is dissipated. Thus, the more elastic the gel, the greater its resistance. In this case, the most elastic and most resistant gel was corn starch, while arrowroot and cassava starches showed similar behaviors and similar resistance. The higher the G' value, the greater the solid character of the gel and the deformations will be elastic or recoverable (Kavanagh & Ross-Murphy, 1998; Rao et al., 1992).

Figure 3. Storage modulus (G') and dissipation module (G'') in relation to the frequency sweep of corn (A), arrowroot starch (B), and cassava starch (C).



Source: Authors.

Corn starch displayed a behavior with stronger elasticity when compared to the other starches, which could also be seen in the preparation of the samples, where, after the gelatinization and cooling process, the corn starch gel was more consistent in its elasticity and resistance than the arrowroot and cassava starch gels.

Ahmad and Williams (2001) studied the rheological behavior of cassava starch gel and concluded that the sample showed pseudoplastic behavior and that, even with the typical gel behavior of $G' > G''$. It was considered a weak gel, indicating that the greater the amount of

amylose in the starch, the greater the G' value. When it comes to a gel, the values of G' were significantly higher than G'' throughout the studied frequency range with both modules being nearly independent of the frequency. The point at which the curves intersect occurs when $G' = G''$, corresponding to a phase angle equal to $\pi / 4$ (Steffe, 1996).

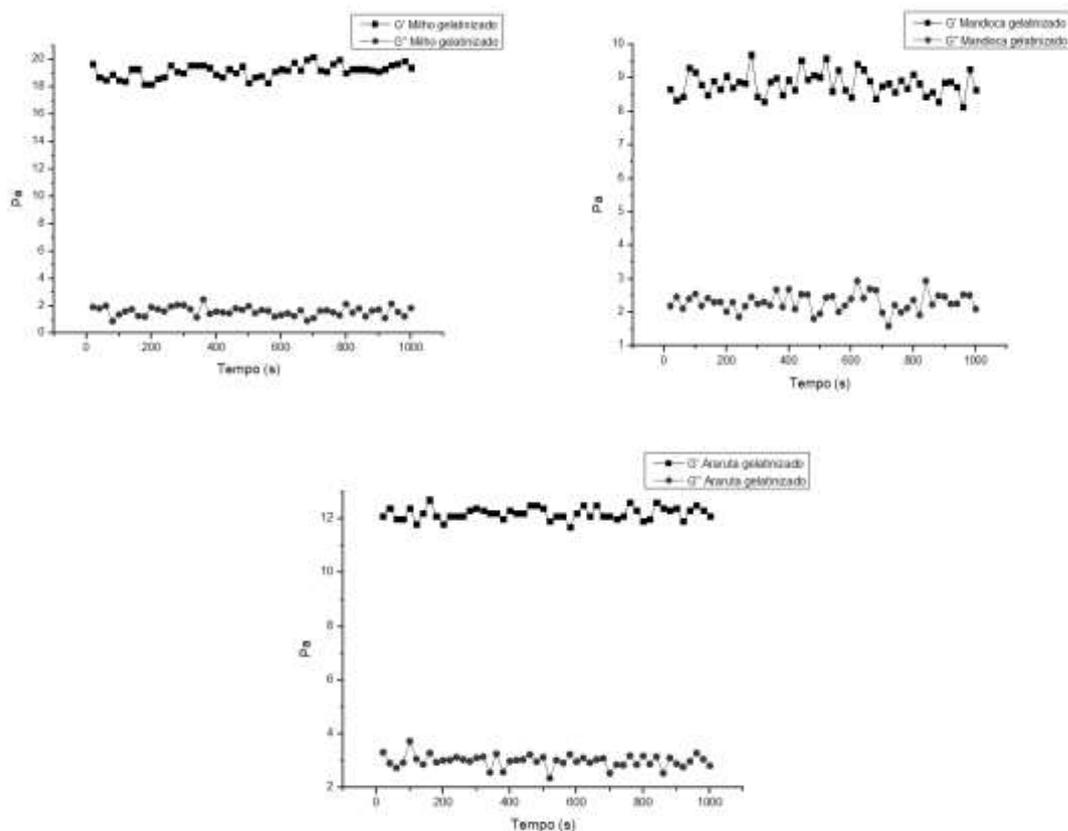
When the G' and G'' curves intersect, the gel is characteristic of a system of tangled polymers, with viscosity stronger than elasticity which is characteristic of weak gel behavior (Karam et al., 2006).

Time sweep is the study in which G' and G'' are determined as a function of time at fixed frequency and temperature. This type of test, often called a gel treatment experiment, is suitable for studying physical structure development in gels (Rao, 2014).

