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Biobased Polyurethane Films Based on Andiroba (*Carapa guianensis*) and Buriti (*Mauritia flexuosa*) Oil Polyols

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ABSTRACT

This study reports the synthesis of new bio-based polyurethane (PU) films using polyols derived from enzymatic glycerolysis of andiroba (*Carapa guianensis*) and buriti (*Mauritia flexuosa*) oils. The glycerolysis process yielded polyols rich in monoacylglycerols (70–78 mol%) with hydroxyl values of 388–397 mg KOH g^{-1}, while preserving phenolic compounds and, in the case of buriti oil, carotenoid-related molecules. Those polyols were blended with PEG 400 to produce solvent-cast PU films, with vegetable polyol contents ranging from 5 to 100% of the total polyol mixture. Urethane formation was confirmed by FTIR, and mechanical testing revealed that formulations containing 50% vegetable polyol exhibited the best performance, with tensile strength reaching up to 15 MPa. UV–Vis analysis demonstrated the presence of carotenoid-related absorption bands in buriti-based PU films, indicating successful incorporation of bioactive compounds into the polymer matrix. To the best of our knowledge, this is the first report of buriti oil-derived polyols used as reactive monomers for PU film formation. These results demonstrate the feasibility of producing PU films from Amazonian vegetable oils via an enzymatic route while preserving intrinsic bioactive functionality, contributing to the development of sustainable polymeric materials.

1 | Introduction

The investigation of renewable resources to produce innovative polymers has received significant attention from the scientific community in recent years. The use of this kind of raw material is supported by their relatively lower environmental impact and interesting features such as bioactivity and biocompatibility, as well as by the increasing demand for sustainable materials aligned with circular economy principles and decarbonization strategies [1–4]. In this context, bio-based polymeric systems have emerged as promising alternatives to partially or fully replace petrochemical counterparts, as well as due to the growing environmental concerns posed by petro-based chemicals.

Among the polymers, polyurethanes (PU) are highlighted for their many applications in daily life, such as mattresses, heat-insulating materials, sealants, paints, resins, bonds, and medical devices [5, 6]. PUs are prepared basically by the reaction of polyols and isocyanates. An important feature of PUs is the possibility of their synthesis from different classes of precursors and additives, allowing tailor-made finished materials. This versatility has allowed significant developments in the substitution of petroleum-based polyols by renewable raw materials [7], such as cellulose [8], sugars [9], proteins [10], and most often vegetable oils (VOs) [11–14], which have become one of the most explored renewable platforms for bio-based PU production in recent years.

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VOs have stood out in the production of PUs for their diversity of species and their availability in the world, which results in a relatively low market price and reduces an eventual competition with the food chain. There are plenty of studies available exploring the use of VOs for PU production, as well as many review articles detailing the most recent advances [13, 15]. VOs based polyols can be applied in adhesives [16], coatings [14, 17, 18], elastomers [19], and foams and thermosets [12, 20], the latter being the most prominent. The transformation of vegetable oils (VOs) into reactive monomers typically requires modification of their main chemical structure, namely triglycerides (TAG). This can be achieved either by functionalizing double bonds (e.g., epoxidation, silanization, acrylation, maleinization) or by interchanging acyl groups through esterification/transesterification reactions such as glycerolysis [13, 15], which may be conducted via chemical or enzymatic processes depending on the desired selectivity and reaction conditions [11, 21].

Glycerolysis is a classic method for the preparation of monoacylglycerols (MAG) and diacylglycerols (DAG) from VOs. MAG and DAG are non-ionic emulsifiers with applications in several fields, such as cosmetics, food products [22] and polymers (alkyd resins and polyurethanes) [11, 23–25]. The reaction is kinetically controlled and is carried out under stoichiometric glycerol excess to shift the equilibrium to MAG [21, 26]. It can be performed using inorganic or lipase-based catalysts. The employment of lipases as catalysts is more recent and has revealed important advantages, such as low reaction temperature (40°C–60°C) and reduction of chemical wastes, which results in a cleaner process [21]. A special feature of enzymatic catalysts is their stereoselectivity that, combined with low temperature conditions, allows the modification of VOs without significant degradation. We have demonstrated it by comparing the carotenoids' integrity in the glycerolysis of buriti oil using the conventional MgO catalyst and Novozym 435, showing that the former caused a substantial degradation of the carotenes in the product while the lipase catalyst allowed the maintenance of those compounds [21]. The preservation of such intrinsic bioactive molecules may contribute to additional functional properties in the resulting materials, particularly when compared to systems obtained via harsher chemical modification routes. This feasibility of obtaining VO derivatives that keep the bioactive components of the raw oil suggests potential for the development of multifunctional materials, including medical devices and food packaging [27–30].

In this work, our attention is focused on two Amazonian VOs, andiroba (*Carapa guianensis*) and buriti (*Mauritia flexuosa*), which are potential candidates for the production of new polymers with bioactive properties. Andiroba is an endemic species from the North region of Brazil that is rich in a non-edible oil that presents bioactive substances (limonoids), and is well reported for medicinal applications, such as antimicrobial [27], anti-inflammatory [31], and repellent properties [32]. Buriti, a palm-tree of high occurrence in the Amazon region, stands out for its high content of antioxidant substances, mainly carotenes and phenolics. β-carotene, α-carotene, γ-carotene, among other apocarotenoids, are found in reasonable amounts in the buriti fruit [33–35], and many polyphenols have already been extracted from the buriti trunk and leaves [36]. Andiroba oil, in a previous work of our group, has already been successfully modified by enzymatic glycerolysis and used in the preparation of flexible PU foams [11]. Buriti oil was already reported as a polymer additive, having the function of antioxidant or plasticizer [28, 29]. Recent reports have described the incorporation of natural phenolic compounds as additives in polyurethane matrices for UV-shielding or antioxidant purposes [37–40]. Moreover, carotenoids have also been incorporated directly as chain extenders in segmented poly(ester-urethane)s, demonstrating their chemical compatibility with polyurethane systems and the feasibility of producing carotenoid-containing PU films [41]. However, unlike additive-based approaches, and to the best of our knowledge, buriti oil-derived polyols have not yet been employed as reactive monomers in polyurethane synthesis, particularly for film formation.

Considering these aspects, the aim of this work is to evaluate the preparation of PU films derived from enzymatically modified andiroba and buriti oils, through the following steps: (i) enzymatic glycerolysis with Novozym 435 to guarantee a minimum degradation of the bioactive compounds; and (ii) production of the PU films by step-growth polymerization of the obtained polyols. In addition, the study investigates whether chromophoric features associated with buriti oil can be retained after polymer network formation.

