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Article

Effect of Glycerol on Thermal Properties of Galactomannan

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Abstract

Galactomannan (Gal), a neutral polysaccharide extracted from the endosperm of legumes, stands out for its applications in the pharmaceutical, food, cosmetic and biomedical industries. The incorporation of additives such as glycerol, which acts as a plasticizer, can modulate the physicochemical properties of this biopolymer through changes in its molecular network. Considering this potential, this research objective was to evaluate the influence of the addition of glycerol on Gal's thermal properties. The material was prepared using heating solutions of pure Gal containing the following glycerol contents: 0.5 and 1.0 mL. Characterizations were performed using Fourier transform infrared spectroscopy, differential scanning calorimetry, and thermogravimetric analysis. The FTIR spectra revealed that the addition of glycerol promoted significant changes in the hydrogen network of Gal, replacing the rigid Gal-Gal interactions with more dynamic Gal-glycerol interactions. This structural modification was corroborated by thermal analyses, which demonstrated the plasticizing effect of glycerol, which inhibits the vitrification characteristic of pure Gal. These results indicate that plasticization with glycerol confers potential to Gal for applications as a biomedical glue, particularly in situations that require greater flexibility and adaptability to biological tissues.

Keywords: glycerol; glue; thermal analysis; infrared spectroscopy

1. Introduction

Galactomannan (Gal) is a natural polymer found mainly in the endosperm of leguminous seeds [1,2]. In general, galactomannan presents in its structure mannose units, which compose the main chain, and galactose units in the branches [3], being characterized by high solubility in solutions [4] and chemical stability in a wide range of pH due to non-ionic character [5]. It can be applied as a thickener/stabilizer in the food industry [6] and in medical applications, like in the diagnosis of invasive fungal infections [7–9] and the diagnosis of Aspergillosis [10].

The interaction of natural glue with different biological systems has been investigated [11,12], as polysaccharides are the raw material of interest for this application. The thermal properties of these polysaccharides, especially the presence of water, are a complex and essential process to determine

the ideal application and destination for the material [13] since it can be submitted to thermal treatments during the preparation or use [14]. Thus, it is interesting to develop new glues of natural origin in medical applications due to their biocompatibility. In civil construction, this material resists thermal expansion and bonds to the substrate [15].

The application of different natural polymers as glue/adhesive is observed in the literature, such as casein [16], soy protein [17], chitosan [18], cellulose [19], collagen [20], and others. It is also known that glycerol-based polymers have potential applications as more ecological adhesives [21]. In this context, this research aims to study the thermal behavior of the glue produced from galactomannan from the seeds of *Adenanthera pavonina* L. and glycerol through the analysis of mass loss events, thermal decomposition, and the process of glue glazing. The structure and composition of polysaccharides are expected to influence the thermodynamic behavior of biopolymers.

2. Materials and Methods

2.1. Galactomannan Source and Extraction

The seeds of *Adenanthera pavonina* L. were collected in the Fortaleza (Ceará) region in Brazil, while reagents and solvents were used without prior purification. The extraction of galactomannan was carried out following the method previously described by Macêdo et al. [6]. The seeds of *Adenanthera pavonina* L. were heated in distilled water for 30 minutes after boiling and then swelled after 24 hours. Subsequently, the obtained endosperm was manually separated from the tegument and the embryo, dried with acetone (purity 100.0 %, Synth), and ground to powder. The powder obtained was stored for posterior preparation of galactomannan/glycerol solutions.

2.2. Galactomannan Solutions of 5.0 %

Galactomannan (Gal) was obtained by solubilizing the endosperm in a solution pre-adjusted to pH 3.0 with acetic acid. Then, four grams of galactomannan were homogenized in 100 mL of 0.1% acetic acid solution. The solution was homogenized (in moderate stirring) and then centrifuged at 3000 rpm for 20 minutes. The precipitate was removed, and 0.2 mL of potassium sorbate (purity \geq 99.0%, Sigma / Aldrich) was added to the supernatant. The resulting solution was stored under refrigeration.

