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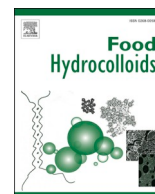
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Extraction, characterization and concentration regimes of the natural gum from *Triumfetta cordifolia* stem bark

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ABSTRACT

Gum extracted from the *Triumfetta cordifolia* plant is traditionally used for its impressive and versatile properties, but many of its intrinsic characteristics still remain poorly documented. This article presents the extraction, purification and physicochemical characterization of the gum present in the stem bark of the plant on the one hand, and the delineation of its concentration regimes in solution on the other. To this end, fresh stem bark was collected and cold extracted in distilled water, followed by ethanol precipitation. The gum was then characterized by elemental analysis, ICP-AES, FT-IR, ATG and GC-FID. Finally, the intrinsic viscosity was determined and concentration regimes delineated using both capillary viscometry and rheometry. Results revealed a monosaccharide composition rich in uronic acids, making *T. cordifolia* gum a polyelectrolyte, with a high mineral charge, and physicochemical properties similar to those of other polyelectrolyte gums. *T. cordifolia* gum solutions showed a relatively high intrinsic viscosity ($24.3 \pm 2.3 \text{ dL g}^{-1}$) and exhibited three distinct concentration regimes (namely dilute, semi-dilute and concentrated) separated by two critical concentrations ($C^* = 0.3 \text{ g L}^{-1}$ and $C^{**} = 1.4 \text{ g L}^{-1}$). These properties open up new prospects for the use of *T. cordifolia* gum as a texturizing and structuring agent for a variety of mixtures and complex preparations such as emulsions.

1. Introduction

In recent decades, interest in natural polysaccharides and their derivatives has increased at the expense of synthetic polymers, which are non-renewable and have adverse effects on the environment and on humans. Such polysaccharides can be of algal, microbial, animal or plant origin (Mohammed, Naveed, & Jost, 2021). Plant polysaccharides have particular advantages because they are abundant in nature, sustainable, renewable, nontoxic, biocompatible, biodegradable, safe and economic (Bhatia, 2016; Neves, Faria, Biswas, & Costa, 2023). In addition, some of them exhibit unique properties that justify their application and their very high demand in many areas such as food (Saha, Tyagi, Gupta, & Tyagi, 2017), cosmetics (Savary, Grisel, & Picard, 2016), pharmaceutical (Hasnain & Nayak, 2019; Khvostov, Tolstikova, Borisov, & Dushkin, 2019), oil and gas (Lapasin & Prich, 1995) or aroma industries (Jouquand, Aguni, Malhiac, & Grisel, 2008; Savary, Hucher, Bernadi,

Grisel, & Malhiac, 2010); a demand further reinforced by certain lifestyles such as veganism. Facing this huge demand, the need to identify and explore new and effective polysaccharide candidates is obvious and urgent. That is why our interest has focused on a readily available agro-resource with interesting properties, but which is often neglected and remains under-exploited: the gum from *T. cordifolia* stem bark.

T. cordifolia is a shrub belonging to Malvaceae family (APG, 2003) that is mainly found in moist parts of tropical Africa (Jiofack, 2012). Some authors reported that several species of the same genus are also found in America, Asia and Oceania (Lay, 1950; Rye, 1994). *T. cordifolia* grows fast, up to 2.5 m tall. It is cultivated either by cuttings or by seed germination with a germination rate of 80–90% (Jiofack, 2012). The fruits with hooked hairs are sometimes dispersed by animal fur, favoring its spread; interestingly, a shrub can be harvested several times during the year. The leaves and mucilage from the stem bark are edible and used in traditional dishes and drinks, either as a main ingredient or as a

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thickener (Chevassus-Agnes, Favier, & Joseph, 1976). In Cameroon, the mucilage is used to cook a sticky sauce known as “nkui”. Previous studies on this mucilage (Saidou, 2012; Woguia, Ngondi, Boudjeko, Rihouey, & Oben, 2012) have shown that it is a polysaccharide composed of neutral sugars and two uronic acids, with interesting macromolecular characteristics ($M_w = 4.07 \times 10^6$ Da; $M_n = 3.05 \times 10^6$ Da; $I_p = 1.3$; $R_g = 184.3$ nm; $[\eta] = 18.3$ dL/g).

Saidou reported that drying the bark at temperatures not exceeding 60 °C does not alter the quality of the gum, which can therefore be exploited in both dry and fresh form (Saidou et al., 2013). He also demonstrated that *T. cordifolia* gum significantly improved the dough swelling and the physical and organoleptic characteristics of doughnuts made with corn and sorghum flours (Saidou, 2012). Recently, *T. cordifolia* gum was highlighted as a promising bio-emulsifier and stabilizer for the formulation of oil-in-water emulsions, with impressive efficiencies at very low concentrations (Fanwa et al., 2023).

Despite these interesting functional properties, a number of intrinsic characteristics of the gum still remain poorly documented, and most of the mechanisms governing its potential are based on untested assumptions. Present research hypothesis is that further characterizations would provide a better understanding and a better control of *T. cordifolia* gum properties and increase its valuation potential. Thus, the aim of this study was to further investigate the intrinsic physicochemical characteristics of the gum with a view to facilitating its application in various fields, thus responding to some extent to the high demand for natural polysaccharides. To this end, a reproducible, reliable and controlled gum extraction protocol was first developed; then, physicochemical properties and sugar composition were assessed on gum powder using a series of analytical methods, and finally, concentration regimes of *T. cordifolia* gum were delineated by viscometric characterization of gum solutions.

2. Material and methods

2.1. Materials and chemicals

T. cordifolia stems were collected at Yaoundé in the Centre region of Cameroon. The plant was identified by a botanist at the Cameroon National Herbarium using voucher specimen number 41920/HNC. Ethanol was purchased from a local reagent dealer and distilled before use. Ultrapure water ($R > 18$ M Ω -cm, TOC <1 ppb, bacteria <1 CFU/mL) was obtained from a Barnstead Easypure UV compact ultrapure water system (Thermo Scientific, Illkirch, France). Sodium azide (NaN₃) and sodium chloride (HCl) were purchased from Sigma Aldrich (Saint-Quentin-Falavier, France) and Carlo Erba (Val-de-Reuil, France) respectively. All chemicals were of reagent grade and were used without further purification.

2.2. Extraction and purification of the gum from *T. cordifolia*

The choice of extraction by kneading and pressing fresh bark was inspired by the method traditionally used in Cameroon to extract this mucilage for food use. *T. cordifolia* gum was extracted from fresh stem barks using cold distilled water at ambient temperature (25–30 °C). The collection of the stems was carried out in a series of batches. Indeed, in order to take account of possible variations in harvests from different periods, and to obtain reliable conclusions from this study, it was judicious not to limit the study to a single batch. In all, 3 sets of collections were carried out over different periods (from January to April 2019, from December 2019 to January 2020 and from February to April 2022) resulting in three batches of gum. For each collection, the stems were immediately transported to the laboratory where they were subjected to a very rigorous and repeatable extraction protocol to obtain the crude extract, including among other washing, scraping and debarking followed by soaking for about 1 h. After soaking, the swollen hydrated gum is separated from the bark by manual kneading and pressing. The

operation is repeated 4 or 5 times until the gum contained in the bark is exhausted (the pressed bark is extracted again in new water until it no longer produces viscous juice). The small residues of bark detached at the same time as the gum were then removed by vacuum filtration and the gum in the filtrate was precipitated by adding ethanol, with renewal of the ethanol (the solvent removed is initially dark, as it carries with it free sugars and other extractables, and becomes increasingly clear after renewal). The extracts obtained from a series of extractions over each harvesting season as defined above were pooled together to constitute a unique batch. Further, each batch was dried, finely ground in a porcelain crucible, packaged in hermetic polypropylene flasks, and kept at 4 °C until use. Fig. 1 summarizes the steps involved in the extraction and purification of the gum.

The extraction yield (τ) was calculated from the raw gum powder according to (Eq. 1)

$$\tau (\%) = \frac{\text{mass of dry gum}}{(\text{mass of dry gum} + \text{mass of dry residual bark})} \times 100 \quad (1)$$

Gum stock solutions were prepared by first dissolving 400 ppm sodium azide (preservative) in distilled water; gum powder was then progressively added by sprinkling under mechanical stirring for 12 h to reach complete dissolution thus ensuring efficient solubilization process.

2.3. Physico-chemical analyses

2.3.1. Elemental analyses

Carbon, hydrogen, nitrogen, and sulfur contents of *T. cordifolia* gum were determined by elemental analysis using a Thermo Scientific Flash 2000 analyzer (Montigny-le-Bretonneux, France) with a quantification limit of 0.4%. Atropine (70.56% C; 8.01% H; 4.84% N; 16.59% O) and BBOT or 2,5-Bis-(5-*tert*-butyl-2-benzoxazol-2-yl) tiophene (72.53% C; 6.09% H; 6.51% N; 7.44% S) were used as standard for calibration. Measurements were carried out in duplicate on the three different batches using the method specified by the manufacturer.

2.3.2. Metal analyses

Mineral composition of each batch of *T. cordifolia* gum was determined using a Thermo Fisher Scientific ICP-AES 6300 ICP-AES analyzer (Courtabœuf, Les Ulis, France). 5 mL of 68% nitric acid were added to 0.1 g of gum sample prior to testing, with a blank test performed on the 68% nitric acid solution. Seven elements including Ca, Na, Mg, Mn, K, Zn, and Fe were identified and quantified using the ICP-AES method under the optimized conditions described by Vievard et al. (2022). For each gum batch, results were obtained as the average of three replicates.