2 | Materials and Methods

2.1 | Materials

Andiroba and buriti oils were purchased from Ferquima (Vargem Grande Paulista—Brazil). Their fatty acid compositions were provided by the supplier. Buriti oil is composed of palmitic acid (16:0, 17%), palmitoleic acid (16:1, 1%), stearic acid (18:0, 2%), oleic acid (18:1, 75%), and linoleic acid (18:2, 5%), with a molar mass of 921 g/mol. Andiroba oil contains oleic acid (18:1, 48%), palmitic acid (16:0, 28%), linoleic acid (18:2, 11%), stearic acid (18:0, 9%), and arachidic acid (C20:0, 1,4%), with a molar mass of 894 g/mol. The commercial lipase immobilized on acrylic resin Novozym 435 from Novozymes was donated by LNF company (Bento Gonçalves—Brazil). Glycerol (Neon, Suzano—Brazil), t-butanol (Neon Comercial, São Paulo—Brazil), 4,4-Diphenyl methane diisocyanate (Dinâmica, Indaiatuba—Brazil), polyethylene glycol 400 g mol^{-1} (Neon, Suzano—Brazil), isopropanol HPLC grade (Êxodo, Hortolândia—Brazil), methanol HPLC grade (Qhemis, Jundiaí—Brazil), monoolein, diolein and triolein standards (Sigma-Aldrich) were used as received. Toluene P.A. (Neon Comercial, Suzano—Brazil) was stored in a glass container with molecular sieves until use.

2.2 | Methods

2.2.1 | Preparation of Polyols From Andiroba and Buriti Oils

Polyols from andiroba and buriti oils were prepared by enzymatic glycerolysis, according to the reaction scheme represented in Figure 1.

Similar reaction procedures were employed for the preparation of both types of polyols, differing only in terms of specific

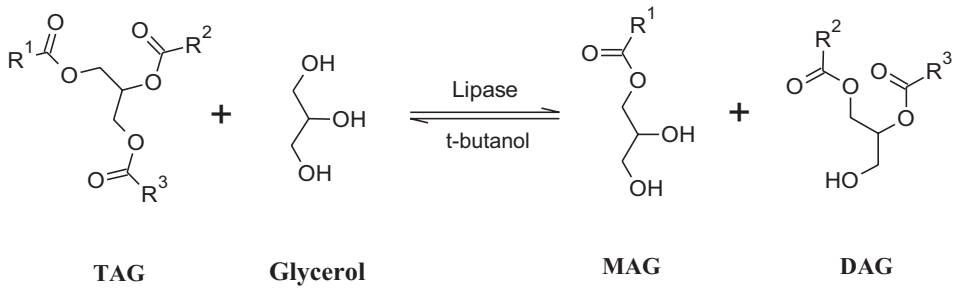


FIGURE 1 | Glycerolysis reaction scheme.

TABLE 1 | Parameters of the glycerolysis of the vegetable oils.

Reaction parameter	Andiroba polyol	Buriti polyol
Temperature [°C]	70	55
Glycerol/oil molar ratio [mol:mol][a]	9:1	6:1
Solvent/substrate ratio [v/w%]	150	150

[a]The employed molar quantity of each oil was calculated using as reference the respective average molecular weight obtained from its fatty acid composition.

TABLE 2 | Reference formulations for polyurethane films (5 g basis, NCO/OH = 1.0).

Composition (OH basis)	Vegetable polyol (g)	PEG 400 (g)	MDI (g)
Andiroba 100:0	2.50	0.00	2.49
Andiroba 75:25	1.78	0.84	2.37
Andiroba 50:50	1.13	1.60	2.26
Andiroba 25:75	0.54	2.30	2.16
Andiroba 10:90	0.21	2.69	2.10
Andiroba 5:95	0.10	2.81	2.09
Buriti 100:0	2.53	0.00	2.47
Buriti 75:25	1.81	0.84	2.35
Buriti 50:50	1.15	1.60	2.25
Buriti 25:75	0.55	2.29	2.15
Buriti 10:90	0.22	2.68	2.10
Buriti 5:95	0.11	2.81	2.08

values of process parameters. The experiments were performed under mechanical stirring in a round bottom flask of 250 mL immersed in a silicone bath at the conditions specified in Table 1, which were determined in a previous work [21], using an experimental design. The reaction time was 24 h for both oils. The reactions were carried out based on the protocol described by da Silva et al. [21], consisting of the following steps: (i) addition of the required amounts of substrate (glycerol + oil) and enzyme (3 wt% of substrate content) to the reaction flask; (ii) addition of the solvent (t-butanol); (iii) heating of the reaction mixture to the specified reaction temperature; and (iv) establishment of the defined stirring condition, which was considered the starting point of the reaction time. After 24 h, the reaction mixture was allowed to cool to room temperature. The immobilized catalyst was then removed by sieving (400 mesh), and the solvent was distilled in a rotary evaporator under reduced pressure at approximately 86°C and −700 mmHg. The resulting mixture was left to stand overnight for phase separation, allowing excess glycerol to decant due to its higher polarity and density. The upper polyol-rich phase was then collected. The approximate experimental quantities were as follows: for andiroba oil, 42 g of oil, 39 g of glycerol and 122 mL of *t*-butanol were used, corresponding to an approximate glycerol-to-oil molar ratio of 9:1; for buriti oil, 50 g of oil, 30 g of glycerol and 120 mL of *t*-butanol were employed, corresponding to an approximate molar ratio of 6:1. These values are consistent with the solvent/substrate ratio (150% vol/wt.) reported in Table 1. Polyols were obtained as yellow (andiroba) or red (buriti) butter and stored in a refrigerator (−18°C) until use. A sample of the polyol was collected for determining MAG, DAG, and TAG through HPLC analysis. The hydroxyl value (IOH) of the polyols was determined according to ASTM E222-17—Method A. Approximately 1.99 g of sample were weighed in duplicate using 250 mL borosilicate glass flasks. The samples were maintained at 98°C for 4 h in a heating bath (Quimis). After the reaction, the mixture was titrated with 0.52 M NaOH solution until the appearance of a persistent pink color. The hydroxyl value was calculated based on the difference between blank and sample titration volumes, considering the normality of the titrant and the sample mass.

2.2.2 | Polyurethane Films Preparation

Prior to PU films synthesis, moisture control measures were adopted. Toluene (P.A.) was stored in a sealed glass container containing molecular sieves until use. The vegetable-based polyols were further dried under vacuum at 40°C for 24 h to eliminate any residual solvent and moisture. PEG 400 was also vacuum-dried for 24 h prior to use. These procedures were carried out to minimize possible side reactions between isocyanate groups and water. For polyurethane film preparation, Part A consisted of mixtures of vegetable-based polyol and PEG 400. The nominal compositions (100:0, 75:25, 50:50, 25:75, 10:90, and 5:95) were defined based on the percentage contribution of each polyol to the total amount of hydroxyl functional groups in Part A. The hydroxyl values were used to calculate the equivalent weights and the corresponding masses required for each formulation. Part B (MDI) was added in an amount calculated to maintain an NCO/OH molar ratio