2.3. Galactomannan/Glycerol Solutions

Twenty grams of Gal 5.00 % were weighed in different flasks, and 0.2 mL of 0.3% potassium sorbate solution was added in both. After that, three solutions with glycerol (purity \geq 99.5%, Sigma/Aldrich) were prepared. Samples were classified according to the quantity of glycerol added: Gal5.00 - solutions without glycerol (0.00 mL), Gal5.05 - solutions with 0.50 mL of glycerol, and Gal5.10 - solutions with 1.00 mL of glycerol. Each solution was heated until it reached the consistency of gum.

2.4. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis was used as an effective tool to gain information on the vibrational of the galactomannan-glycerol solutions (Gal5.00 – Gal5.10). FTIR spectra were determined using a Fourier transform infrared spectrometer (SHIMATZU FTIR-283B). The gums were ground with spectroscopic grade potassium bromide (KBr) powder and then pressed into pellets for measurement in the wavenumber range of 400 and 4000 cm^{-1} .

2.5. Thermogravimetry (TG)

TG and Derivative Thermogravimetry (DTG) analysis were used to characterize thermal decomposition and evaluate the stability of films. The TG measurements were carried out in synthetic atmosphere air N2 (flow rate of 50 mL min⁻¹) using a Shimadzu TGA-50H equipment calibrated with

Indium as standard. The analyses started at 30 °C to 600 °C, with a heating rate of 10 °C min⁻¹. The samples were weighed (5 ± 0.1 mg) in alumina crucibles.

2.6. Differential Scanning Calorimetry (DSC)

DSC was used to investigate thermal transitions. The DSC measurements were carried out in an N2 atmosphere (flow rate of 50 mL·min⁻¹) using Shimadzu DSC-50 calibrated with Indium as standard. The analyses started at 30 °C to 400 °C with a heating rate of 10 °C min⁻¹. The samples were weighed (5 ± 0.1 mg) in alumina pans. The enthalpy was calculated using the area of the peaks between the onset temperature and the end set temperature.

3. Results

The characteristic vibrations of the galactomannan and galactomannan-glycerol solutions (Gal5.00, Gal5.05, and Gal5.10) were analyzed in the spectral region from 4000 to 500 cm⁻¹ by FTIR (Figure 1).

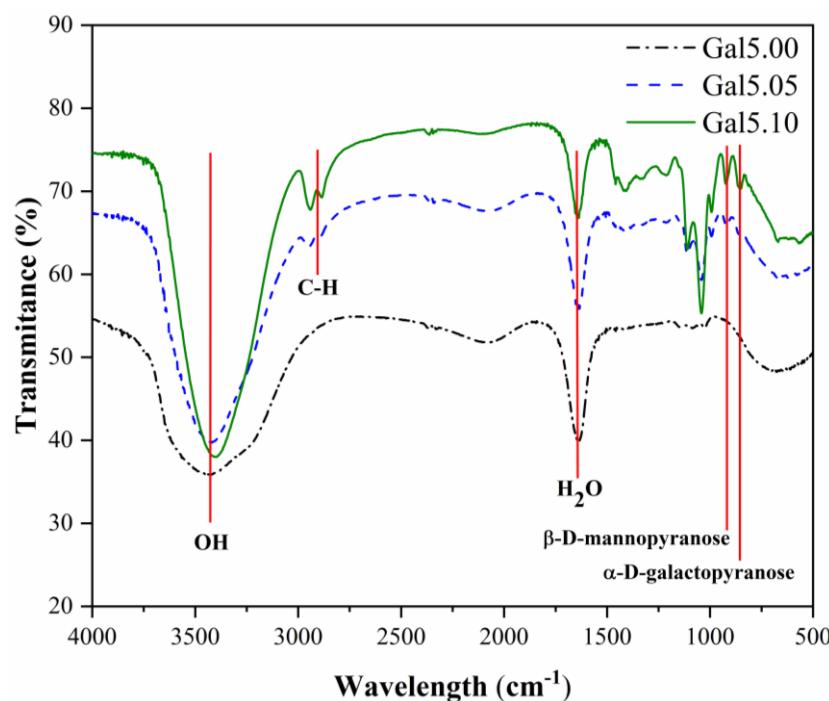


Figure 1. Spectra infrared of the samples Gal5.00, Gal5.05, and Gal5.10.