2.3.3. Fourier Transform Infrared spectroscopy

FT-IR spectra of *T. cordifolia* gum were recorded in ATR mode in the spectral range of 700–4000 cm⁻¹ using a PerkinElmer Frontier spectrometer (Waltham, MA, USA) equipped with a ZnSe crystal with its usual blind region 2000–2200 cm⁻¹. A tiny amount of *T. cordifolia* gum powder was directly pressed onto the crystal and analyzed without further preparation. Spectrum software version 10.4.00.0190 was used for data processing.

2.3.4. Thermal analyses

The thermogravimetric (TG) and differential thermogravimetric (DTG) analyses of *T. cordifolia* gum were conducted using a Setaram Setsys 1200 instrument (Caluire-et-Cuire, France). The thermal properties were determined on approximately 10 mg of sample; experiments were carried out either under air or under nitrogen atmospheres according to the following heating program: initial isotherm (20 °C; 5 min) – heating ramp (from 20 to 900 °C; 20 °C min⁻¹) – final isotherm (900 °C; 10 min). The dry-matter of stock solutions was assessed using an isotherm at 110 °C for 60 min under air atmosphere on test samples of 80–110 mg (Enev, Sedláček, Řihák, Kalina, & Pekař, 2022). Sample mass

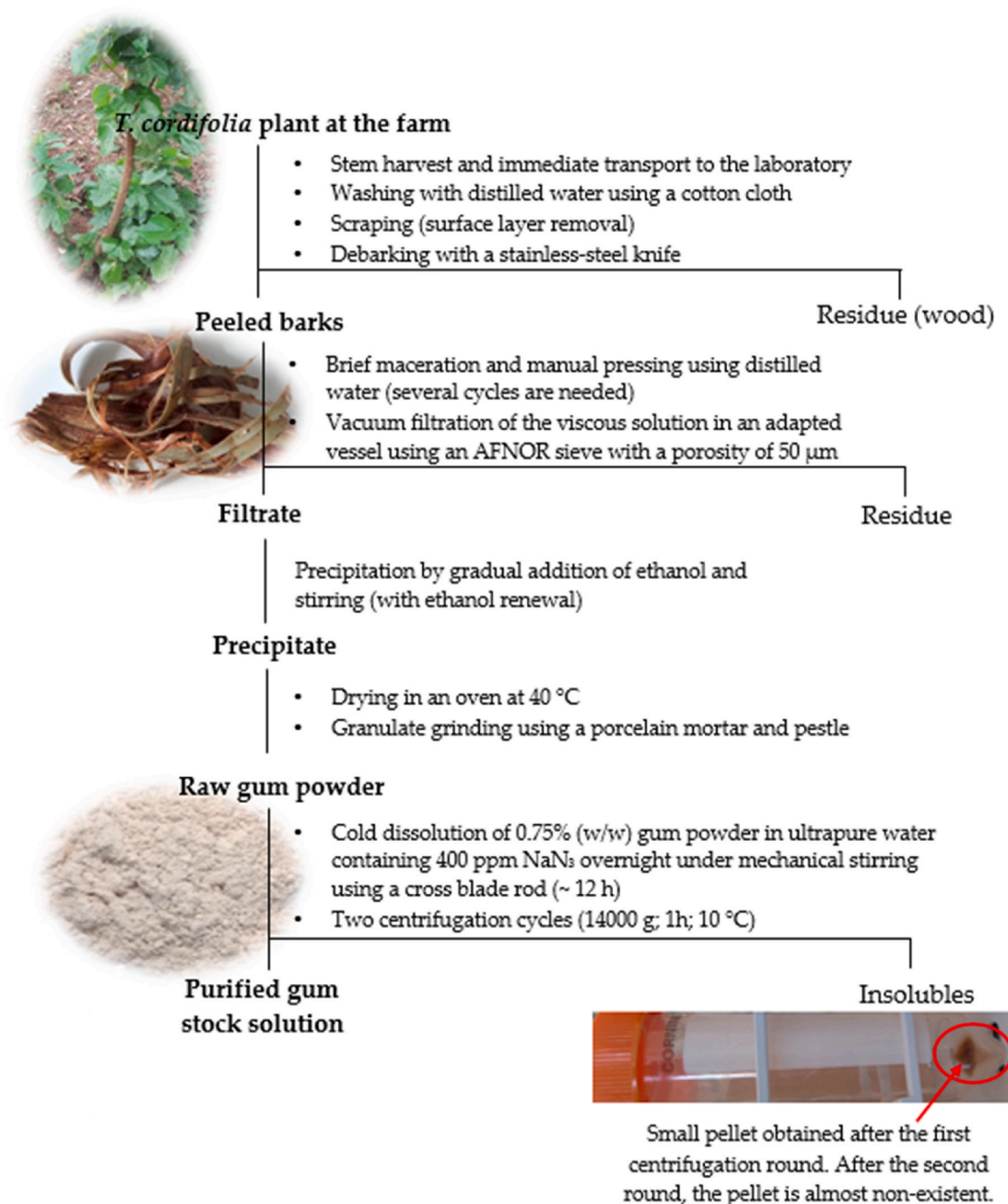


Fig. 1. Experimental procedure for the extraction and purification of *T. cordifolia* gum: from stem collection to high-purity gum.

loss was monitored as a function of temperature (and time). Experiments were performed in duplicate.

2.4. Sugar analysis

The monosaccharide composition of *T. cordifolia* gum was determined and quantified by gas chromatography coupled to a flame ionization detector (GC-FID) using a 8860 gas chromatograph system from Agilent. Sample preparation was inspired by the method used by Dardelle et al. (2010). A 2 mM inositol solution was used as an internal standard. 2 mg of gum sample were first hydrolyzed with 2 M trifluoroacetic acid (TFA) for 2 h at 110 $^{\circ}\text{C}$; TFA was then washed twice using a 50% v/v isopropanol-water mixture and samples were

lyophilized. The released monosaccharides were subjected to methanolysis by incubation in 1 M methanolic HCl at 80 $^{\circ}\text{C}$ overnight. The resulting O-methyl glycosides were converted into their trimethylsilyl derivatives by heating the samples at 110 $^{\circ}\text{C}$ for 20 min in a silylating mixture containing pyridine, 1, 1,1,3,3,3-hexamethyldisilazane and chlorotrimethylsilane. Samples were then dried and suspended in cyclohexane before being injected in a CP-Sil 5 CB column from Agilent. A five-step temperature program (isotherm for 3 min at 40 $^{\circ}\text{C}$; ramp up to 160 $^{\circ}\text{C}$ at 15 $^{\circ}\text{C min}^{-1}$; ramp up to 220 $^{\circ}\text{C}$ at 1.5 $^{\circ}\text{C min}^{-1}$; ramp up to 280 $^{\circ}\text{C}$ at 20 $^{\circ}\text{C min}^{-1}$; isotherm for 3 min at 280 $^{\circ}\text{C}$) was optimized for the separation of monosaccharides commonly found in plant cell wall such as rhamnose (Rha), galactose (Gal), arabinose (Ara), fucose (Fuc), xylose (Xyl), glucuronic acid (GlcA), mannose (Man), galacturonic acid

(GalA) and glucose (Glu).

A total of 3 gum samples obtained from different crops were analyzed and 3 replicates were performed for each sample. Results were expressed as the relative molar percentage.

2.5. Rheological measurements

2.5.1. Capillary viscometry

The viscosity of low-concentration solutions of *T. cordifolia* gum (0.008% w/w – 0.04% w/w) were measured at 20 °C using an Ubbelohde capillary viscometer AVS 470 (SCHOTT Instruments GmbH, Mainz, Germany). The relative viscosity as a function of NaCl concentration was first plotted to determine the threshold value for screening the charge effect of the polyelectrolyte. Reduced viscosity was then plotted as a function of gum concentration based on Huggins equation (Eq. 2) and extrapolation to infinite dilution led to the intrinsic viscosity $[\eta]$ of the gum (Marani, Hjelm, & Wandel, 2013).

$$H_{\text{red}} = k'[\eta]^2 C + [\eta] \quad (2)$$

where k' is the Huggins constant, $[\eta]$ the intrinsic viscosity and C the polymer concentration.

2.5.2. Rheometry

A DHR rheometer (TA instruments, New Castle, DE, USA), equipped with an aluminum cone–plate geometry (diameter 60 mm; angle 2.006; truncation gap 53 μm) and piloted by TRIOS software, was used to assess the zero-shear viscosity of *T. cordifolia* gum solutions with relatively high concentrations (0.06%–0.8%). Temperature was controlled at 20 °C using a Peltier system with an accuracy of ± 0.1 °C. Flow sweep measurements were performed from 10^{-3} s $^{-1}$ up to 100 s $^{-1}$. Prior to measurement, the instrument and geometry have been properly calibrated in accordance with the manufacturer's recommendations. Once loaded, the sample was kept at rest for 300 s (conditioning) before starting the experiment. To ensure accurate results, a minimum of two replicates were carried out for each sample.

3. Results and discussion

As the literature on *T. cordifolia* gum is exhaustive, many of the properties discussed in this work will be compared with those of other natural gums, mainly polyelectrolytes.

