

THREE-DIMENSIONAL HYDRATED NETWORKS: AN INTEGRATED ANALYSIS OF THE MECHANICAL AND RHEOLOGICAL PROPERTIES OF CARRAGEENAN AND XANTHAN GUM GELS IN MULTICOMPONENT SYSTEMS

REDES HIDRATADAS TRIDIMENSIONAIS: UMA ANÁLISE INTEGRADA DAS PROPRIEDADES MECÂNICAS E REOLÓGICAS DE GÉIS DE CARRAGENANA E GOMA XANTANA EM SISTEMAS MULTICOMPONENTES

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Abstract

Hydrocolloids are highly hydrophilic substances that, when dispersed or dissolved in water, increase the viscosity of the system. Chemically, they are polysaccharides (such as gum arabic, xanthan gum, guar gum, carrageenan, carboxymethylcellulose, starch, and pectin) or proteins. This study investigated the mechanical and rheological properties of binary systems (hydrocolloid + water) and ternary systems (hydrocolloid + protein or sugar + water). Mechanical properties were assessed through texture analysis, while rheological properties were determined by frequency sweep tests under low stress and constant temperature. Average values for force (6.00 N), consistency (60.79 N·s), apparent elastic stress (0.08 N/cm²), and apparent elasticity (0.28 N/cm·s) were obtained for both systems. A 0.3% xanthan gum solution showed predominantly viscous behavior ($G'' > G'$) up to around 10 Hz, beyond which elastic behavior became dominant. The elasticity of xanthan-containing solutions was significantly influenced by sugars and other hydrocolloids. Increasing concentrations of sucrose and ovalbumin enhanced elasticity. Solutions containing carrageenan or xanthan gum tend to become both elastic and viscous as the concentrations of these components increase. It was also observed that the addition of ovalbumin and

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glucose to carrageenan solutions did not alter their properties, which is a favorable result, as these additives can act as protective agents without compromising the functionality of carrageenan.

Keywords: Analysis of Variance, Elasticity, Hydrocolloids, Rheological Properties, Sugars, Texture.

Resumo

Hidrocoloides são substâncias altamente hidrofílicas que, quando dispersas ou dissolvidas em água, aumentam a viscosidade do sistema. Quimicamente, são polissacarídeos (como goma arábica, goma xantana, goma guar, carragenana, carboximetilcelulose, amido e pectina) ou proteínas. Este estudo investigou as propriedades mecânicas e reológicas de sistemas binários (hidrocoloide + água) e ternários (hidrocoloide + proteína ou açúcar + água). As propriedades mecânicas foram avaliadas por meio de análise de textura, enquanto as propriedades reológicas foram determinadas por testes de varredura de frequência sob baixa tensão e temperatura constante. Valores médios de força (6,00 N), consistência (60,79 N·s), tensão elástica aparente (0,08 N/cm²) e elasticidade aparente (0,28 N/cm·s) foram obtidos para ambos os sistemas. Uma solução de goma xantana a 0,3% apresentou comportamento predominantemente viscoso ($G'' > G'$) até cerca de 10 Hz, frequência a partir da qual o comportamento elástico passou a predominar. A elasticidade das soluções contendo xantana foi influenciada significativamente por açúcares e outros hidrocoloides. O aumento das concentrações de sacarose e ovalbumina incrementou a elasticidade. Soluções contendo carragenana ou goma xantana tendem a apresentar características tanto elásticas quanto viscosas à medida que as concentrações desses componentes aumentam. Observou-se também que a adição de ovalbumina e glicose a soluções de carragenana não alterou suas propriedades, o que constitui um resultado favorável, visto que esses aditivos podem atuar como agentes protetores sem comprometer a funcionalidade da carragenana.

Palavras-chave: Açúcares, Análise de variância, Elasticidade, Hidrocoloides, Propriedades reológicas, Textura.



INTRODUCTION

Hydrocolloids are highly hydrophilic substances, soluble or that disperse in water, increasing the viscosity of the system. From a chemical point of view, they are polysaccharides (arabic gum, guar gum, carrageenan gum, xanthan gum, carboxymethylcellulose, starch, pectin) and proteins (Ibanes and Ferreira, 2003). Hydrocolloid mixtures are commonly used to impart new and improved rheological properties to food products, in addition to providing cost reduction (Phillips & Williams, 2000).