In most viscoelastic materials, linearity is observed when working with very small deformations. In this case, it can be said that the elastic effects follow Hooke's law and the viscous effects obey Newton's law (Alfrey & Gurnee, 1956). Hooke's Law is a law of physics that determines the deformation suffered by an elastic body from a force, whereas Newton's law comprises static and dynamic behaviors.

The time scan indicates time-dependent structural changes. The behavior of corn starch gels and arrowroot and cassava starches (Figure 4) indicated that the storage module (G') was higher in all materials; however, in corn starch and arrowroot starter there was greater stability between molecules, that is, small deformations of the physical structures of the gels had occurred.

Figure 4. Storage modules (G') and dissipation module (G'') in relation to the time scan of corn starch (A), arrowroot starch (B), and cassava starch (C).

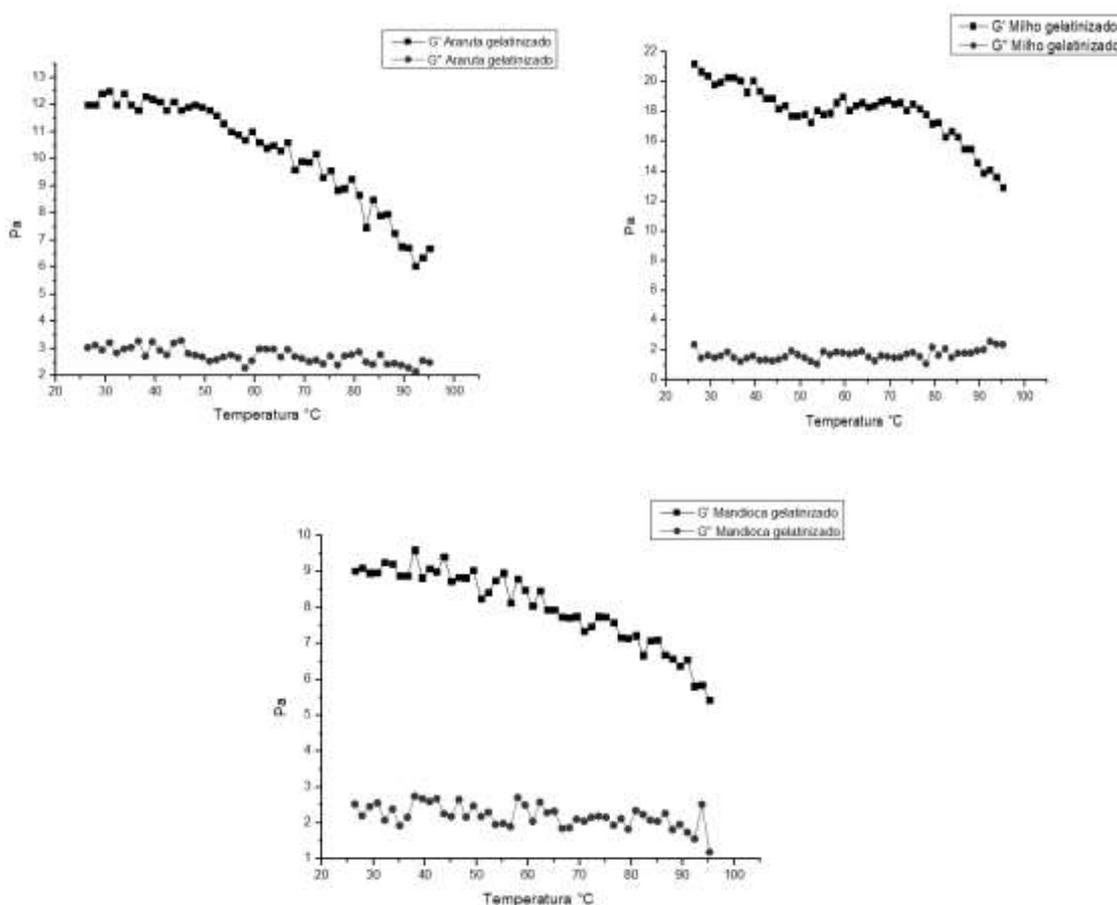


Source: Authors.

In the temperature scan (Figure 5), G' and G'' are determined as a function of temperature, at a fixed frequency. This test is suitable for the study of gel formation during the cooling of a heated dispersion (Rao & Cooley, 1993), gelatinization of starch dispersions during heating and formation of protein gels.

The storage modules for corn, arrowroot, and cassava starches showed an increase, but with time, there was a tendency for all temperatures to decrease. Similar results were obtained by Singh et al. (2006), in their study of the gelatinization kinetics of corn starch paste by rheology.

