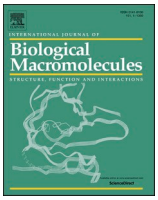
International Journal of Biological Macromolecules 298 (2025) 139890

Contents lists available at ScienceDirect

International Journal of Biological Macromolecules

journal homepage: www.elsevier.com/locate/ijbiomac



Cell wall polysaccharides from macauba pulp (*Acrocomia aculeata* L.): Fractionation and characterization of their chemical and rheological properties

Sérgio Henrique Toledo e Silva [a,b,*], Stephanie Bader-Mittermaier [b], Lidiane Bataglia Silva [c], Carlos Augusto Colombo [d], Roseli Aparecida Ferrari [e], Peter Eisner [a,b]

[a] Technical University of Munich (TUM), TUM School of Life Sciences Weihenstephan, Alte Akademie 8, 85354 Freising, Germany
[b] Fraunhofer Institute for Process Engineering and Packaging IVV, Giggenhauser Str. 35, 85354 Freising, Germany
[c] University of Campinas, Cidade Universitária Zeferino Vaz, 13083-970 Campinas, Brazil
[d] Campinas Agronomic Institute (IAC), Av. Dr. Theodureto de Almeida Camargo 1500, 13075-630 Campinas, Brazil
[e] Institute of Food Technology (ITAL), Av. Brasil 2880, 13070-178 Campinas, Brazil

ARTICLE INFO

Keywords:
Bocaiuva
By-products
Gums

ABSTRACT

Macauba fruit pulp (*Acrocomia aculeata*) is an emerging oil source. After de-oiling, the macauba pulp meal (MPM) offers a dietary fiber content of 40–50 %, which mainly comprises cell wall polysaccharides (CWP). The present work aimed to assess the potential of MPM as an innovative source of sustainable food polysaccharides. To this end, the macauba CWP were fractionated into water-soluble galactoglucomannans (21.7 %), calcium- and ester-bound pectins (3.4 %), loosely-bound xyloglucans (27.6 %), strongly-bound xylans (6.5 %), and a cellulose-rich fraction (39.3 %). The galactoglucomannans produced shear-thinning aqueous dispersions with an increase in consistency index from $3.03{\cdot}10^{-2}$ to $3.58{\cdot}10^{1}$ Pa·s^{n} by increasing the concentration from 1.0 to 5.0 %. The galactoglucomannans dispersions showed semi-dilute behavior, evidenced by relaxation times ranging from $1.24{\cdot}10^{-2}$ to 1.17 s for concentrations from 2.5 to 10.0 %. Macauba pectins and xyloglucans showed weak gel behavior, with an increase in yield stress from $3.20{\cdot}10^{-1}$ to $1.04{\cdot}10^{2}$ Pa and from $7.01{\cdot}10^{-2}$ to $1.35{\cdot}10^{2}$ Pa for dispersions at 2.5 to 10.0 %, respectively. 2.5 to 5 times higher concentration of macauba polysaccharides is needed to obtain rheological behavior similar to guar and xanthan gum. The thickening and gelling properties of macauba CWP highlight their potential as thickeners and stabilizers for the food industry.

1. Introduction

Macauba is an oil-bearing palm member of the *Arecaceae* family, native to tropical and subtropical Americas [1,2]. Macauba palms occur naturally in marginal soils of five Brazilian biomes (Amazon, Cerrado, Atlantic Forest, Pantanal, and Caatinga). Macauba palms present oil productivity superior to many oil crops like canola, peanut, soybean, and sunflower [2]. The global demand for vegetable oil is estimated to reach 250 million tons by 2030, mainly driven by the energy and food sectors [3]. The African palm (*Elaeis guineensis*) and soybean are the main sources of vegetable oils. These oil sources are frequently criticized due to their negative environmental impact and unsustainable agriculture practices [4]. The diversification of raw materials for the vegetable oil market is thus strategic for both food and non-food sectors. Macauba palms are also suitable for intercropping and agroforestry systems which reduces the risk of rain forest clearance [2,5]. For those reasons, macauba palm has recently spurred the interest of scientists and private companies as a new sustainable source of vegetable oil.

The pulp of macauba fruits has a high oil content of 40 % in dry matter on average [6–8], which is superior to flaxseed and hemp seed,

Abbreviations: MPM, Macauba pulp meal; CWP, Cell wall polysaccharides; WSF, water-soluble fraction; CSF, chelator-soluble fraction; LHF, loosely-bound hemicellulose fraction; SHF, strongly-bound hemicellulose fraction; CLF, cellulose-rich fraction; EDTA, ethylenediaminetetraacetic acid; AIR, alcohol insoluble residue; SDF, soluble dietary fiber; IDF, insoluble dietary fiber; TDF, total dietary fiber; LVR, linear viscoelastic region; f, frequency; DM, Dry matter; Tanδ, Loss factor.

* Corresponding author at: Technical University of Munich (TUM), TUM School of Life Sciences Weihenstephan, Alte Akademie 8, 85354 Freising, Germany.
E-mail addresses: sergio.toledo@ivv.fraunhofer.de (S.H. Toledo e Silva), stephanie.mittermaier@ivv.fraunhofer.de (S. Bader-Mittermaier), carlos.colombo@sp.gov.br (C.A. Colombo), roseliferrari@ital.sp.gov.br (R.A. Ferrari), peter.eisner@ivv.fraunhofer.de (P. Eisner).

https://doi.org/10.1016/j.ijbiomac.2025.139890
Received 5 October 2024; Received in revised form 11 December 2024; Accepted 13 January 2025
Available online 14 January 2025

and comparable to rapeseed and sunflower [9]. The oil from Macauba pulp is economically attractive owing to its composition similar to olive oil [10]. After oil extraction, a dietary fiber-rich pulp meal is obtained [11], which is primarily composed of cell wall polysaccharides (CWP) and could be exploited as a new source of functional ingredients [12].

CWP are a heterogeneous group of long-chain polymers comprising pectin, hemicelluloses, and cellulose [13,14]. These polysaccharides are valuable functional food ingredients owing to their thickening and gelling properties [15], contributing to improve texture, enhance stability, retain moisture, and extend the shelf life of food products [14,16]. For these reasons, CWP such as citrus pomace pectins, chemically modified cellulose derivatives, and galactomannans from seed storage tissues are extensively used as food additives [17]. Nevertheless, the food industry continuously seeks inexpensive and functional ingredients obtained from sustainable sources. In this context, the underutilized macauba pulp meal shows high potential as a new source of polysaccharides, which can only be fully exploited if the composition and rheological characteristics of its CWP are elucidated.

Prior research focused on the molecular and compositional properties of water-soluble polysaccharides from macauba pulp. According to Silva et al. [18], the water-soluble polysaccharides from macauba pulp mainly comprised a galactoglucomannan with an average molecular weight of 350 kDa, whereas Denagbe et al. [19] reported a linear acetylated glucomannan with molecular weight from 1.1 to 82 kDa and high emulsion stabilization properties. However, the potential of these polysaccharides as food ingredients remains unfulfilled, as fundamental data on their rheological properties still need to be determined. Moreover, quantifying and characterizing the other polysaccharide fractions from macauba pulp meal, like pectins, hemicelluloses, and cellulose fractions, are imperative to design strategies for its complete valorization. In addition, the ability to modify the rheology of food systems is the primary criterion for selecting polysaccharides for food applications [20]. In this context, providing a comprehensive characterization of the rheological properties of new polysaccharide sources is imperative to assess their potential for application as food ingredients.