Figure 2 (a, b) shows the TG-DTG curves of Gal5.00, Gal5.05 and Gal5.10 samples. Gal5.0, two mass loss events were verified, while for Gal5.05 and Gal5.1, three events were observed.

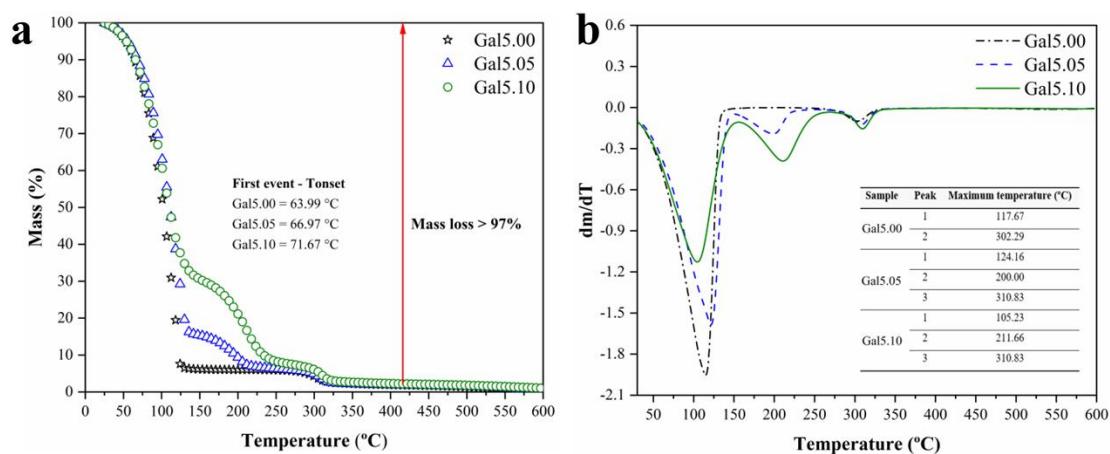


Figure 2. Thermogravimetry and Derivative thermogravimetry curves of the samples Gal5.00, Gal5.05, and Gal5.10.

The DSC results for the samples Gal5.00, Gal5.05, and Gal5.10 are present in Figure 3. The DSC curve of the Gal5.0 sample shows an endothermic peak at 157.97 °C and an exothermic peak at 303.57 °C, respectively. The endothermic peak is related to the water loss process, and the exothermic peak is associated with thermal degradation, considering that these processes occur at similar temperatures in TG (Figure 2a). Figure 4 presents a schematic representation of the interaction of galactomannan with and without the presence of a plasticizer.