3.1. Extraction yield and chemical composition of *T. cordifolia* gum

The gum was extracted from *T. cordifolia* stem barks to exhaustion, according to the protocol described in section 2.2., with a yield of 51% (w/w) relative to dry mass of whole bark. This yield value was expected, since the chemical composition of *T. cordifolia* stem bark revealed that it contains $54.96 \pm 2.15\%$ (w/w) gums per mass of dry matter (Saidou, 2012), thus confirming that the process used in this study allowed the

extraction of almost all the gum contained in the bark. Table 1 compares some data of *T. cordifolia* gum extracts from different locations using different extraction conditions. The yield value obtained in this study is consistent with the yield of 50.15% obtained by Saidou (2012) but much higher than the yield of 27% obtained by Woguia et al. (2012). This may be due to the drastic thermal conditions employed in the latter case (80 °C), which could have potentially caused the hydrolysis of some polysaccharide fractions (Lu, Li, Qiao, Qiu, & Liu, 2018).

Furthermore, the extraction protocol implemented in this study resulted in virtually protein-free gum samples and did not require an additional step to remove protein as in the Saidou's study (Saidou, 2012) whose samples contained protein impurities, which had to be eliminated by a three-step purification process (hydrolysis of proteins using pepsin; precipitation using trichloroacetic acid and centrifugation). The absence of protein confirms to some extent the emulsifying and stabilizing potential of the gum reported in our previous study (Fanwa et al., 2023) and reveals that this potential is related to the intrinsic properties of the gum, without being favored by the action of proteins as for acacia gum for example.

On the other hand, the ash content ($5.4 \pm 0.9\%$) was much lower than the value of 17.1% obtained by Saidou (2012). Indeed, the extraction protocol at 50 °C on dry bark used by the author might introduce free minerals in the gum extracts. Only after a 48-h dialysis did the ash content drop to 10.5% (Saidou, 2012). Thus, it appears that the cold extraction performed in this study would have limited the solubilization of minerals so that the minerals present in the cold extract are essentially those incorporated into the polymer (structural minerals).

From the above considerations, it is clear that the extraction protocol implemented in this study is efficient and advantageous. In addition to a good yield, the cold extraction protocol on fresh bark also presented multiple interests: it takes fewer steps, preserves the gum from eventual degradation and obtains a gum with high purity by limiting the yield of undesirable extractives (proteins, free minerals). It is worth mentioning that centrifuging gum extracts at 14000 g for 1 h produced an insignificant pellet at the bottom of the conical tube (Fig. 1), thus attesting to the high purity of the gum which contains only a very negligible amount of insoluble matter.

Table 2 gives the elemental composition (C, H, N, S) of *T. cordifolia* gum, compared with other polysaccharides. The results reveal that there was no disparity between batches, and that *T. cordifolia* gum contained 37% C and 5.8% H (averages). Nitrogen and sulfur could not be detected as they were absent or present in trace amounts, thus confirming the virtual absence of sulfurous or nitrogenous entities such as proteins, as indicated above. The remaining 54% can be attributed to oxygen (O) and structural minerals of the gum. It appears that the carbon to hydrogen ratio is consistent with those of other polysaccharides such as *A. senegal*, *X. campestris*, guar and locust bean gums (Espinosa-Andrews, Báez-González, Cruz-Sosa, & Vernon-Carter, 2007; Kaity & Ghosh, 2013; Mendes et al., 2011; Pal et al., 2007 Mal, & Singh, 2007).

The presence of nitrogen in *A. senegal* is obvious since its structure

Table 1

Comparison of the influence of collection place and extraction mode of *T. cordifolia* gum on its extraction yield, protein and ash contents.

Place of collection	Conditions of extraction	Mode of extraction	Yields (%)	Protein content (%)		Ash content (%)		References
				Crude gum	Purified gum	Crude gum	Purified gum	
Yaoundé	Cold (~25 °C)/fresh bark (long strands)	Kneading	51	Traces ^a	n.d.	5.4 ± 0.9^b	n.d.	This study
Ngaoundéré	50 °C/dried bark (pieces of 1 × 05 cm)	Mechanical stirring	50.15	0.3	Traces ^a	17.1	10.5	Saidou (2012)
Yaoundé	80 °C/defatted dried bark (powder)	Infusion	27.4	n.d.	n.d.	n.d.	n.d.	Woguia et al. (2012)

n.d. = not determined.

^a Deduced from elemental analyses.

^b Deduced from TGA.

Table 2Effect of variation in harvesting period on carbon, hydrogen, nitrogen, and sulfur contents of *T. cordifolia* gum and comparison with those of other common gums.

Gum (reference)		Elemental content				
		Carbon (% w/w)	Hydrogen (% w/w)	Nitrogen (% w/w)	Sulfur (% w/w)	C/H ratio
<i>T. cordifolia</i> (this study)	Batch 1	37.62	5.69	N.D.	N.D.	6.6
	Batch 2	36.73 ± 0.17	5.81 ± 0.25	N.D.	N.D.	6.3
	Batch 3	36.58 ± 0.06	5.87 ± 0.34	N.D.	N.D.	6.2
	Average	37.0 ± 0.46	5.79 ± 0.07	N.D.	N.D.	6.4
<i>A. senegal</i> (Espinosa-Andrews et al., 2007)		42.64	6.20	0.64	n.d.	6.9
<i>X. campestris</i> (Mendes et al., 2011)		35.7	5.7	0.6	n.d.	6.3
Guar gum/ <i>Cyamopsis tetragonoloba</i> (Pal et al., 2006)		39.1	6.4	0.16	n.d.	6.1
Locust bean gum (Kaity & Ghosh, 2013)		38.9	7.2	n.d.	n.d.	5.4

n.d. = not determined.

N.D. = not detected i.e. below the quantification threshold (<0.4%).

includes an arabinogalactan-protein complex (Picton, Bataille, & Muller, 2000; Williams & Phillips, 2003). In contrast, the presence of nitrogen in xanthan gum was related to some impurities (proteins) coming from the production process (Mendes et al., 2011).

The mineral composition of *T. cordifolia* gum is given in Table 3 (Balaghi, Mohammadifar, & Zargaraan, 2010; Inegbedion, Okojie, & Egharevba, 2020; Janaki & Sashidhar, 1998; Klaic, Nunes, Moreira, Vendruscolo, & Ribeiro, 2011; Saidou, 2012; Yebeyen, Lemenih, & Feleke, 2009). For all batches, divalent metals are predominant, in particular calcium and magnesium, with averages of 23.2 mg/g and 10.6 mg/g respectively. The mineral composition of the different batches was very homogeneous, with the exception of sodium and potassium contents which were very high in batch 2 compared to the other two. Such a disparity (more pronounced for Na) suggests that the harvest site was enriched in these two metals (by spreading ash for example, a practice sometimes used by farmers) prior to harvesting batch 2. Calcium, magnesium and potassium are also the major metals identified by Saidou (2012) although the concentrations are not in the same range for calcium and potassium which are respectively 3 times lower and 2 to 6 times higher respectively than those obtained in this study. Such a discrepancy may be linked to the geographical position, the composition of the soil, the climate, the harvest season, the age of the plant ... (Al-Assaf, Phillips, & Amar, 2009; Idris, Williams, & Phillips, 1998). Indeed, the gum samples characterized in these two studies come from different geographical regions of Cameroon, namely Ngaoundéré, in the Adamaoua region in the North of the country and Yaoundé in the Centre region of the country.

Table 3Influence of harvesting period variation on the mineral content of *T. cordifolia* gum and comparison with that of other polyelectrolyte gums.

Gum		Mineral content (mg/g)							References
		Ca	Fe	K	Mg	Mn	Na	Zn	
<i>T. cordifolia</i>	Batch 1	23,44 ± 1,77	0,03 ± 0,01	1,20 ± 0,08	10,65 ± 0,83	0,56 ± 0,04	0,08 ± 0,02	0,06 ± 0,01	This study
	Batch 2	21,61 ± 1,70	0,03 ± 0,01	3,39 ± 0,17	10,24 ± 0,84	0,53 ± 0,03	4,79 ± 0,17	0,05 ± 0,01	
	Batch 3	24,59 ± 1,42	0,10 ± 0,01	1,64 ± 0,02	10,75 ± 0,57	0,43 ± 0,01	0,05 ± 0,01	0,07 ± 0,01	
	Average	23.21 ± 1.23	0.05 ± 0.03	2.08 ± 0.95	10.55 ± 0.22	0.51 ± 0.06	1.64 ± 2.23	0.06 ± 0.01	
<i>T. cordifolia</i>		8.67 ± 0.12	0.49 ± 0.14	7.46 ± 1.01	7.66 ± 0.33	0.02 ± 0.0003	0.21 ± 0.02	0.14 ± 0.006	Saidou (2012)
Karaya		11.62 ± 1.94	0.04 ± 0.004	6.35 ± 1.32	0.13 ± 0.04	0.14 ± 0.006	2.06 ± 0.08	0.009 ± 0.003	Janaki and Sashidhar (1998)
Xanthan ^a		<0.11–10.9		1.35–54.8	0.22–0.89		28.7–50.5		Klaic et al. (2011)
Acacia		7	0.041	9.5	2.01	0.0482	0.14	0.003	Inegbedion et al. (2020)
Tragacanth ^b		4.84–9.76	0.02–0.08	0.76–2.66	0.02–0.05	N.D.	0.03–0.17	N.D.	Yebeyen et al. (2009)
								(0.14–8.10) × 10 ⁻²	Balaghi et al. (2010)

N.D. = not detected.