Therefore, the primary aim of the present study was to advance the knowledge of macauba CWP and evaluate their potential as a new source of polysaccharides for food ingredients. To this end, the CWP of de-oiled macauba pulp were sequentially fractionated into a water-soluble fraction (WSF), a chelator-soluble fraction (CSF), a loosely-bound hemicellulose fraction (LHF), a strongly-bound hemicellulose fraction (SHF), and a cellulose-rich fraction (CLF). The polysaccharide composition of those fractions was assessed by neutral sugar profile and chemical composition analyses. This approach allows the identification of the major polysaccharides, thus filling the knowledge gap on the composition of macauba pulp cell wall. The potential of macauba polysaccharides to modify the rheology of food systems was investigated by determining fundamental rheological parameters. Steady shear measurements were employed to evaluate the thickening potential of the macauba polysaccharide fractions, whereas their gelling and structuring properties were investigated employing small deformation oscillatory measurements. By comparing the rheological properties of Macauba CWP with commercially available hydrocolloids, target polysaccharides for food applications can be identified. Moreover, the framework the recovery of Macauba CWP can be conceptualized based on the results from polysaccharide fractionation. Therefore, the functional characterization of macauba CWP is reported for the first time, contributing to the complete valorization and sustainable use of macauba pulp meal as a novel source of functional polysaccharides to be used as food thickeners, stabilizers, and texturizers.

2. Material and methods

2.1. Chemicals

Analytical grade acetone (purity ≥99.5 %), ethanol (purity ≥99.5 %), ethylenediaminetetraacetic acid (EDTA, purity≥98 %), *n*-hexane (purity ≥98 %), hydrochloric acid (purity 37 %), potassium hydroxide (purity ≥85 %), sodium hydroxide (purity ≥97 %), sodium azide (purity ≥99.5 %), sulfuric acid (purity ≥95 %), and monosaccharide chromatographic standards arabinose, fucose, galactose, glucose, mannose, rhamnose, and xylose were purchased from Sigma-Aldrich (Steinheim, Germany). Refined soybean oil, neutral detergent, and sodium hypochlorite were purchased in a local supermarket. Food grade guar gum and xanthan gum were purchased at Würzteufel GmbH Gerwürzmanufaktur (Empfingen, Germany).

2.2. Harvesting and pre-processing of macauba fruits

Macauba fruits were harvested from a native population in the municipality of Dourado, state of São Paulo (Brazil), located at a latitude of 22°06′00″ south, a longitude of 48°19′03″ west, at an altitude of 706 m, and a meteorological pattern of savanna climate, with an average annual temperature and precipitation of 22.4 °C and 1310 mm, respectively. Bunches of mature fruits were cut from the mother tree and allowed to fall over a plastic canvas to prevent contact with the ground. The fruits were selected according to hull integrity, washed with in neutral detergent solution, rinsed with water, immersed in 200 ppm of sodium hypochlorite for 5 min for disinfection, and then rewashed with water to remove the hypochlorite solution. The cleaned and sanitized fruits were dried in a greenhouse under local environmental conditions for two weeks at temperatures ranging from 19 to 29 °C. After drying, the fruits were mechanically separated into hulls, pulp, and endocarp (inner shell) using a pulper machine (Saturno, Sete Lagoas, Brazil) operated batch-wise (20 kg/h). The pulp was dried overnight at 45 °C and then refrigerated (14 °C) until use.

2.3. Preparation of de-oiled macauba pulp meal (MPM)

The dried macauba pulp was pressed using an endless screw press extractor ERT 60II (Scott Tech, Vinhedo, Brazil). The average press cake temperature was 45 °C. De-oiling was completed in a 2.5 L laboratory Soxhlet apparatus (500 g of press cake) with *n*-hexane for 24 h, sufficient for the completion of 15 extraction cycles and reduction of the oil content below 1 %. The MPM was de-solventized under a fume hood overnight.

2.4. Isolation of macauba cell wall polysaccharides

The cell wall material present in MPM was isolated as alcohol insoluble residue (AIR) based on the method described by Montoya-Arroyo et al. [21]. For this, the MPM was suspended in 10 volumes of boiling aqueous ethanol at a concentration of 80 % (*v*/v) and stirred at 300 rpm for 1 h. The solid fraction was separated from the alcohol phase by filtration in a Buechner funnel with a Whatman™ paper filter n° 1. This procedure was repeated five times to obtain a clear alcohol extract. The residue of the five aqueous ethanolic extractions was washed four times with 250 mL of 80 % aqueous ethanol (v/v) at 75 °C, and subsequently stirred overnight in 250 mL of pure acetone. Afterwards, the dispersed solid was filtered and de-solventized at room temperature for 24 h. The yield of AIR from MPM was determined gravimetrically from triplicate trials ($n = 3$).

2.5. Fractionation of cell wall polysaccharides

The cell wall polysaccharides from macauba AIR were fractionated according to their solubility and their binding strength to the cell wall matrix into a water-soluble fraction (WSF), a chelator-soluble fraction (CSF), a loosely-bound hemicellulose fraction (LHF), a strongly-bound hemicellulose fraction (SHF), and a cellulose-rich fraction (CLF). The fractionation procedure described in Sections 2.5.1 to 2.5.5 consisted of sequential solid-liquid extractions adapted from Schieber et al. [22] and

Li et al. [23], as illustrated in Fig. 1. Extraction was performed in a double jacket glass reactor (2 L) coupled to a water bath for isothermal conditions and a mechanical stirrer for homogenous mixing (250 rpm). The yield of the macauba cell wall fractions was determined gravimetrically from three independent fractionation trials ($n = 3$).

2.5.1. Water-soluble fraction (WSF)

100 g of AIR was suspended in 2 L of demineralized water and extracted at 40 °C with continuous stirring (250 rpm) for 60 min. The liquid phase was separated from the solid residue by centrifugation (15,000 *g*, 20 min, 25 °C), followed by filtration of the supernatant with a Buechner funnel and Whatman™ paper n° 1. The extraction procedure was repeated two times with 2 L of demineralized water at 40 °C with continuous stirring (250 rpm) for 60 min, and the solid residue was washed four times with 250 mL of demineralized water. The filtrates were pooled, concentrated at 35 °C in a rotary evaporator to a final volume of 2 L, dialyzed (Servapor® dialysis tubings 50 mm, molecular weight cut-off from 12 to 14 kDa, SERVA Electrophoresis GmbH, Heidelberg, Germany) against 20 volumes of demineralized water (changed twice a day) at 5 °C for three days, and lyophilized (Beta 1-8 LSCplus, Martin Christ GmbH) to yield the WSF.

2.5.2. Chelator-soluble fraction (CSF)

The residue from WSF was extracted three times with 1 L of 50 mmol/L NaOH and 0.5 mmol/L EDTA solution for 60 min at 30 °C under continuous stirring (250 rpm). The liquid phase was separated from the solid residue by centrifugation (15,000 *g*, 20 min, 25 °C), followed by filtration of the supernatant with a Buechner funnel and Whatman™ paper n° 1. The extraction procedure was repeated two times with 1 L of 50 mmol/L NaOH and 0.5 mmol/L EDTA solution for 60 min at 30 °C under continuous stirring (250 rpm), and the solid residue was washed four times with 250 mL of demineralized water. The filtrates were pooled and neutralized to pH 6.5 with 37 % (*v*/v) HCl, then concentrated to a final volume of 1 L, dialyzed, and freeze-dried as described in Section 2.5.1 to yield the CSF.

2.5.3. Loosely-bound hemicellulose fraction (LHF)

The residue from CSF was dispersed in 1 L of 1 mol/L KOH for 3 h at 25 °C under continuous stirring (250 rpm). The liquid phase was separated from the solid residue by centrifugation (15,000 *g*, 20 min, 25 °C), followed by filtration of the supernatant with a Buechner funnel and Whatman™ paper n° 1. The extraction procedure was repeated two times with 1 mol/L KOH for 3 h at 25 °C under continuous stirring (250 rpm), and the solid residue was washed four times with 250 mL of demineralized water. After three extraction cycles, the filtrates were pooled, neutralized, concentrated, dialyzed, and freeze-dried as described in Section 2.5.2 to produce the LHF.