The presence of sugars has also been related to the increase in thermostability of proteins and subsequent gelation, probably due to the establishment of hydrogen bonds between sugars and water, which requires greater thermal energy to overcome the barrier energy that hinders the unfolding of protein molecules (Antipova and Semenova, 1997).

Polysaccharides are very versatile biopolymers that can be found in nature in the most diverse forms, performing different functions. Polysaccharides contribute to the structural integrity and mechanical forces of solutions, thus forming three-dimensional hydrated networks (Lapasin et al., 1999).

Gels formed from hydrocolloids are referred to as "physical gels", because the junction zones are formed through physical interaction, for example, by hydrogen bonding, hydrophobic association, cationic crosslinking, etc., and differ from synthetic polymeric gels that normally consist of polymer chains crosslinked by covalent bonds (Phillips & Williams, 2000).

Rheology is the science that studies the flow and deformation of materials. Currently it has become a basic tool for fluid handling, sizing of pumps and agitators and fermenter and heat exchanger designs, among other aspects (Gabas et al., 2012). Knowledge of rheological properties (viscosity, plasticity, elasticity, etc.) requires knowledge directed at maintaining the shape of the material, control of senses and flow, type of container to be used, among other applications.



MATERIALS AND METHODS

MATERIALS

The solutions were prepared using xanthan gum (Product Number G1253), Carrageenan type I commercial grade, suitable for gel preparation (Product Number C1013), Glucose - Anhydrous ACS Reagent (Product Number G5767), Sucrose (Product Number 80389) and Albumin obtained from Chicken egg grade II (power of 66-88%; Agarose gel electrophoresis; Product Number A5253). All materials were purchased from Sigma-Aldrich (St. Louis, USA) and ultrapure water (conductivity approximately 0.05 $\mu\text{S}/\text{cm}$; electrical resistivity approximately 18.2 $\text{M}\Omega\cdot\text{cm}$; Master System P&D Gehaka, Brazil).

GEL PREPARATION

Gel formation was adapted from the methodology of Christ et al. (2005) and Calliari et al. (2011). A 100 mL beaker with a diameter of approximately 47 mm was used. The samples were weighed on an analytical balance (Model Ay220 from Shimadzu, Japan) with an uncertainty of ± 0.02 .

For binary systems, a quantity of water between 10 and 20 mL was placed to prevent the polysaccharides from sticking to the walls of the beaker, the concentration of Carrageenan or Xanthan Gum was added in percentages of 0.5; 1.0; 1.5; 2.0 and 3.0%, and water was added to complete 100% of the solution.

For ternary systems, the same proportions of hydrocolloids were used, adding sucrose in proportions of 5.0; 10.0; 15.0 and 20.0%, or glucose in the same proportions, or ovalbumin in proportions of 0.42; 0.83; 1.67 and 3.33%. After homogenization in a mixer (Ultra-Turrax, Ika T25 Digital, Germany) for 20 minutes at 10,000 rpm, ultrasound was applied (Ultrasonic processor Heischer, Germany) for 30 minutes at 60% amplitude, followed by an ultrathermostatic bath (Quimis, Brazil) at 80°C for 30 minutes and cooling for 24 hours before analysis.



TEXTURE ANALYSIS

Texture analysis was performed on a TA.XT Plus texture analyzer (Stable Micro Systems, United Kingdom), equipped with a 25 mm diameter cylindrical probe. The parameters evaluated were: force (F), consistency (Cs), cohesiveness (Co) and viscosity index (Iv). From these data, the apparent elastic stress (σ) and apparent elasticity (Ea) were calculated using the equations:

$$\sigma = F / A \quad e \quad Ea = \sigma \times t$$

Where F is the penetration force (N), A is the probe area (cm²) and t is the deformation time (s).