^a 3 samples studied.^b 6 samples studied.

Compared to other polyelectrolyte gums, *T. cordifolia* gum contains more minerals. These minerals, which are incorporated in the gum as cations, would be attached to the polymer mainly by the charged glucuronic groups (–COO–) present in large quantities, as revealed by the monosaccharide profile in Table 4.

3.2. Monosaccharide composition

Results of the sugar analysis revealed that *T. cordifolia* gum consisted mainly of two neutral sugars (Rha and Gal) and two uronic acids (GalA and GlcA) accounting for around 60% and 39% of gum samples respectively. 1% of gum extracts was shared between Glc, Xyl and Man. The order of predominance was as follows: Rha > Gal ≈ GalA > GlcA > Glc > Xyl > Man (Table 4). The high uronic acid content reveals that the gum carries ionizable groups (–COOH/–COO[–]) making it an anionic polyelectrolyte. It is worth mentioning that arabinose was detected at trace levels in two different sample replicates, while trace amounts of fucose were detected in one sample replicate.

As shown in Table 4, the monosaccharide profile obtained in this study is very close to the composition reported by Woguia et al. (2012) for another sample of *T. cordifolia* gum, showing as before the predominance of Rha (41%), followed by Gal (24%), GalA (17.5%) and GlcA (10%); the remainder being shared between Glc, Man, Ara and Xyl.

It is difficult to compare these results to data reported by Saidou (2012) since he did not characterize the whole gum, but rather separate extracts obtained from 5 successive extractions of the gum from *T. cordifolia* stem bark. However, looking at the composition of these

Table 4
Comparison of the monosaccharide composition of *T. cordifolia* gum from different sources.

Monosaccharide	Rha	Gal	GalA	GlcA	Glc	Xyl	Man	Ara	Reference
Mol %	35.5 ± 0.4	24.9 ± 0.7	25.3 ± 0.3	13.4 ± 0.7	0.6 ± 0.1	0.26 ± 0.01	0.15 ± 0.04	n.d.	This study ^a
	41	24	17.5	10	3	1	2	1.5	Woguia et al. (2012)

^a Average of 3 batches.

extracts, it appears that the composition of Saidou's sample is close to that analyzed in this study and that reported by woguia et al. (2012) from a qualitative point of view, but differs significantly from a quantitative point of view since all his extracts showed a predominance of glucose (which is very low in the other samples), followed by GalA and GlcA respectively. The discrepancy may result from the difference in the place of harvest for example, since Woguia collected the gum in the same city as this study (Yaoundé, Centre region of Cameroon) while Saidou collected it in Ngaoundéré (Adamaoua region, North-Cameroon). Indeed, plant materials are often subject to a certain variability due to the influence of several factors such as the season and the place of harvest, the soil composition ...

Overall, all these studies agree on the high uronic acid content of *T. cordifolia* gum, particularly galacturonic acid which is reminiscent of pectins, but the relatively high glucuronic acid content rules out this hypothesis, as pectins generally contain little or no glucuronic acid (Belkheiri et al., 2021; Burana-osot, Soonthornchareonnon, Chaidedgumjorn, & Hosoyama, 2010; Lara-Espinoza et al., 2021; Marcon, Vriesmann, Wosiacki, Beleski-Carneiro, & Petkowicz, 2005; Méndez et al., 2022; Martínez-Abad, Martínez-Sanz, López-Rubio, & Fabra, 2022; Van Audenhove et al., 2021; Vidal, Williams, O'Neill, & Pellerin, 2001; Zhang, Waterhouse, Cui, Sun-Waterhouse, & Wu, 2023). *T. cordifolia* gum can therefore be considered as a conventional polyelectrolyte gum.

On the other hand, the low dispersion of values around the mean of the three gum batches of this study (which is characterized by the standard deviation as shown in Table 4), reveals very good reproducibility, suggesting that variation in harvesting period had no impact on gum composition.

3.3. Physico-chemical and thermal properties of *T. cordifolia* gum

FT-IR spectroscopy was used to highlight the main functional groups and chemical bonds of *T. cordifolia* gum. The spectra of different batches are superimposed, as shown in Figure A1 (Appendix A). Peak assignments are shown in Table 5 (Balan et al., 2019; Espinosa-Andrews, Sandoval-Castilla, Vázquez-Torres, Vernon-Carter, & Lobato-Calleros, 2010; Kora & Arunachalam, 2012; Liu et al., 2021; Moravej, Alavi, Azin, & Salmanian, 2020; Singh, Sharma, & Pal, 2011; Zhu & Zheng, 2021).

The broad band at 3339 cm⁻¹, characteristic of the hydroxyl (O-H)

stretching vibration involved in intermolecular hydrogen bonding, is frequently observed in polymeric structures. There are three bands characteristic of acetyl group vibrations including 1724 cm⁻¹ (carbonyl C=O stretching vibration), 1246 cm⁻¹ (C-O stretching of acetyl) and 1374 cm⁻¹ (C-H bending in -O(C=O)CH₃) which suggest the presence of acetylated moieties in the gum. This agrees with the results of NMR analyses reported by Saidou (2012) showing peaks of acetyl groups. Similar vibrational bands are found in the spectra of karaya and tragacanth which also include acetylated groups in their structures (Hasnain & Nayak, 2021).

Peaks at 1603 and 1417 cm⁻¹, attributable to carboxylic groups (COO⁻), indicate the presence of uronic acids (Zhang, Du, Xu, & Zhang, 2009) which is consistent with the monosaccharide profiles of the gum reported by Saidou (2012) and Woguia et al. (2012) including high amounts of glucuronic and galacturonic acids. These peaks are also found in the spectra of xanthan, acacia, karaya and tragacanth which are all anionic polyelectrolyte gums. The spectra of galactomannans do not show such bands, but rather a characteristic peak of galactose and mannose ring stretching that appears at 1647 and 1648 cm⁻¹ for guar and locust bean gum respectively. Finally, the region between 1200 and 900 cm⁻¹ constitutes the fingerprint, including the characteristic bands of pyranose and glycosidic linkages that are common to all polysaccharides (Table 5).

Data from the ATG and DTG thermograms of *T. cordifolia* gum are shown in Table 6a (under nitrogen atmosphere) and 6b (under air). The thermal degradation of *T. cordifolia* gum powder shows 3 wt losses under inert nitrogen atmosphere (Table 6a), and 5 wt losses under oxidizing air atmosphere (Table 6b). In both cases, the thermal behavior is similar up to around 400 °C. The initial stage with maximum around 105 °C corresponds to the desorption of water molecules (Akbar, Iqbal, Massey, & Masih, 2012) with a moisture content of 15%. The second stage, starting at around 215 °C with a maximum at 312 °C under nitrogen and 299 °C under air atmosphere, displays 64–68% weight loss which corresponds to the decomposition of the *T. cordifolia* gum backbone. This stage includes the loss of combined or structural water, the depolymerization of the gum through chain scission and cleavage of glycosidic bonds, as well as the release of carboxylic groups from uronic acids (Soares, Lima, Oliveira, Pires, & Soldi, 2005; Zohuriaan & Shokrolahi, 2004). Under nitrogen, this occurs as a single broad peak on the DTG curve while under air atmosphere, some of these phenomena seem to stand out, with

Table 5
Assignments of FT-IR adsorption frequencies of *T. cordifolia* gum in comparison with those of some natural gums.

Gum	Assignments							References
	$\nu(\text{O-H})$ s	$\nu(\text{C-H})$ s	$\nu(\text{C=O})$ s/ b	COO ⁻ assym/sym (uronic acids)	Ring-s (Gal and Man)	$\nu(\text{C-H})$ b	C-O-C (pyranose and glycosidic linkage)	
<i>T. cordifolia</i>	3340	2940	1724, 1246	1604, 1418		1374	1149, 1039	This study
Xanthan	3421	2933	1735, 1252	1611, 1404			1160, 1076	Moravej et al. (2020)
Karaya	3444	2928	1728	1624, 1416			1034	Zhu and Zheng (2021)
	3442	2935	1741, 1256	1620, 1429		1379	1153, 1041	Singh et al. (2011)
Acacia	3330	2925	/	1600, 1420			1150, 1020	Espinosa-Andrews et al. (2010)
Guar	3431	2924	/	/	1647	1381	~ 1150, 1024	Balan et al. (2019)
Locust bean	3352	2940	/	/	1648	1379	1140, 1076	Liu et al. (2021)
Tragacanth	3435	2926	1740, 1258	1626, 1429		1379	1153, 1076, 1040	Kora and Arunachalam (2012)

Table 6

Effect of harvesting period variation on the thermal behavior of *T. cordifolia* gum and comparison of TGA/DTG decomposition stages with those of some natural gums in nitrogen (a) and air (b) atmospheres.