2.5.4. Strongly-bound hemicellulose fraction (SHF)

To obtain the SHF, the residue from LHF was extracted three times in 4 mol/L KOH at 25 °C for 90 min under continuous stirring (250 rpm). The liquid phase was separated from the solid residue by centrifugation (15,000 *g*, 20 min, 25 °C), followed by filtration of the supernatant with a Buechner funnel and Whatman™ paper n° 1. The extraction procedure was repeated two times with 4 mol/L KOH at 25 °C for 90 min under continuous stirring (250 rpm), and the solid residue was washed four times with 250 mL of demineralized water. After three extraction cycles, the filtrates were pooled, neutralized, concentrated, dialyzed, and freeze-dried as described in Section 2.5.2 to produce the SHF.

2.5.5. Cellulose-rich fraction (CLF)

The insoluble residue from the SHF extraction was dispersed in 5 volumes of demineralized water, neutralized, dialyzed, and freeze-dried as described in Section 2.5.2 to obtain the CLF.

2.6. Composition of macauba polysaccharide fractions

The dry matter and ash content of the macauba AIR and polysaccharide fractions were determined in a thermogravimetric system (TGA 601, Leco Corporation, St. Joseph, USA) at 105 °C and 550 °C, following the official methods L 18.00-12 and L 15.00-7, respectively [24]. The protein content was determined by the Dumas combustion method [25] using a nitrogen analyzer FP 528 (Leco Corporation, St. Joseph, USA) and a conversion factor of N x 6.25. The soluble dietary fiber (SDF) and insoluble dietary fiber (IDF) contents were determined according to the enzymatic-gravimetric method [26]. All analyses were performed in triplicate ($n = 3$).

2.7. Neutral sugar composition

As polysaccharides have characteristic sugar compositions, the determination of neutral sugar profile is imperative to identify the main polysaccharides present in cell wall fractions. Macauba polysaccharide fractions were hydrolyzed by the Saeman procedure (sulfuric acid hydrolysis: 12 mol/L, 1 h, 30 °C, followed by a hydrolysis at a concentration of 1 mol/L sulfuric acid, 3 h, 100 °C) and the resulting monosaccharides (arabinose, fucose, galactose, glucose, mannose, rhamnose, xylose) were determined by high-performance anion-

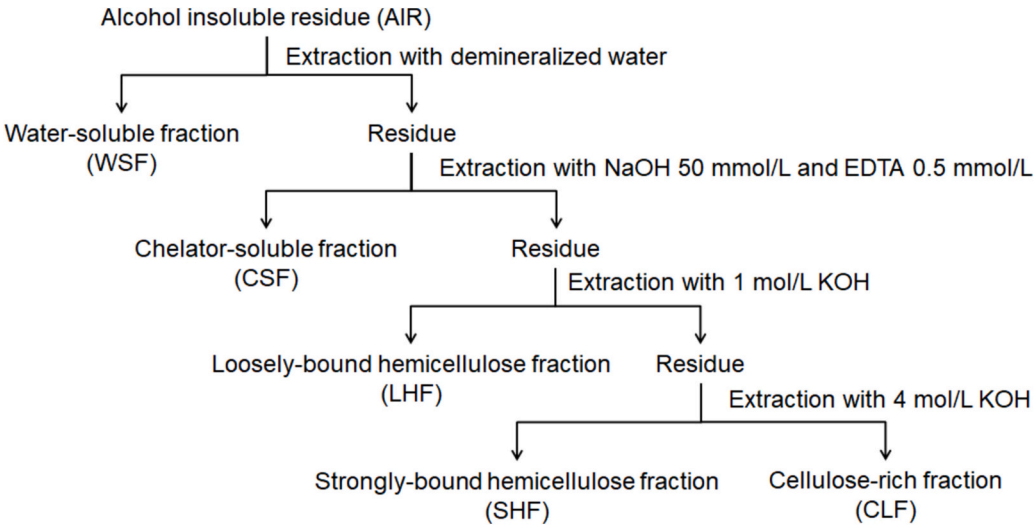


Fig. 1. Sequential fractionation of macauba cell wall polysaccharides according to their solubility and their binding strength to the cell wall matrix.

exchange chromatography with pulsed amperometric detection [27]. The total uronic acid was determined by spectrophotometric quantification [27]. The neutral sugar composition was performed as a single determination ($n = 1$).

2.8. Rheological characterization

2.8.1. Sample preparation

Freeze-dried macauba polysaccharide fractions were suspended in demineralized water containing 0.02 % sodium azide (m/v) to prevent microbial growth. Suspensions of commercial guar gum and xanthan gum at 1.0 % (m/v) were also prepared for comparison. To ensure complete hydration and solubilization, the suspensions were stirred overnight at 150 rpm and room temperature. The concentration of the suspensions was adjusted to 0.5, 1.0, 2.5, 5.0, and 10.0 % (m/v) of total dietary fiber (TDF), determined as the sum of SDF and IDF contents, to compensate for the variable chemical composition of macauba polysaccharide fractions. The concentration range for evaluation of rheological properties was based on the work from Tabarsa et al. [28].

2.8.2. Rheological measurements

All rheological measurements were performed on a stress-controlled rheometer (Physica MCR 301, Anton Paar GmbH, Graz, Austria) equipped with Rheoplus software version 3.40 (Anton Paar GmbH). A cylindric measuring system (CC27-SN24807) was used for suspensions at 0.5 and 1.0 % TDF. A 5 cm plate-plate system (PP50-SN23165) and a shear gap of 1 mm were employed for suspensions at 2.5, 5.0, and 10.0 % TDF. A minimum torque limit was fixed at 0.1 μNm to ensure the reliability of the data. All measurements were performed at 20 °C and in triplicate ($n = 3$).

Flow behavior is directly related to the thickening properties of polysaccharides, as it determines how they increase viscosity and resist flow under different shear conditions [29]. The flow behavior of WSF, CSF, LHF, and SHF was characterized through unidirectional steady shear measurements, with a concentration range from 0.5 to 5.0 % TDF, except for the CLF which exhibited a gel-like behavior, even at low concentrations. Above this range, the samples showed high consistency and gel-like behavior. Before the measurements, the samples were pre-sheared at 5 s^{-1}, followed by 1 min rest and then subjected to a shear ramp from 1 to 1000 s^{-1}. The obtained flow curves were fitted to the Newton and Ostwald – de Waele (power law) models as described in Eqs. (1) and (2), respectively.

$$\tau = \eta \cdot \gamma \tag{1}$$

$$\tau = k \cdot \gamma^{n} \tag{2}$$

where τ is the shear stress (Pa), γ is the shear rate (s^{-1}), η is the shear viscosity (Pa·s), k is the consistency index (Pa·s^n), and n is the flow behavior index (dimensionless).

Viscoelastic behavior is directly related to the ability of polysaccharides to form structured systems like gels and weak gels [30]. Oscillatory measurements were employed to assess the viscoelastic properties of all macauba polysaccharide fractions at concentrations from 2.5 to 10.0 % TDF. Below this range, most samples showed predominantly liquid behavior as determined in preliminary tests. Amplitude sweeps were employed to assess the linear viscoelastic region (LVR) of macauba polysaccharide fractions and were performed by increasing the shear strain logarithmically from 0.01 to 100 % at a constant angular frequency of 10 rad/s. From the amplitude sweep rheological spectra, the viscoelastic moduli (G′ LVR and G″ LVR) and the yield stress at the limit of LVR were calculated using Rheoplus software.

Frequency sweeps were employed to assess the time-dependent rheological behavior of macauba polysaccharide fractions in the non-destructive deformation range. The measurements were performed by decreasing the angular frequency logarithmically from 100 to 0.1 rad/s at a constant shear strain of 0.1 %, previously determined to be within the LVR. The slope of the double logarithmic modulus (G′ and G″) – frequency (f), and the relaxation times, expressed as the inverse of the frequency at which the viscoelastic moduli cross (G′ = G″), were also calculated using Rheoplus software. The loss factor (tanδ) was calculated according to Eq. (3).

$$tan\delta = \frac{G''}{G'} \tag{3}$$

2.9. Statistical evaluation of data

Results were reported as mean values ± standard deviations. The average values of 2 corresponding groups were compared using Tukey's test at a significance level of 5 % ($p \leq 0.05$). The statistical analysis of data was performed using OriginPro version 2022b software (OriginLab Corporation, Northampton, USA).