RHEOLOGICAL ANALYSIS

The rheological properties were determined by controlled deformation rheometer (AR 1500ex, TA Instruments, USA), using parallel plate geometry of 40 mm diameter, with a gap of 1 mm. The analyses were performed at 25°C. Initially, an oscillation stress sweep was performed to determine the linear viscoelastic zone. A stress of 1.0 Pa was selected for all samples. Then, a frequency sweep (0.1 to 100 Hz) was performed to obtain the storage (G') and loss (G'') moduli.

STATISTICAL ANALYSIS

Analysis of variance (ANOVA) was employed to evaluate the influence of different hydrocolloids, sugars and proteins on the mechanical and rheological properties of the gels. A significance level of 5% ($p < 0.05$) was adopted.



RESULTS AND DISCUSSION

BINARY SYSTEMS

Texture analysis

Table 1 presents the average values of force (F), consistency (Cs), apparent elastic stress (σ) and apparent elasticity (Ea) for the binary systems Carrageenan + H₂O and Xanthan Gum + H₂O.

Table 1

Apparent Elasticity Values of Carrageenan (CA) and Xanthan Gum (GX) in Binary System.

CA (% m/m)	F (N)	Cs (N·s)	σ (N/cm ²)	Ea (N/cm·s)
0,5	6,00	60,79	0,18	0,22
1,0	29,64	308,00	0,88	1,09
1,5	68,33	718,24	2,03	2,52
2,0	159,56	1660,52	4,75	5,89
3,0	332,67	3408,37	9,90	12,28
GX (% m/m)				
0,5	0,18	1,64	0,0054	0,0066
1,0	0,34	3,94	0,0101	0,0125
1,5	0,55	6,82	0,0164	0,0203
2,0	0,81	10,76	0,0241	0,0299
3,0	1,91	26,71	0,0568	0,0705

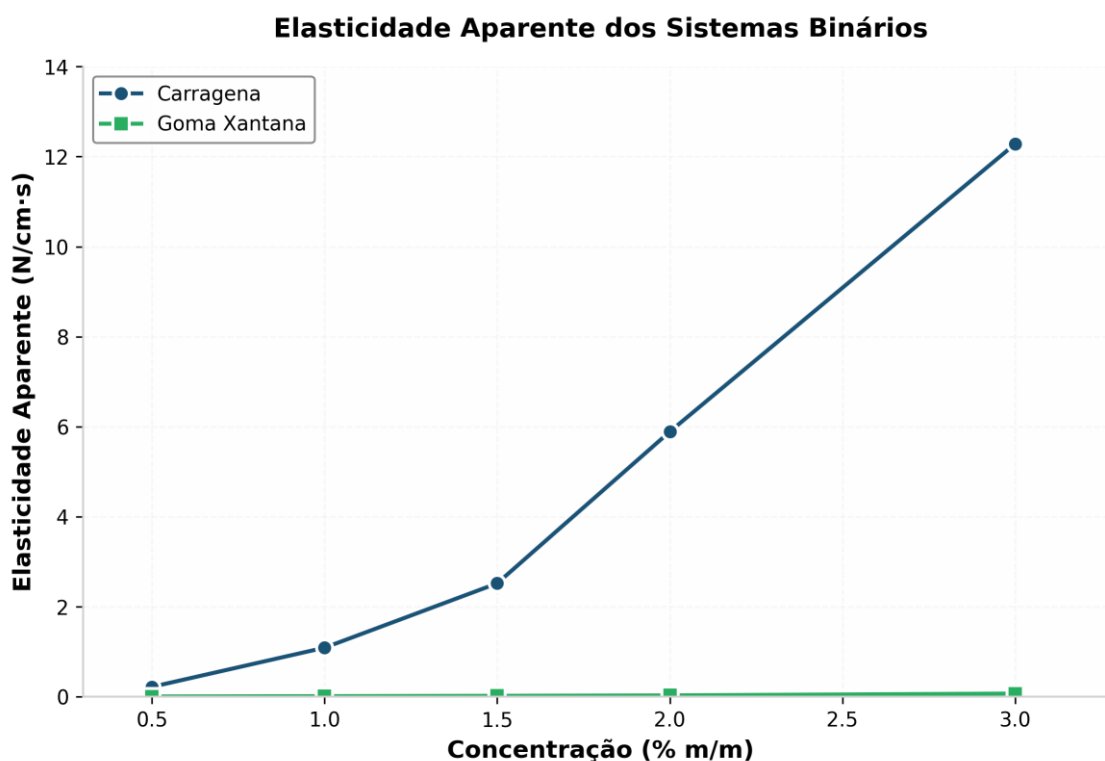
Source: Zódima (2025).