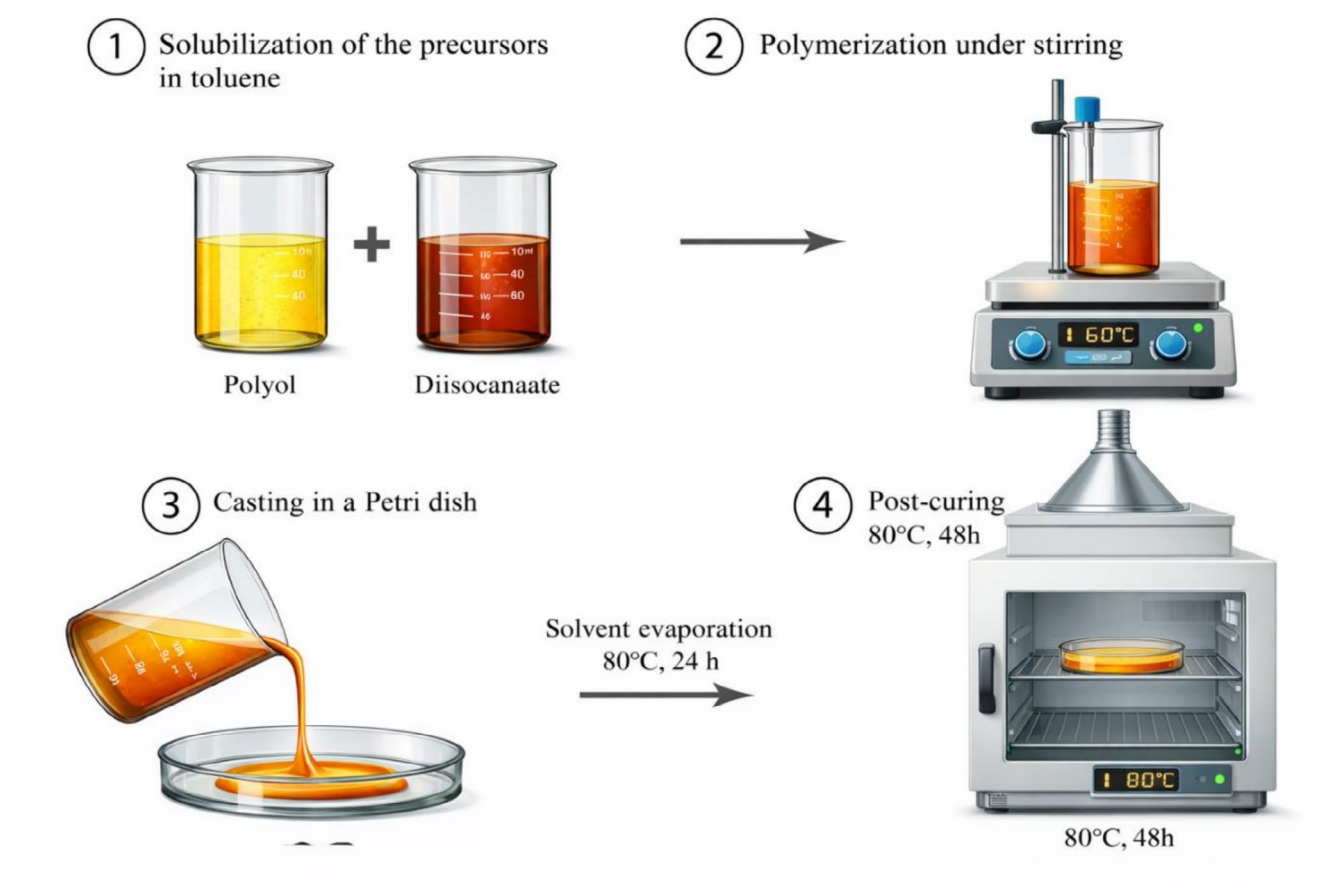


FIGURE 2 | Schematic illustration of the solvent casting method used for the preparation of the polyurethane films. [Color figure can be viewed at wileyonlinelibrary.com]

of 1.0. Table 2 presents the resulting reference formulations for reactive samples (Part A + Part B) of approximately 5 g. The actual total mass of the reactive samples ranged from approximately 4 to 10 g, depending on the composition and the scale of the experimental batch, but the proportions of reactants shown in Table 2 were kept in all cases. To reduce viscosity and facilitate homogenization, 5 mL of toluene was added to each beaker (Part A and Part B). The beaker containing Part A was placed on a heating plate at 60°C until complete melting and dilution of the polyol. Afterwards, Part B was slowly added to Part A under agitation, and the reactive mixture was stirred for 10 min. The mixture was poured into a 15 cm diameter petri dish and placed in a stove at 80°C for 24 h to allow solvent evaporation. After this period, the formed PU film was unmolded and placed back in the stove for an additional time of 48 h at 80°C for curing. No bubble formation was observed during solvent evaporation and curing, suggesting negligible CO_2 evolution from isocyanate–water side reactions under the adopted processing conditions. The film thickness was measured using a digital caliper prior to tensile testing, resulting in an average thickness of 393 ± 54 μm. This procedure is represented in the scheme of Figure 2.

2.2.3 | Instrumental Analyzes

The andiroba and buriti polyols compositions were determined by high performance liquid chromatography (HPLC), in an Agilent 1260 Infinity equipment (Zorbax Eclipse Plus C18 column, 250×4.6 mm, 5 μm particle size and diode-array detector at 205 nm). The mobile phase was a gradient of isopropanol and methanol: water mix (90:10 vol/vol), according to the methodology described by da Silva et al. [8]. Calibration curves of monoolein (R^2 = 0.9912), diolein (R^2 = 0.9999), and triolein (R^2 = 0.9936) standards were used to determine the concentrations of MAG, DAG, and TAG.

The antioxidant activity was evaluated through the determination of total phenols by the Folin–Ciocalteu assay. The quantification of phenolic compounds was performed by means of comparison with a gallic acid calibration curve, prepared using dilutions of a stock solution of 0.4 mg/mL of gallic acid in methanol in the range of 10 to 100 μg/mL. In a black 96-well microplate (Fluotrac 200, Greiner bio-one), 25 μL of the prepared solutions, 125 μL of 10% Folin–Ciocalteu reagent, and 100 μL of 7.5% sodium carbonate were added. After mixing, the tubes remained resting in the dark for 60 min and then, after gentle homogenization, the readings of the absorption measurements were carried out in a spectrophotometer (SpectraMax M3) at a wavelength of 760 nm, thus obtaining a standard analytical curve. The oil and polyol samples were diluted at a concentration of 2 mg/mL and submitted to the same experimental conditions as the analytical standard. Approximately 5% (vol/vol) of surfactant Tween 80 was added to all samples to improve the solubility since oily samples are poorly soluble in methanol. Three replicates of each sample

