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The wettability of carrageenan-based edible coatings on chicken breasts[1]

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ABSTRACT - The aim of the present study was to characterise the surface properties of chicken breasts, and evaluate the wettability properties of edible coatings based on carrageenan and glycerol, using varying concentrations of the biopolymer and plasticiser. The surface of the chicken breasts was characterised as of low energy, with a surface tension of 54.56 mN/m, whose polar and dispersive components were 39.79 mN/m and 14.77 mN/m, respectively. The results showed that with an increase in the carrageenan concentration of the film-forming solution there was a reduction in the values for adhesion and spreading. Coatings with a higher glycerol concentration also obtained a lower spreading coefficient. Solutions 1 (carrageenan 1% and glycerol 0%), 2 (carrageenan 1% and glycerol 1%) and 3 (carrageenan 1% and glycerol 2%) did not differ statistically, obtaining the highest values for spreadability. Solution 1 (carrageenan 1% and 0% glycerol) presented the highest numerical values. For carrageenan coatings on chicken breasts, the best spreading coefficient was -4.49 mN/m.

Key words: Plasticiser. Contact angle. Surface properties.

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INTRODUCTION

There is great demand in the food industry for the development of non-thermal preservation techniques and/or the introduction of new ingredients to extend the shelf life and/or improve the quality of poultry products. (ALA; SHAHBAZI, 2019). Although there are a large number of synthetic and chemical preservatives that are used to maintain the quality of food during storage, consumers are currently seeking foods with characteristics closer to those of natural products, leading to greater interest in developing products prepared with natural additives (JEON; KAMIL; SHAHIDI, 2002).

Edible coatings can preserve food quality by delaying lipid oxidation, inhibiting microbial growth, and reducing sensory spoilage and moisture loss, as edible films reinforce natural layers to prevent moisture loss, allowing the transfer of gases such as carbon dioxide (CO_2) and oxygen (O_2) to be controlled (ATIENO *et al*., 2019; CARRIÓN-GRANDA *et al*., 2018; KUMAR; BHATNAGAR, 2014; SABERI *et al*., 2016).

Polysaccharides and proteins are the hydrocolloid matrices that are currently most used in producing edible films and coatings (FALGUERA *et al*., 2011). Edible coatings based on polysaccharides, such as carrageenan, have good barrier properties. However, despite being highly permeable to water vapour compared to other films, such as plastic, they are still able to delay water loss from food by functioning as the first layer to lose water (ALVES *et al*., 2011; MARTINS *et al*., 2012).

However, the effectiveness of edible coatings largely depends on the wettability of the coating on the surface to which it is applied. When the coating is ideal, spreadability on the surface to be coated is practically spontaneous (CASARIEGO *et al*., 2008). This property affects the thickness and uniformity of the coating and, consequently, influences its properties after drying (ALCANTARA *et al*., 2019).

The wettability of a coating is directly influenced by the surface energy or surface tension of the two surfaces, as these factors affect the forces that control the wetting and coating of surfaces (DAVID; NEUMANN, 2014). Surface tension is a result of adhesive forces that promote the spread of a liquid on a solid surface, and cohesive forces, which promote its contraction. The wetting behaviour of the resulting solutions depends mainly on the balance between these two forces (LIMA *et al*., 2010; RIBEIRO *et al*., 2007; SOUZA *et al*., 2010).

Previous studies have reported the wettability and surface properties of solutions used to form coatings, and their corresponding application in fruit, vegetables and fish (ALCANTARA *et al*., 2019; CASARIEGO *et al*., 2008; CERQUEIRA *et al*., 2009; LIMA *et al*., 2010; RIBEIRO *et al*., 2007; SOUZA *et al*., 2010). However, there are no reports of these properties being investigated in chicken meat. The aim of the present study therefore was to characterise the surface of chicken breasts and evaluate the effects of concentrations of carrageenan and plasticiser on the wettability properties of the resulting coating solutions.

MATERIAL AND METHODS

Materials

The materials used to prepare the coating solutions were carrageenan from the red seaweed, *Hypnea musciformis*, and glycerol 99.5% PA ACS (VETEC, Brazil) used as a plasticiser. The chicken fillets were purchased from stores in Fortaleza.