(a)														
Gum		T range (°C)	1st peak		2nd peak			3rd peak		Residue (%)	References			
			Δm (%)	T _{max} (°C)	Δm (%)	IDT	T _{max} (°C)	Δm (%)	T _{max} (°C)					
<i>T. cordifolia</i>	Batch 1	20–900	14.5 ± 0.2	107.7 ± 0.2	66.0 ± 2.1	214.7 ± 0.5	313.3 ± 0.7	7.5 ± 0.3	711.9 ± 2.7	12.0 ± 2.1	This study			
	Batch 2		14.5	103.3	63.6	214.7	310.3	4.3	689.0			17.6		
	Batch 3		16.0 ± 0.3	105.1 ± 1.7	63.5 ± 0.9	216.6 ± 5.1	311.8 ± 0.8	6.8 ± 1.9	704.9 ± 7.1			13.7 ± 3.0		
	Average		15.0 ± 0.7	105.4 ± 1.8	64.4 ± 1.2	215.3 ± 0.9	311.8 ± 1.3	6.2 ± 1.4	702.0 ± 9.6			14.4 ± 2.3		
			xanthan	−900	~ 10	~ 75	~ 56	232	290			~ 10	~ 770	24
Karaya	30–900	13	55	52	160	279	30	463/655	5	Patra et al. (2015)				
Guar	30–800	9.94	30–252	58.26	252	252–346	23.0	346–800	8.79	Thombare et al. (2018)				
<i>A. Senegal</i>	30–900	19	84	~ 62	230	313	~ 6	~ 700–750	12.78	Sharma et al. (2021)				
Locust bean	25–900	12.6	~ 75	54.1	200	291	24.2	596/611	9.1	Kaur et al. (2020)				
Tragacanth	−800	18.3	69.4	54.0	242.9	308.0	24.3	442.5	1.5	Gupta et al. (2013)				
(b)														
Gums		T range (°C)	1st peak		2nd peak			3rd and 4th peaks		5th peak		Residue (%)	References	
			Δm (%)	T _{max} (°C)	Δm (%)	IDT (°C)	T _{max} (°C)	Δm (%)	T _{max} (°C)	Δm (%)	T _{max} (°C)			
<i>T. cordifolia</i>	Batch 1	20–900	13.6 ± 0.6	110.0 ± 0.6	70.0 ± 1.9	212.8 ± 1.5	301.4 ± 0.3	9.5 ± 1.4	454.8 ± 7.2/481.6 ± 1.4	2.4 ± 0.2	678.6 ± 2.4	4.6 ± 1.6	This study	
	Batch 2		15.4 ± 0.2	103.4 ± 0.2	69.0 ± 1.9	216.8 ± 0.4	295.2 ± 0.9	7.9 ± 1.9	425.5 ± 1.1/461.5 ± 2.1	2.7 ± 0.2	669.4 ± 3.7			5.0 ± 0.4
	Batch 3		14.9 ± 0.4	102.5 ± 0.7	65.3 ± 3.1	212.3 ± 3.0	299.7 ± 0.6	10.7 ± 3.4	445.5 ± 2.9/481.5 ± 7.4	2.4 ± 0.1	677.6 ± 2.2			6.7 ± 1.1
	Average		14.7 ± 0.8	105.3 ± 3.4	68.1 ± 2.0	213.9 ± 2.0	298.8 ± 2.6	9.4 ± 1.2	441.9 ± 12.2/474.9 ± 9.5	2.5 ± 0.1	675.2 ± 4.1			5.4 ± 0.9
			xanthan	40–800	~ 15	~ 92	~ 40	~ 210	288	32.3	~ 450/−525			~ 11
Karaya	30–700	~ 18	58	53	170	275	25	498/560	~ 1	632	~ 3	Patra et al. (2015)		
Guar ^a	25–900	14.9	69	72.6	250	317	9.6	503	/	/	2.9	Cunha et al. (2007)		
Acacia	−800	~ 14	~ 51	~ 52	~ 150	251	~ 22	415	~ 4	611	~ 8	Kasperski (2013)		
Locust bean	−600	~ 10	~ 60	72	~ 250	317		~ 500	/	/		Simões et al. (2014)		
Tragacanth	−700	10.75	~ 80	54.5	227	288	24	422/~ 510	~ 2	~ 675	4.79	Singh and Sharma (2014)		

^a The sample GG2 purified with successive dissolution and precipitation was considered.

the appearance of shoulders on the DTG curve, as shown in Figure A2b (Appendix A). After backbone fragmentation, a final stage occurs under nitrogen atmosphere at around 702 °C, with 6% weight loss corresponding to further degradation of the gum fragments which produce at the end around 14% of a black residue consisting of pyrolytic carbon and minerals. The two intermediate peaks observed in air (3rd and 4th peaks at 442 and 475 °C with 9% weight loss), but absent in a nitrogen atmosphere, correspond to oxidation phenomena such as combustion which led to gaseous products and residual carbonaceous material such as graphitic carbon (Singh et al., 2011). Finally, the last stage occurring under air atmosphere at around 600 °C (maximum around 675 °C) with a mass loss of 2.5% corresponds to the burning off of the graphitic carbon. As an illustration, ATG and DTG of graphene display a single graphitic burn stage occurring in the same range between 550 °C and 750 °C (Chabot, Kim, Sloper, Tzoganakis, & Yu, 2013). The final stage of *T. cordifolia* gum under air atmosphere produced around 5% light-grey ash residue consisting of minerals.

Table 6a (Gupta et al., 2013; Kaur, Ghildiyal, & Soni, 2020; Kumar, Srivastava, & Behari, 2007; Patra, Vojtová, & Martinová, 2015; Sharma, Bhushette, & Annapure, 2021; Thombare et al., 2018) and Table 6b (Cunha, Paula, & Feitosa, 2007; Kasperski, 2013; Patra et al., 2015; Qiu

et al., 2016; Simões, Maricato, Nunes, Domingues, & Coimbra, 2014; Singh et al., 2011; Singh & Sharma, 2014) compare the thermal behavior of *T. cordifolia* gum with those of other natural gums. These tables show that the different stages during the thermal degradation of *T. cordifolia* gum are common to many polysaccharides. The temperature at the maximum (T_{max}) of the first stage is higher for *T. cordifolia* gum, reflecting a greater propensity of the hydrophilic moieties of *T. cordifolia* gum to establish hydrogen bonds with absorbed water molecules. For all the listed gums, the major degradation (40–73%) occurs at the second stage with an initial decomposition temperature (IDT) between 150 and 254 °C. Further decomposition takes place as a third stage under nitrogen atmosphere (Table 6a). This last step is sometimes absent depending on the experimental conditions and the sensitivity of equipment. Under air atmosphere (Table 6b), the final decomposition stage of guar and locust bean gums which are galactomannans occurs with a maximum around 500 °C, while for xanthan, acacia, karaya and tragacanth which are all polyelectrolyte gums like *T. cordifolia* gum, there is an additional step with a maximum between 611 and 710 °C attributed to the combustion of carbonaceous material. The absence of this stage in the thermal profile of galactomannans may be due to the fact that their combustion would essentially generate

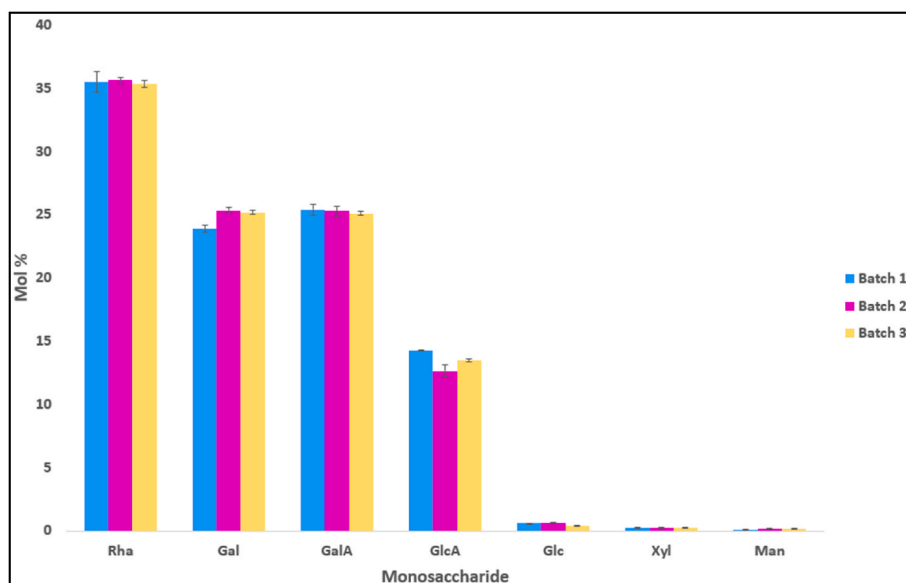


Fig. 2. Relative molar percentages of monosaccharides in *T. cordifolia* gum.

gaseous materials and ashes, with no residual carbonaceous material.

From the above considerations, it is clear that *T. cordifolia* gum is an available bio-resource which can be extracted with a good yield and high purity. It is a polyelectrolyte gum, with a high mineral filler, with good thermal stability and physicochemical properties similar to those of other natural gums. The gum extracts from three harvests were remarkably homogeneous, with no significant divergence in their characteristics. To have a better knowledge of this gum, it is necessary to characterize its properties in solution.

The data discussed in the rest of this study are those obtained from batch 1, with the exception of intrinsic viscosity, which is the average obtained over the 3 gum batches.