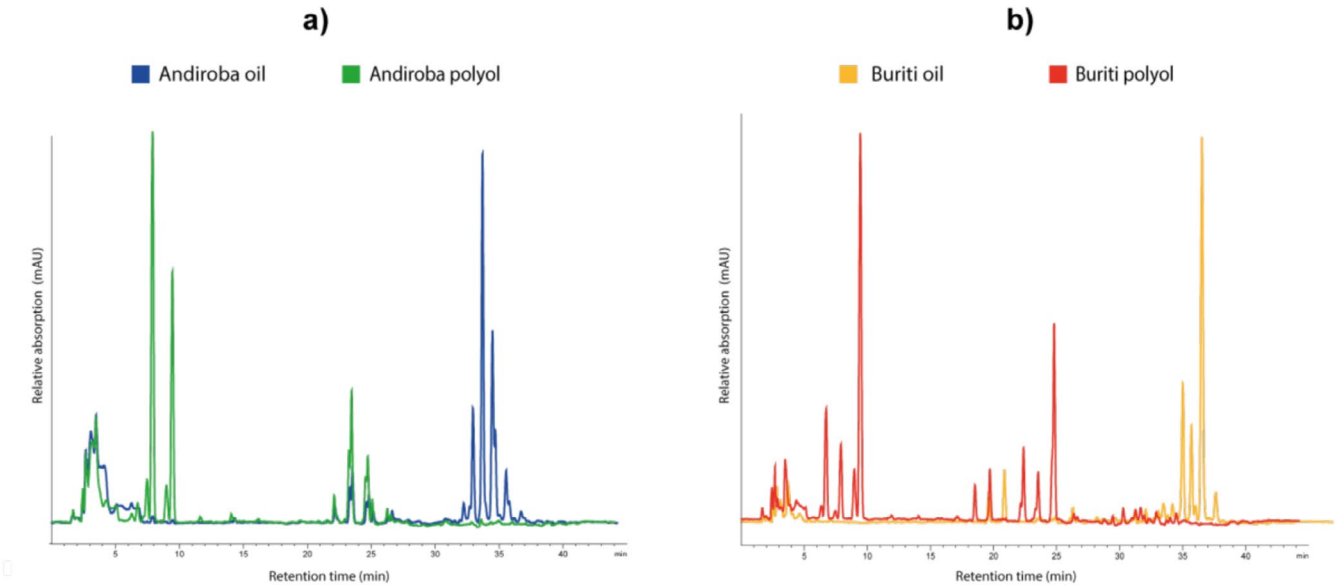


FIGURE 3 | HPLC chromatograms of the vegetable oils and their respective polyols prepared through enzymatic glycerolysis: (a) andiroba oil and andiroba polyol prepared at 70°C, gly/oil 9:1 (mol:mol), and solv/subs ratio of 150 (vol/wt%); (b) buriti oil and buriti polyol prepared at 55°C, gly/oil 6:1 (mol:mol), and solv/subs ratio of 150 (vol/wt%). [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 3 | Compositions of Buriti and Andiroba based polyols.

Parameter	Buriti oil	Buriti polyol	Andiroba oil	Andiroba polyol
MAG [mol%]	0	78	0	70
DAG [mol%]	4	22	8	30
TAG [mol%]	96	0	92	0
IOH [mg KOH g^{-1}]	—	388	—	397

were prepared. Pure solvent was used as blank. Results were expressed as milligrams of gallic acid equivalents (GAE) per 100 g of sample.

Fourier transformed infrared (FTIR) spectroscopy measurements were made in a Frontier Perkin Elmer spectrophotometer using attenuated total reflection analysis (ATR) mode, wavenumber range of 4000 to 700 cm^{-1}, 16 scans, and resolution of 4 cm^{-1}.

Thermogravimetric analysis of PU samples was conducted in a TA Instruments SDT Q600 equipment, applying nitrogen atmosphere of 100 mL·min^{-1}, heating rate of 20°C min^{-1} and temperature range from 25°C to 700°C.

PU films were analyzed using a UV–Vis spectrophotometer (T80, PG Instruments). Film samples with similar thickness (~0.4 mm) were placed directly in the optical path, and spectra were recorded in the 370–600 nm wavelength range.

Tensile properties were investigated by means of an EMIC DL30000 machine equipped with a 100 N load cell. The measurements were performed at room temperature using rectangular specimens with 15 mm width and approximately 0.4 mm thickness, with a crosshead speed of 200 mm × min^{-1} and an initial length of 25 mm. At least seven specimens were tested for each PU composition.

2.2.4 | Statistical Analyses

The obtained experimental data were statistically analyzed in MS Excel using the supplement based on R programming language ActionStat (EstatCamp, São Carlos-Brazil), using ANOVA and Tukey test for mean comparisons. In all cases a p value lower than 0.05 was considered significant.

3 | Results and Discussion

3.1 | Polyol Characterization

The comparison of the HPLC chromatograms shown in Figure 3 indicates that the glycerolysis reaction was effectively shifted toward the production of MAG and TAG at the employed conditions. This is shown by the disappearance of the retention peaks corresponding to triglycerides (TAG) in the region of 30–35 min, in the chromatograms of the pure andiroba and buriti oils, and the appearance of peaks at 7–11 min (MAG) and 20–25 min (DAG) for the respective polyols. Table 3 shows the molar percentages of mono and diester in the polyols based on the areas of these two later peaks, and the hydroxyl value. In agreement with the use of a molar excess of glycerol, MAG was the major component in both polyols. Higher yields of MAG are relevant in the context of this work, since these molecules contain the two active hydroxyl groups required for the step-growth formation of PUs. Conversely, it is important to minimize DAG quantities, since they act as chain terminators, resulting in a potentially low degree of polymerization and, consequently, in a polymer with poor mechanical properties for most common applications.

Samples of vegetable oils and polyols were analyzed by Folin–Ciocalteu assay to evaluate the presence of phenolic compounds. The results are presented in Table 4. The determination of these compounds indicates antioxidant activity since phenolic compounds can donate hydrogen or electrons besides their capacity to form stable radical intermediates [36], which prevents oxidation and consequently extends the shelf life of the oil. The concentration of phenolic compounds found for both oils can be considered low in comparison to other values reported in the literature. For andiroba oil, Novello et al. [42] reported total phenolic compounds in the range from 11.15 mg GAEq/100 g to 35.67 mg GAEq/100 g for the oil extracted using pressurized n-butane as solvent, and Teixeira et al. [43] reached a concentration of 24.99 mg GAEq/Kg for the oil extracted using hexane. Regarding buriti oil, Marcelino et al. [44] obtained a concentration of 21.57 ± 0.83 mg GAEq/100 g for a commercial sample extracted by cold pressing, while Koolen et al. [36] achieved even higher concentrations for extracts obtained from fruits (378.07 ± 3.12 mg GAEq/100 g) using extraction methods involving hexane: dichloromethane and methanol: water. It can be seen from these data that the final concentration of phenolic compounds in the oil is strongly dependent on the extraction method and conditions. Based on that, it is inferred that the low values of phenolic compounds in the commercial oils employed in this work may be related to the method of extraction of the oil. However, no detailed information regarding this process was given by the oil supplier, in order to confirm this hypothesis.

On the other hand, despite the low concentration of phenolic compounds in the pure oils, it is observed that there is no difference in total phenolic compounds between pure oil and polyol for both samples. This demonstrates the efficiency of the enzymatic method in preserving the bioactive compounds of the pure oil, either from andiroba or buriti. The presence of phenolic compounds in the polymeric matrix can help them in the protection of the film, retarding the oxidation process of the material. However, it is important to note that, in the present study, antioxidant behavior was inferred indirectly from total phenolic content in the oils and polyols. Direct antioxidant assays on the final polyurethane films were not performed, and therefore the effective radical-scavenging capacity of the polymeric network remains to be quantitatively demonstrated in future investigations.

TABLE 4 | Total phenolic compounds in andiroba oil, buriti oil, andiroba polyol, and buriti polyol.

Sample	Total phenolics [mg GAEq/100 g]
Andiroba oil	2.77 ± 0.21
Andiroba polyol	2.94 ± 0.17
Buriti oil	2.57 ± 0.06
Buriti polyol	2.60 ± 0.42

3.2 | Polyurethane Characterization

3.2.1 | Appearance and Consistency

Figure 4 shows photos of samples of the PU films prepared with andiroba and buriti polyols, PEG 400 and MDI, whose visual aspect ranged from translucent to matte, with the buriti polyol-based PU films having more intense colors. The films with 100% polyol, of both buriti and andiroba, were brittle to the point of making it impossible to keep the film integrity during its removal from the dishes used as molds. The PU films with 75% polyol, although removable from the molds, were still too brittle to be cut into specimens for mechanical testing. The other films showed elastomeric behavior and were easily removed from the molds with the aid of light heating.