3. Results and discussion

3.1. Composition of macauba cell wall polysaccharide fractions

The yield and composition of macauba cell wall fractions are presented in Table 1, and their neutral sugar composition is shown in Table 2. The yield of AIR was 62.1 ± 0.3 %, whereas a total of 85.7 ± 1.4 % of the AIR were recovered in the different polysaccharide fractions. Similar recovery was previously reported for the fractionation of CWP from orange albedo [31], cocoa pulp [32], apples, cherries, and strawberries [33]. Therefore, the sequential fractionation protocol used in the present work exhibited similar performance for the fractionation of macauba pulp CWP as for a wide range of other cell wall matrices.

The AIR was mainly comprised of CLF, LHF, and WSF with yields of 33.7, 24.2, and 17.9 %, respectively. In all fractions, TDF was the main component, but the content of SDF and IDF differed according to the binding strength of the polysaccharides to the cell wall matrix. Higher SDF contents were obtained in fractions containing polysaccharides

Table 1
Yield and proximate composition of macauba alcohol insoluble residue (AIR) and polysaccharide fractions obtained after sequential fractionation into water-soluble fraction (WSF), chelator-soluble fraction (CSF), loosely-bound hemicellulose fraction (LHF), strongly-bound hemicellulose fraction (SHF), and cellulose-rich fraction (CLF), ($n = 3$).

Fraction	Yield	SDF	IDF	Protein	Ash
	(g/100 g AIR)	(g/100 g DM)	(g/100 g DM)	(g/100 g DM)	(g/100 g DM)
AIR	100.0	20.9 ± 1.4^{d}	59.1 ± 0.3^{b}	06.4 ± 0.1^{c}	02.9 ± 0.2^{c}
WSF	17.9 ± 0.7^{c}	90.4 ± 1.6^{a}	< 0.5*	02.8 ± 0.1^{e}	00.9 ± 0.1^{d}
CSF	03.4 ± 0.1^{e}	44.9 ± 0.6^{b}	01.2 ± 0.1^{e}	18.0 ± 0.1^{a}	13.9 ± 0.1^{a}
LHF	24.2 ± 0.3^{b}	16.8 ± 1.3^{e}	16.0 ± 0.8^{d}	15.7 ± 0.1^{b}	02.9 ± 0.1^{c}
SHF	06.5 ± 0.2^{d}	36.9 ± 0.9^{c}	52.4 ± 2.6^{c}	04.8 ± 0.1^{d}	03.3 ± 0.1^{b}
CLF	33.7 ± 0.4^{a}	06.4 ± 0.8^{f}	86.7 ± 1.6^{a}	01.4 ± 0.1^{f}	00.5 ± 0.1^{e}

Values followed by different superscript letters in the same column are significantly different ($p < 0.05$) according to Tukey's test. *Below quantification limit and was not included in the statistical analysis.

Table 2
Content of total uronic acid and neutral sugar composition of macauba alcohol insoluble residue (AIR) and polysaccharide fractions obtained after sequential fractionation into water-soluble fraction (WSF), chelator-soluble fraction (CSF), loosely-bound hemicellulose fraction (LHF), strongly-bound hemicellulose fraction (SHF), and cellulose-rich fraction (CLF) ($n = 1$).

Fraction	Total uronic acid (g/100 g DM)	Neutral sugars (g/100 g DM)						
		Arabinose	Fucose	Galactose	Glucose	Mannose	Rhamnose	Xylose
AIR	04.0	1.8	0.2	2.8	52.9	12.8	0.2	16.7
WSF	04.2	1.2	0.1	3.6	18.0	59.5	0.3	02.0
CSF	20.6	8.6	0.1	8.4	05.7	01.6	0.8	11.9
LHF	02.4	1.9	0.2	1.9	42.3	00.5	0.2	12.8
SHF	03.7	1.9	1.0	4.2	12.4	03.6	0.3	48.2
CLF	03.1	1.6	0.1	2.7	70.7	06.5	0.1	10.2

DM: dry matter.

loosely bound to the cell wall matrix, as observed for WSF (90.5 %) and CSF (44.9 %). On the other hand, the IDF content progressively increased with the increase in polysaccharide binding strength to the cell wall matrix, as observed for LHF (16.0 %), SHF (52.4 %), and CLF (86.7 %). Accordingly, the LHF and SHF were obtained after treatment with 1 M KOH and 4 M KOH, respectively, thus a progressive increase in chaotropic effect was required to extract these fractions [34]. This indicates a stronger binding strength to the cell wall matrix, primarily via hydrogen bonds and potentially ester linkages [35].

Mannose, glucose, and galactose were the most abundant monosaccharides in the WSF, with 59.5 %, 18.0 %, and 3.6 %, respectively. Therefore, the WSF mainly comprises galactoglucomannans, consistent with the results previously reported by Silva et al. [18] and Denagbe et al. [19]. Different from macauba, the polysaccharide composition of water extracts from other fruit pulps of the Aracaceae family showed high contents of high methoxylated homogalacturonans, arabinan and arabinogalactan side chains as reported for açaí [36] and buriti [37]. The CSF showed the highest content of uronic acid (20.6 %) and rhamnose (0.8 %), in addition to appreciable amounts of galactose (8.4 %) and arabinose (8.6 %). This monosaccharide profile is consistent with the composition reported for pectic polysaccharides extracted either with chelator agents or dilute alkali [23,31,38]. The small ratio of rhamnose to uronic acid ($\frac{Rha}{UA}$) of approximately 0.04, shows a little contribution of the rhamnogalacturonan domains to the pectin population [38,39]. In addition, the higher contents of arabinose and galactose in relation to rhamnose also indicate the presence of highly branched rhamnogalacturonan domains containing arabinogalactan side chains [38,39]. Therefore, the results show that the CSF is composed of a mixture of homogalacturonans from the middle lamella with a small proportion of highly branched rhamnogalacturonans bound to hemicellulose via ester linkages. Those pectin-like polysaccharides are found in low content in the macauba cell wall matrix, as evidenced by the low yield of the CSF (3.4 %).

The LHF and SHF comprised the loosely- and strongly bound hemicellulose fractions of macauba pulp, respectively. The LHF was predominantly composed of glucose (42.3 %), followed by xylose (12.8 %), demonstrating a predominance of xyloglucans with minor contributions of heteroxylans [23,36,40]. On the other hand, the main neutral sugar in SHF was xylose (48.2 %), followed by glucose (12.4 %) and low contents of galactose (4.2 %) and mannose (3.6 %), thus indicating the presence of heteroxylans, with minor contributions of xyloglucans and heteromanans [23,31,41,42]. The SHF also showed the highest content of fucose (1.0 %), which indicates the presence of fucogalactoxyloglucan [43]. A large variability was reported for the composition of hemicelluloses in fruits of Aracaceae family. In açaí pulp, the main polysaccharide was a linear xylan [44], whereas linear xylans, arabinans, calose, and mixed linkage beta-glucans were present in hemicellulose extracts from buriti pulp [37]. Similar to macauba, the hemicelluloses in the pulp of tucumã mainly comprised xylans and xyloglucans [36]. As expected, glucose was the main neutral sugar in CLF (70.7 %) owing to its high cellulose content [31,32,45].

Hitherto, previous research has mainly focused on the chemical characterization of the water-soluble galactoglucomannans from macauba pulp. In the present work, the successful fractionation and quantification of other polysaccharide fractions present in macauba pulp is reported for the first time. Their neutral sugar composition was also shown, which allowed the identification of galactoglucomannans, pectins, xyloglucans, xylans, and cellulose in the macauba cell wall matrix. The sample material of the present work was sourced from a large natural palm population. This differs from previous studies on macauba CWP, in which fruits were harvested from a limited number of palms, thus representing the composition of single genotypes. Therefore, our results showed the diverse composition of macauba CWP, potentially reflecting the complexity and heterogeneity observed in a larger palm population. Therefore, our methods can be replicated to macauba fruits sourced from different locations to gain a deeper understanding on the influence of genetic variability and climate conditions on the composition of macauba cell wall polysaccharides.