Extracting the carrageenan

The polysaccharides were extracted by first thawing the algae, followed by drying in an air circulation oven at 25 °C. Grinding was carried out using a knife mill (Tecnal –Wiley TE-650/1). The resulting powder was subjected to aqueous extraction under mechanical agitation (1.5% m/v) at 25 °C for 24 h. The aim of this step is to eliminate pigments and contaminants.

The material was subjected to re-extraction in a water bath (Microprocessor - model Q215M2 - Quimis) for four hours at 80 °C to obtain gel-forming polysaccharides (PS). The PSs were precipitated (24 h at 4 °C) by the addition of commercial ethanol (1:3 v/v). To completely remove the alcohol, the polymer underwent a process of dialysis using cellulose membranes (Sigma-Aldrich, 76 mm) in 2.5 litres of distilled water with approximately six to eight changes every three hours. The material was freeze-dried (Líotop, series; 39413, Brazil) at a pressure of between 400 and 300 µHg and a temperature of -50 ºC for 75 hours.

Preparing the coatings

The edible coatings were prepared as per the methodology proposed by Souza *et al*. (2010) with adaptations. The stock solutions were initially prepared using three different concentrations of carrageenan (1.0%, 2.0% and 3.0% (m/v)) dissolved in distilled water (v/v). These solutions were stirred for one hour at 40 ºC on a magnetic stirrer. Each stock solution was then divided up, and glycerol at different concentrations—0.0%, 1.0% and 2.0% (m/v)—was added, after which the solutions were stirred for 30 minutes.

Preparing the samples of chicken breast

Chicken breasts were obtained from the local market in Fortaleza, Ceará, Brazil, and taken in thermal boxes to the Laboratory for Biomass Technology (LTB) of Embrapa Agroindústria Tropical (Brazil). The samples were kept refrigerated at 4 °C until analysis. To measure the contact angles, the chicken breasts were cut into small samples, approximately 5.0 cm long by 2 cm wide and 1.5 cm thick.

Determining the critical surface tension

The critical surface tension of the chicken breasts was determined using the Zisman method (1964). According to this method, for surfaces considered to be of low energy, i.e. when the surface tension is less than 100 mN/m, the contact angles formed by a liquid on such a surface are a linear function of the surface tension of that liquid.

Given a liquid of known surface tension and polar and dispersive components, the contact angle (θ) of one drop of this liquid with a surface can be described in terms of coefficients of reversible adhesion.

$$W_a = W_a^d + W_a^p \leftrightarrow W_a = 2\times\left(\sqrt{\gamma_S^s \times \gamma_L^d} + \sqrt{\gamma_S^p \times \gamma_L^p}\right) = \gamma_L \times (1+\cos\theta) \quad (1)$$

Where $\left(\gamma_S^p\right)$ and $\left(\gamma_S^d\right)$ are the polar and dispersive contributions from the surface of the solid under study, respectively. Rearranging Equation 1 gives the following equation:

$$\frac{1+\cos\theta}{2} \times \frac{\gamma_L}{\sqrt{\gamma_L^d}} = \sqrt{\gamma_S^p} \times \sqrt{\frac{\gamma_L^p}{\gamma_L^d}} + \sqrt{\gamma_S^d} \quad (2)$$

The contact angle of three pure compounds, Milli-Q water, toluene (VETEC, Brazil) and formamide (VETEC, Brazil) on the surface of the chicken breasts, together with their respective dispersive values and polar components, were determined and used to calculate both the independent variable $\left(\sqrt{\frac{\gamma_L^p}{\gamma_L^d}}\right)$ and the dependent variable $\left(\frac{1+\cos\theta}{2}\right) \times \frac{\gamma_L}{\sqrt{\gamma_L^d}}$ of Equation 2. The experimental data were then plotted on a graph as $\left(\frac{1+\cos\theta}{2}\right) \times \frac{\gamma_L}{\sqrt{\gamma_L^d}}$ versus $\left(\sqrt{\frac{\gamma_L^p}{\gamma_L^d}}\right)$, the values of $\left(\gamma_S^p\right)$ and $\left(\gamma_S^d\right)$ were then calculated.