3.2.2 | Fourier Transform Infrared Spectroscopy

Figure 5 shows the FTIR spectra of all the prepared PUs. Stretching vibrations of carbonyl (amide I) are observed at 1712 cm^{-1}, evidencing urethane formation [45]. The bands at 1540 cm^{-1} and 1513 cm^{-1} are also related to urethane groups, more specifically to C—H and N—-H vibrations, respectively [46, 47]. The band at 3300 cm^{-1} was attributed to the axial deformation of —NH of urethane groups [48, 49]. The formulations with 100% polyol showed residual bands centered at 2280 cm^{-1}, indicating the presence of unreacted isocyanate groups. This behavior may be attributed to the predominance of secondary hydroxyl groups in monoacylglycerols, which exhibit lower reactivity toward isocyanates compared to primary hydroxyl groups, as widely discussed in polyurethane reaction mechanisms and

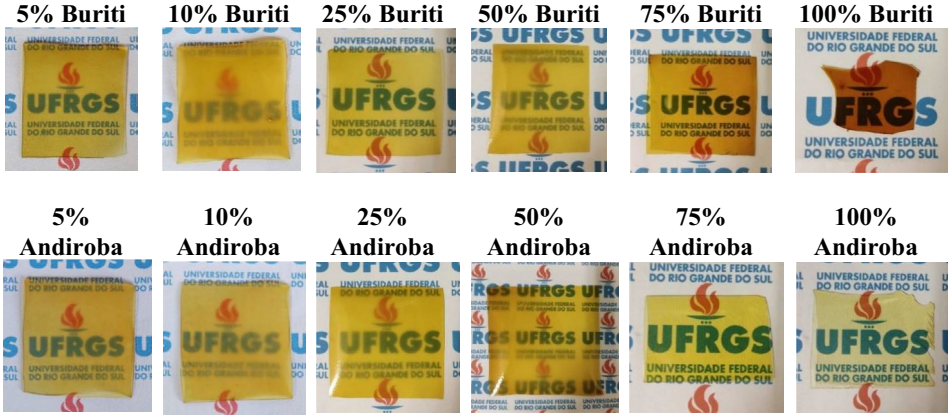


FIGURE 4 | Visual aspect of the polyurethane films from andiroba and buriti polyols. [Color figure can be viewed at wileyonlinelibrary.com]

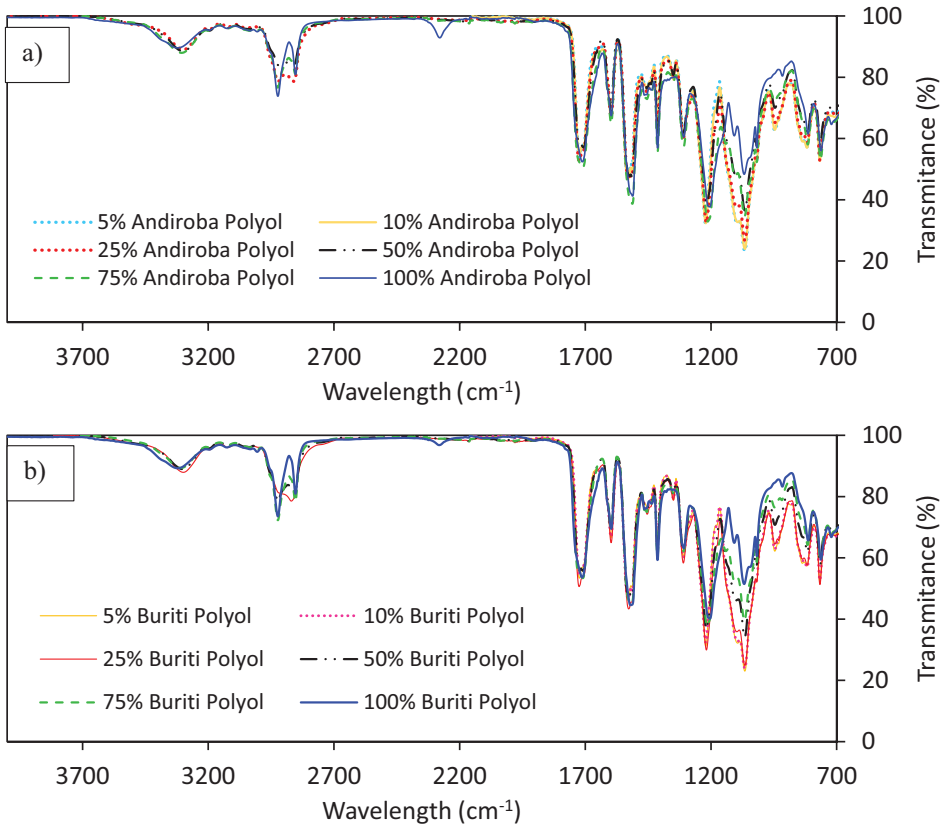


FIGURE 5 | FTIR of polyurethane films from (a) andiroba and (b) buriti polyols. [Color figure can be viewed at wileyonlinelibrary.com]

segmented PU design principles [50]. In addition, the absence of PEG 400 reduces molecular mobility and diffusion during the step-growth polymerization process, limiting effective interaction between NCO and OH groups. The higher viscosity and hydrophobic character of these systems may further restrict chain mobility and segmental rearrangement, thereby contributing to incomplete conversion, a phenomenon also reported in bio-based PU systems containing structurally irregular or bulky natural components [40, 51]. The bands between 2950 and 2850 cm^{-1} result from the asymmetric vibrations from the —CH_2 and —CH_3 groups derived from vegetable oil fatty acids [48]. *Stretching vibrations of* C—O groups from polyol and PEG segments are observed in the range between 1065 and 1217 cm^{-1} (~1065 cm^{-1}, ~1104 cm^{-1}, and ~1217 cm^{-1}) [52].

3.2.3 | Thermogravimetric Analysis

Figure 6 shows mass loss (TG) and derivative mass loss (DTG) curves for the 12 compositions of PUs films. The PU films with 100% and 75% of vegetable polyol present the two common PU thermal decomposition stages. The first, between 240°C and 330°C, is related to the urethane/urea bonds [48, 53, 54]. The second, from 300°C to 550°C, is the result of multiple processes, including the scission of the polyol chains and other thermo-oxidation reactions at higher temperatures [48, 53, 54]. On the other hand, for the PUs with 25%, 10%, and 5% of vegetable polyol, the decomposition of the PEG segments becomes the predominant thermal event, generating an intermediate decomposition event in the interval around 250°C–450°C, which is also found in the pure PEG analysis, followed by a shoulder that extends up to 550°C. Besides these degradation peaks, the TGA thermograms of all samples show a mass loss event at lower temperatures (up to 150°C), related to moisture vaporization. The intensity of this moisture loss event is proportional to the PEG content of the sample, due to the high hygroscopicity of this component. Table 5 presents the temperatures corresponding to 5%, 10%, and 50% of mass loss (T_5%, T_{10}%, and T_{50}%), and the residual char residue at 800°C for each sample, both in terms of dry sample. In general, the films with lower vegetable polyol content exhibited greater thermal stability, as evidenced by the progressive increase in T_5% and T_{10}% values for the compositions from 100% to 10% polyol. This behavior may be attributed to the fact that the vegetable-based polyols are composed mainly of esterified fatty acids and bioactive molecules with lower degradation temperatures, compared to PEG.