The results showed that galactoglucomannans and xyloglucans were the main extractable polysaccharides from macauba pulp cell wall material, whereas cellulose was the main component of the insoluble residue. In this sense, the present work provides the starting point for further purification of macauba cell wall polysaccharides to elucidate their molecular properties such as molecular weight distribution, linkage pattern analysis, substitution with functional groups, and degree of branching. As such, the results herein reported pave the way in elucidating the composition of macauba pulp polysaccharides, which coupled with the characterization of their rheological properties, will assist in designing strategies for the complete valorization and application of macauba pulp meal in the food industry.

3.2. Flow behavior of macauba cell wall polysaccharide fractions

Steady shear measurements with aqueous suspensions of WSF, CSF, LHF, and SHF were conducted to assess their potential as thickening agents for food formulations. The results are presented in Fig. 2 and the rheological parameters are provided in Table 3.

The WSF, CSF, and LHF showed Newtonian behavior for suspensions at 0.5 %, whereas shear-thinning behavior was observed for concentrations above 1.0 %, as indicated by the flow behavior index (n) below 1 and a good fit to the Ostwald – de Waele model. Polysaccharides like galactoglucomannans, homogalacturonans and xyloglucans assume random coil conformation in solution and their flow behavior is concentration and shear-rate dependent [46]. At sufficiently low concentrations, the polysaccharide molecules occupy a discrete hydrodynamic domain and do not interact with each other, characterizing a dilute regime with Newtonian behavior.

As the concentration increases and reaches a critical value, the individual polysaccharide chains overlap each other, and form entangled networks. In this semi-dilute regime, the entangled polysaccharides tend to impede flow, thus deviating from Newtonian behavior [47]. With the increase in shear rate, the polysaccharide chains orient themselves in the

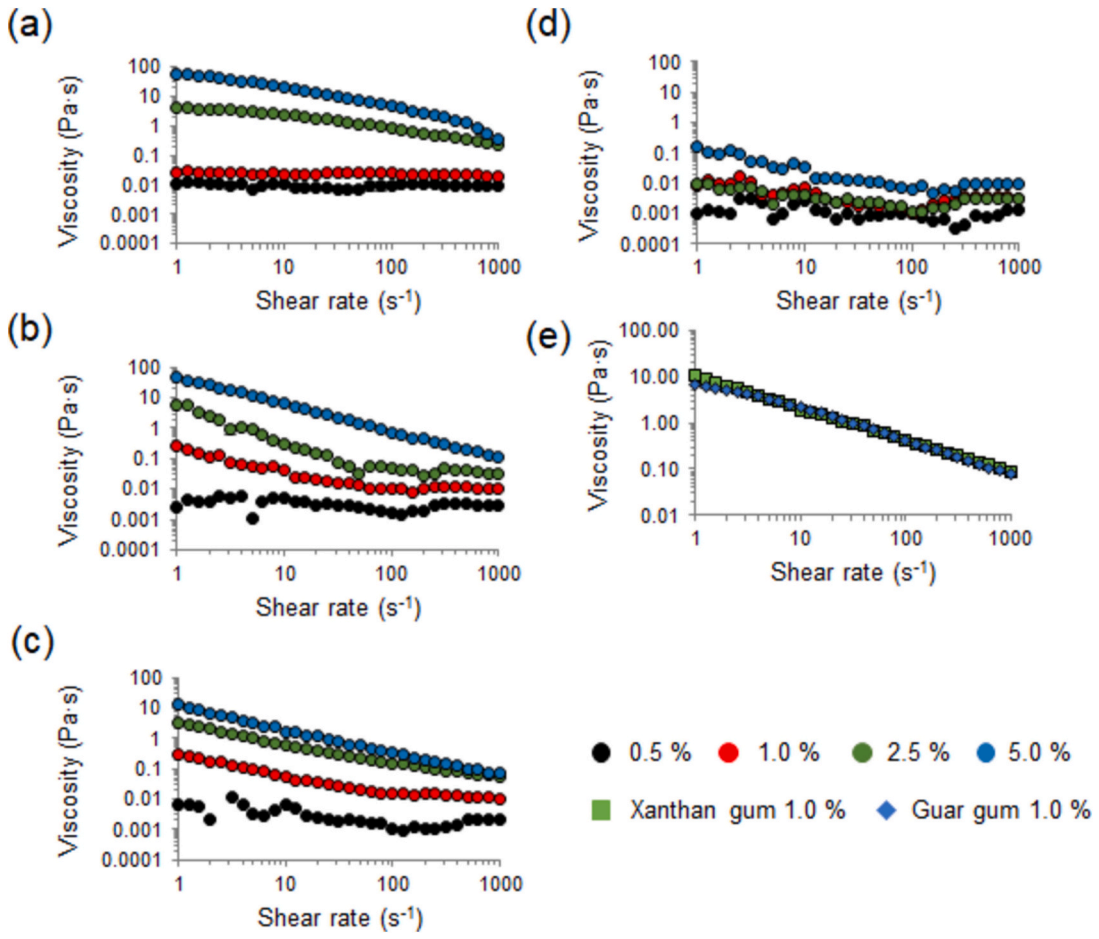


Fig. 2. Viscosity of (a) water-soluble fraction (WSF), (b) chelator-soluble fraction (CSF), (c) loosely-bound hemicellulose fraction (LHF), (d) strongly-bound hemicellulose fraction (SHF), and (e) commercial hydrocolloids as a function of shear rate and total dietary fiber content.

Table 3
Flow behavior parameters calculated from steady shear measurements of macauba polysaccharide fractions after sequential fractionation into water-soluble fraction (WSF), chelator-soluble fraction (CSF), loosely-bound hemicellulose fraction (LHF), strongly-bound hemicellulose fraction (SHF), and commercial hydrocolloids (n = 3).

Fraction	Content (% TDF)	Shear viscosity (Pa·s)	Consistency index (Pa·s^n)	Flow behavior index	R^2	Model
WSF	0.5	$(9.10 \pm 0.25)\cdot10^{-3\ a}$	–	–	1.00	Newton
	1.0	–	$(3.03 \pm 0.09)\cdot10^{-2\ g}$	$(9.60 \pm 0.10)\cdot10^{-1\ a}$	0.99	Ostwald – de Waele
	2.5	–	$(3.35 \pm 0.47)\cdot10^{0\ d}$	$(7.00 \pm 0.40)\cdot10^{-1\ b}$	0.98	Ostwald – de Waele
	5.0	–	$(3.58 \pm 0.26)\cdot10^{1\ b}$	$(6.10 \pm 0.30)\cdot10^{-1\ c}$	0.95	Ostwald – de Waele
CSF	0.5	$(2.50 \pm 0.09)\cdot10^{-3\ c}$	–	–	0.96	Newton
	1.0	–	$(2.33 \pm 0.07)\cdot10^{-1\ f}$	$(3.90 \pm 0.10)\cdot10^{-1\ d}$	0.95	Ostwald – de Waele
	2.5	–	$(3.38 \pm 0.49)\cdot10^{0\ d}$	$(1.30 \pm 0.10)\cdot10^{-1\ h}$	0.94	Ostwald – de Waele
	5.0	–	$(4.41 \pm 0.63)\cdot10^{1\ b}$	$(1.10 \pm 0.10)\cdot10^{-1\ h}$	0.99	Ostwald – de Waele
LHF	0.5	$(1.90 \pm 0.01)\cdot10^{-3\ d}$	–	–	0.96	Newton
	1.0	–	$(3.38 \pm 0.13)\cdot10^{-1\ e}$	$(3.80 \pm 0.20)\cdot10^{-1\ d}$	0.97	Ostwald – de Waele
	2.5	–	$(3.66 \pm 0.25)\cdot10^{0\ d}$	$(3.20 \pm 0.10)\cdot10^{-1\ e}$	0.97	Ostwald – de Waele
	5.0	–	$(1.12 \pm 0.02)\cdot10^{1\ c}$	$(2.40 \pm 0.10)\cdot10^{-1\ g}$	0.99	Ostwald – de Waele
SHF	0.5	$(1.09 \pm 0.01)\cdot10^{-3\ e}$	–	–	0.88	Newton
	1.0	$(2.90 \pm 0.18)\cdot10^{-3\ b}$	–	–	0.98	Newton
	2.5	$(3.37 \pm 0.10)\cdot10^{-3\ b}$	–	–	0.99	Newton
	5.0	$(9.27 \pm 0.38)\cdot10^{-3\ a}$	–	–	0.98	Newton
Guar Gum	1.0	–	$(1.13 \pm 0.10)\cdot10^{1\ a}$	$(2.32 \pm 0.02)\cdot10^{-1\ g}$	0.99	Ostwald – de Waele
Xanthan Gum	1.0	–	$(9.23 \pm 0.64)\cdot10^{0\ a}$	$(2.83 \pm 0.01)\cdot10^{-1\ f}$	0.99	Ostwald – de Waele