The critical surface tension $\left(\gamma_C\right)$ of the chicken breasts was estimated by extrapolating from the Zisman graph (1964). The critical surface tension $\left(\gamma_C\right)$ was defined as follows:

$$\gamma_C = \frac{\lim}{\theta \to 0} \gamma_{LV} \quad (3)$$

Wettability

Wettability was evaluated by determining the spreading coefficient (W_S) and those of the work of adhesion (W_a) and cohesion (W_C). The equilibrium spreading coefficient (W_S) is defined by Equation 4.

$$W_s = W_a - W_c = \gamma_{SV} - Y_{LV} - Y_{SL} \quad (4)$$

and represents the work of adhesion and cohesion, as defined in Equations 5 and 6, respectively.

$$W_a = \gamma_{LV} + \gamma_{SV} - \gamma_{SL} \quad (5)$$

$$W_C = 2 \times \gamma_{LV} \quad (6)$$

When in equilibrium, the contact angle can be considered an intensive property, as it is independent of the amount of liquid (gas or solid) used in the measurement. The contact angle of a drop of liquid on a solid surface is defined by the mechanical equilibrium of the droplet under the action of three interfacial tensions, namely: solid vapour tension, solid liquid tension and liquid vapor tension (DALTIN, 2011). The surface tension of the coating solution was measured following the platinum ring method (MACY, 1935).

Wettability of the carrageenan coatings on chicken breasts

The coating solution was removed employing a 1-mL glass syringe (Fortuna® Optima) which was then used to drip the solution onto the surface of the samples. To determine the shape of the droplet, computer image processing was used. The diameter of the needle (0.815 mm) was measured using a digital micrometer (Mitutoyo, Japan).

Each chicken breast was cut into rectangular pieces, approximately 5.0 cm long by 1.5 cm thick, fixed in a Petri dish and kept at 20 ± 1 °C. The samples were fixed, and videos were taken of each solution being dripped onto the surface of the chicken breasts. The videos were recorded using a PixeLINK Nikon camera (Japan) connected to a contact angle meter (MCAT-Digidrop, GBX Instrumentation Scientifique, France). The contact angles were measured at the moment the droplet become fixed to the surface of the chicken breasts, using the equipment from GBX Instrumentation Scientifique, and the Visiodrop software, which employs the polynomial method.

The surface tensions of the coating solutions were measured using a KRUSS K6 analogue tensiometer (Germany), which is based on the DuNoüy ring method. Twenty repetitions of the contact angle and surface tension measurements were made, at 20.0 ± 0.5 °C.

Statistical analysis

The Microsoft® Excel® 2010 software and the Statistica 7.0 software were used to plot the Zisman graphs and carry out the Analysis of Variance (ANOVA).

RESULTS AND DISCUSSION

Surface tension and critical surface tension on the surface of the chicken breasts

The independent and dependent variables (Equation 2) were calculated from the values of the contact angles on the surface of the chicken breasts of three pure compounds, water (A), formamide (B) and toluene (C) (Figure 1). The angles were combined with the values for surface tension and its components in the pure compounds as reported by Busscher *et al.* (1984) and Jańczuk and Białlopiotrowicz (1989), and are shown in Table 1.

Equation 7 shows the regression equation of the data obtained with the pure compounds on the surface of the samples. This equation was used to calculate the surface tension components (polar and dispersive) of the chicken breasts.

$$\frac{1+\cos\theta}{2}\times\frac{\gamma_L}{\sqrt{\gamma_L^d}}=(6.3083)\times\sqrt{\frac{\gamma_L^p}{\gamma_L^d}}+(3.844)R^2=0.9964 \quad (7)$$

The high correlation coefficient (R^2 = 0.9964) suggested a strong linear correlation between the dependent and independent variables. To validate the global model and determine the statistical significance of the parameters in Equation 7, ANOVA analysis of variance was used at a significance value of $p < 0.05$ (Figure 2).