Regarding the percentage char residue, a gradual increase was observed with the increase of PEG content. This may be attributed to the fact that PEGs are known to be decomposed by chain scission and dehydration processes, which can lead to the formation of carbonaceous residues under inert atmosphere [55]. Voorhees et al. [56] described the formation of multiple PEG degradation products and hydroxytelechelic fragments that undergo carbonization at temperatures above 300°C. In contrast, vegetable oils and their derivatives contain fatty acid esters and bioactive molecules that are more volatile and degrade more completely under the same conditions, resulting in lower or no residual mass [57, 58]. A similar effect was reported by Kim et al. [59], who observed a decrease in char content as the content of castor oil increased while lignin-rich formulations (aromatic) produced higher char yields.

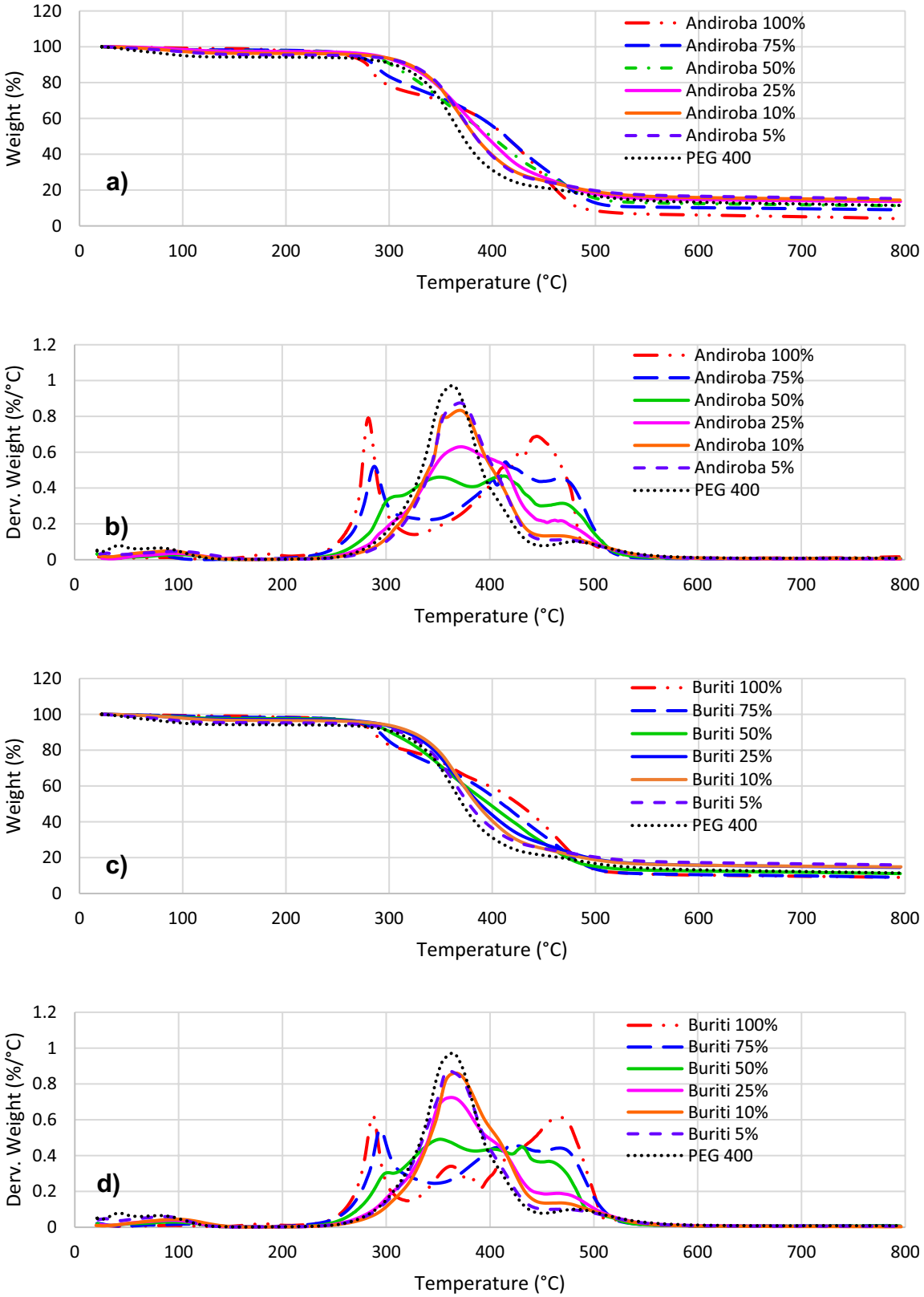


FIGURE 6 | Thermogravimetric curves of the polyurethane films from (a) andiroba polyol and their (b) first derivative curves, and the thermogravimetric curves of the PU films from (c) buriti polyol and their respective (d) first derivative curves. [Color figure can be viewed at wileyonlinelibrary.com]

3.2.4 | UV Spectra of the Polyurethane Films

Figure 7 shows the spectra of PU films prepared with andiroba and buriti polyols in the wavelength range from 400 to 600 nm. The films analyzed had a thickness of 393 ± 54 μm. An absorption band in the region typical of carotenoids (450–500 nm) [35, 60] is clearly visible in the films with 100% and 75% buriti polyol, corroborating the hypothesis that the enzymatic route allows

TABLE 5 | Thermal decomposition temperatures at different percent weight losses (T_5%, T_{10}%, and T_{50}%) and char residue for polyurethane films and PEG 400.

Sample	T_5% [°C]	T_{10}% [°C]	T_{50}% [°C]	Char residue at 800°C [%]
Andiroba 100%	265.8	279.9	413.6	5.2
Andiroba 75%	275.3	288.0	414.4	9.7
Andiroba 50%	291.2	307.1	400.7	12.1
Andiroba 25%	303.6	323.7	396.2	14.4
Andiroba 10%	311.9	329.8	385.7	15.6
Andiroba 5%	313.0	332.1	385.8	16.5
Buriti 100%	273.7	285.8	426.2	
Buriti 75%	279.7	292.8	411.1	9.7
Buriti 50%	291.2	307.9	399.5	10.0
Buriti 25%	305.7	325.4	390.5	12.1
Buriti 10%	313.6	332.1	387.3	15.5
Buriti 5%	303.7	323.7	379.7	15.8
PEG 400	300.2	320.8	373.5	16.4

Note: All parameters (T_5%, T_{10}%, T_{50}%, and percent char residue) were expressed in terms of the dry sample weight.

