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Active Edible Coating for Lipid Oxidation Control in Brazil Nuts

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ABSTRACT

The aim of this study was to evaluate the effect of adding a lipid antioxidant (tocopherol) and a surfactant (soy lecithin) to carboxymethyl cellulose (CMC) films. CMC film-forming solutions containing sorbitol, soy lecithin, and different tocopherol concentrations (0.125% and 0.250% tocopherol mix) were produced. The solutions were characterized for their rheological, optical, antioxidant, phenolic content, and stability, and were then applied as edible coatings to whole and broken Brazil nuts. The mass gain and degree of lipid oxidation of the coated and control nuts were evaluated. Special emphasis has been given to the antioxidant properties of the solution, as they are decisive for the use of the solutions as a coating to prevent or retard lipid oxidation. The solutions had a pseudoplastic character; the viscosity varied as the temperature changed, and the modules G' and G'' depended on the applied frequency ranges. The coated nuts showed oxidative stability against the T4 coating formulation during the storage period. It was concluded that the T4 coating has the potential to be used as active packaging for foods with a high lipid content to prevent or delay oxidation processes.

1 | Introduction

Oxidation, free radical formation, and elimination reactions occur not only in the human body, but also in all living organisms and biological systems. Thus, food is not different, and auto-oxidation, lipid peroxidation, and other types of oxidation are extremely common. Antioxidants added to foods have the same mission as endogenous antioxidants in the human body: they must protect foods against these attacks and preserve their organoleptic, textural, and food safety properties (Carocho et al. 2018).

Lipid oxidation is the major cause of food spoilage. This can be slowed down by adding antioxidants to foods or by using vacuum or modified atmosphere packaging. A recent approach involved the application of antioxidants in active packaging (Ganiari et al. 2017; Ali et al. 2025).

Food antioxidants prevent the auto-oxidation and oxidation of foods, stabilizing lipids and other food compounds, preventing the cascade of oxidative reactions, and interrupting these reactions when they cannot be avoided (Carocho et al. 2018).

Oxidation control is an important challenge for the Brazil nut processing industry, as almonds are exposed to factors that trigger oxidation during shelling and processing (Zajdenwerg et al. 2011).

In this context, the aim of this study was to evaluate the effect of the addition of lecithin and tocopherols on the rheological properties of carboxymethyl cellulose (CMC) coating solutions, examine in detail the antioxidant characteristics of the solutions, and examine the effects of the application of these coating solutions on whole Brazil nuts and Brazil nuts injured during processing. These data are important for evaluating the possible applications of these coatings in foods with high lipid content for the prevention or delay of lipid oxidation.

2 | Material and Methods

2.1 | Material

The raw materials used in producing the films were all food grade and were supplied or donated by companies that produce/distribute food ingredients. The sodium CMC used in this study was CMC Cekol 30,000 (minimum purity of 99.5%), kindly donated by Vogler Company Ingredients and Cp Kelco sorbitol in solid form (powder) were purchased from Pryme Foods Company. Vitamin E was used in the form of a tocopherols mix (Tocopherol Mix 95% DSM Nutritional Products) of composition α- tocopherol: 0%–15%; β-tocopherol: < 5%; γ-tocopherol: 55%–75%; and δ-tocopherol: 20%–30%, which was kindly donated by the company Tovani Benzaquen. The Brazil nuts used were kindly donated by the Cooperativa Mista de Guariba-MT (COMIGUA), located in the Guariba district of Colniza-MT. The access to biodiversity (Brazil nut) was registered at SISGEN—National System for Managing Genetic Heritage and Associated Traditional Knowledge (AE870E9).

The analyses were carried out in partnership with the laboratories of IFGoiano—Campus Rio Verde (LaBBio, Food Biotechnology, Postharvest of fruits and vegetables, Postharvest of vegetable products, Central Analytical), UFG—Campus Samambaia (LabMulti and CRTI) and UFMT—Campus Cuiabá.

2.2 | Preparation of the Film-Forming Solutions

The coating solutions were prepared according to a method previously described by Larrauri et al. (2016) method with some modifications. They were prepared by mixing 1.0% ($m\,m^{-1}$) CMC, 1.9% ($m\,m^{-1}$) sorbitol, and 97.6% ($m\,m^{-1}$) distilled water as a control.

First, sorbitol was dissolved in distilled water under constant stirring for 5 min (solution A), then CMC was added to solution A and stirred for 55 min at 40°C (solution B or control solution). After the stirring step of solution B was completed, lecithin was added, followed by tocopherol when necessary, and the solution (now solution C) was homogenized in three steps of 4 min of stirring using a 4-blade mixer with 600 watts of power (Philco, PMX 600, China). After the solutions were finished, they were degassed for 15 min in an ultrasonic bath at 40°C to eliminate air bubbles present in the solutions resulting from the homogenization process.

In solutions with added antioxidants, partial replacement of water in the composition of the coating solution was considered, as outlined in Table 1.

2.3 | Physical Stability of Film-Forming Solutions

To verify the physical stability of simple (CMC + sorbitol and CMC + sorbitol + lecithin) and emulsified (CMC + sorbitol + lecithin + vitamin E) film-forming solutions for coating Brazil nuts, the centrifugation stability (accelerated test) and creaming index were analyzed in triplicate immediately after the preparation stage.

2.3.1 | Centrifugation Stability

To verify the stability of the coating solutions through centrifugation, 10 g of each formulation of the film-forming solutions were weighed in 15 mL falcon tubes in triplicate, and the samples were subjected to centrifugation at 3000 rpm (or 1214X g) for 30 min, with 20 s for acceleration and 30 s for deceleration (Solab, SL-701, Piracicaba, Brazil).

2.3.2 | Creaming Index

The stability of the solutions through the Creaming Index (CI) was evaluated by considering the methodology of Lorenzo et al. (2008), with adaptations. Aliquots of 25 mL of each coating solution were transferred immediately after the preparation step to 25 mL graduated cylinders, sealed, and stored at room temperature for 168 h. The height of the initial phase was checked periodically (every 24 h) during solution storage. The CI was calculated as a percentage of the height of the cream layer (lipid-rich

TABLE 1 | Formulation of film-forming solutions for edible coating.

Solution formulation	CMC (%)	Sorbitol (%)	Soy lecithin (%)	Vit E (%)	Water (%)
Control 1– C1	1.0	1.9	0	0	97.1
Control 2– C2	1.0	1.9	0.19	0	96.91
Control 3– C3	1.0	1.9	0.19	0.125	96.78
Control 4- C4	1.0	1.9	0.19	0.250	96.66

layer) in relation to the total height of the film-forming solution, both in cm.

2.4 | DPPH Free Radical Scavenging Capacity of Film-Forming Solutions

Determination of the antioxidant activity of the solutions by the DPPH radical capture method was carried out according to the methodology described by Brand-Williams et al. (1995), with some modifications. Approximately 100 mg of the coating solution was dissolved in 10 mL of distilled water to prepare extracts. The mixture was then stirred in a mechanical shaker at 25°C for 20 h. Then, 100 μL of the aqueous extract obtained from the solutions was transferred to tubes containing 3.9 mL of DPPH ethanolic solution, and the resulting mixture was homogenized and left to stand for 30 min at room temperature, protected from light, and then read using a spectrophotometer. The amount of DPPH radicals that were not captured was determined by measuring absorbance at 515 nm. The results are expressed in μM Trolox per gram of sample (μM Trolox g^{-1}).

2.5 | Free Radical Scavenging Capacity ABTS of Film-Forming Solutions

Determination of the antioxidant activity of the solutions by the ABTS radical capture method was carried out according to the methodology described by Rufino et al. (2010) previously, with modifications. Approximately 100 mg of coating solution was dissolved in 10 mL of distilled water to prepare the extracts. The mixture was stirred in a mechanical shaker at 25°C for 20 h. Then, 30 μL of the aqueous extract obtained from the solutions was transferred to test tubes containing 3.0 mL ABTS radical, and the mixture was homogenized and left to stand for 6 min protected from light and at room temperature for subsequent reading in a spectrophotometer. The absorbance was measured at 734 nm to determine the amount of uncaptured ABTS radicals. The results were expressed in μM Trolox per gram of sample (μM Trolox g^{-1}).

2.6 | Total Phenolic Compounds of Film-Forming Solutions

The total phenolic content was determined according to the methodology of Singleton et al. (1998) described previously. Approximately 100 mg of coating solution was dissolved in 10 mL of distilled water to prepare the extracts. The mixture was shaken in a digital incubator with orbital shaking (Thoth, Shaker 6430, Piracicaba, Brazil) at 25°C for 20 h. Then, 200 μL of the aqueous extract obtained from the solutions was transferred to tubes containing 1.9 mL of freshly prepared Folin–Ciocalteu reagent solution diluted 10 times in distilled water. Subsequently, 1.9 mL of aqueous Na_2CO_3 solution (60 g L^{-1}) was added, and the resulting mixture was homogenized and left to stand for 2 h at room temperature, protected from light, for subsequent reading using a spectrophotometer at a wavelength of 725 nm. The results are expressed in mg of Gallic Acid equivalents per 100 gram of sample (mg GAE 100 g^{-1}).