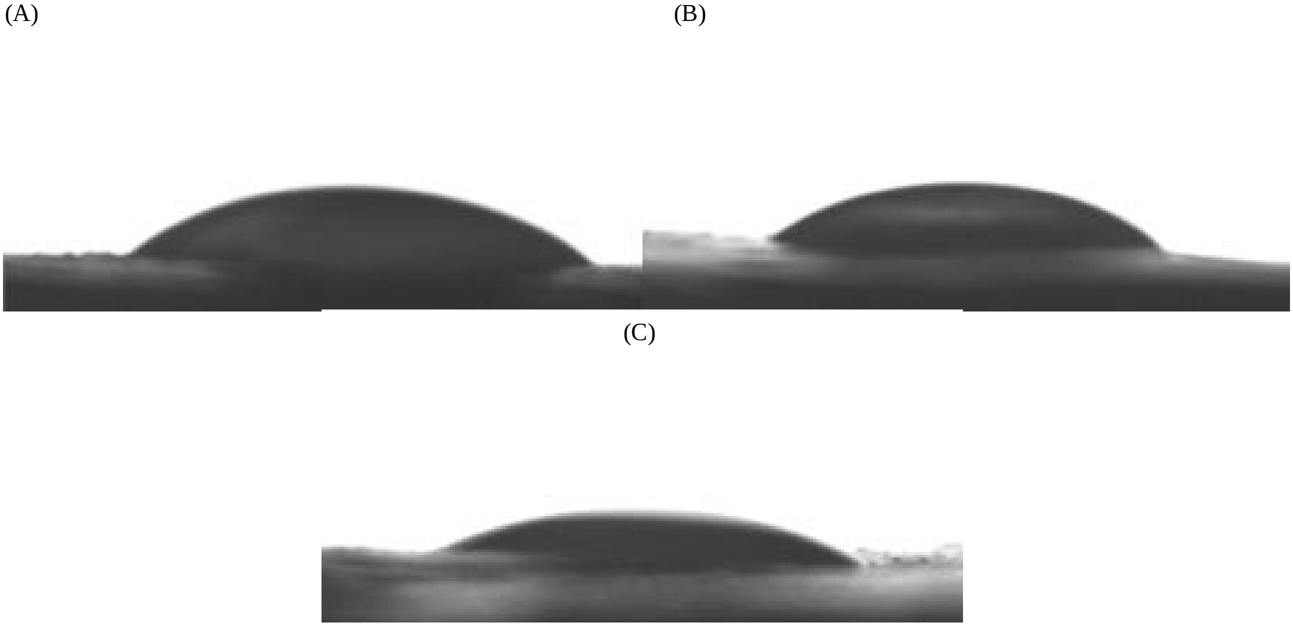
By extrapolating the Zisman graph for the surface of the chicken breasts (Figure 2), it was possible to determine a value for the critical surface tension (CST) of -9.9487 and surface tension of 54.56 mN/m. As the CST value is less than 100 mN/m, the Zisman method can be used to estimate the critical surface tension by extrapolating the Zisman graph.

The coefficient of determination (R^2 = 0.9780) shows that 97.80% of the variation in cos θ was explained by the model. This indicates a strong linear association between the cosine of the contact angle (θ) and the surface tension of the liquid under test (Equation 8). Global validation of the model and the significance of the parameters of Equation 8 were determined by ANOVA at p < 0 .05.

$$\cos\theta=(-0.0039)\gamma_L+(1.0388)R^2=0.978 \quad (8)$$

The data are consistent with the results of Dann (1970), and showed that the values for critical surface tension were lower than the values for surface tension. Furthermore, the results found in this study were consistent with those of other authors in characterising the surface of vegetables, fruit and fish (CASARIEGO *et al*., 2008; DO VALE *et al*., 2020; LIMA *et al*., 2010; RIBEIRO *et al*., 2007; SOUZA *et al*., 2010). Those authors showed that the values for critical surface tension were lower than those for surface tension.

Figure 1 - Drops on the surface of chicken breasts: (A) Water. (B) Formamide. (C) Toluene



(A)

(B)

(C)

Table 1 - Surface tension components of the liquids used to characterise the surface of chicken breasts. (A) Data from Busscher *et al.* (1984). (B) Data from Jańczuk and Białlopiotrowicz (1989)

Compound	γ_L (mN/m)	γ^d_L (mN/m)	γ^p_L (mN/m)
WaterA	72.10	19.90	52.20
FormamideA	56.9	23.50	33.40
TolueneB	28.5	27.5	33.40