It is observed that with increasing polysaccharide concentration there is a significant increase in apparent elasticity. Carrageenan showed considerably higher values than xanthan gum, even at identical concentrations. This behavior can be explained by the absorption power of carrageenan (one part of carrageenan absorbs 30 parts of water), which allows increased economic yield and favors the formation of a firmer gel (Nussinovitch, 1997). Carrageenan is a high molecular weight polysaccharide, characterizing the difference in values with xanthan gum.



Figure 1

Apparent elasticity of binary systems as a function of hydrocolloid concentration.



Source: Zódima (2025).

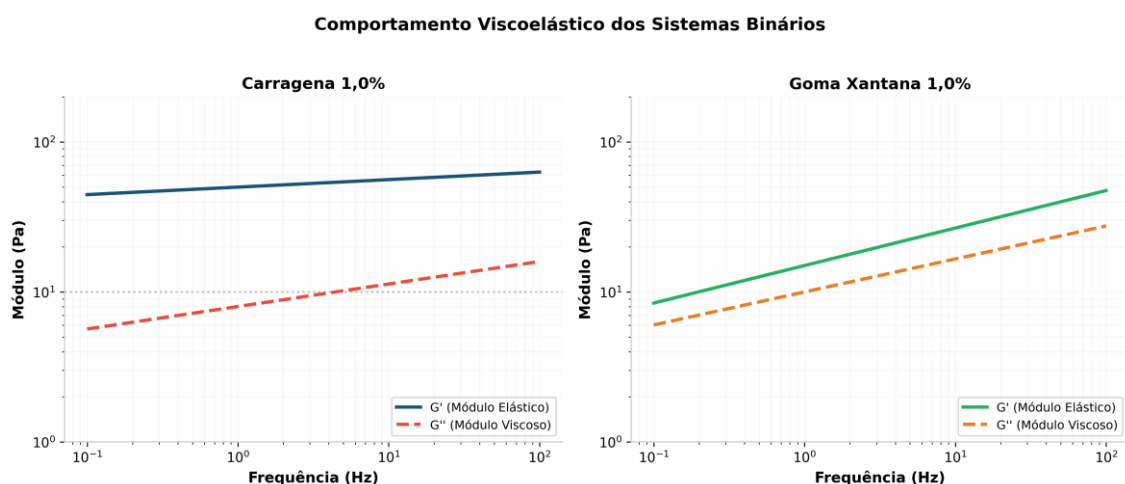
Rheological analysis

Figures 2 present the mechanical spectra of the binary systems. For 1.0% carrageenan, it is observed that the storage modulus (G') showed higher values than the loss modulus (G'') throughout the analyzed frequency range, characterizing a predominantly elastic behavior and gel definition.



Figure 2

Viscoelastic behavior of binary systems: G' and G'' moduli as a function of frequency. Closed symbols: G' ; Open symbols: G'' .



Source: Zódima (2025).

For 0.5% xanthan gum, viscous behavior predominates over elastic behavior up to approximately 10 Hz, above which elastic character becomes dominant. For higher concentrations, elastic behavior predominates throughout the entire frequency range, characteristic of a weak gel (Takeuchi, 2008). According to Brandão et al. (2010), xanthan gum exhibits high viscosity in aqueous solutions at low concentrations (0.05-1%), which can be used to describe the increase in G' and G'' values.

TERNARY SYSTEMS

Influence of sugars

Tables 2 and 3 present the texture analysis results for the ternary systems Carrageenan + Glucose + H₂O and Xanthan Gum + Sucrose + H₂O, respectively.

**Table 2**

Texture Analyses of Carrageenan + D-Glucose + H₂O.