2.7 | Rheological Behavior of Film-Forming Solutions

The viscosity of the solutions was measured by determining the flow curves at 25°C and 40°C, considering the estimated immersion temperature of the nuts in the film-forming solution and the drying temperature of the solution coating the nuts. The tests were performed using a controlled tension oscillatory rheometer (Physica MCR101, Ostfildern, Germany). The measurements were performed in triplicate in a cone-plate geometry of 6 cm in diameter and 2° angle with controlled temperature. The solutions were evaluated 48 h after preparation. The scans were performed in three stages: the first stage (ascent 1) with an increasing deformation rate from 0.1 to 300 s^{-1}, the second (descent) with a decreasing rate from 300 to 0.1 s^{-1} and finally, the third (ascent 2) with an increasing rate again from 0.1 to 300 s^{-1}. The apparent viscosity of the emulsions was calculated as the ratio between the shear stress (σ) and the deformation rate ($\dot{\gamma}$), with adjustment of the Power Law model.

$$\sigma\ (\text{Pa}) = k * \dot{\gamma}^{n} \quad (1)$$

where μ is the viscosity (Pa.s), k is the consistency index (Pa.s^n), and n is the behavior index.

Oscillatory strain sweep tests were performed to determine the linear range using the controlled strain rate mode. Shear rate from 10^{-4} min^{-1} to 3000 min^{-1} (50 s^{-1}), with an initial deformation of 0.01 and a final deformation of 10% using a cone-plate geometry of 6 cm in diameter at temperatures of 25°C and 40°C. Oscillatory tests of frequency sweeps between 0.1 and 100 Hz, with a tension of 0.2 Pa, were carried out at temperatures of 25°C and 40°C to evaluate the mechanical behavior of the film-forming solutions.

2.8 | Color Parameters of Film-Forming Solutions

A colorimeter (Konica Minolta, Chroma Meter Cr-400, Osaka, Japan) was used to analyze the colors of the solutions. The instrumental color parameters Luminosity L* (variation from light to dark), chromaticity a* (chromaticity on the green to red color axis), chromaticity b* (chromaticity on the blue to yellow color axis), and saturation (chroma-C *) and hue (h°) parameters, as well as their color variations in relation to the control solution (C1): delta L (ΔL), delta a* (Δa*), delta b* (Δb*), and delta E (ΔE) were determined for all prepared solutions. The solutions were evaluated in triplicate for each treatment, with seven readings taken for each, using an accessory to analyze liquids on a white background.

2.9 | Coating of Brazil Nuts

The coating of whole Brazil nuts and Brazil nuts damaged during processing (broken) was carried out by immersion in coating solutions C1 and C4 (prepared according to item 2.2) as per Table 2, chosen based on their antioxidant capacities and phenolic compound content.

The coating was applied using the methodology described by Kowalczyk et al. (2017), with modifications outlined in the flowchart (Figure 1). Uncoated nuts were used as control treatments (T0 control treatment for whole nuts and T0q control treatment for broken nuts).

The Brazil nuts were dipped in the coating-forming solutions for 30 s and dried in an oven at 40°C for 30 min on a nonstick surface. The nut coating process was repeated thrice to improve the coating coverage efficiency. The coated and uncoated seeds were stored in a temperature-controlled environment under simulated commercial conditions (23°C ± 2°C) without light (Kowalczyk et al. 2017).

2.10 | Mass Loss of Coated Nuts

Mass loss was monitored during storage. The mass of the nuts (approximately 25 g per replicate), coated and uncoated, broken, and whole, was quantified on the 1st (initial day), 7th, 14th, 21st, and 28th days of storage. The difference between the initial and final weights of the nuts was considered as the total mass loss during this storage interval and calculated as percentages on a fresh weight basis.

A first-order kinetic equation was used to fit the mass loss (migration) data of the different nut samples (T0, T1, T4, T0q, and T4q) as a function of the storage time, as shown by Takeuchi (2008). The mass loss in relation to initial mass results were expressed in g g^{-1}.

$$X(t) = X_{eq} + A\exp(-kt) \quad (2)$$

where $X(t)$ is the measured property (mass loss) as a function of time, X_{eq} is the steady-state value of the property (equilibrium mass), t (days) is time, k is the migration rate (g day^{-1}), and A is a fitting parameter.

2.11 | Mechanical Behavior of Coated Brazil Nuts

The mechanical behavior of coated and uncoated (control) Brazil nuts was evaluated through compression and shear tests performed using a texturometer (TA Instruments, TA-XT Plus, Texture Analyzer Stable Microsystems, Surrey, England). Compression tests were performed to evaluate coated and uncoated whole and broken nuts. Both tests were performed using a normal compression force with a 500 N load cell. The texturometer was calibrated with a 5000 g standard mass.

To apply compression force to the Brazil nuts, a cylindrical geometry with a diameter of 2 mm was used, in which each nut was individually placed on the texturometer platform, stabilized in relation to the length (X-axis), and compressed by the geometry until rupture, which was observed as the point of maximum force on the force (N) × distance (m) curve. The blade and guillotine geometry in "V" (HDP/WBV: Warner Bratzler) was used for shearing. Blade Set with "V" slot blade for USDA Standard.

The analysis parameters of the mechanical tests were pre-test, test, and post-test speeds of 1, 0.1, and 10 mm s^{-1}, respectively; initial analysis force (trigger force) of 0.05 N and deformation of 50% in relation to the initial height of the sample for piercing and deformation of 75% in relation to the initial height of the sample for shearing. The energy absorbed by the sample at rupture was calculated by integrating the area under the force (N) × the compression distance (m) curve. Fifteen samples of Brazil nuts were used for each treatment, and the analyses were performed on days 01, 15, and 30 of storage at 25°C.

TABLE 2 | Schematisation of the formulation of coating solutions applied to Brazil nuts.

Formulation of coating solutions	CMC (%)	Sorbitol (%)	Soy lecithin (%)	Vit E (%)	Water (%)
T1 (whole nuts)	1.0	1.9	0	0	97.1
T4 (whole nuts)	1.0	1.9	0.19	0.250	96.66
T4q (broken nuts)	1.0	1.9	0.19	0.250	96.66

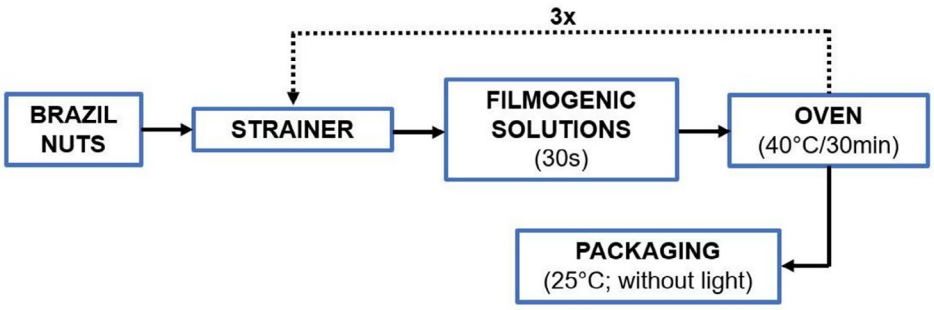

FIGURE 1 | Flowchart of the active edible coating process for Brazil nuts.

2.12 | Stability of Coated Brazil Nuts Oils

Coated and uncoated (control) Brazil nuts were induced to accelerate the lipid oxidation process in an environment (incubator) at a controlled temperature (40°C), according to the methodology used by Kang et al. (2013), with modifications. The lipid oxidation of each group of products was measured on the 1st, 14th, and 28th days of storage of Brazil nuts and evaluated by the formation of primary and secondary products of lipid oxidation present in the extracted oils through analyses of the values of peroxides and conjugated dienes (CDs).

Oil extraction from the nuts was carried out according to the methodology described by Bligh and Dyer (1959), which is a cold extraction method. The nuts were crushed and subjected to extraction, and the extracted oils were subjected to oxidative stability analysis.

2.12.1 | Peroxide Value

The oil peroxide value (PV) from Brazil nuts was obtained according to the official AOCS Cd 8-53 methodology (AOCS 1989a), with some modifications. An amount of 0.5 g of oil from each sample was weighed and 10 mL of acetic acid/chloroform solution (3:2) was added. After stirring, 0.5 mL of a previously prepared saturated aqueous solution of potassium iodide (KI) was added. The solution was then stirred and stored in the dark for 1 min. After the resting period, 10 mL of distilled water was added to the solution and titrated with a standard sodium thiosulfate solution ($Na_2S_2O_3$) at 0.01 N. The PV, given in mEq peroxide kg^{-1} oil, was determined using Equation (3):

$$\mathrm{PV}\left(\frac{m\text{Eq peroxide}}{\text{kg oil}}\right) = \frac{(V - V_0) * N * 1000}{m_{\text{oil}}} \tag{3}$$

where V is the volume (mL) spent titrating the sample, V_0 is the volume (mL) spent titrating the blank, N is the normality of the $Na_2S_2O_3$ solution, and m is the mass (g) of the oil sample.