Figure 2 - Adjustment of the experimental data based on Equation 7

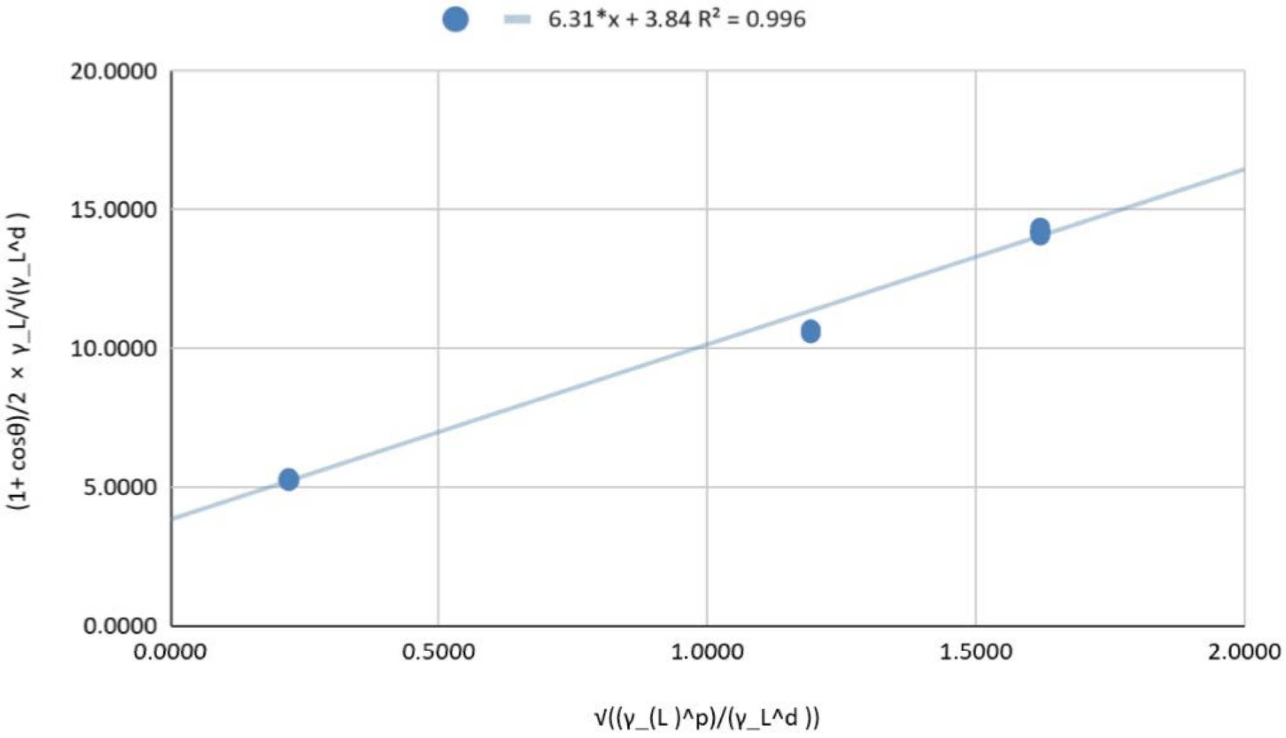
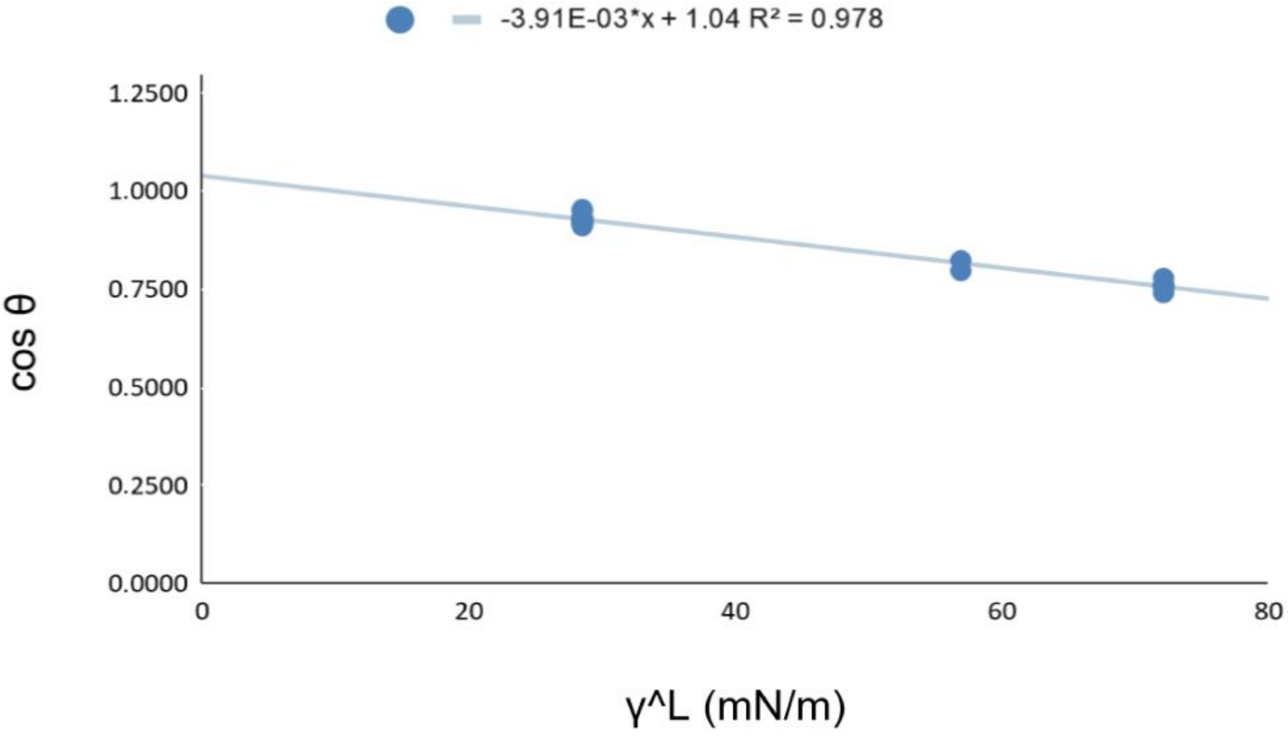