3.4. Rheological properties and delineation of concentration regimes of *T. cordifolia* gum solutions

The results of FT-IR analyses in section 3.2. confirmed the presence of charged carboxylic moieties from uronic acids as previously reported by Saidou (2012) and Woguia et al. (2012). Such ionizable entities make *T. cordifolia* gum a polyelectrolyte. Polyelectrolytes are polymers bearing ionizable groups which, in polar solvents, dissociate into a macro-ions or poly-ions surrounded by small ions of opposite charge called counter-ions (Barrat & Joanny, 1996). Polyelectrolyte solutions are the site of important electrostatic interactions. In particular, the repulsion between like-charged monomers causes a significant expansion of the polymer, resulting in an extended rod-like conformation (Lefebvre & Doublier, 2005; Odijk, 1982; Staño, Nova, Uhlík, & Košovan., 2020). To minimize this effect, polyelectrolyte solutions are usually studied in the presence of added electrolytes acting as counter-ions. The addition of a monovalent salt induces a screening of the charge effects of the polyelectrolyte, which becomes less extended and adopts its true conformation, similar to that of a neutral polymer. For these reasons, solutions of *T. cordifolia* gum were characterized in the presence of NaCl, in order to study the gum in its true conformation, free from tensions due to the polyelectrolyte effect. The first step consisted in determining the salt concentration required to ensure a total screening of the charge effects of the polyelectrolyte gum. By plotting the relative viscosity of a *T. cordifolia* gum solution as a function of NaCl concentration, 0.2 M was defined as the optimum screening concentration for subsequent measurements (curve not shown).

Due to its polymeric nature, the rheological properties of *T. cordifolia* gum solutions depend very much on concentration. Therefore, both low-

concentration (0.008% w/w – 0.04% w/w) and high-concentration solutions (0.06%–0.8% w/w) were distinctly studied.

For low-concentration solutions, the reduced viscosity was plotted as a function of gum concentration according to the Huggins equation (Eq. 2). By extrapolation to zero concentration or infinite dilution, the ordinate intercept gave the intrinsic viscosity ($[\eta]$), while the slope was used to calculate the Huggins coefficient k' (Figure A3, appendix A), which reflects the quality of the solvent and predicts polymer conformation. For *T. cordifolia* gum, these values were found to be 24.3 ± 2.3 dL g⁻¹ and 0.4 ± 0.1 respectively. The intrinsic viscosity is consistent with the value of 18.3 dL g⁻¹ obtained by Saidou (2012) at 30 °C using size exclusion chromatography in 0.1 M NaNO₃ electrolyte, since viscosity is supposed to decrease when temperature is reduced. Mark-Houwink-Sakurada equation establishes a proportionality relationship between intrinsic viscosity and molecular weight of polymers. Based on this relationship, Morris (1990) reported that random coil

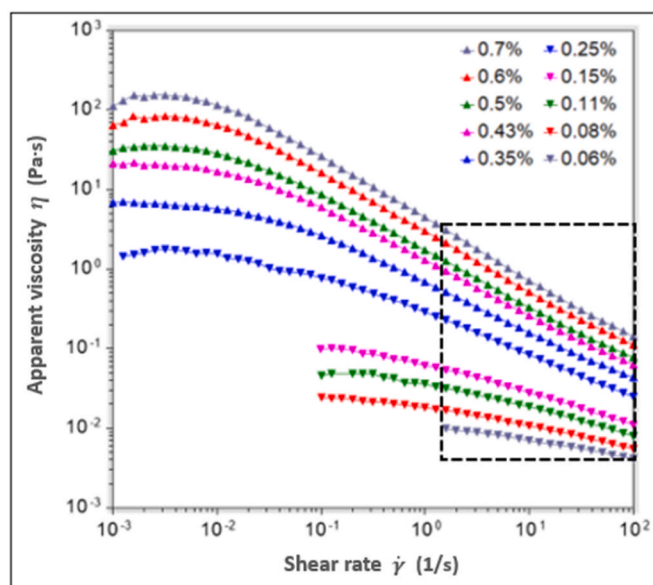


Fig. 3. Apparent viscosity curves of *T. cordifolia* gum solutions at different concentrations: solutions are shear-thinning and the power law index decreases with gum concentration.

polysaccharides with high molecular weight have intrinsic viscosities in the range 5–25 dL g⁻¹. This is consistent with the high molecular weight value of 4.07×10^6 Da reported by Saidou (2012) for *T. cordifolia* gum. The Huggins coefficient of 0.4 ± 0.1 reveals good solvent conditions (polymer-solvent interactions are favored over polymer-polymer self-interactions), with a flexible and extended conformation. Indeed, flexible polymer chains have k' values between 0.2 and 0.8, and exhibit good solvation when this value is close to 0.3 and more generally when it is between 0.25 and 0.5 (Marani et al., 2013; Pamies et al., 2008; Hernández Cifre, del Carmen López Martínez, & García de la Torre, 2008).

For high-concentration solutions, the viscosity curves of *T. cordifolia* gum solutions were plotted as a function of gum concentration (Fig. 3). All these curves show a Newtonian plateau at the beginning followed by a decrease in viscosity as the shear rate increases. Although the shear-thinning region does not appear in totality (since the plots were limited to shear rates of 10^2 s⁻¹ as the interest was in the Newtonian viscosity), it is clear that rheo-fluidification is very limited at lower concentrations and becomes gradually pronounced as concentration increases, slopes becoming steeper as highlighted by the square on the figure.

Analyses of these rheograms using the Carreau-Yasuda model (the best fit model given by the TRIOS™ software) gave access to the rate index or power law index n which measures the degree of shear thinning of the samples ($n < 1$ for shear-thinning, $n = 1$ for Newtonian and $n > 1$ for shear-thickening fluids). The rate index decreased from 0.9 ± 0.01 for the 0.06% gum solution to 0.2 ± 0.001 for 0.7% gum solution. Such a decrease of the power law index reflects an intensification of the shear-thinning behavior as the gum concentration increased. The relative viscosity of these solutions was calculated, using as a reference, the dynamic viscosity value $\eta_0 = 1.0120$ mPa s obtained by Yan-Sheng et al. (2009) for a 0.208 mol/kg NaCl solution at 293.15 K (20 °C).

Combining the data for low-concentration solutions (characterized by capillary viscometry) and high-concentration solutions (characterized by rheometry) enabled the bi-logarithmic plot, over a wide concentration range, of specific viscosity as a function of the dimensionless coil overlap parameter $C[\eta]$ which characterizes the space-occupancy, C being proportional to the number of coils present and $[\eta]$ being proportional to the volume occupied by each coil (Goycoolea, Morris, Richardson, & Bell, 1995). Such a Utracki and Simha diagram led to the delineation of concentration regimes of *T. cordifolia* gum as shown in Fig. 4.

The log-log plot of $\eta_{sp} = f(C[\eta])$ for *T. cordifolia* gum solutions

highlights two breaks in slopes at C^* and C^{**} separating three concentration regimes appearing as straight lines with very good correlation coefficients, namely: the dilute regime ($C < C^*$) with a slope of 1.2; the semi-dilute regime ($C^* < C < C^{**}$) for which the slope was equal 3.2 and the concentrated regime ($C > C^{**}$) with a slope of 4.6. These concentration regimes are typical for polymer solutions as clearly illustrated by (Nabilah, Sitanggang, & Purnomo, 2022).

Graessley (1980) showed the importance of distinguishing between entanglements of chains and simple chain overlap, bringing into play a third critical concentration C_e , such as $C^* < C_e < C^{**}$ delimiting the semi-dilute unentangled regime and the semi-diluted entangled regime. The concentration C_e can be determined by extrapolating the two extreme straight lines of the Utracki-Simha plot linking the dilute and concentrated regimes as shown by (Launay, Cuvelier, & Martinez-Reyes, 1997; Ross-Murphy, 1994).

Then, the concentration $C^* = 0.34$ g L⁻¹ marks the transition from the dilute to the semi-dilute regime. This means that for $C < 0.34$ g L⁻¹, *T. cordifolia* chains are isolated from each other and free to move independently in all directions. Above this concentration, they begin to interact with each other. The break in slope marks the transition to the semi-dilute regime, in which the chains overlap more as the concentration increases, until they begin to entangle at $C_e = 0.61$ g L⁻¹ which marks the transition from the semi-dilute unentangled regime to the semi-dilute entangled regime (Graessley, 1980). As the concentration increases, the polymer chains become more interpenetrated and, above $C^{**} = 1.43$ g L⁻¹, they form a network that tends towards a solid state, characteristic of the concentrated regime.

It is worth mentioning that the overlap concentration of *T. cordifolia* gum is such that $C^*[\eta] = 0.74$, suggesting a random coil conformation. In agreement with the Flory-Fox theory, Graessley showed that for random coil polymers, the critical overlap concentration C^* satisfies Eq. 4.

$$C^* = \frac{0.77}{[\eta]} \quad (4)$$

Moreover, from Fig. 4, the value of the specific viscosity at concentration C^* is such that $\eta_{sp} \approx 1$. This trend is common for both neutrals and polyelectrolytes in which overlapping generally occurs when the viscosity of the solution is roughly twice the solvent viscosity (Colby, 2010; Doderò, Vicini, Alloisio, & Castellano, 2019).