2.12.2 | Conjugated Dienes Value

The CD value was determined according to the official AOCS Ti 1a-64 methodology (AOCS 1989b). Approximately 200 mg of each oil sample was weighed into a 10 mL volumetric flask, and the volume was completed with isooctane (solution A). After that, the solution was vortexed, and 1 mL of this solution was transferred into another 10 mL volumetric flask, completing the volume again with isooctane (solution B). The absorbance of solution B at 233 nm was measured in a spectrophotometer using isooctane as a blank. The percentage of CDs was calculated using Equation (4):

$$\mathrm{CD} = 1.0769 * \frac{\mathrm{Abs}_{233\ \mathrm{nm}}}{\mathrm{Conc.}} \tag{4}$$

where Conc. is the concentration of oil ($g\,L^{-1}$) in the isooctane solution.

2.13 | Statistical Analysis

All analyses were performed at least in triplicate, and the data were statistically evaluated using analysis of variance (ANOVA). In the case of significant differences, Tukey's mean comparison test was applied ($p < 0.05$). A linear regression test ($p < 0.05$) was performed to analyze the mass loss in Brazil nuts.

3 | Results and Discussion

3.1 | Physical Stability of Film-Forming Solutions

3.1.1 | Centrifugation Stability

Centrifugation stability was used to accelerate gravitational effects on the emulsified film-forming solution, such as phase separation, aggregation, *and creaming* (Mori Cortés et al. 2019). None of the formulations used in this study showed phase separation (coalescence, creaming, or flocculation) after centrifugation (Figure 2). Therefore, both solutions were stable in this test and were suitable for further studies. Thus, considering the industrial applicability, emulsified film-forming solutions can be prepared and used to coat nuts while maintaining a uniform concentration of the active antioxidant compound during processing.

This test indicated a good relationship between the amount of surfactant (soy lecithin) and lipids (tocopherols) in the solution, because the amount of surfactant used in the solution was sufficient to prevent phase separation during centrifugation. Variables such as viscosity, interfacial tension, and particle diameter are key factors that control the gravitational stability of emulsified solutions. The stability of the solutions may be associated with the small size of the lipid droplets and the interfacial tension (Lawrence and Rees 2012).

3.1.2 | Creaming Index

None of the solutions showed phase separation (creaming) during the study, and they were stable at room temperature for 7 days (Figure 3). This is an important factor for future applications, in which film-forming solutions can be prepared without the need for use on the same day of preparation. Lorenzo et al. (2008) also observed similar behavior in the creaming test of low-lipid oil-in-water (o/w) emulsions, and they remained stable for more than 7 days.

The high concentration of CMC in the solution may have contributed to its stability against creaming by increasing the viscosity of the continuous phase. Incorporation of hydrocolloids is a suitable alternative for stabilizing low-lipid o/w emulsions against creaming (Lorenzo et al. 2008).

The test also indicated a good relationship between the levels of surfactant (soy lecithin) and lipids (tocopherols) used because the amount of surfactant used in the solution was sufficient to form a strong bond with the lipids present and prevent them from coalescing and causing phase separation in the solution at a steady state during the study period. Lecithin is naturally present in

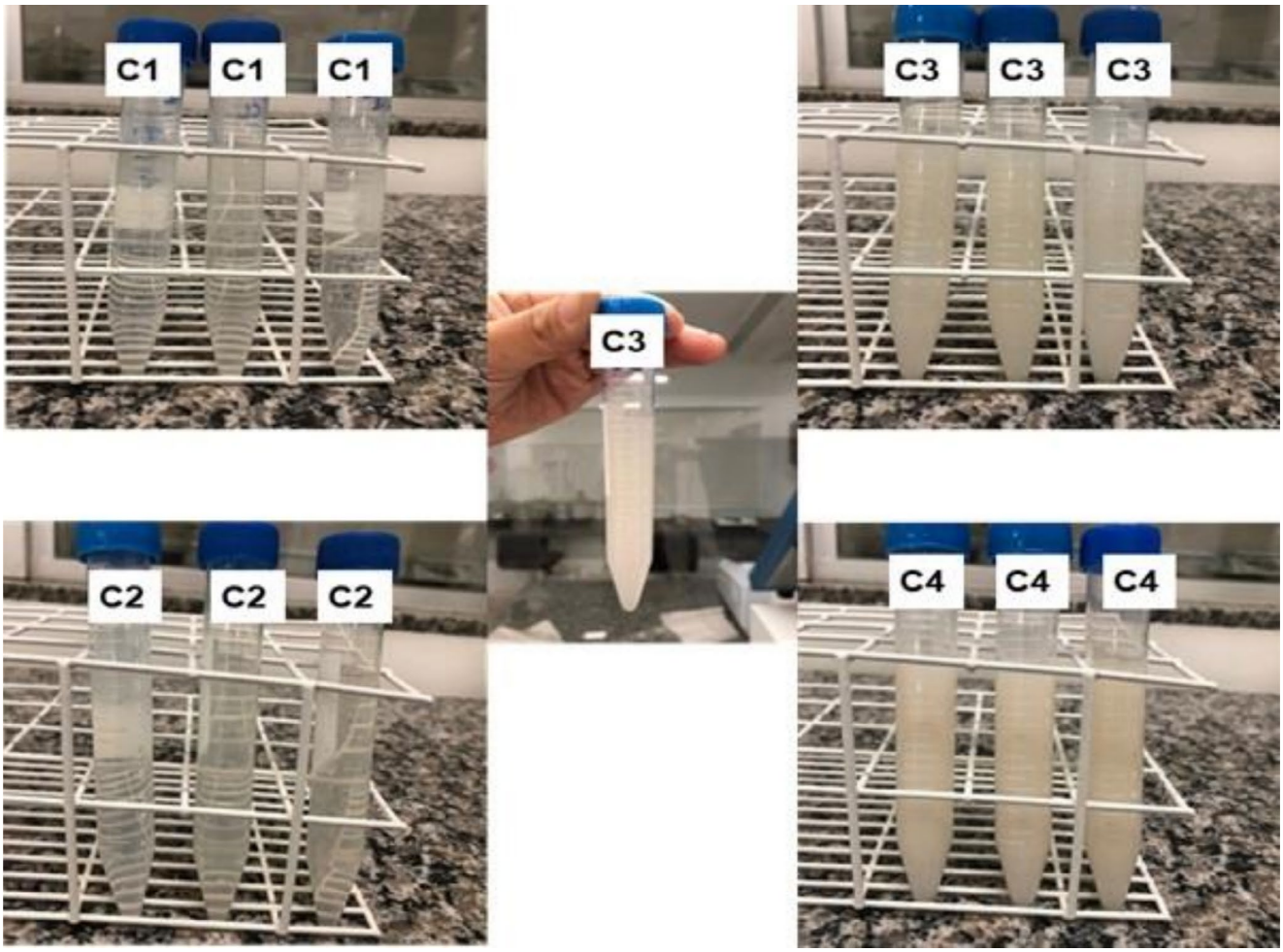

FIGURE 2 | Images of film-forming solutions C1 (CMC + sorbitol), C2 (CMC + sorbitol + soy lecithin), C3 (CMC + sorbitol + soy lecithin + 0.125% tocopherols), and C4 (CMC + sorbitol + soy lecithin + 0.25% tocopherols) after centrifugation test.

natural oils and helps to stabilize the tocopherols present in these oils through the interaction of the hydroxyl group of the vitamin E ring with the phosphate group of lecithin (Bongiorno et al. 2006).

3.2 | Bioactive Compounds

The antioxidant activities of the coating solutions are listed in Table 3.

The addition of tocopherols to the matrices of the coating solutions led to greater antioxidant capacities for the two different free radical capture methods studied. There were no differences in the antioxidant activities between solutions C3 and C4 (Table 3).

It can be observed that the addition of lecithin increased ($p < 0.05$) the antioxidant capacity for the ABTS radical capture method, and when tocopherols were added, these solutions showed significant improvements ($p < 0.05$) for this property due to the presence of antioxidant compounds in these formulations, the tocopherols. In terms of activity, α-tocopherol is the most active isoform of Vitamin E, followed by β-, γ-, and finally δ-tocopherol. These compounds have a chroman ring and a hydrocarbon chain with methyl residues (Figure 4).

Martelli et al. (2017) also observed a synergistic effect between lecithins and tocopherols in film-forming solutions, with greater antioxidant activities found in films containing these compounds.

The incorporation of tocopherols into the solution significantly increased the content of phenolic compounds in the solutions, in agreement with the antioxidant activities, as expected because they have a phenolic hydroxyl in their chemical structure that attributes antioxidant activity to these compounds (Figure 4).