Figure 3 - Extrapolation of the Zisman graph for the surface of the chicken breasts, with a confidence interval of 95%



The surface tension value of the chicken breast found in this study (54.56 mN/m) has a polar component of 39.79 mN/m and a dispersive component of 14.77 mN/m. The higher value of the polar component is mainly due to the high water content (Table 2) of the muscle and also the polarity of the amino acids that make up the proteins.

The dispersive component of surface tension is associated with the nonpolar components that are present; the low lipid content of chicken breast directly contributes to a reduction in the nonpolar interactions that the surface can be involved in, which may explain the low value of the dispersive component. Similar results were found in Nile tilapia, which have a lipid content of approximately 2.68 ± 0.11% (ALCÂNTARA *et al*., 2019). These surface characteristics can influence the spreading and coverage of liquids, and consequently the adhesion of coatings on their surface (SOUZA *et al*., 2010).

Wettability of the carrageenan coatings on chicken breasts

Wettability is one of the main parameters to be determined when evaluating the ability of a solution to coat a surface. Adhesion forces that arise between the solution and the surface help to spread the coating; on the other hand the cohesion forces that are present as a result of the interactions that occur between the polymer chains promote contraction of the solution, and have an antagonistic effect on the adhesive forces, with the wettability depending on the balance between these forces. Therefore, to determine wettability, and thereby optimise the composition of a coating, it is necessary to know the adhesion (W_a), cohesion (W_c), and spreading (W_s) coefficients (RIBEIRO *et al*., 2007; SOUZA *et al*., 2010).

The spreading coefficient (W_s) is the result of the difference between the forces of W_a and W_c and is the parameter used to evaluate the ability of a solution to coat a surface. This was the parameter used in the present study.

The adhesion, cohesion and spreading coefficients were determined for solutions with different concentrations of carrageenan (1%, 2% and 3% (m/v)) and glycerol (1% and 2% (m/v)).

Figure 4 shows the contact angles formed by the drops of coating solution on the surface of the chicken breasts. From the value of the angles, it was possible to calculate the parameters of adhesion (W_a), shown in Table 3; cohesion (W_c), shown in Table 4; and spreading (W_s), shown in Table 5, and consequently evaluate the effects of different concentrations of carrageenan and glycerol on these parameters. In the absence of glycerol, the carrageenan concentration had a negative effect on the value of the adhesion coefficient (Table 3). The same effect was seen in the presence of glycerol, where the carrageenan concentration also had a significant negative effect ($p <0 .05$) on adhesion in the glycerol concentrations under evaluation.