TDF: total dietary fiber content. Values followed by different superscript letters in the same column are significantly different ($p < 0.05$) according to Tukey's test.

direction of flow, resulting in their disentanglement with a consequent reduction in viscosity [48]. Therefore, the shift from Newtonian to shear-thinning behavior demonstrates a transition from dilute to semi-dilute regime for WSF, CSF, and LHF suspensions and indicates that the critical concentration of those suspensions is between 0.5 and 1.0 %. Similarly, Newtonian behavior at concentrations below 1.0 % was reported for pectins extracted from okra [48] and chicory root [49], as well as xyloglucans extracted from apple pomace [50]. Newtonian behavior was also reported for mandarin pectins at concentrations below 1.5 % [51]. Shear thinning profile is a desired behavior, allowing

products like sauces, yogurts, and dressings to flow easily when stirred, poured, spread, or swallowed but remain thick and stable at rest [52]. Shear thinning behavior is also relevant in modern applications, such as foods for dysphagia patients [53] or food obtained by 3D printing [54].

Macauba WSF showed higher viscosity than polysaccharides fractions from cherry [38] *Porphyridium cruentum* [55], and quince fruits [56] obtained with conditions similar to those employed in the present work. Under similar extraction conditions, the thickening properties of macauba CSF was higher than pectins from Chinese quince fruits [56], similar to pectins from Okra [48], and tomatoes [23], but lower than pectins from mandarin citrus peel [51] and chicory root [49]. The thickening properties of macauba LHF was comparable to loosely-bound hemicelluloses from tomato cell walls [23], higher than reported for Chinese quince fruits [56] and apple pomace, but lower compared to xyloglucans from tamarin seed [50].

The increase in WSF, CSF, and LHF concentration from 0.5 to 5.0 % increased the consistency index by 4 orders of magnitude and reduced the shear dependency, indicated by the decrease in flow behavior index. The WSF and CSF showed the greatest thickening potential, evidenced by the highest, but similar ($p > 0.05$) consistency index at concentrations of 2.5 % and 5.0 %.

However, the CSF showed higher shear dependence than WSF, evidenced by a significantly lower ($p < 0.05$) flow behavior index at concentrations from 1.0 to 5.0 %. The CSF also showed higher shear dependence than LHF at 2.5 and 5.0 %. Comparable persistent length was reported for pectins with lower molecular weight than Konjac glucomannans, potentially indicating higher stiffness for the pectin chains [57–59]. Therefore, it is possible that the higher stiffness of the pectin chains, particularly of the homogalacturonan domains, among other factors, allows their molecules to align more quickly to the flow direction, resulting in a stronger reduction in viscosity upon shearing. Moreover, differences in molecular weight, net charge, conformation, degree of branching, and substitution might also result in differences in rheological behavior. As previously mentioned, the results from the present work provide the starting point for further purification of macauba cell wall polysaccharides to determine their molecular weight, net charge, and degree of branching and substitution of the polysaccharides in WSF, CSF, and LHF. Thereby, in future research the structure-function relationship of macauba CWP could be elucidated to gain a mechanistic understanding on the differences in their rheological properties.

The rheological behavior of SHF suspensions differed from the other macauba polysaccharide fractions, in which Newtonian behavior was observed over the concentration range of 0.5–5.0 %. The suspension at 0.5 % showed the lowest viscosity, with torque close to the device detection limit. The increase in concentration from 0.5 to 5.0 % resulted in a marginal increase in viscosity, as shown in Table 3. Therefore, the critical concentration of the polysaccharides in SHF was not achieved even at 5.0 % suspensions. Moreover, the SHF, rich in xylans, showed the least thickening capacity among the investigated fractions, and similar low viscosity was reported for strongly-bound hemicelluloses from Chinese quince fruits [56], tomatoes [23], and alkali-soluble arabinoxylans from wheat [60].

Macauba polysaccharide solutions have lower thickening capacity compared to commercial hydrocolloids. As indicated in Table 3, achieving comparable rheological properties requires 2.5 to 5 times higher macauba polysaccharides concentrations than guar gum and xanthan gum. Therefore, future research should explore cost-benefit analysis for using macauba polysaccharide ingredients in various food systems. This is relevant given the higher concentration required to achieve similar rheological performance compared to commonly used hydrocolloids.

The thickening properties of polysaccharides are due to non-specific intermolecular entanglements above the critical concentration [16]. Differences in thickening properties between polysaccharides are attributed to variations in molecular weight, chain flexibility, electrostatic charge, hydrodynamic volume, and degree of ramification and substitution [16,61]. Polysaccharides commonly used as food thickeners are xanthan gum, galactomannans such as guar gum, locust bean gum and tara gum, and carboxymethylcellulose [62]. Thickening agents contribute to obtaining the desired consistency, mouthfeel, and texture. In addition, the increase in viscosity and the formation of colloidal suspensions also reduce syneresis, control the growth of ice and sugar crystals, prevent creaming and settling, and stabilize foams [63]. As a consequence, thickening polysaccharides are employed in a wide range of applications like frozen dairy desserts, juices and soft drinks, salad dressings, whipped creams, and bakery products [57].

The results show that macauba galactoglucomannans (WSF), pectins (CSF), and xyloglucans (LHF) were able to form entangled networks at concentrations as low as 1.0 %, thus showing high potential as food thickeners. Moreover, the characteristic sugar ratios of the polysaccharide fractions have a strong influence on their molecular and rheological properties. For instance high mannose to glucose ratios $\left(\frac{Man}{Glu}\right)$ correlates with increased viscosity in glucomannans [64], whereas higher contents of galactose relates to lower thickening power [65]. For comparison, macauba WSF showed higher $\frac{Man}{Glu}$ ratio than Konjac glucomannan. High ratios of arabinose and galactose to rhamnose ($\frac{Ara+Gal}{Rha}$) are associated with increased viscosity owing to enhanced entanglement of rhamnogalacturonan-I side chains [66]. For comparison, macauba CSF showed higher $\frac{Ara+Gal}{Rha}$ ratios than pectins extracted from lemon and orange peels [66]. The macauba polysaccharide fractions (WSF, CSF, LHF) can be classified as medium viscosity hydrocolloids, as their consistency factor at 1.0 % suspensions was in the same range as medium viscosity carboxymethylcellulose [67]. The galactoglucomannans in WSF are particularly interesting as a new food hydrocolloid, owing to their thickening properties, high yield and high SDF content. Therefore, future research addressing the optimization of the extraction and isolation of macauba galactoglucomannans can leverage the utilization of macauba pulp meal as a new source of food hydrocolloids. Given the rising interest in macauba pulp cultivation [68], it can be anticipated that macauba pulp meal will be an inexpensive source of polysaccharides. Similarly, commonly used food hydrocolloids are obtained from side streams such as pectins [69] and cellulose derivatives [70]. Nevertheless, the final cost of macauba polysaccharide ingredients will also depend on the extraction and isolation steps employed for their recovery [71]. The optimization of these processing steps and the provision of mass and energy balance is fundamental to assessing whether macauba polysaccharides will be cost-competitive compared to existing thickeners and stabilizers.