3.3 | Rheological Behavior of Film-Forming Solutions

The graphs in Figure 5a–f show the results of rheological analyses of the film-forming solutions. Flow curves were obtained for the film-forming solutions at two temperatures: 25°C and 40°C. As can be seen in the graphs (Figure 5a,b), the intersection between the G' (elastic mode) and G'' (viscous mode) curves

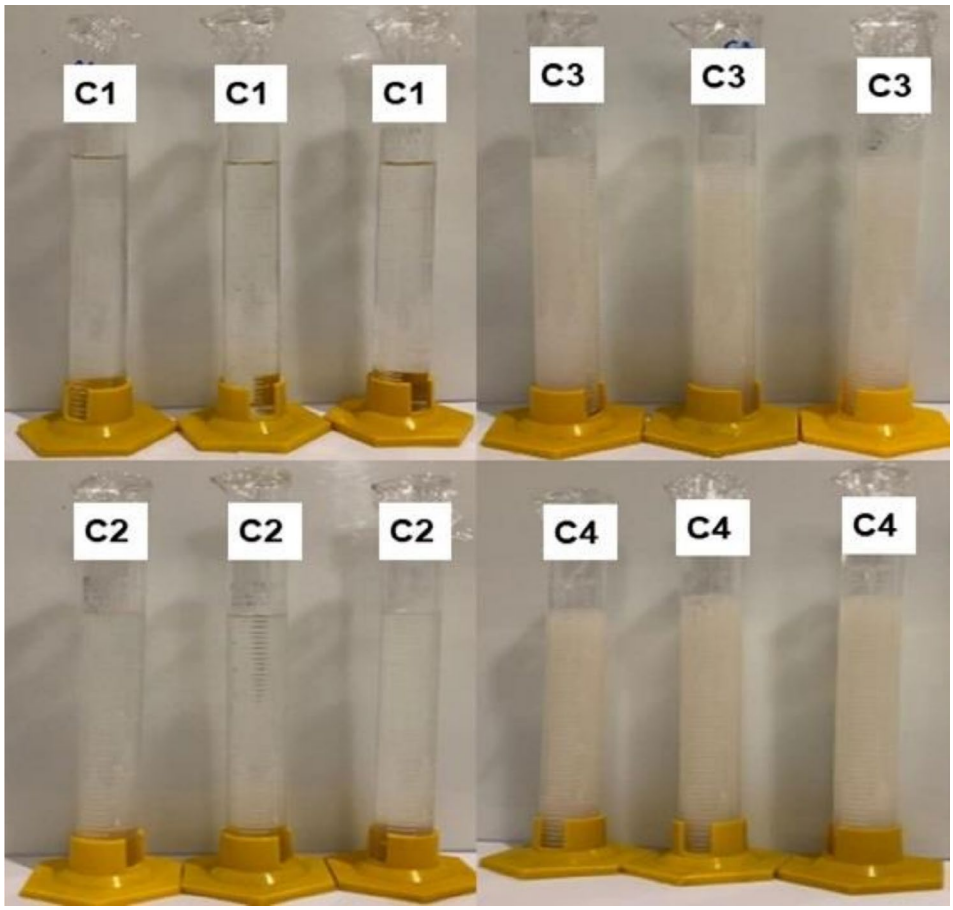

FIGURE 3 | Images of film-forming solutions C1 (CMC + sorbitol), C2 (CMC + sorbitol + soy lecithin), C3 (CMC + sorbitol + soy lecithin + 0.125% tocopherols), and C4 (CMC + sorbitol + soy lecithin + 0.25% tocopherols) on the 7th day of the creaming test.

TABLE 3 | Antioxidant activity and total phenolic compounds for film-forming solutions: C1 (CMC + sorbitol), C2 (CMC + sorbitol + soy lecithin), C3 (CMC + sorbitol + soy lecithin + 0.125% tocopherols), and C4 (CMC + sorbitol + soy lecithin + 0.25% tocopherols).

Formulations	DPPH (μM Trolox g^{-1})	ABTS (μM Trolox g^{-1})	Total phenolic compound (mg GAE $100\,g^{-1}$)
C1	87 ± 4b	174 ± 12c	10.4 ± 0.1c
C2	98 ± 6b	189 ± 10b	10.5 ± 0.5c
C3	420 ± 7a	284 ± 20a	49.8 ± 0.3b
C4	991 ± 2a	404 ± 20a	62 ± 2a

Note: Mean values within the same column with the same letters are not statistically different for Tukey's test at one level ($p < 0.05$).

indicates that the solution behaved like a concentrated solution and did not exhibit gel formation.

The film-forming solutions showed a tendency $G'' > G'$ and were predominantly viscous, which was expected because this behavior is more characteristic of liquids. Both G' and G'' depend on the applied frequency.

The complex mode (G^*) is the ratio between the viscous behavior (G'') and elastic behavior (G'). The solutions behaved similarly for the different temperatures studied, with an increase in the values found for G^* with an increase in the value of the applied frequency (Figure 6a,c).

Regarding the complex viscosity, the solutions showed a tendency to decrease in viscosity with increasing frequency, indicating that with increasing frequency, there was a greater disturbance in the structure of the film-forming solution and a greater tendency for the molecules to order. The behavior was similar at both temperatures under study.

The frequency sweep was performed at two different frequencies with the intention of characterizing the coating solutions in different applications: a 10 Hz sweep was performed to size the solutions for application in slower processes, such as food coatings, and a 100 Hz sweep was performed to size the solutions for application in faster, large-scale processes, such as pumping.

Because the n values for the Power Law model were less than 1, CMC film-forming solutions with and without the addition of tocopherols were considered pseudoplastic fluids because they presented a pseudoplastic character when $n < 1$. However, the C4 solution presented a characteristic closer to that of a Newtonian fluid ($n = 1$) than the other films, presenting the highest n values for the two temperatures studied (power law).

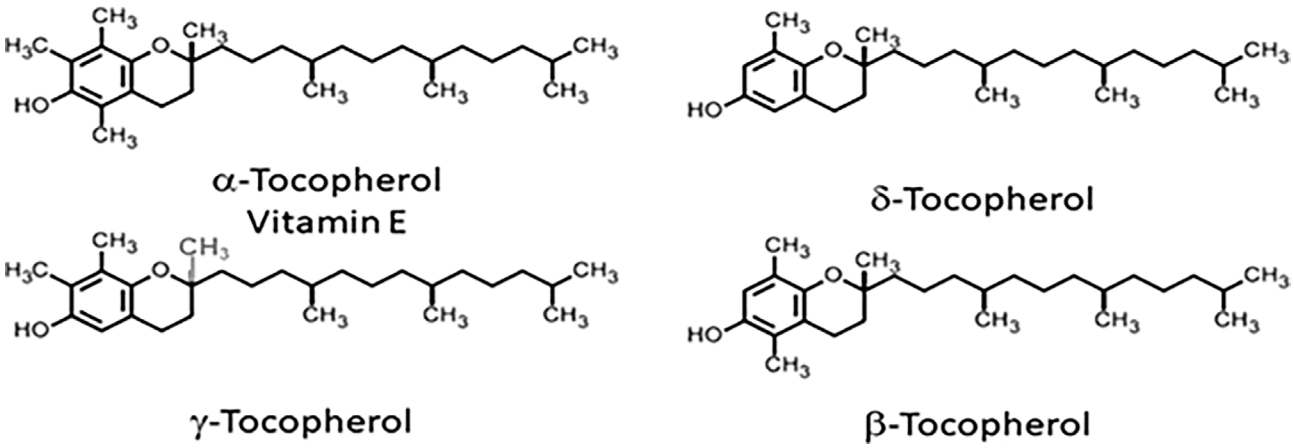

FIGURE 4 | Chemical structures of tocopherols (Azzi 2019).

Ghasemlou et al. (2011) also observed pseudoplastic behavior in film-forming solutions with oleic acid.

According to the curves presented in Figure 7a,b, it is observed that at a temperature of 25°C, solutions C2 and C3 required higher stress values for deformation, while film-forming solution C4 (with a higher concentration of tocopherols) required lower stress for shearing to occur when compared to the other solutions. For the highest analysis temperature (40°C), solutions C1 and C2 behaved similarly, presenting similar behavior curves for the variation in shear stress with increasing deformation rate. By analyzing the curves at 40°C, it was observed that film-forming solution C4 required less shear stress than the other solutions. In general, increasing the temperature from 25°C to 40°C led to a decrease in stress and viscosity.

Figure 7c,d presents the values of viscosity versus the deformation rate of the film-forming solutions. The figure shows that for all solutions, the apparent viscosity decreased considerably with an increase in the shear rate.

An increase in the process speed (deformation or shear rate) decreased the apparent viscosity (Figure 7e). The C4 solutions exhibited the lowest apparent viscosity values at the two temperatures analyzed (25°C and 40°C).

3.4 | Color Parameters of Film-Forming Solutions

Table 4 lists the color parameter values of the coating solutions.

The incorporation of lecithins and tocopherols reduced the brightness and hue of the solution. It also increased the chromaticity value a*, indicating a tendency toward reddish coloration. However, solutions C2 and C3 (Table 4) did not differ, indicating that a lower tocopherol concentration did not influence this parameter.

The incorporation of lecithin caused an increase in the b* chromaticity of the solutions, highlighting their yellow coloration. When tocopherols were added, the solutions exhibited a reduction in b* chromaticity values. The higher the concentration of tocopherols, the lower the value of b*, causing a slight reduction in yellow and a tendency toward blue. Similar behavior was observed for the saturation parameters of the films.

Solution C4 showed greater color variation than the control solution, followed by solutions C2 and C3 (Table 4).

3.5 | Mass Loss of Coated Brazil Nuts

In Figure 8, mass loss behavior during storage time in a digital incubator (25°C) for coated and uncoated whole Brazil nuts (T4, T1, and T0) and coated and uncoated broken Brazil nuts (T4q and T0q) can be observed, respectively.

It can be seen that there was an increase in mass loss for all samples during the storage period. Weight loss occurs mainly due to water loss through transpiration and loss of carbon reserves due to respiration (Sogvar et al. 2016). The function of edible coatings is to form an extra layer that results in reduced transpiration, and therefore, limited mass loss (Guerreiro et al. 2015). It can be stated from this that the coatings formed thin films because they did not limit the mass transport of the nuts during storage.