Considering the simplified extrapolation limiting unentangled and entangled regimes, it could be deduced that the behavior of *T. cordifolia* gum solutions is such as the slope changes from 1.2 before entanglement

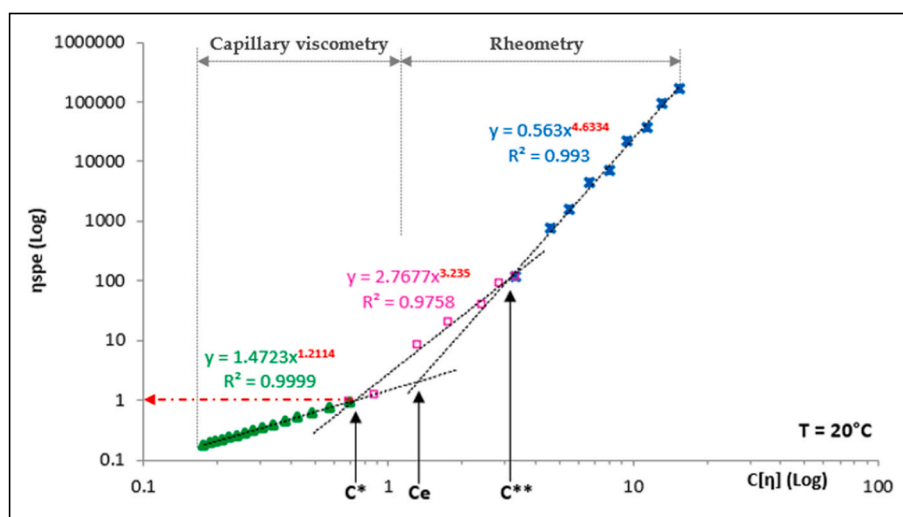


Fig. 4. Concentration regimes of *T. cordifolia* gum solutions: dilute, semi-dilute and concentrated regimes are characterized by different slopes and are delimited by the critical concentrations C^* and C^{**} respectively, while the critical concentration C_e marks the onset of entanglement.

to 4.6 after entanglement occurred, which is consistent with the prediction for neutral polymers in good solvents and θ solvents respectively. Indeed, several authors have reported that the dynamics of neutral polymers in semi-dilute unentangled solutions is such that $\eta_{sp} \sim C^{1.3}$ or $\eta_{sp} \sim C^2$ when dealing with good solvents and θ solvents respectively while their dynamics in entangled solutions is such that $\eta_{sp} \sim C^{4.7}$ or $\eta_{sp} \sim C^{3.9}$ for θ solvents and good solvents respectively (Colby, 2010; O'Connell, et al., 2023). The dynamics of polyelectrolyte solutions without salt is such that $\eta_{sp} \sim C^{0.5}$ in semi-dilute unentangled regime and $\eta_{sp} \sim C^{1.5}$ in entangled regimes (Colby, 2010). This confirms that the addition of NaCl salt in *T. cordifolia* gum solutions effectively changed the polyelectrolyte conformation to a less extended conformation close to those of neutral polymers.

The critical concentrations and slopes as well as general intrinsic characteristics of *T. cordifolia* gum were compared with those of other natural gums under similar conditions, as shown in Table 7 (Chauhan, Verma, Doley, & Ojha, 2019; Cuvelier & Launay, 1986; Doublier & Wood, 1995; Goycoolea et al., 1995; Launay et al., 1997; Mohammadifar, Musavi, Kiumarsi, & Williams, 2006; O'Connell, et al., 2023; Richardson, Willmer, & Foster, 1998; Saidou, 2012).

The intrinsic viscosity of *T. cordifolia* gum is lower than that of xanthan gum, close to that of tragacanth gum and higher than that of other natural gums. The C^* obtained in this study for *T. cordifolia* gum (0.34 g dL⁻¹) is three times higher than the value obtained by Saidou (2012) for the same gum (0.12 g dL⁻¹). Although its determination was made on the basis of a limited number of experimental points, it is logical to have such discrepancy as Saidou's measurements were carried out in water, without added salt. Indeed, since the polyelectrolyte chains adopt an extended conformation in salt-free solutions, they will reach a total space-occupancy earlier than compacted chains found in polyelectrolyte solutions with added salt.

The C^* and C^{**} values for *T. cordifolia* gum obtained in this study are of the same order of magnitude as those observed for xanthan under similar conditions, and lower than those observed for other gums. This could be explained by the fact that *T. cordifolia* and xanthan gums have relatively higher molecular weights than the others. Indeed, Knoll and Prud'homme (1987) showed that as molecular weight increases, polymer chains in solution occupy a larger volume and consequently entanglement occurs earlier, i.e. at lower concentrations. As an illustration, O'Connell et al. (2023) obtained characteristic concentrations very close to those of *T. cordifolia* and xanthan gums for a locust bean gum sample with a very high molecular weight, but discarded from the value obtained by Richardson et al. (1998) for a low-molecular weight sample of the same gum (Table 7).

In addition to the lower C^* values, the third slope characteristic of the concentrated regime is higher for *T. cordifolia* gum (4.6) and xanthan gum (4.2) than for the other gums. Such a high slope in the concentrated regime suggests a propensity for hyper-entanglements as evoked by Morris et al., 1981; Cutler, Ross-Murphy, Rees, and Price (1981) and supported by other authors (Andrade, Azero, Luciano, & Gonçalves, 1999) who obtained a slope of 4.8 for a study on galactomannans. Such a hyper-entanglement hypothesis could justify the self-pouring character as well as the Weissenberg effect which are very pronounced on concentrated solutions of *T. cordifolia* gum. Further studies are needed to verify the extent of these hyper-entanglements and the nature of the specific associations that would govern them.

The C^{**}/C^* ratio gives an idea of the extent of the semi-dilute regime. Generally, the gums listed in Table 7 have an average C^{**}/C^* ratio close to 4, showing that all these gums have approximately the same extension of the semi-dilute regime. However, this extent is sharp compared to predictions of $C_e/C^* \approx 10$, stated for neutral polymers in good solvents, assuming an extent of one decade only for the semi-dilute unentangled regime (Colby, 2010). Such a discrepancy means that, in reality, the dynamics of the semi-diluted regime would be faster than expected. As the system has already reached a total space-occupancy (at C^*), the tightening of polymer chains (similar to cooked noodles) in the medium following the increase in concentration would rapidly lead to entanglements that would evolve towards the formation of a concentrated system, hence the compression observed for the semi-diluted regime. It's worth mentioning that the introduction of the third critical concentration C_e , has led to some confusion in the literature where it is sometimes used instead of C^{**} (Czarnecka, Wojsiński, Ciach, & Sajkiewicz, 2021; O'Connell et al., 2023; Zhang, Dai, Dong, Qiu, & Ji, 2016) or sometimes substituted by C^* (Andrade et al., 1999; Ganter, Milas, Correa, & Rinaudo, 1992; Kapoor, Milas, Taravel, & Rinaudo, 1994; Morris et al., 1981).

The behavior of Arabic gum is very particular. It showed the lowest intrinsic viscosity value and the highest C^* , and neither C_e nor C^{**} could be found for this gum due to its resistance to entanglements. Indeed, the log-log plot of η_{sp} vs $C[\eta]$ for this gum provided identification of C^* at $C[\eta] \approx 1$ with a slope of 1.4 in the dilute region, but above this point, there was a steady increase in slope rather than the sharp break and subsequent linearity expected for entangled coils. The absence of entanglements was confirmed by the viscosity curve of concentrated solutions of this gum which showed a very limited shear-thinning behavior as the shear rate increased (even a very high concentrations of the order of 50%) rather than the pronounced rheo-fluidification usually observed for interpenetrated coils as the result of shear-induced

Table 7

Comparison of some intrinsic parameters and critical concentrations of *T. cordifolia* gum with those of some natural gums.

Gum (experimental conditions)	Variable							References	
	$[\eta]$ (dL·g ⁻¹)	k'	C^* (g·dL ⁻¹)	C^* [η]	C^{**} (g·dL ⁻¹)	C^{**}/C^*	Slope		M_w (Da)
<i>T. cordifolia</i> (NaCl 0.2 M; 20 °C)	24.3 ± 2.3	0.4 ± 0.1	0.03	0.74	0.14	4.3	1.2-3.2-4.6		This study
<i>T. cordifolia</i> (NaNO ₃ 0.1 M; 30 °C ^a /H ₂ O; 22 °C ^b)	18.3 ^a		0.01 ^b					4.07 × 10 ⁶	Saidou (2012)
Xanthan (NaCl 0.1 M; 25 °C)	49.3		0.03	1.4	0.11	3.9	1.25-2.1-4.2	2 × 10 ⁶	Cuvelier and Launay, 1986
Karaya (KCl, 0.2%)			0.1		0.42	4.2		0.95 × 10 ⁶	Cauhan et al., 2019
Guar (Eau 25 °C)	11.1	0.8	0.063	0.7	0.23	3.6	1.1-1.8-3.9	1.3 × 10 ⁶	Doublier and Wood, 1995
H ₂ O, 25 °C)	12.7	0.45	0.06	0.76	0.28	4.7	1.2-1.9-4.0	1.8 × 10 ⁶	Launay et al. (1997)
Arabic (NaCl 0.1 M; 20 °C)	0.19		5.26	1			1.4-/-/-	~4 × 10 ⁵	Goycoolea et al. (1995)
LB (H ₂ O, 25 °C)	13.79	0.41	0.1	1.38	0.13	4.0	1.16-2.28	8.12 × 10 ⁵	Richardson et al. (1998)
H ₂ O, 25 °C)	16.6 ± 0.3		0.03	0.52			1.0-1.7-3.0	1.5 × 10 ¹⁰	O'Connell et al. (2023)
H ₂ O, 25 °C)	14.1	0.64	0.05	0.76	0.25	4.7	1.2-1.9-4.0	2.3 × 10 ⁶	Launay et al. (1997)
Tragacanth (NaCl 0.1 M, 25 °C)	19.2	0.3						8.4 × 10 ⁵	Mohammadifar et al., 2006

disentanglements. It was concluded that the individual molecules of Arabic gum in concentrated solutions are squashed together like balls of foam rubber crammed into a jar, rather than inter-penetrating like coils (Goycoolea et al., 1995). Unlike Arabic gum, macromolecular chains in *T. cordifolia* gum solutions become more interpenetrated as the concentration increases, resulting in the shear-thinning behavior shown in Fig. 3. Indeed, shear-thinning behavior occurs when shear-induced disentanglements become predominant over the re-entanglements. The more a system is interpenetrated, the more it will exhibit an accentuated shear-thinning behavior when sufficient shear rates are applied. Moreover, the power law index $n = 0.9$ obtained for the 0.06% gum solution (close to the critical concentration C_c) is very close to the value $n = 1$ expected for Newtonian fluids. This means that *T. cordifolia* gum solutions are quasi-newtonian before the onset of entanglements and become non-Newtonian (shear-thinning) when the system is sufficiently interpenetrated.