Table 2 - Chemical composition of the chicken breasts

Component	Content (%)
Moisture	77.93 ± 0.14
Protein	19.64 ± 0.23
Lipids	1.14 ± 0.27
Ash	1.00 ± 0.02
Carbohydrates	0.28 ± 0.24

Coating 1 (1% carrageenan and no plasticiser) showed the highest values for W_a ($p < 0.05$) in relation to the other coatings under test (Table 3).

For coatings 1, 4 and 7 with the absence of glycerol, the increase in carrageenan concentration caused statistically significant variations ($p < 0.05$) in the values of the cohesion coefficient (W_c). In coating 3 (1% carrageenan and 2% glycerol), the increase in glycerol concentration significantly reduced the value of W_c ($p < 0.05$). On the other hand, in the presence of the plasticiser, the coatings with higher concentrations of carrageenan (8 and 9) showed the opposite effect, significantly increasing the values of W_c, differing from coating 7, which contained no glycerol (Table 4).

For the spreading coefficient (W_s), the coatings containing 1% carrageenan showed statistically similar values, albeit significantly different from the other formulations; Coatings 1, 2 and 3 are therefore the most suitable for use on chicken breasts. Since the addition of glycerol had no significant effect on the spreading coefficient, Coating 1 gave the best economic result.

In their study on the rheological properties of carrageenan-based films, Sun *et al*. (2018) showed that the addition of the plasticiser improved the flexibility, elasticity and mechanical properties of the films and reduced the intermolecular forces of attraction. This explains the lower values of the cohesion coefficients between coatings 1, 2 and 3, as these have proportionally higher concentrations of glycerol. The high values for W_s are probably linked to the high polarity of the carrageenan solution, as both the coatings and the surfaces of the chicken breasts have strong polar components, promoting greater interaction, which improves the adhesion and spreading of the coating (DEHGHANI; HOSSEINI; REGENSTEIN, 2018; SOUZA *et al*., 2010). The glycerol, however, had a negative effect on adhesion and spreading. The carrageenan concentration also had a negative effect on the spreadability of the coatings. These results are similar to those found in other studies (ALCÂNTARA *et al*., 2019; CASARIEGO

Figure 4 - Contact angle of the coating solutions on the surface of the chicken breasts