The T0q and T4q treatments showed the highest percentage of mass loss (% g g^{-1}) at equilibrium, estimated for up to 28 days. The lowest mass loss values at equilibrium were observed for T0, T1, and T4, which were not different from those of T4q (Table 5). The lowest percentage of mass at equilibrium indicates greater moisture loss during storage.

The lowest mass loss rate (% g g^{-1}) was observed for T4, probably because of the greater barrier to mass transfer by the coating containing lecithin and tocopherol. Conversely, T4q, which has the same coating but broken nuts, did not have a reduced mass loss rate compared to T0, T1, and T0q.

Application of edible coatings containing lipids can be effective as a barrier to mass loss due to the hydrophobic character of lipids (Galus and Kadzińska 2015). Hashemi et al. (2017), using higher concentrations of lipids (1%–6%) in coating solutions, observed a direct correlation between lipid

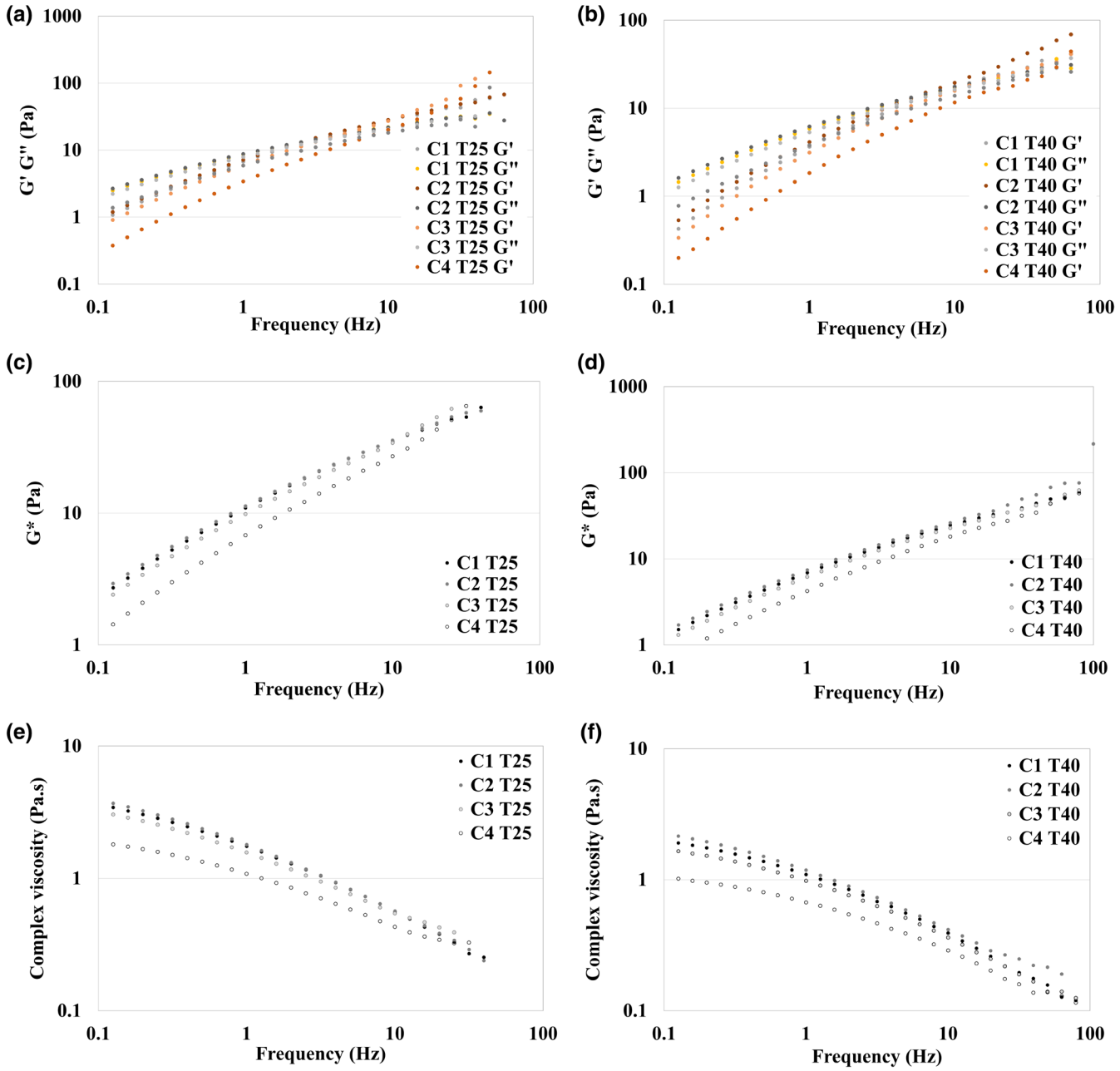

FIGURE 5 | Frequency sweeps of coating solutions C1 (CMC + sorbitol), C2 (CMC + sorbitol + soy lecithin), C3 (CMC + sorbitol + soy lecithin + 0.125% tocopherols), and C4 (CMC + sorbitol + soy lecithin + 0.25% tocopherols) at 25°C (a, c, e), and at 40°C (b, d, f).

concentration and the reduction in mass loss of fresh apricots during storage.

At the end of the 28-day storage period at 40°C under constant air circulation, the mass losses (% g g^{-1}) of the Brazil nuts were 0.55%, 0.54%, and 0.58% for the T0, T1, and T4 treatments, respectively, and 0.7% and 0.62% for the T0q and T4q treatments, respectively (Table 5). Brazil nuts were subjected to drastic storage conditions, which are considered accelerated lipid oxidation tests. The recommended storage for peeled Brazil nuts is at room temperature, approximately 25°C, vacuum packed, and protected from light, which are factors that delay lipid oxidation reactions.

3.6 | Stability of Coated Brazil Nut Oils

Table 6 presents the values for the levels of primary compounds (peroxides) and secondary compounds (dienes) from the oxidation of oils extracted from Brazil nuts subjected to accelerated oxidation.

It was observed that on day 1, the beginning of the evaluation of the evolution of lipid oxidation, the values of peroxide index and CDs already presented different values, probably due to the natural variation of Brazil nuts. The trend in the evolution of lipid oxidation was calculated based on the percentage increase on the 14th and 28th days in relation to the initial value quantified