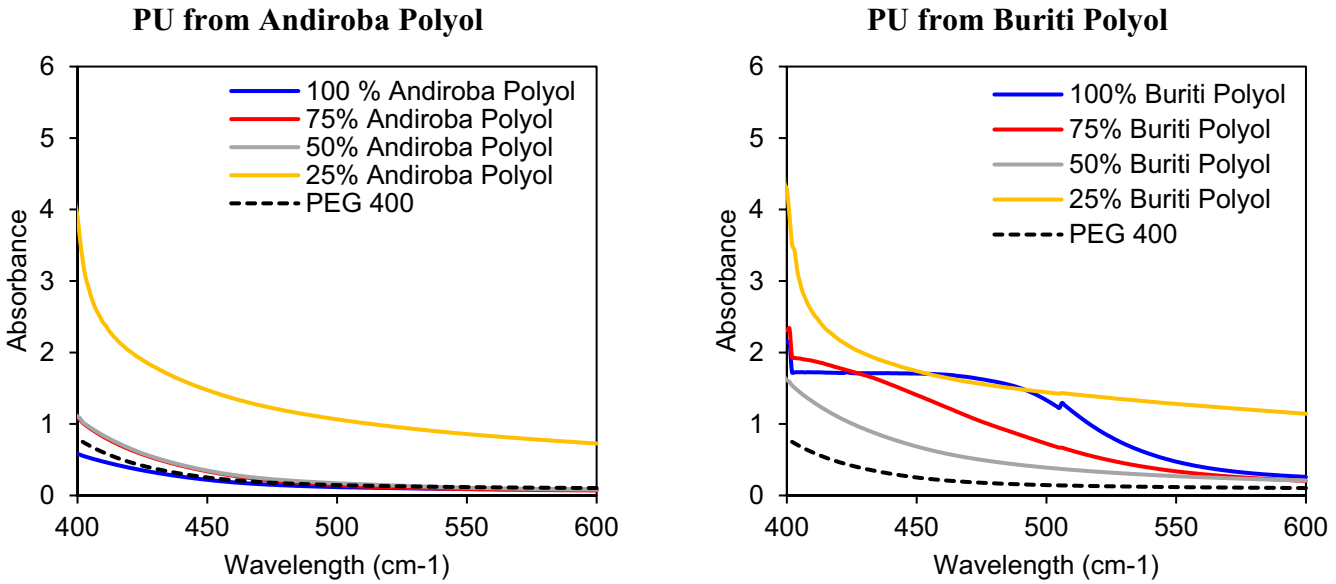


FIGURE 7 | UV spectra of the polyurethane films prepared from andiroba and buriti polyols. [Color figure can be viewed at wileyonlinelibrary.com]

producing PUs containing active compounds present in the vegetable oils from which they are derived. This observation indicates that conjugated chromophoric structures originally present in buriti oil remain detectable after polyurethane network formation, suggesting that the mild enzymatic modification preserves oil-derived bioactive molecules prior to step-growth polymerization. A similar preservation of conjugated structures has been reported in bio-based WPU systems incorporating natural chromophores, where the retention of UV-active moieties after film formation was directly associated with the chemical stability of the bioactive compound during polymerization [51]. The spectra of the PU films with 50% and 25% of buriti polyol presented no carotenes absorption, which can be related to their low concentration in these films. This behavior may be associated with a dilution effect within the polymer matrix, in which the chromophore concentration becomes insufficient to generate detectable absorbance in the analyzed thickness range. The PU films prepared with andiroba polyol presented no absorption for any composition in the considered range of wavelength, in agreement with the fact that andiroba oil has no constituents with chromophore groups. The retention of carotenoid-related absorption bands is particularly relevant when compared to recent studies in which natural phenolic or chromophoric compounds were incorporated into polyurethane systems or sustainable coating materials to impart UV-shielding properties for packaging and protective applications [37, 39, 40, 61]. In those

systems, antioxidant molecules such as tannic acid or curcumin were externally introduced into the PU matrix. In contrast, the present work employs polyols derived from enzymatically modified oils, meaning that the chromophoric compounds are associated with the renewable precursor prior to polymer formation rather than added as post-functionalizing agents. This strategy suggests that the conjugated structures may become physically entrapped within the crosslinked network during polymerization. A similar strategy involving carotenoid incorporation into polyurethane backbones has been previously reported in segmented PUs, where xanthophylls were used as chain extenders and remained detectable after polymer formation [41]. However, in that case, carotenoids were intentionally introduced as discrete monomers, whereas in the present work, the chromophoric compounds originate intrinsically from the renewable oil precursor. From a packaging perspective, the presence of retained carotenoid absorption bands indicates potential light-filtering capability in the blue-visible region, which may contribute to the oxidative stability of light-sensitive products, similarly to what has been described for multifunctional bio-based WPU films designed for active food packaging applications [37, 51].

Although carotenoid-related absorption bands were clearly detected, this spectroscopic evidence indicates retention of chromophoric compounds rather than a direct measurement of antioxidant performance. Additional antioxidant assays, such as DPPH or ABTS, performed on extracts obtained from the PU films would be required to quantitatively confirm radical-scavenging activity.

3.2.5 | Mechanical Tests

Table 6 shows the results of the mechanical tests carried out with the andiroba and buriti PU films. The PU films containing 75% or 100% of vegetable polyol were brittle and therefore could not be evaluated. The brittleness observed at high vegetable polyol contents may suggest that excessive incorporation of monoacylglycerol-derived segments can disrupt the balance between hard and soft domains, reducing cohesive energy density within the polyurethane network. Similar embrittlement behavior has been reported in bio-based PU systems when structurally irregular or bulky natural components exceed the threshold required for effective microphase organization [40, 51]. The observed differences between the compositions with 5% and 10% PUs of vegetable polyol from both oils, for the tensile strength property, were not statistically significant according to the Tukey test ($\alpha = 0.05$). However, the statistical comparison of the results for the films with 25% and 50% of vegetable polyol shows an increase in tensile strength for compositions with a higher percentage of vegetable polyol, although at a given percentage of polyol the tensile strength was independent of the polyol type. The increase in tensile strength can be attributed to the presence of the smallest chain polyol, which gives greater rigidity to the polymeric films. In addition to chain-length effects, the molecular architecture of the vegetable-based polyols must be considered. Enzymatic glycerolysis predominantly yields monoacylglycerols containing secondary hydroxyl groups and long hydrophobic fatty acid chains. Secondary hydroxyl groups exhibit lower reactivity toward isocyanates compared to primary hydroxyls, potentially leading to a less homogeneous network structure even at an NCO/OH molar ratio of 1.0. Moreover, the long aliphatic chains derived from fatty acids may act as internal flexibilizing segments, which can influence microphase separation and reduce hard-segment packing efficiency. According to the stress vs. strain curves in Figure 8, only the formulations with 50% of vegetable polyol presented a well-defined interval of elastic response. The presence of a defined elastic region at this composition may indicate an optimal balance between soft aliphatic segments derived from fatty chains and urethane hard segments formed by MDI, consistent with partial microphase organization typically observed in segmented polyurethane systems. In segmented polyurethane systems, mechanical performance is strongly governed by the degree of phase separation and hydrogen bonding between hard segments, as extensively discussed by Yilgör et al. [50]. Efficient urethane–urethane hydrogen bonding promotes hard-domain cohesion and elastomeric behavior, whereas structural irregularities reduce segmental organization and lead to plastic mechanical responses. Comparing the values of mechanical properties reported in Table 3 to those of low-density polyethylene (LDPE), a flexible

TABLE 6 | Mechanical properties of the polyurethane films and their comparison based on Tukey's test.