Figure 5. Storage modulus (G') and dissipation module (G'') in relation to the temperature sweep of corn starch (A), arrowroot starch (B), and cassava starch (C).



Source: Authors.

The values of G' and G'' both showed dependence on temperature, with the values of both decreasing with increased temperatures. The decrease in G' values, after peak temperature, can be attributed to the swelling to several times its previous size, the rupture of the starch granule, and simultaneous leaching of amylose. A three-dimensional network is formed by leached amylose (Tester & Morrison, 1990).

Biliaderis (1991); Tester and Morrison (1990) suggest that high transition temperatures indicate greater stability of the amorphous region and low degree of branched chains. High gelatinization temperatures explain the high stability of the starch granule region.

Note that with increasing temperature, the G' value decreases for corn, arrowroot, and cassava starches, indicating that the gel structure was destroyed during prolonged heating (Tsai et al., 1997). It was observed that with the progressive increase in temperature, the gels

studied showed low stability, characteristic of weak gels, but still maintaining $G' > G''$. There was greater instability for arrowroot and cassava starches. This can be explained by the loss of water during the process, which leads to faster retrogradation, due to the increased presence of amylose, and, consequently, syneresis occurs, or the expulsion of water from the molecule. The behavior of corn starch indicated stability at higher temperatures, until syneresis occurs, when compared to the starches studied.

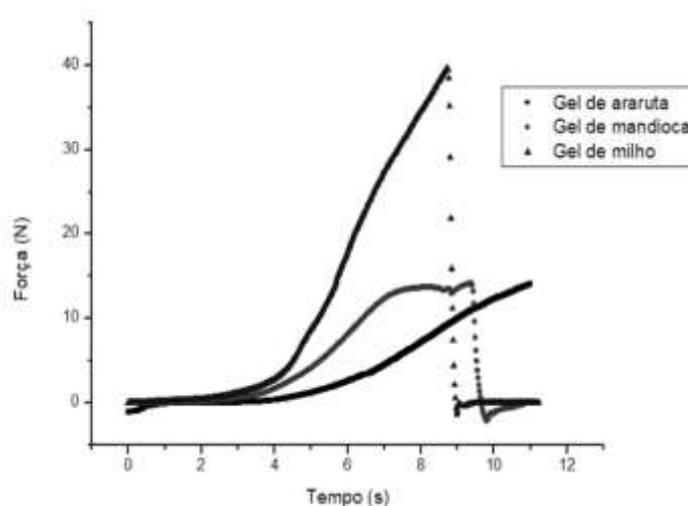
The high gelatinization temperatures result from a high degree of crystallinity, resulting from greater structural stability. This makes the granule resistant to gelatinization. The greater the resistance of the gel in relation to high temperatures, the greater its elasticity, with arrowroot, cassava, and corn starch showing increasing order, respectively.

3.4 Extrusion of gels

Texture is of great importance for the development of new products and tests with new ingredients, either for process optimization or texture profile determination (Szczesniak, 2002). The textural properties are closely related to the deformation, disintegration, and food flow under the application of a given system of forces (Geise, 1995).

Figure 6 shows that the higher the peak, the greater the material's resistance and the greater its ability to resist extrusion. Corn starch showed the greatest resistance in comparison to arrowroot and cassava starches.

Figure 6. Strength of corn, arrowroot, and cassava starch gels in relation to time.



Source: Authors.

The resistance of these gels is due to the amylose content present in each starch. Studies have shown that the amylose content of corn starch is approximately 27% and that of arrowroot and cassava starches 23.9% and 18%, respectively (Peroni, 2003; Leonel et al., 2001; Bobbio & Bobbio, 1995).

Sandhu and Singh (2007) studied the properties of corn starch and showed that the content of amylose is directly related to resistance, with the firmness of the gel being caused by retrogradation and associated with the syneresis and crystallization of amylopectin. Irani et al. (2019) and related the resistance of the gel to the starch retrogradation. In general, starches that form more resistant gels have a higher amylose content and long chain of amylopectin.

However, corn starch showed more resistance and elasticity, supporting greater compression forces. This is a desirable property for products in which crispness is necessary. Arrowroot and cassava starches showed similar behaviors because of their lower amylose content and consequently lower resistance and reduced elasticity, making them suitable for baked goods.

4. Conclusion

Corn, arrowroot, and cassava starches are non-Newtonian fluids. They are characterized independent of time as dilatants in non-gelatinized liquid mixtures and as pseudoplastics in gel form. Dynamic rheological tests showed that these are elastic fluids with weak gels, with corn starch being the most resistant, and that these characteristics are directly influenced by the structure and composition of the granules. It is suggested to evaluate the functional properties of starches, in order to compare their gels, and to use them as ingredients in food formulations.

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