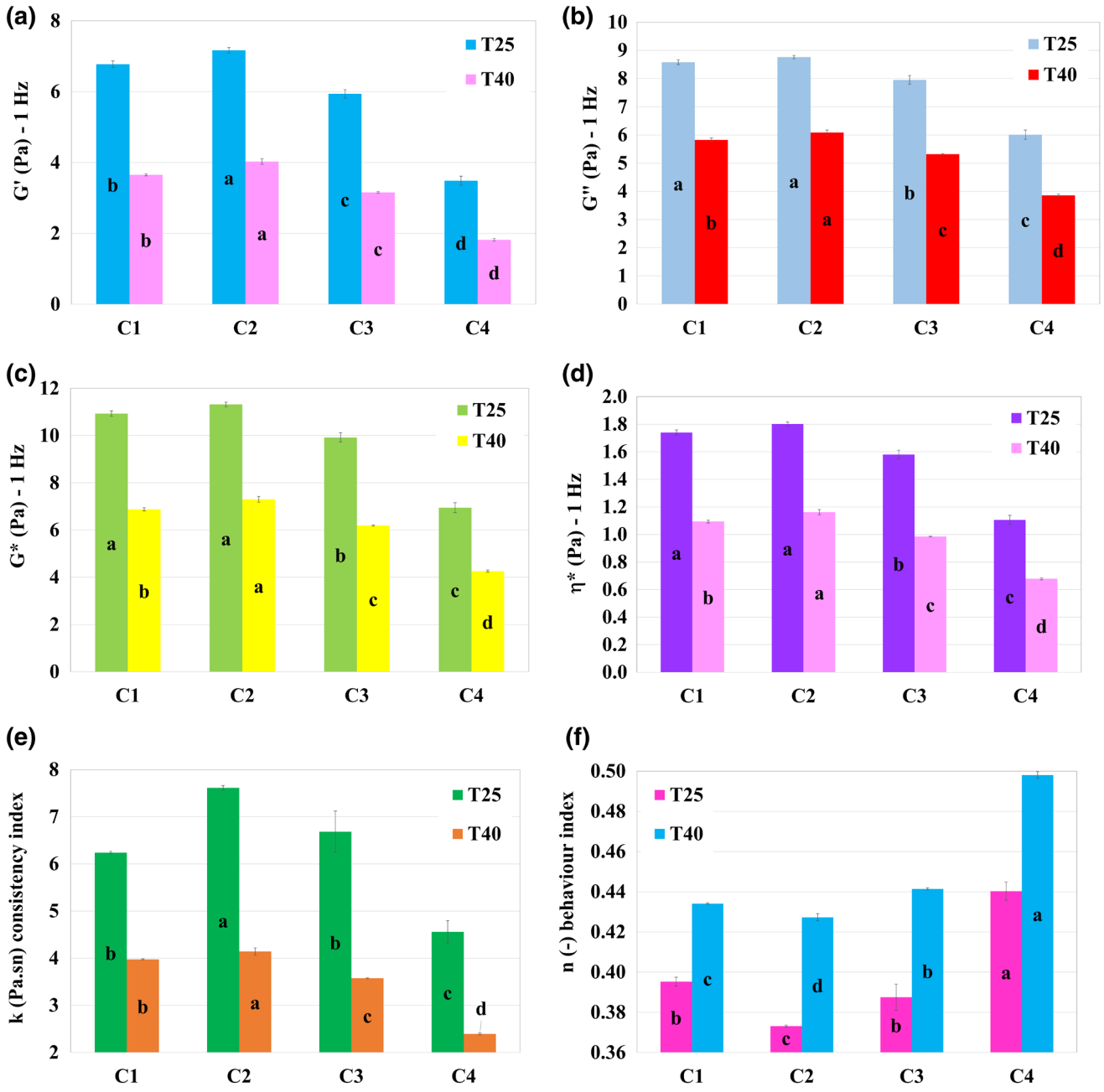

FIGURE 6 | Effect of temperature on storage modulus (a), loss modulus (b), complex modulus (c), complex viscosity (d), consistency index (e), and behavior index (f) of coating-forming solutions C1 (CMC + sorbitol), C2 (CMC + sorbitol + soy lecithin), C3 (CMC + sorbitol + soy lecithin + 0.125% tocopherols), and C4 (CMC + sorbitol + soy lecithin + 0.25% tocopherols) at 25°C and 40°C. Mean values within the same graph with the same letters are not statistically different at same temperature for Tukey's test at one level ($p < 0.05$).

on day 1 to avoid statistical biases due to initial values on day 1 for treatments T0, T1, and T4 for whole Brazil nuts and T0q and T4q for broken Brazil nuts.

In Figure 9, the evolution curves of lipid oxidation can be observed as a function of the number of days of storage under extreme temperature conditions (accelerated *shelf-life test*) for the indices of peroxides and CDs.

For whole Brazil nuts, it can be observed that there was a growing increase in the values found for the primary oxidation compounds investigated, the peroxides. The uncoated Brazil nuts (T0) had an increasing triggering of peroxide formation, presenting a growing difference in the peroxide content found for the different periods analyzed during the storage period.

For the coated Brazil nuts without the addition of tocopherols (T1), the oxidation rate was practically constant on the second and last days of the analysis, presenting similar peroxide levels.

In the T4 treatment, with the addition of tocopherols, there was no variation in the production of peroxides in the nuts subjected to accelerated oxidation, maintaining constant levels throughout the storage period, indicating that the coating blocked the oxidation of the nuts. When analyzing

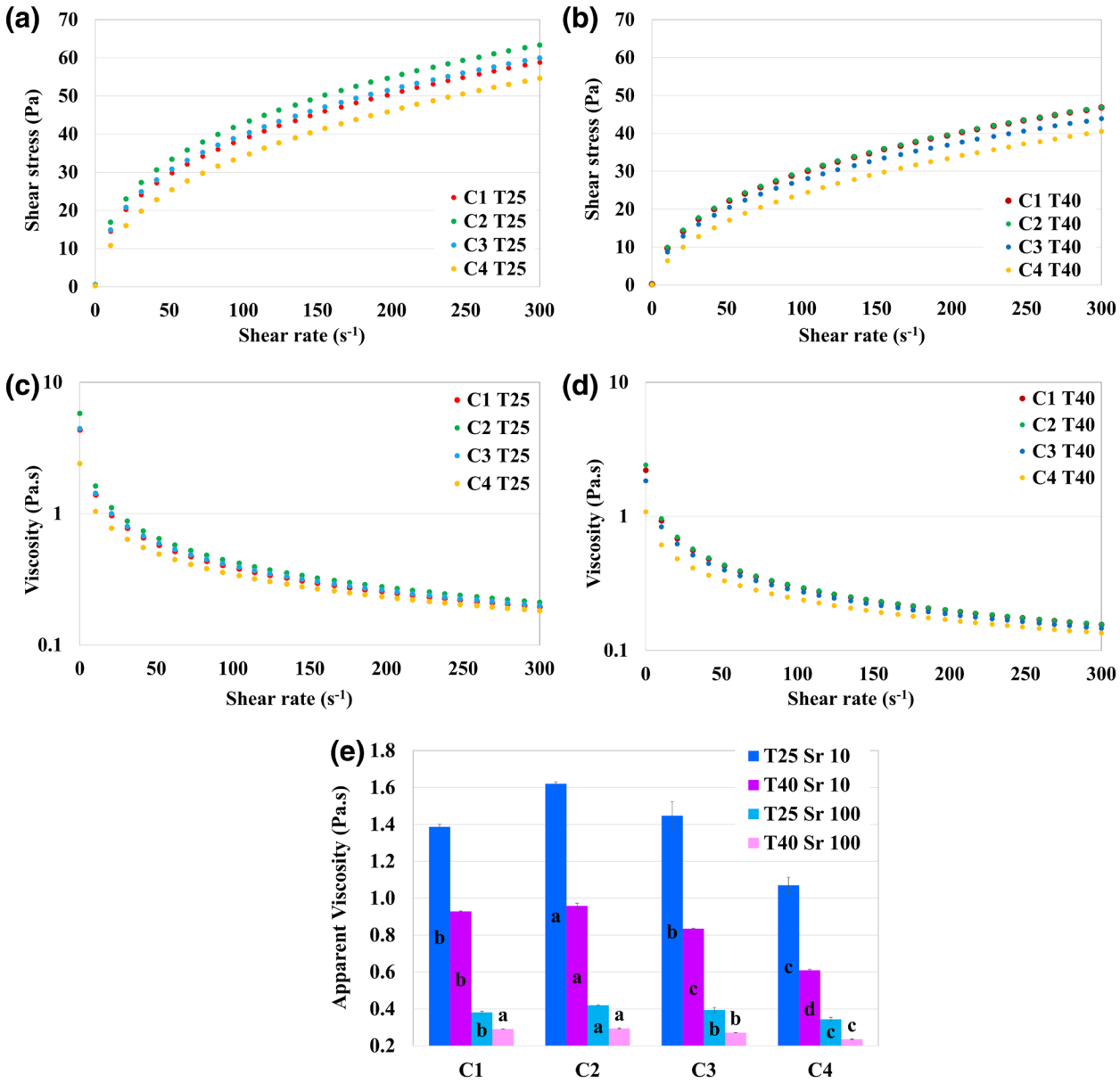

FIGURE 7 | Curves for shear stress at 25°C (a) and 40°C (b); viscosity at 25°C (c) and 40°C (d) and apparent viscosity values (e) at different shear rates (10 and 100 s^{-1}) for coating-forming solutions C1 (CMC + sorbitol), C2 (CMC + sorbitol + soy lecithin), C3 (CMC + sorbitol + soy lecithin + 0.125% tocopherols), and C4 (CMC + sorbitol + soy lecithin + 0.25% tocopherols). Mean values within the same graph with the same letters are not statistically different for Tukey's test at one level ($p < 0.05$).

TABLE 4 | Results for the color parameters of the coating solutions C1 (CMC + sorbitol), C2 (CMC + sorbitol + soy lecithin), C3 (CMC + sorbitol + soy lecithin + 0.125% tocopherols), and C4 (CMC + sorbitol + soy lecithin + 0.25% tocopherols) by the CIELab System.

	Formulation			
Parameter	**C1**	**C2**	**C3**	**C4**
Luminosity (L*)	45.06 ± 0.09a	36.1 ± 0.2c	37.5 ± 0.1b	32.17 ± 0.09d
Chromaticity a*	−0.38 ± 0.02c	−0.13 ± 0.01b	−0.13 ± 0.02b	0.12 ± 0.03a
Chromaticity b*	1.01 ± 0.03b	3.35 ± 0.01a	−0.80 ± 0.03c	−1.59 ± 0.03d
Chroma (C*)	1.08 ± 0.02c	3.36 ± 0.01a	0.81 ± 0.04b	1.6 ± 0.8d
Hue (h°)	69.6 ± 0.8d	87.9 ± 0.2a	81 ± 1c	85.2 ± 0.9b
Delta E	—	43.5 ± 0.3b	30 ± 1c	87 ± 1a

Note: Mean values within the same line with the same letters are not different for Tukey's test at one level ($p < 0.05$).

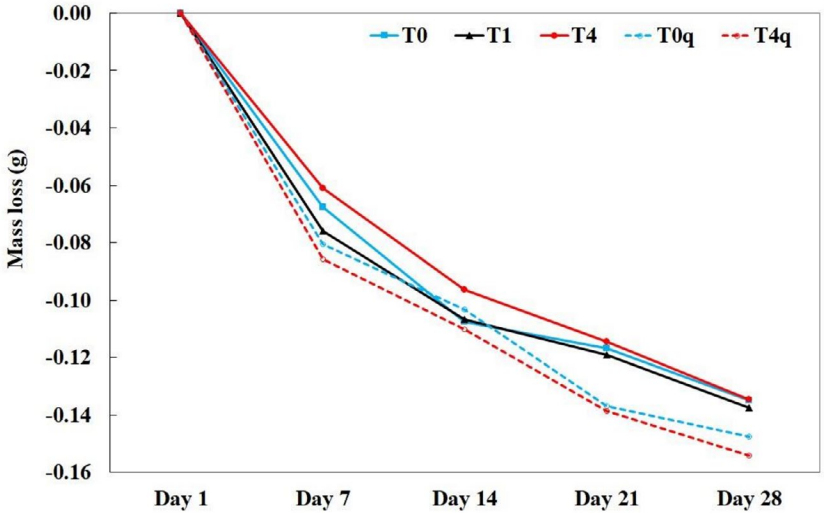

FIGURE 8 | Mass loss curves for whole Brazil nuts and broken Brazil nuts T0 = Brazil nuts without coating; T1 = Brazil nuts with coating (CMC + sorbitol) and T4 = Brazil nuts with coating (CMC + sorbitol + soy lecithin + 0.25% tocopherols), T0q = broken Brazil nuts without coating and T4q = broken Brazil nuts with coating (CMC + sorbitol + soy lecithin +0.25% tocopherols).