CA (% m/m)	F (N)	Cs (N·s)	σ (N/cm ²)	Ea (N/cm·s)
0,5 + GL 5%	4,81	56,11	0,14	0,18
1,0 + GL 5%	26,88	277,23	0,80	0,99
1,5 + GL 5%	84,21	851,89	2,51	3,10
2,0 + GL 5%	164,00	1685,24	4,88	6,05
3,0 + GL 5%	362,52	3590,56	10,79	13,38

Source: Zódima (2025).

It is observed that the addition of D-glucose promoted a slight increase in the values of apparent elasticity. The values began to be noticeable from concentrations of 1.5% carrageenan with 5.0% glucose, due to the high concentration of carrageenan in the solution. D-glucose, when placed in an aqueous base, isomerizes into D-fructose, with no great variation in the values, as they have approximately equal molar mass.

Table 3

Texture Analyses of Xanthan Gum + Sucrose + H₂O.

GX (% m/m)	F (N)	Cs (N·s)	σ (N/cm ²)	Ea (N/cm·s)
0,5	0,22	2,32	0,0065	0,0081
1,0	0,49	5,62	0,0146	0,0181
1,5	0,94	10,67	0,0280	0,0347
2,0	1,83	19,54	0,0545	0,0675
3,0	3,34	44,97	0,0994	0,1233

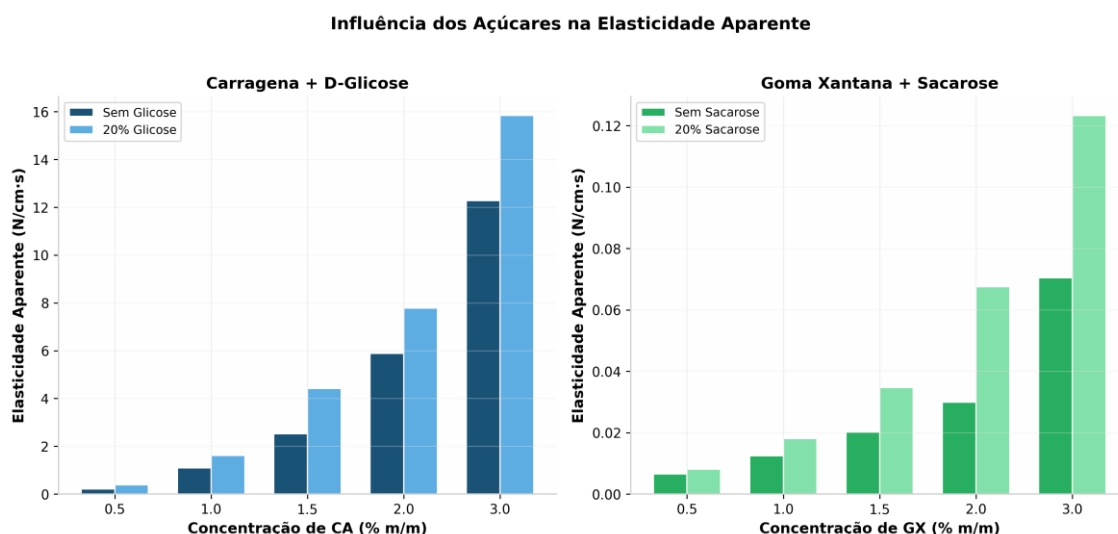
Source: Zódima (2025).

The addition of sucrose to xanthan gum also resulted in an increase in the values of force, consistency and apparent elasticity. This behavior can be explained by the fact that D-glucose is a constituent part of sucrose (Ferreira et al., 2009). Sucrose is a disaccharide widely used in the formulation of dairy products and can cause major changes in the microstructure and rheological properties of these products, depending on the concentration added (Antipova et al., 1999).



Figure 3

Influence of sugars on the apparent elasticity of ternary systems.



Source: Zódima (2025).

Influence of ovalbumin

Tables 4 and 5 present the results of the addition of ovalbumin to the carrageenan and xanthan gum systems, respectively.

Table 4

Texture Analyses of Carrageenan + Ovalbumin + H₂O.