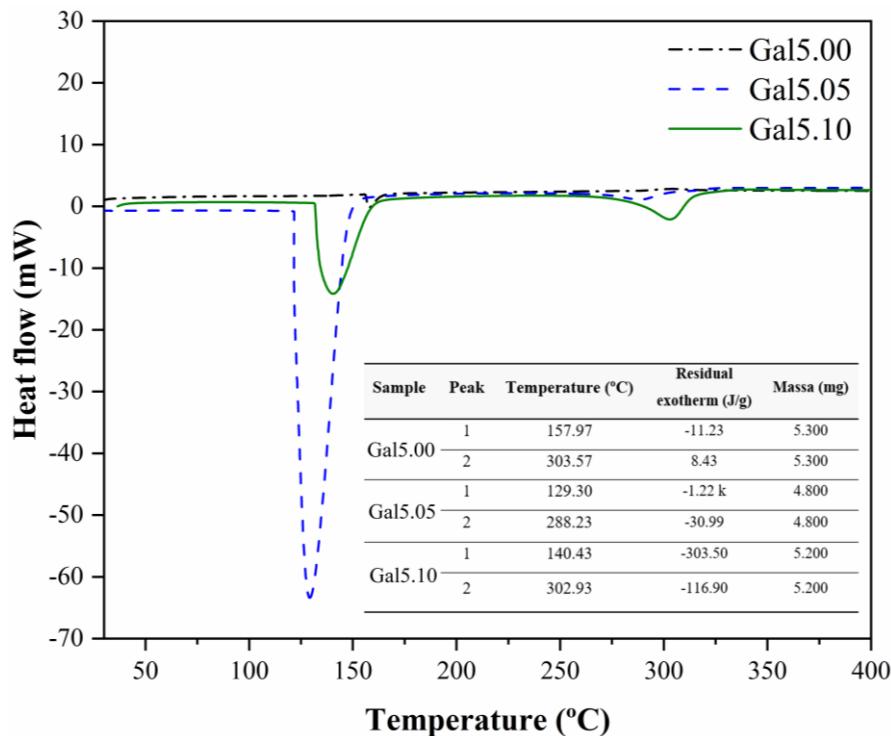


Figure 3. Differential scanning calorimetry curves for samples Gal5.00, Gal5.05, and Gal5.10.