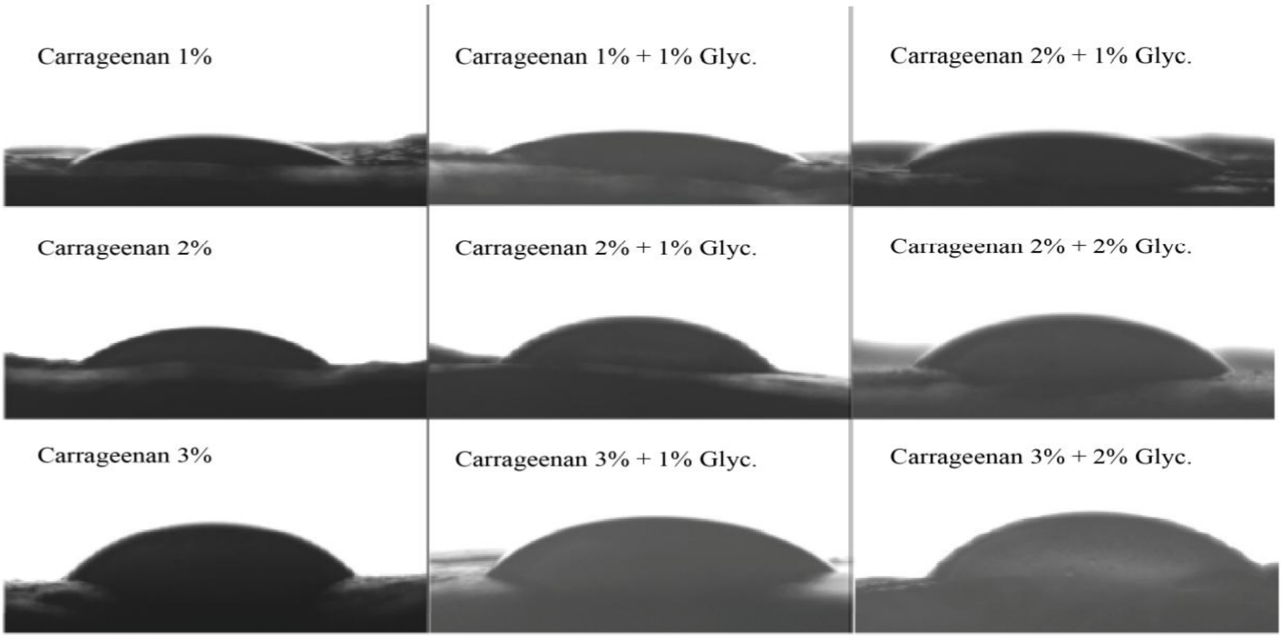


Table 3 - Adhesion values (W_a) of the carrageenan and glycerol coatings on the chicken breasts

Coating	Carrageenan (% m/v)	Glycerol (% m/v)	Wa (mN/m)
1	1	0	98.78 ± 0.83 a
2	1	1	95.57 ± 0.59 b
3	1	2	87.22 ± 0.85 d
4	2	0	91.59 ± 1.07 c
5	2	1	90.36 ± 1.25 c
6	2	2	88.35 ± 1.55 d
7	3	0	81.83 ± 1.13 e
8	3	1	83.49 ± 2.19 ef
9	3	2	82.26 ± 1.52 f

Different letters indicate a statistically significant difference (Tukey’s test, $p < 0.05$)

Table 4 - Cohesion values (W_c) of the carrageenan and glycerol coatings on the chicken breasts

Coating	Carrageenan (% m/v)	Glycerol (% m/v)	Wc (mN/m)
1	1	0	101.6 ± 1.48 a
2	1	1	100.2 ± 0.27ab
3	1	2	91.0 ± 1.41 d
4	2	0	98.2 ± 0.27 b
5	2	1	99.0 ± 0.83 ab
6	2	2	100.2 ± 0.53 ab
7	3	0	90.2 ± 0.62 d
8	3	1	94.4 ± 2.9 c
9	3	2	95.0 ± 1.22 c

Different letters indicate a statistically significant difference (Tukey’s test, $p < 0.05$)

Table 5 - Spreading coefficient (Ws) of the carrageenan and glycerol coatings on the chicken breasts

Coating	Carrageenan (% m/v)	Glycerol (% m/v)	Ws (mN/m)
1	1	0	-3.55 ± 0.79 c
2	1	1	-4.33 ± 0.58 c
3	1	2	-4.49 ± 0.85 c
4	2	0	-6.53 ± 1.07 b
5	2	1	-8.53 ± 1.25 d
6	2	2	-11.69 ± 1.55 a
7	3	0	-7.88 ± 1.13 b
8	3	1	-11.47 ± 2.19 a
9	3	2	-12.38 ± 1.52 a

Different letters indicate a statistically significant difference (Tukey's test, $p < 0.05$)

et al., 2008; DO VALE *et al*., 2020; SOUZA *et al*., 2010). Alcantara *et al*. (2019) found that increasing the concentration of chitosan in the coating solution reduces the spreading coefficient on the surface of Nile tilapia, a similar result to that found by Souza *et al*. (2010) in salmon fillets. Do Vale *et al*. (2020) reported that higher concentrations of glycerol in chitosan-based coatings reduced the spreadability of the coatings in sawfish. Therefore, the behaviour of the coatings, as well as the W_a, W_c and W_s coefficients on the surface of the chicken breasts seen in the present study, were consistent with earlier findings on other matrices reported in the literature.

CONCLUSIONS

1. Determining the surface tension (54.56 mN/m) and critical surface tension (-9.9487) of the chicken breasts showed this to be a low energy surface, which allows the Zisman method to be applied to determine the wettability of carrageenan and glycerol coatings on chicken breasts;
2. It was found that higher concentrations of carrageenan have a negative effect on the adhesion coefficient, increasing the cohesion coefficient and reducing spreading on the surface. Coatings 1 (carrageenan 1% and glycerol 0%), 2 (carrageenan 1% and glycerol 1%) and 3 (carrageenan 1% and glycerol 2%) were the most suitable for use on chicken breasts;
3. Wettability proved to be a good method of optimising biopolymer-based coatings on chicken breasts.

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