TABLE 5 | Mass loss kinetics parameters (Equation 1) obtained for the different coating treatments and whole or broken nuts (T0 = nuts without coating; T1 = nuts with coating [CMC + sorbitol]; T4 = nuts with coating [CMC + sorbitol + soy lecithin + 0.25% tocopherols]; T0q = broken nuts without coating; and T4q = broken nuts with coating [CMC + sorbitol + soy lecithin + 0.25% tocopherols]).

Formulations	X_{eq} (% g g^{-1})	A	k (% g day^{-1})	R^2
T0	−0.55 ± 0.04ab	0.61 ± 0.04b	0.11 ± 0.01a	0.995
T1	−0.54 ± 0.03a	0.60 ± 0.03b	0.113 ± 0.005a	0.992
T4	−0.58 ± 0.04ab	0.62 ± 0.04b	0.083 ± 0.005b	0.996
T0q	−0.7 ± 0.1c	0.8 ± 0.1a	0.11 ± 0.01a	0.985
T4q	−0.62 ± 0.04bc	0.68 ± 0.04ab	0.11 ± 0.02a	0.986

Note: Mean values within the same column with the same letters are not statistically different for Tukey's test at one level ($p < 0.05$).

TABLE 6 | Peroxide value (PV) and conjugated dienes (CD) content for the oils extracted from the nuts T0 = nuts without coating; T1 = nuts with coating (CMC + sorbitol); and T4 = nuts with coating (CMC + sorbitol + soy lecithin + 0.25% tocopherols) during the storage period.

Storage time (days)	Brazil nuts condition	T0	T1	T4
Peroxide value (mEq O_2 kg^{-1})				
Day 01	Whole nuts	1.91 ± Cc	3.78 ± Bb	5.38 ± Aa
Day 14		3.92 ± Bb	5.93 ± Aa	5.90 ± Aa
Day 28		7.64 ± Aa	5.97 ± Ab	6.25Aab
Day 01	Broken nuts	3.86 ± Cb	—	5.74 ± Ba
Day 14		6.57 ± Ba	—	7.95 ± Aa
Day 28		8.33 ± Aa	—	8.41 ± Aa
Conjugated dienes (g L^{-1})				

(Continues)

TABLE 6 | (Continued)

Storage time (days)	Brazil nuts condition	T0	T1	T4
Day 01	Whole nuts	0.174 ± Cc	0.242 ± Ca	0.210 ± Cb
Day 14		0.267 ± Bc	0.250 ± Bb	0.293 ± Ba
Day 28		0.329 ± Ab	0.330 ± Ab	0.340 ± Aa
Day 01	Broken nuts	0.159 ± Cb	—	0.215 ± Ba
Day 14		0.174 ± Bb	—	0.185 ± Ba
Day 28		0.348 ± Aa	—	0.298 ± Ab

Note: Mean values within the same column with the same uppercase letters are not statistically different at one level ($p < 0.05$), and mean values within the same row with the same lowercase letters are not statistically different for Tukey's test at one level ($p < 0.05$).

the incorporation of tocopherols in the oxidative stability of mayonnaise, Alizadeh et al. (2019) observed that for the first 2 months of storage, there was an increase in the PV of mayonnaise oil, which stabilized over the months.

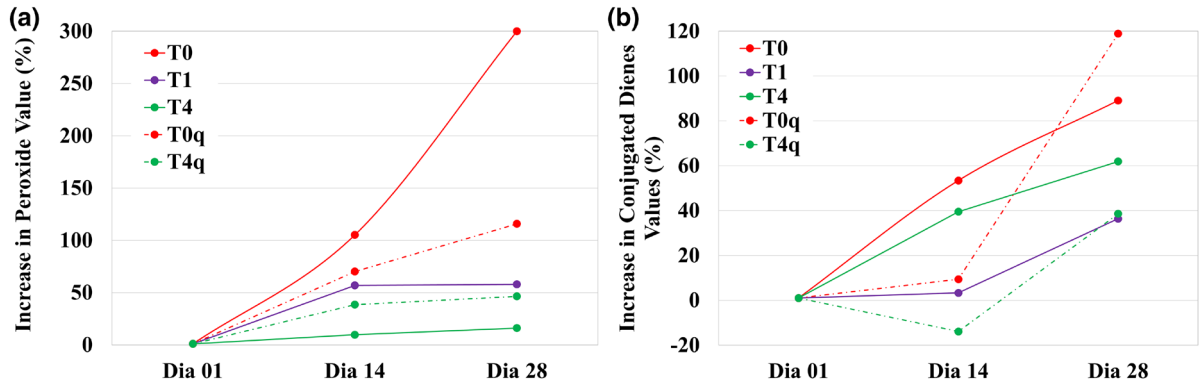

FIGURE 9 | Lipid oxidation evolution curves after 1, 14, and 28 days for the peroxide value (a) and conjugated dienes (b) for whole and broken or injured Brazil nuts T0 = Brazil nuts without coating; T1 = Brazil nuts with coating (CMC + sorbitol) and T4 = Brazil nuts with coating (CMC + sorbitol + soy lecithin + 0.25% tocopherols); q = broken Brazil nuts.

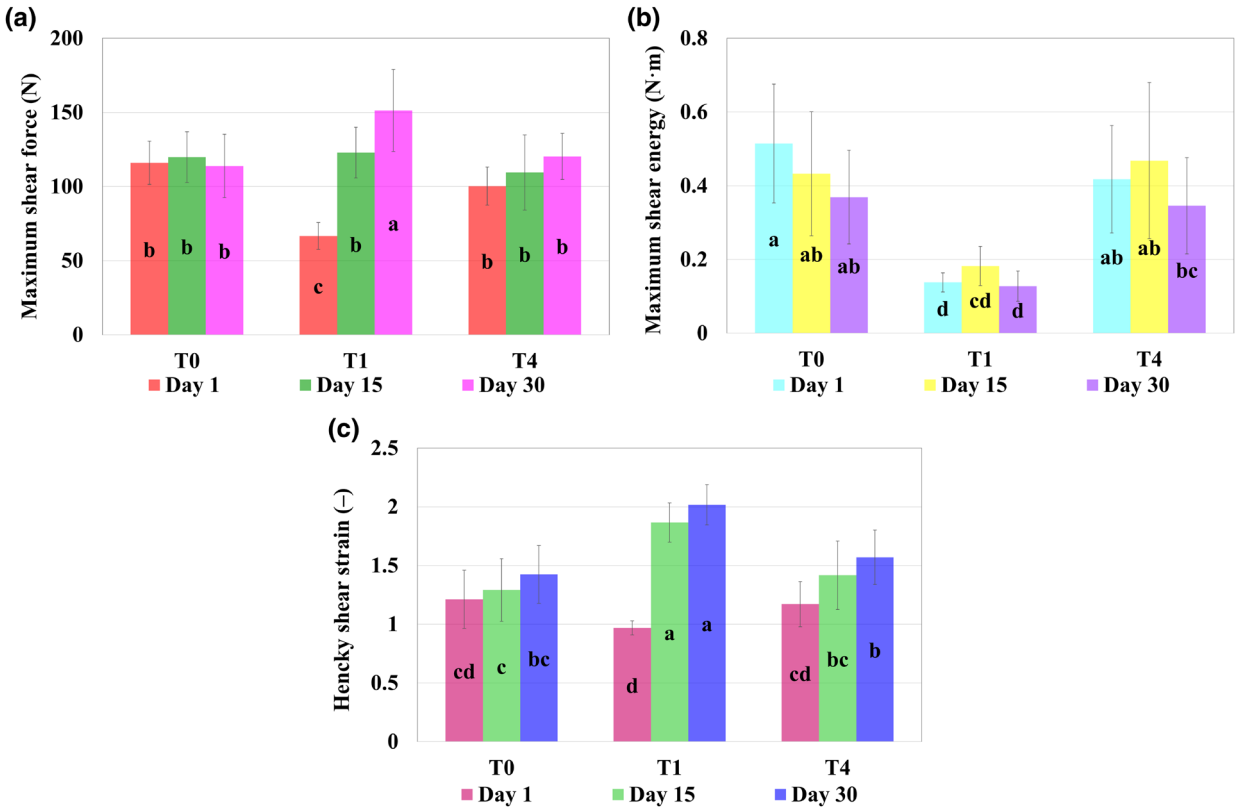

FIGURE 10 | Maximum shear force (a), shear energy (b), and Hencky shear strain (c) for whole Brazil nuts T0 = Brazil nuts without coating; T1 = Brazil nuts with coating (CMC + sorbitol); and T4 = Brazil nuts with coating (CMC + sorbitol + soy lecithin + 0.25% tocopherols). Mean values within the same graph with the same letters are not statistically different for Tukey's test at one level ($p < 0.05$).