Composition	Tensile strength [MPa]	Elongation at rupture [%]	Yield strength [MPa]	Young's modulus [Mpa]
Andiroba 5%	2.0 ± 1.2^{d}	120.9 ± 54.5^{cd}	—	1.7 ± 0.9^{b}
Andiroba 10%	2.9 ± 0.6^{d}	207.8 ± 63.6^{bc}	—	1.6 ± 0.4^{b}
Andiroba 25%	6.0 ± 2.1^{bc}	289.3 ± 52.4^{a}	—	1.9 ± 0.4^{b}
Andiroba 50%	14.7 ± 1.6^{a}	205.9 ± 34.4^{bc}	6.6 ± 2.0	9.3 ± 1.2^{a}
Buriti 5%	3.4 ± 0.5^{cd}	163.4 ± 35.5^{cd}	—	2.2 ± 0.5^{b}
Buriti 10%	2.1 ± 0.6^{d}	153.9 ± 34.1^{cd}	—	1.5 ± 0.4^{b}
Buriti 25%	6.4 ± 1.4^{b}	262.6 ± 35.0^{ab}	—	2.4 ± 0.5^{b}
Buriti 50%	15.5 ± 2.7^{a}	213.1 ± 40.8^{bc}	6.3 ± 1.7	9.4 ± 2.0^{a}
PEG 400	1.0 ± 0.3^{d}	40.3 ± 21.7^{d}	—	—

Note: The results of Tukey's test at the 5% level of significance are expressed in terms of the superscript letters beside each data point. Means followed by a common letter are not significantly different.

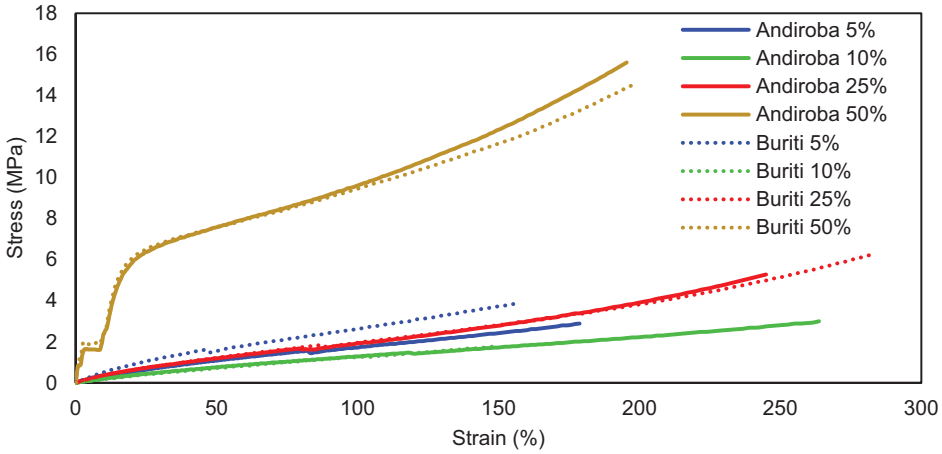


FIGURE 8 | Stress–Strain curves for the polyurethane films. [Color figure can be viewed at wileyonlinelibrary.com]

material extensively used in food packing, for example, the formulations with 50% of polyol presented: similar tensile strength (11.93 ± 0.36 MPa) [62]; a slightly inferior value of yield strength (10 MPa) [63]; and a great difference in maximum elongation (512.31% ± 29.45%) and Young's Modulus (88.44 ± 5.73 MPa) [62]. Although the tensile strength values approach those of LDPE, the significantly lower elongation at break indicates a predominantly plastic behavior rather than elastomeric performance. This suggests limited chain extensibility and restricted molecular rearrangement, possibly associated with reduced soft-segment crystallinity and constrained hard-domain cohesion. Similar trade-offs between strength and elongation have been observed in multifunctional bio-based WPU films incorporating natural aromatic compounds, where steric hindrance and hydrogen-bond disruption influenced mechanical response [39, 51]. This comparison indicates the rigidity of the PU films based on buriti and andiroba polyols is under the typical values for food packing applications. Nevertheless, from a structural standpoint, the observed behavior reflects the intrinsic architecture of oil-derived monoacylglycerol polyols, whose asymmetry and hydrophobic tails modify classical hard/soft segment distribution. Further improvements in mechanical performance could be achieved by increasing hydroxyl functionality or incorporating chain extenders with higher reactivity, thereby enhancing crosslink density and phase segregation, as suggested in segmented PU design principles [50]. However, the mechanical behavior of these PU films can be improved with the use of polyols of higher hydroxyl value than PEG 400, which should be the subject of further studies.

Although, as discussed in the next section, the films containing 100% or 75% of buriti polyol showed poor mechanical performance, the retention of carotenoid-related chromophoric features within the polymeric network represents an innovative aspect of this study. This statement is supported by literature reports describing the antioxidant behavior of these substances, which may delay weather-induced degradation or act as natural colorants in food packaging materials, particularly for light-sensitive products such as fatty foods [30, 64]. However, in the present study, antioxidant functionality in the PU films was inferred from spectroscopic and compositional analyses rather than directly quantified through radical-scavenging assays. Besides, more specifically in the case of materials aimed at the food sector, the use of natural substances is valuable for reducing health risks [30]. Thus, the obtained results open new perspectives for the use of buriti polyol in the development of films for food packing, both as a monomer for the polymeric matrix and as color and antioxidant additive. However, future development efforts in this direction should focus on the optimization of the process or additional purification steps to ensure the absence of toxic solvent residues in the product.

4 | Conclusion

Vegetable-based polyols were successfully obtained through enzymatic glycerolysis of andiroba and buriti oils, preserving their phenolic content and, in the case of buriti, carotenoid-related compounds. The enzymatic route proved effective in maintaining bioactive molecules under mild reaction conditions. These polyols were employed as reactive components in the synthesis of polyurethane films via solvent casting. FTIR analysis confirmed urethane formation, and mechanical testing demonstrated that formulations containing 50% vegetable polyol exhibited the best balance between tensile strength and elasticity. Notably, UV–Vis analysis revealed that carotenoid-related absorption bands remained detectable in buriti-based polyurethane films, indicating successful incorporation of naturally occurring bioactive compounds into the polymeric network.

Although further studies are required to quantitatively assess antioxidant performance directly on the PU films and to optimize mechanical properties, the present results demonstrate the feasibility of producing bio-based polyurethane films containing retained oil-derived bioactive constituents, as evidenced by total phenolic content in the polyols and carotenoid-related absorption in the final materials, through an enzymatic approach. This work contributes to the development of sustainable polymeric materials derived from Amazonian resources.

Author Contributions

João A. P. da Silva: conceptualization (lead), data curation (lead), formal analysis (lead), investigation (lead), methodology (lead), project administration (lead), resources (lead), supervision (lead), validation (lead), visualization (lead), writing – original draft (lead), writing

– review and editing (lead). **Amanda L. Ferreira:** investigation (equal). **Felipe F. C. Tavares:** conceptualization (equal), data curation (equal), methodology (equal), writing – review and editing (equal). **Chana M. da Silva:** data curation (equal), investigation (equal), methodology (equal), supervision (equal). **Cesar L. Petzhold:** conceptualization (equal), data curation (equal), formal analysis (equal), investigation (equal), methodology (equal), supervision (equal), writing – original draft (equal), writing – review and editing (equal). **Nilo S. M. Cardozo:** conceptualization (lead), data curation (lead), formal analysis (lead), funding acquisition (lead), investigation (lead), methodology (lead), project administration (lead), resources (lead), supervision (lead), writing – original draft (lead), writing – review and editing (lead).

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Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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