CA (% m/m)	F (N)	Cs (N·s)	σ (N/cm ²)	Ea (N/cm·s)
0,5	11,28	135,85	0,34	0,42
1,0	24,91	268,22	0,74	0,92
1,5	62,04	709,56	1,85	2,29
2,0	103,20	1113,33	3,07	3,81
3,0	148,31	1611,08	4,41	5,47

Source: Zódima (2025).

When ovalbumin protein is added to carrageenan, the values of force, consistency and consequently apparent elasticity decrease compared to the binary system. This phenomenon can be explained by the formation of foam during mixing, which influences the final force values due to the shape of the analysis performed.



Table 5

Texture Analyses of Xanthan Gum + Ovalbumin + H₂O.

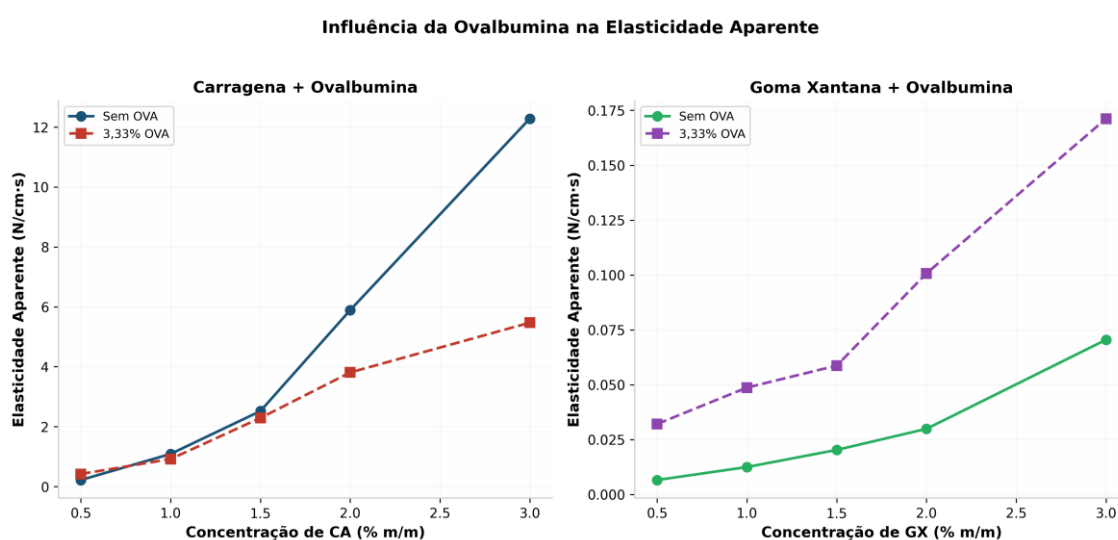
GX (% m/m)	F (N)	Cs (N·s)	σ (N/cm ²)	Ea (N/cm·s)
0,5	0,87	11,62	0,0259	0,0321
1,0	1,32	13,70	0,0393	0,0487
1,5	1,59	20,99	0,0473	0,0587
2,0	2,73	33,77	0,0813	0,1007
3,0	4,64	59,74	0,1381	0,1712

Source: Zódima (2025).

Contrary to what was observed with carrageenan, the addition of ovalbumin to xanthan gum resulted in an increase in elasticity values. Xanthan gum is compatible with most colloids used in foods (Nussinovitch, 1997), which can explain the different behavior observed.

Figure 4

Influence of ovalbumin on the apparent elasticity of ternary systems.



Source: Zódima (2025).

Rheological analysis of ternary systems

In the rheological analyses of the ternary systems, it was observed that the viscoelastic properties of mixed and pure biopolymer gels differed considerably from each other due to intermolecular interactions between the different components of the mixture and possible formation of an interpenetrating three-dimensional network (Takeuchi, 2008).



For carrageenan solutions with D-glucose, it was observed that as the glucose concentration increases, the G' and G'' values also gradually increase. For the highest carrageenan concentrations, G' remained greater than G'' throughout the frequency range, characterizing a weak gel, according to Steffe's (1996) classification.

The addition of sucrose to xanthan gum promoted significant changes in the rheological behavior. At higher concentrations, a greater difference was observed between the G' and G'' values, confirming the gel behavior. This result is in agreement with Barbosa (2013), Takeuchi (2008) and Braga (2006), who also observed that for lower sucrose concentrations there was practically no significant variation in the modulus values.