From the above considerations, the characteristics of *T. cordifolia* gum are interesting in view of the potential properties in formulation, like its thickening capacity. The parameters discussed in this section give an idea of the conformation of this gum, but further tests are needed to confirm its true conformation. All the critical parameters obtained in this study are in line with the main theories and predictions reported in literature, attesting to their accuracy. Such a delineation of concentration regimes of *T. cordifolia* gum has never been reported before.

4. Conclusion

In this work, we were able to develop an efficient, robust and reproducible method for extracting gum from *T. cordifolia* stem bark. Apart from the strong variation in sodium and potassium levels that is most probably a result of farming practices, we were able to observe the constancy in the overall mineral and elemental composition, thermal behavior, and intrinsic viscosity of the gum. The concentration regimes were delineated for the first time, with a transition from dilute to semi-dilute at 0.34 g.L^{-1} and a transition from semi-dilute to concentrated at 1.43 g.L^{-1} . Such a delineation revealed interesting mechanisms that govern the mechanics of *T. cordifolia* gum solutions under different conditions. Compared to other natural gums, *T. cordifolia* belongs to the class of gums with high viscosity and strong entanglement capacity, which can lead to a relatively structured network at moderate

concentrations. These unique properties open the way to a wide range of applications. Such outcomes confirm our initial research hypothesis.

Further studies would include structural, linkage and conformational characterizations to elucidate the primary to tertiary structures of *T. cordifolia* gum. Rheological measurements to assess the viscoelastic properties of *T. cordifolia* gum solutions are also under consideration.

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CRediT authorship contribution statement

Michèle N. Fanwa: Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Funding acquisition, Formal analysis, Conceptualization. **Arnaud M.Y. Cheumani:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Conceptualization. **Bruno Gügi:** Validation, Methodology, Investigation, Formal analysis. **Nicolas Hucher:** Writing – review & editing, Validation, Supervision, Methodology, Conceptualization. **Catherine Malhiac:** Writing – review & editing, Validation, Supervision, Methodology, Conceptualization. **Maurice K. Ndikontar:** Writing – review & editing, Supervision, Funding acquisition. **Michel Grisel:** Writing – review & editing, Validation, Supervision, Project administration, Methodology, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Appendix A

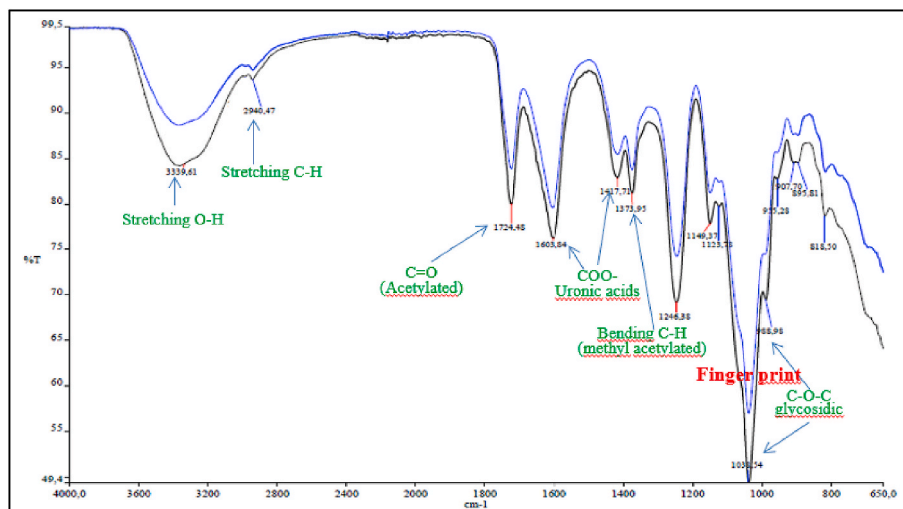


Fig. A1. FT-IR spectra of *T. cordifolia* gum (overlying two batches).

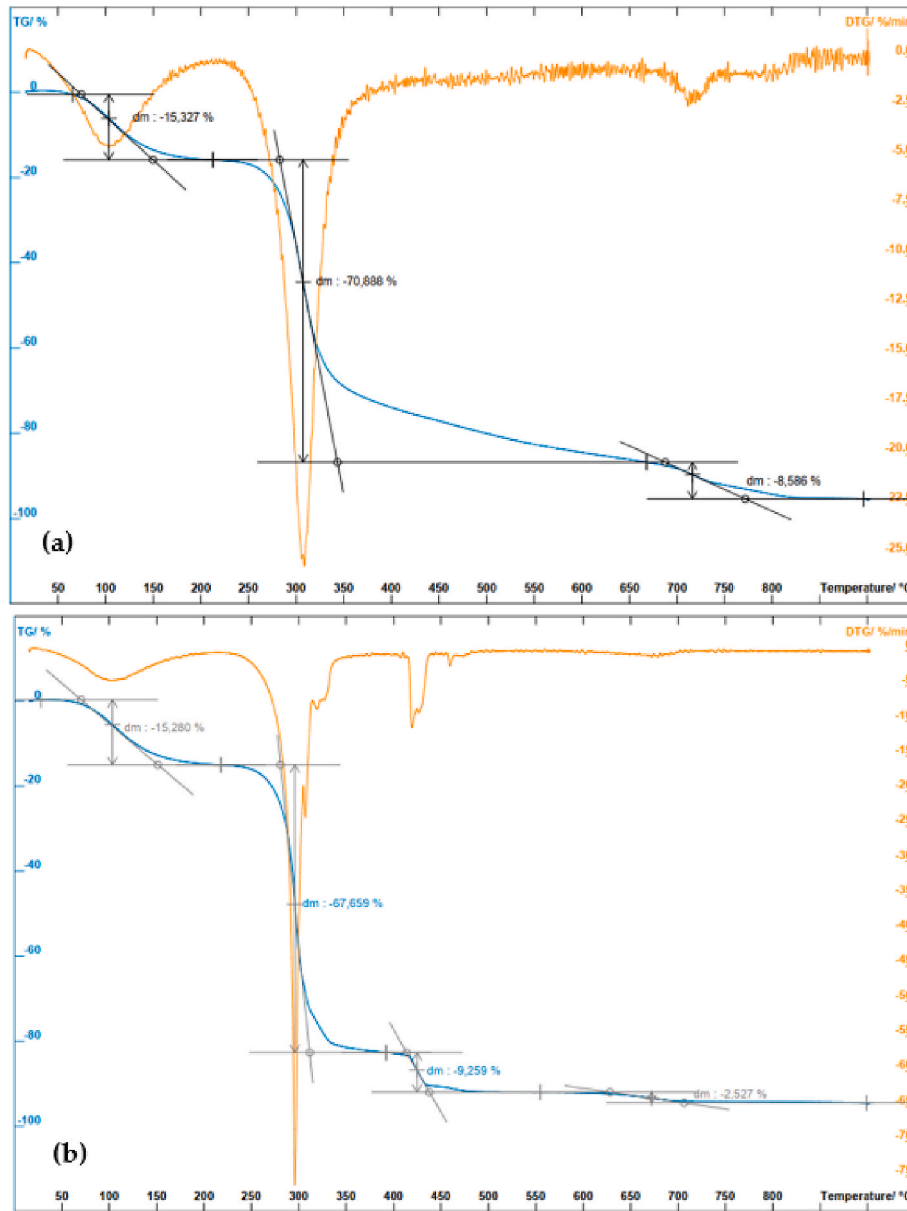


Fig. A2. Examples of TG and DTG curves of *T. cordifolia* gum under nitrogen (a) and air (b) atmospheres.

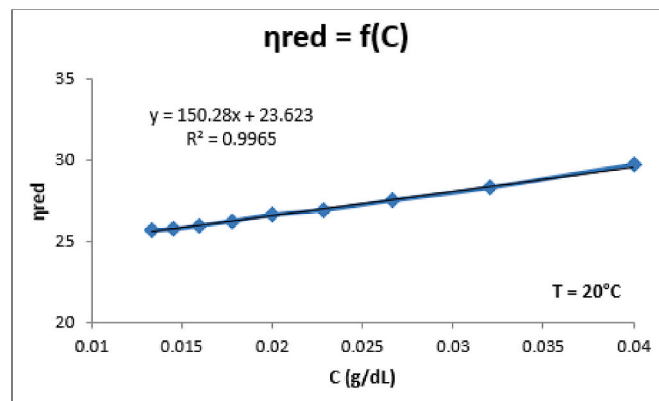


Fig. A3. Illustration of the evolution of the reduced viscosity of *T. cordifolia* gum as a function of gum concentration.

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