3.3. Viscoelastic properties of macauba cell wall polysaccharide fractions

Amplitude and frequency sweep measurements were used to investigate the structuring and network formation properties of macauba polysaccharide fractions. The results are depicted in Figs. 3 and 4, and summarized in Tables 4 and 5.

In amplitude sweeps, the LVR of polysaccharide dispersions is characterized by the G′, G″, and yield stress values [72]. The results from frequency sweeps are expressed as the slopes of G′ and G″ as a function of frequency, representing the stability of the molecular interactions with the change in time scale [73]. Moreover, the relaxation times and the loss factor (tanδ) are also obtained from frequency sweeps to assess the sol-gel transition and the elastic character of the hydrogels [48,74]. The tanδ provides a measurement of the viscoelastic degree of polysaccharide dispersions and the lifetime of intermolecular interactions stabilizing the polymeric network [30,75].

The WSF had the broadest LVR and required the highest energy input to induce plastic deformation, as evidenced by higher yield stress values. For concentrations ranging from 2.5 to 10.0 %, the WSF dispersion showed a sol-gel transition and a high frequency dependency of both G′ and G″, evidenced by the intercept of the G′ and G″ curves and the highest

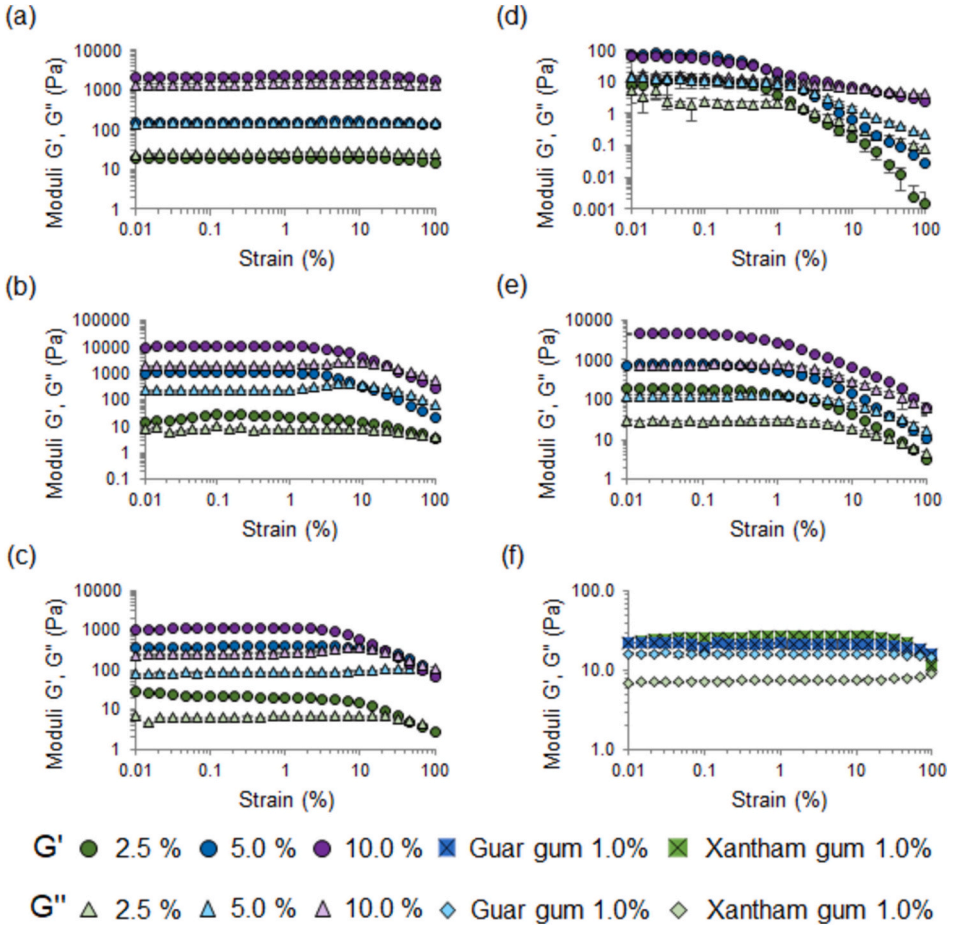


Fig. 3. Amplitude sweep spectra of (a) water-soluble fraction (WSF), (b) chelator-soluble fraction (CSF), (c) loosely-bound hemicellulose fraction (LHF), (d) strongly-bound hemicellulose fraction (SHF), (e) cellulose-rich fraction (CLF), and (f) commercial hydrocolloids. Circles and crossed squares represent the storage modulus (G′) and triangles and diamonds represent the loss modulus (G″).

slope values of both parameters (Table 5). Therefore, the WSF suspensions showed typical behavior of random coil polymers at a semi-dilute regime, behaving like a viscoelastic liquid at low frequencies, but having a predominant elastic character at high frequencies [76]. The increase in WSF concentration from 2.5 to 10.0 % shifted the sol-gel transition, resulting in longer times needed for disruption of chain entanglements as indicated by the increase in characteristic relaxation times from 0.0124 to 1.17 s. Comparable profile was obtained for commercial guar gum, as depicted in Fig. 4f, and similar results were reported for fenugreek [76], tara gum, and locust bean gum [77]. These results highlight the potential of macauba galactoglucomannans for applications as thickening agents owing to their higher effect on increasing the viscosity instead of producing gel networks.

Different from WSF, the other macauba polysaccharide fractions showed gel-like behavior, evidenced by G′ higher than G″ moduli when amplitude and frequency sweep measurements were conducted. At the LVR, stiffer gel-like suspensions were produced by the CSF, indicated by the highest G′ values at concentrations of 5 and 10 % [78]. On the other hand, the LHF showed the highest yield stress values at 5 and 10 %, thus resulting in gels with the highest structural strength [72]. The increase in CSF and LHF content from 2.5 to 10 % improved structural strength of the suspensions as indicated by the increase in 3 and 4 orders of magnitude in yield stress, respectively. These results indicate that the increase in LHF concentration resulted in higher improvement of structural strength (higher yield stress), while the increase in CSF concentration resulted in the increase in gel elasticity (higher values of G′).

The mechanical spectra of both CSF and LHF suspensions showed predominant elastic behavior at all tested concentrations, as G′ was higher than G″ over three cycles log of frequency decay. Both polysaccharide fractions showed weak gel character [48], as indicated by tanδ in the range of 0.08 to 0.70 and 0.05 to 0.30 for CSF and LHF, respectively. Moreover, the increase in concentration improved the stability of the intermolecular associations, as evidenced by the reduction in both G′ and G″ slopes [73]. Higher stability of the molecular interactions was obtained for the CSF in which the increase in concentration from 2.5 to 10.0 % resulted in a more pronounced decrease in the G′ slope. These results highlight the potential of macauba pectins (CSF) and xyloglucans (LHF) as structuring agents. A weak gel character was also reported for pectins from tomatoes [23] and blueberry pomace [79], whereas poor structuring ability was reported for a xyloglucan-rich fraction extracted from tamarind seeds [80]. Moreover, weak gel behavior was also observed for the suspensions containing xanthan gum. Xanthan gum is commonly used as a food stabilizer owing to its ability to undergo weak intermolecular association, forming weak gels with junction zones that can be readily disrupted at low shear rates [61]. Polysaccharides forming weak gels are beneficial in stabilizing suspended solids in dispersions and drinks [81]. Weak gels can also stabilize emulsions and foams by limiting the movement of oil droplets and gas bubbles. In frozen foods, weak gel character can also restrict water mobility and improve freeze-thaw stability [16]. Therefore, both Macauba CSF and LHF could be used as food stabilizers. However, future studies exploring the cost-benefit of using macauba polysaccharides in food systems must be carried out. This is relevant owing to the higher concentration needed to achieve similar rheological behavior compared to commercial hydrocolloids like xanthan gum.