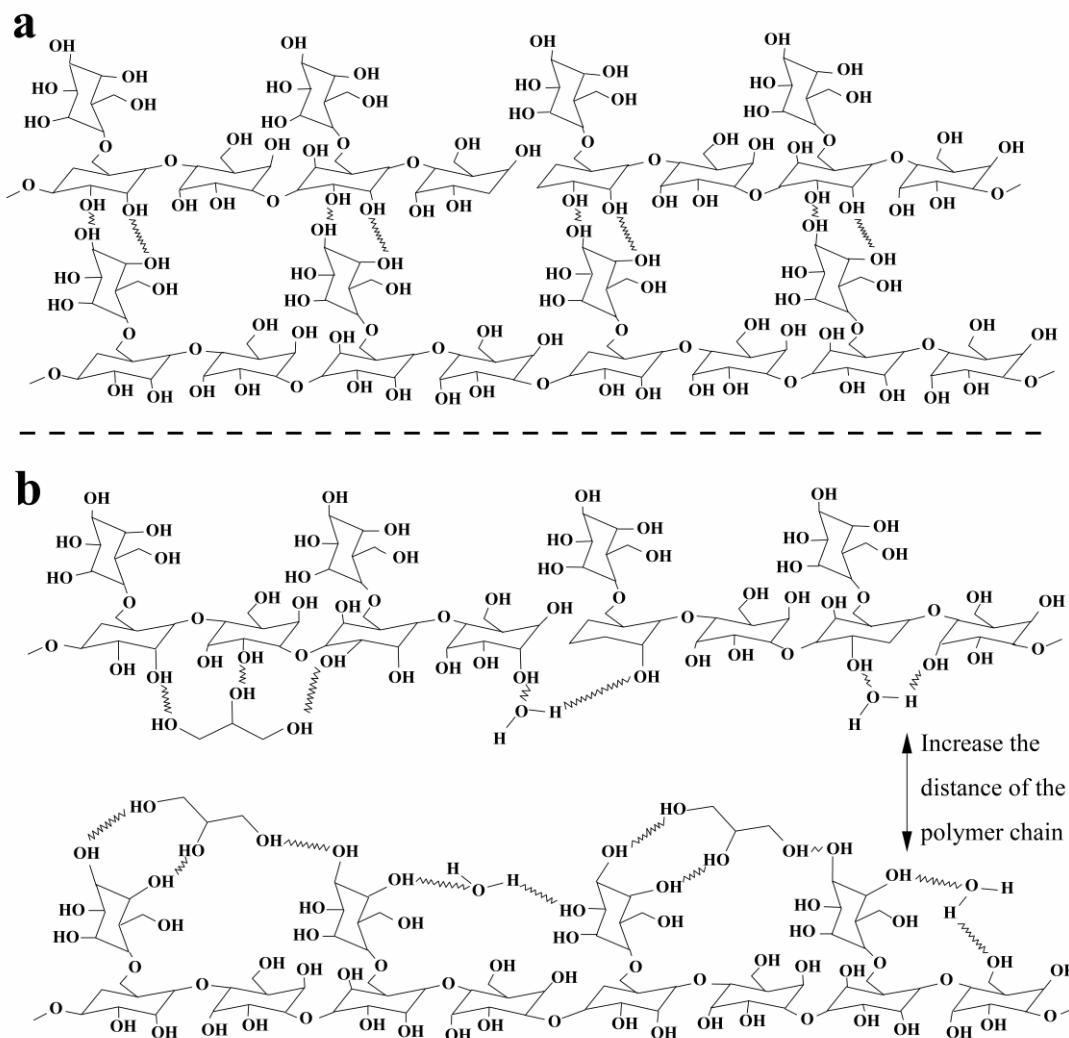


Figure 4. Schematic structure of galactomannan: a. in the absence of plasticizer; b. in the presence of plasticizer.

4. Discussion

According to the FTIR spectrum (Figure 1), the stretching in the region 3381–3430 cm^{-1} has been attributed to OH groups [22]. The 2081, 2073, and 2089 cm^{-1} peaks indicate combination bands, whereas the bands observed at 1637 and 1645 cm^{-1} correspond to the associated water [23]. An absorption band was detected in the region around 1483 - 1409 cm^{-1} , attributed to CH₂ deformation [24]. The peaks between 800 and 1200 cm^{-1} represented the highly coupled C-C-O, C-OH, and C-O-C stretching modes characteristic of carbohydrate polymers [23,24]. The region between 2953 and 2886 cm^{-1} is attributed to C-H stretching modes [24]. The characteristic absorption bands of galactomannan that indicate the presence of β -D-mannopyranose units and α -D-galactopyranose units were observed around 922 and 854 cm^{-1} [23]. The galactomannan bands in the Gal sample could not be analyzed due to their low concentration and the fact that the spectrum was obtained in solution.

The effect of adding glycerol on Gal is evidenced in the FTIR spectra of the samples by changes in the characteristic peaks observed at regions between 2953 and 2886 cm^{-1} and 1409 and 854 cm^{-1} . Other researchers have observed this behavior previously. According to the literature [25,26], the increasing glycerol concentration increases the area corresponding to the O-H stretching vibration region, associated with free, inter, and intra-molecular bound hydroxyl groups (3430-3381 cm^{-1}). This phenomenon arises from glycerol's hydrophilic nature and the formation of hydrogen bonds between the hydroxyl groups of Gal and glycerol [25]. Thus, glycerol does not merely physically blend with

Gal but actively modifies its hydrogen-bonding network by replacing Gal-Gal interactions with Gal-glycerol interactions. These changes indicate effective plasticization, as the introduction of glycerol disrupts the native intermolecular interactions between Gal chains.

Moreover, the bands at 2953, 2888, and 2886 cm^{-1} correspond to the stretching vibration of the C-H bond, which only appears in the glycerol solutions, confirming the more intense C-H stretching vibration in these solutions. Changes in the frequency range between 1409 and 800 cm^{-1} are also observed when glycerol is added to galactomannan solutions. For example, bending the C-O-H bond at 1409 cm^{-1} and the stretching vibration of the C-O bond at 1111 cm^{-1} may be attributed to a primary alcohol. The band at 1046 cm^{-1} corresponds to the stretching vibration of C-O, which appears in all samples; however, it increases intensity for galactomannan solutions with 0.5 and 1.0 mL of glycerol [25].

For the TG-DTG curves (Figure 2) of the Gal5.00 sample, the first event starts near 26.61 $^{\circ}\text{C}$, with a loss percentage of approximately 95% and a maximum temperature of 117.67 $^{\circ}\text{C}$, which can be attributed to water loss according to other works in galactomannans [27–29]. The second event begins at 267.77 $^{\circ}\text{C}$, with a maximum temperature of 302.29 $^{\circ}\text{C}$ and a loss of approximately 4% of the mass, suggesting the disintegration of the galactomannan macromolecular chains, followed by the degradation of the hexose rings according to the literature [30–35].