For the broken nuts (T0q and T4q), the coating provided stability in the formation of peroxides when the second and last analysis times were observed, a factor that was not observed for the nuts stored without coating. Similar findings were reported by Kwon et al. (2015) and Alizadeh et al. (2019) in their studies, in which the samples containing tocopherols presented oils with lower PV at the end of the storage period compared to the control samples. The literature also shows a similar behavior for the incorporation of essential oils into compound emulsions (Nourbehesht et al. 2018).

Bonilla et al. (2018) also found positive effects for active coatings with boldo extract on Brazil nuts and found that the coatings protected the nuts against oxidation by the oxygen barrier effect and the addition of boldo extract. Sabaghi et al. (2015) observed the effective inhibition of lipid oxidation during the storage of walnut seeds using coatings made of chitosan ($10\,g\,L^{-1}$) combined with green tea extract ($5\,g\,L^{-1}$).

Regarding the values found for CDs of whole Brazil nuts, it was observed that there was a similarity in behavior between

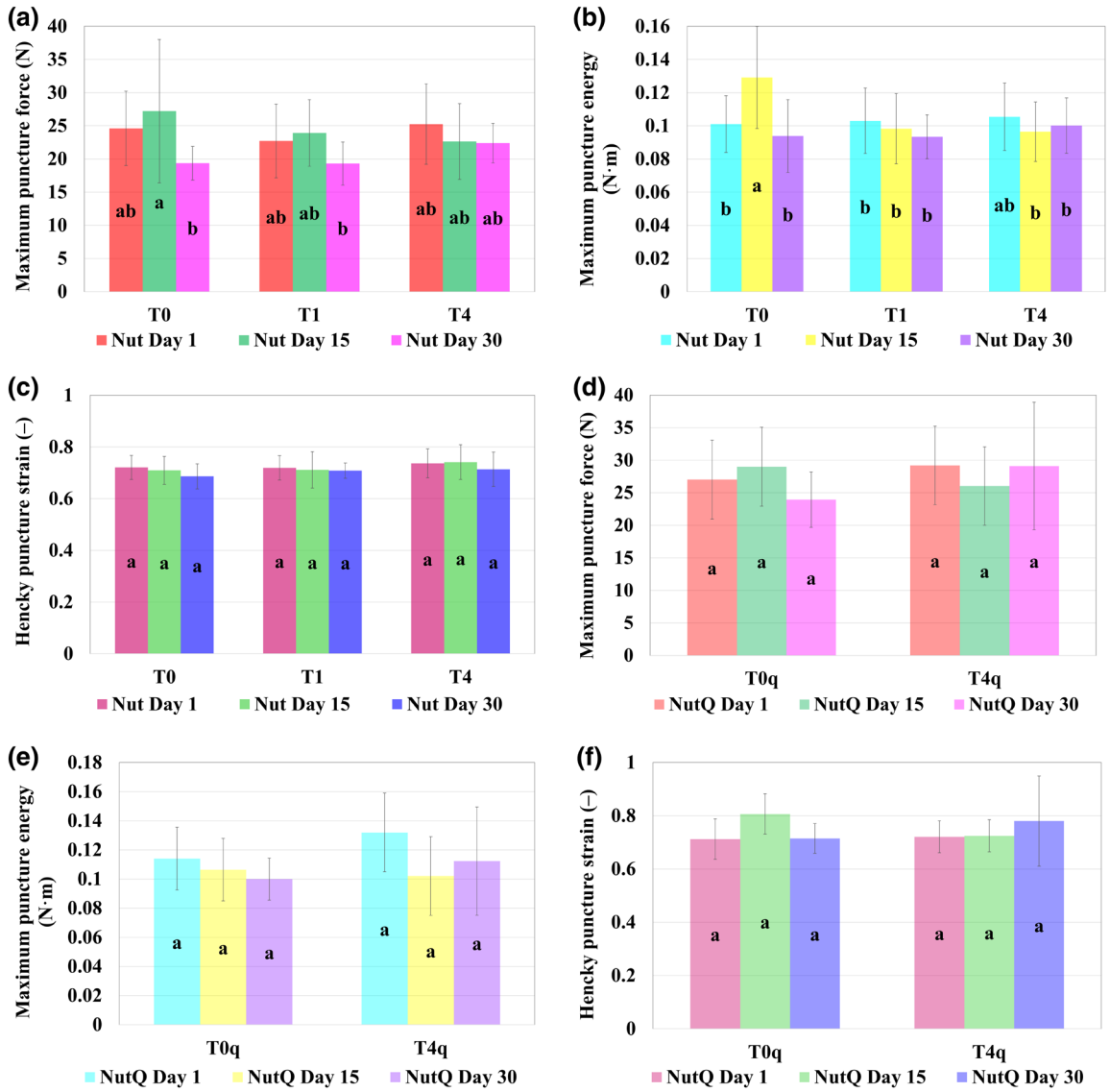

FIGURE 11 | Maximum puncture force, puncture energy, and Hencky puncture strain for whole nuts (a, b, and c, respectively), and maximum puncture force, puncture energy, and Hencky puncture strain for broken nuts (d, e, and f, respectively). T0 = uncoated Brazil nuts; T1 = coated Brazil nuts (CMC + sorbitol); T4 = coated Brazil nuts (CMC + sorbitol + soy lecithin + 0.25% tocopherols); T0Q = broken Brazil nuts without coating; and T4Q = broken Brazil nuts with coating (CMC + sorbitol + soy lecithin + 0.25% tocopherols). Mean values within the same graph with the same letters are not statistically different for Tukey's test at one level ($p < 0.05$).

the different treatments used for the different times analyzed, all of which presented higher diene content throughout the acceleration period. The increasing trend in diene content is due to the breakdown of lipid peroxides present in the oils into secondary fragments and is attributed to the acceleration of oxidation reactions throughout the storage period (Sørensen et al. 2010).

The same behavior was not observed for the broken T4q nuts, which did not follow a trend for evaluating the diene content. This behavior may be related to their initial oxidation state because nuts suffer physical damage during the shelling process in the agroindustry. Therefore, they are more susceptible to triggering oxidation reactions because of the larger surface area exposed to the oxidation process caused by breakage during shelling. It is also noteworthy that the exposed areas (areas that suffered damage) were not uniform, and the damage resulted in nuts of different sizes and shapes, varying from nut to nut, which may lead to different effects on oxidation.

Considering that the oils present in the coating were also quantified in the analyses and that they underwent ultrasonic treatment in solution, the PV and DC values found for the treatments containing tocopherols on the first day of analysis may be the result of the initial oxidation state of the nuts added to the ultrasonic treatment of the coating solutions because the ultrasonic treatment applies extreme physical forces that can decompose lipid molecules and generate highly reactive radicals (Hosseini et al. 2015).

3.7 | Mechanical Behavior of Coated Nuts

Figures 10 and 11 present the results of the analyses of the mechanical behavior of the coated whole nuts and control nuts for the shear tests and the puncture tests.

There were no differences in maximum puncture force between the different days of analysis, both for whole nuts, broken nuts, and coated nuts. For punching energy, no significant effects of applying coatings to the nuts were evident, with no significant differences observed for the different days of analysis.

As with the other tests, the deformation rate of the whole nuts and the broken nuts also did not show significant differences in the coating application for the different analysis times. The application of the edible coating did not influence the mechanical puncture properties of the coated Brazil nuts, in which the coated nuts behaved similarly to the uncoated nuts for the mechanical puncture properties evaluated during the storage period. For the shear properties analyzed for the whole nuts, it can be noted that the edible coating with added tocopherol (T4) did not influence the shear behavior of the nuts for the different analyses performed during the storage period, presenting behavior similar to that of the control nuts (T0).

4 | Conclusions

The addition of tocopherols positively affected the antioxidant capacity and phenolic content of the solutions and did not influence the mechanical behavior of coated Brazil nuts in tensile and puncture tests. The solution containing tocopherols on whole nuts provided stability for peroxide formation during storage. They appear to be promising coatings for applications in foods with high lipid content to prevent or delay oxidation.

Author Contributions

Jessyca Pinheiro da Silva: conceptualization, investigation, formal analysis, validation, visualization, writing – original draft. **Isabelly Campos Carvalho de Cabassa:** investigation. **Tainara Leal de Sousa:** investigation. **Danusa Silva da Costa:** validation, visualization, writing – review and editing. **Evandro Luiz Dall'Oglio:** resources. **Leonardo Gomes de Vasconcelos:** data curation, formal analysis, resources, validation, writing – original draft. **Geovana Rocha Placido:** conceptualization, formal analysis, resources, validation, visualization, writing – original draft. **Mariana Buranelo Egea:** data curation, formal analysis, resources, validation, visualization. **Katiuchia Pereira Takeuchi:** conceptualization, formal analysis, data curation, funding acquisition, project administration, supervision, writing – review and editing.

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

Research data supporting this publication is available upon reasonable request from the corresponding author.

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