STATISTICAL ANALYSIS

Analysis of variance (ANOVA) was employed to evaluate the influence of hydrocolloid concentration on the apparent elasticity of the binary systems. The linear behavior was chosen for each binary system, using the lack of fit, t-test and residual analysis.

The parameters found in the residual analysis are presented in Table 6.

Table 6

Parameters found in the residual analysis.

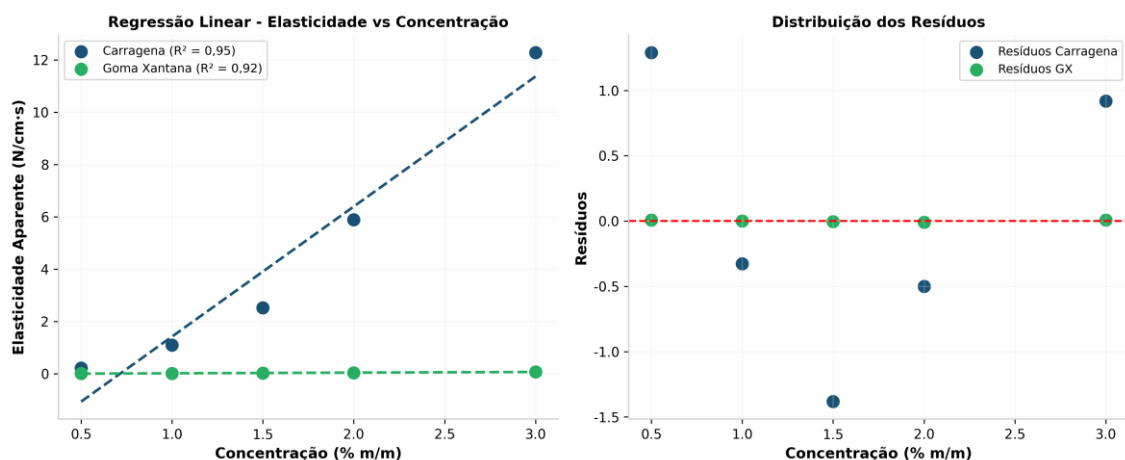
Hidrocoloide	Modelo	R ²	Correlation
Goma Xantana	$Y = 2,54x - 0,01$	0,92	0,95
Carragena	$Y = 510,98x - 3,70$	0,95	0,97

Source: Zódima (2025).



Figure 5

Linear regression of apparent elasticity versus concentration and distribution of residuals.



Source: Zódima (2025).

The analysis of variance showed that there was no significant influence of glucose on the apparent elasticity of carrageenan, nor of sucrose on xanthan gum, for the adopted significance level. Therefore, the information from the binary system can be considered valid for the analysis of these ternary systems.

FINAL CONSIDERATIONS

This work presents a contribution to the study of the mechanical and rheological behavior of gels formed by hydrocolloids and sugars. In the mechanical analysis, through texture analysis, it was possible to study the behavior of the gels and know their proper applications.

For binary systems, even at low carrageenan concentrations it is already possible to perceive the influence on elasticity, but for solutions with xanthan gum, only at concentrations of 1.5% did the elasticity values begin to show significant changes.

For ternary systems, when adding D-glucose to solutions with carrageenan, a slight influence on the values of apparent elasticity is perceived. When adding ovalbumin to carrageenan, a decrease in the values was observed, evidencing a possible structural incompatibility between these hydrocolloids.



Solutions containing xanthan gum are more influenced by other hydrocolloids and sugars, and for each increase in sucrose and ovalbumin concentration the influence on elasticity can already be perceived, since xanthan gum shows great compatibility with most biopolymers.

The rheological studies contributed to the knowledge of gel behavior, providing information complementary to texture analysis. It was possible to classify the solutions as dilute solutions, concentrated solutions, or weak/strong gels, according to the concentration and type of hydrocolloid used.

It was noted that the addition of ovalbumin and glucose to carrageenan did not significantly alter its properties, which is positive, as these additives can act as protective agents without compromising the functionality of carrageenan.

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