Three mass loss events were observed in similar temperature ranges for the TG-DTG curves of the Gal5.05 and Gal5.1 samples. The first curve, as in Gal5.0, is associated with the volatilization of water. Both Gal5.05 and Gal5.1 show the beginning of mass loss at 25.95 $^{\circ}\text{C}$. Maximum temperatures for Gal5.05 and Gal5.1 are 124.16 $^{\circ}\text{C}$ and 105.23 $^{\circ}\text{C}$, respectively. It was observed that the mass loss in both samples differs due to the presence of glycerol; Gal5.0 presented a water loss of 84%, and Gal5.1 presented a water loss of 66%. The event is associated with glycerol volatilization, which started at approximately 147.5 $^{\circ}\text{C}$ and 153.3 $^{\circ}\text{C}$ for Gal5.05 and Gal5.10, and maximum temperature of 200 $^{\circ}\text{C}$ and 211.66 $^{\circ}\text{C}$, respectively, with mass losses of 8.92% and 20.5%, respectively [30,32,33]. The third event is attributed to the thermal decomposition of galactomannan. This process occurs in the same temperature range and presents around the same gal mass.

No exothermic transition was found in samples Gal5.05 and Gal5.10 (Figure 3). Peak temperatures for the second endothermic transition occurred at 288.23 $^{\circ}\text{C}$ for G05 and 302.93 $^{\circ}\text{C}$ for Gal5.1. The absence of an exothermic peak at the thermal degradation temperature of samples Gal5.05 and Gal5.10 may be related to the masking of the exothermic peak by the overlap of the endothermic peak associated with the volatilization of glycerol. At the initial temperatures, a change in the heat capacity, characterized by the formation of a curve without the formation of peaks, is observed in Gal5.00. It is suggested that the occurrence of this curve may indicate the process of glue glazing. In the other samples, these transitions are not observed. It is estimated that this occurred due to the presence of the plasticizer.

The water and glycerol inserted in the glue act as a plasticizer, reducing the interaction between the polymer chains. These plasticizers' absence or low concentrations promote the vitrification of galactomannan chains. At present, water and glycerol interact intermolecularly, making hydrogen bonds with the -OH groups of Gal, preventing chain approximation (Figure 4) and vitrification. Consequently, after the volatilization of the water, glycerol still exists in Gal5.05 and Gal5.10; consequently, the polymer chains will remain in a malleable state instead of vitrifying.

5. Conclusions

Spectroscopic and thermal data agree that glycerol modifies the Gal hydrogen bond network, replacing Gal-Gal interactions with more dynamic Gal-glycerol interactions. This mechanism not only explains the inhibition of vitrification but also supports the use of Gal-based materials with tunable thermomechanical properties for specific applications. In the context of biomedical adhesives, glycerol plasticization possibly improves flexibility and long-term adhesion, in addition to preserving biocompatibility. To prove its efficacy as a biomedical adhesive, complementary studies are essential.

Author Contributions: All authors contributed to the study conception and design: Beatriz da Silva Batista (conceptualization, data curation, original draft, formal analysis); Rômicy Dermondes Souza (conceptualization, data curation, original draft, methodology); Maria Alexandra de Sousa Rios (investigation, formal analysis, validation); Lincoln Almeida Cavalcante (conceptualization, formal analysis); Walajhone Oliveira Pereira (formal analysis, methodology); Selma Elaine Mazzetto (investigation, methodology); Filipe Amaral (investigation, data curation, validation); Fernando Mendes (supervision validation, review editing); and, Ana Angélica Mathias Macêdo (project administration, resource, funding acquisition investigation, validation, review editing).

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Conflicts of Interest: The authors declare no conflicts of interest.

Abbreviations

The following abbreviations are used in this manuscript:

Gal	Galactomannan
Gal5.00	Gal solutions (5.00 %) without glycerol (0.00 mL)
Gal5.05	Gal solutions (5.00 %) with glycerol (0.50 mL)
Gal5.10	Gal solutions (5.00 %) with glycerol (1.00 mL)
FTIR	Fourier Transform Infrared Spectroscopy
TG	Thermogravimetry
DTG	Derivative Thermogravimetry
DSC	Differential Scanning Calorimetry

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