Compared to CSF, LHF, and CLF, the SHF produced weak gels with

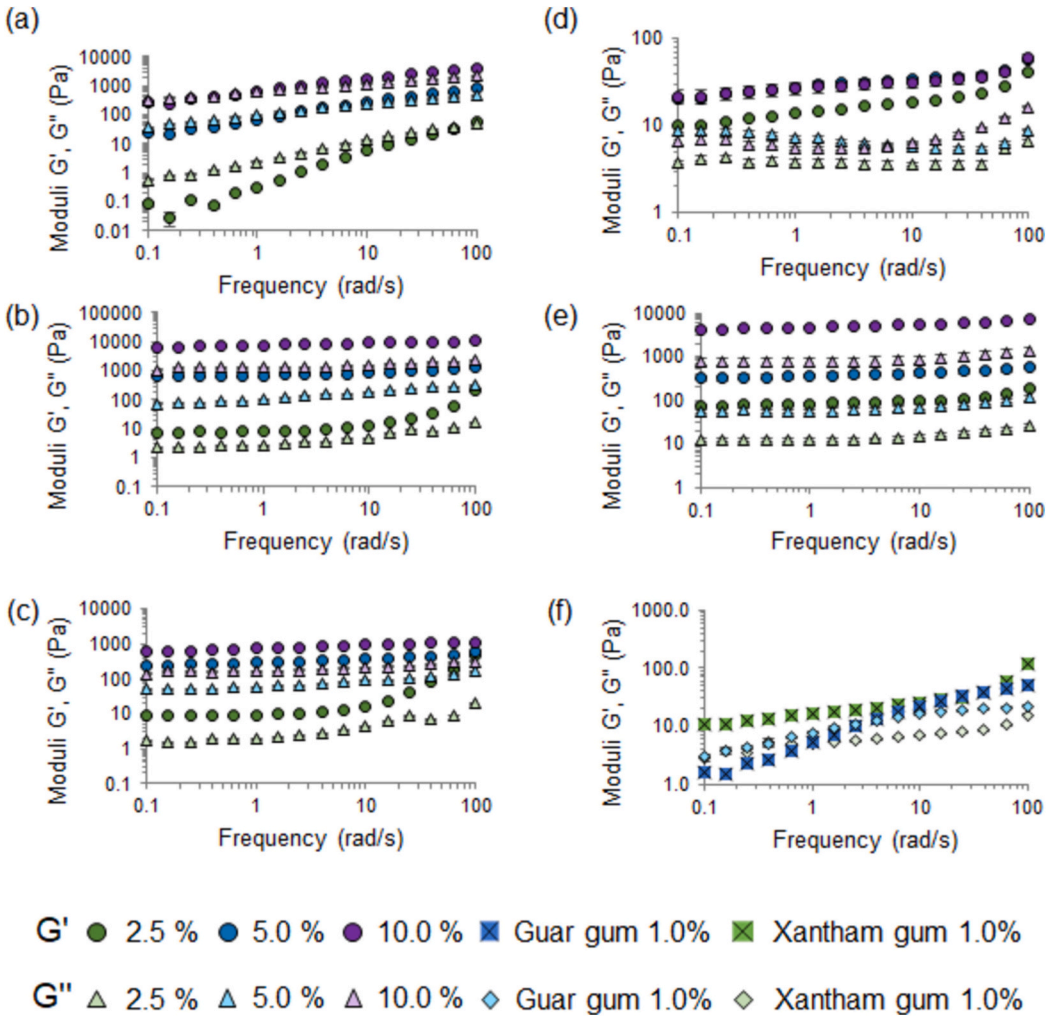


Fig. 4. Mechanical spectra of (a) water-soluble fraction (WSF), (b) chelator-soluble fraction (CSF), (c) loosely-bound hemicellulose fraction (LHF), (d) strongly-bound hemicellulose fraction (SHF), (e) cellulose-rich fraction (CLF), and (f) commercial hydrocolloids. Circles and crossed squares represent the storage modulus (G′) and triangles and diamonds represent the loss modulus (G″).

the lowest structural strength at concentrations from 2.5 to 5.0 %, evidenced by the lowest yield stress values from amplitude sweep measurements. The increase in concentration of the SHF suspensions resulted in a marginal increase in G′ and G″, similar to the viscosity results obtained from steady shear measurements (Section 3.2). The mechanical spectra of the SHF showed weak gel behavior, with a tanδ of 0.1–0.8. As previously discussed, the SHF mainly comprised heteroxylans with a minor contribution from xyloglucans and pectic polysaccharides. Similar to present work, arabinoxylans from barley insoluble fibers [82], wheat bran [83], and spent grains [84] showed weak gel behavior and low gel stability.

The CLF suspensions exhibited yield stresses lower than the WSF at all concentrations and inferior to the CSF and LHF at 5.0 and 10.0 %. These results indicate that despite the high G′ and G″, the CLF showed limited ability to withstand deformation, and relatively low energy input levels were necessary to induce plastic deformation and loss of structural integrity. In the LVR, the CLF suspensions produced weak gels (tanδ 0.13–0.20) with a low frequency dependence of G′ and G″. As discussed in Section 3.1, the CLF mainly comprises cellulose with low content of hemicelluloses and pectic polysaccharides. Cellulose-rich suspensions can retain water and form entanglements to build networks with gel-like properties [85], which are influenced by the size, structure, and density of the cellulose fibers [86]. Macauba CLF showed G′ comparable to citrus fiber from lemons [87] and higher than cellulose-rich suspensions from oat fibers [88].

Polysaccharide gelling involves the formation of a three-dimensional network by means of intermolecular association or junction zones [89]. Polysaccharides form physical gels because their junction zones are stabilized via non-covalent interactions such as hydrogen bonds, hydrophobic association, and cation-mediated cross-linking [63]. The strength and stability of junction zones depends on the type and structure of the polysaccharides [90]. For instance, the presence of acetyl groups in glucomannans suppress intermolecular association via hydrogen bonds [91], in agreement with the poor gelling properties observed for macauba galactoglucomannans (WSF). Homogalacturonans form junction zones via cooperative attraction or entanglement of the rigid chain structures [92], driven by the combination of intermolecular hydrogen bonds and interactions between charged groups and water molecules [93]. Xyloglucans associate through the interaction of hydrophobic patches of the polysaccharide chains [94], which is facilitated by the substantial stiffness of the cellulose-like backbone [91]. Accordingly, the results also showed that the CSF and LHF, rich in pectins and xyloglucans, respectively, were able to form weak gels, in which the polysaccharide chains undergo weak intermolecular association that can be readily disrupted at low shear rates [61]. In this case, the macauba polysaccharide fractions might be used in low concentrations to form a macromolecular network that yields and flows, rapidly recovering their gel-like character when the stress is removed [92].

Table 4
Storage and lost moduli at the linear viscoelastic region and yield stress of macauba polysaccharide fractions obtained after sequential fractionation into water-soluble fraction (WSF), chelator-soluble fraction (CSF), loosely-bound hemicellulose fraction (LHF), strongly-bound hemicellulose fraction (SHF), cellulose-rich fraction (CLF), and commercial hydrocolloids ($n = 3$).

Fraction	Concentration (% TDF)	G′ LVR (Pa)	G″ LVR (Pa)	Yield stress (Pa)
WSF	02.5	$(1.81 \pm 0.05)\cdot10^{1\ j}$	$(2.63 \pm 0.05)\cdot10^{1\ f}$	$(6.89 \pm 0.02)\cdot10^{0\ g}$
	05.0	$(1.57 \pm 0.27)\cdot10^{2\ g}$	$(1.54 \pm 0.03)\cdot10^{2\ e}$	$(2.54 \pm 0.05)\cdot10^{2\ b}$
	10.0	$(2.14 \pm 0.29)\cdot10^{3\ c}$	$(1.37 \pm 0.17)\cdot10^{3\ b}$	$(8.02 \pm 1.06)\cdot10^{2\ a}$
CSF	02.5	$(2.58 \pm 0.72)\cdot10^{1\ i}$	$(8.09 \pm 2.05)\cdot10^{0\ h}$	$(3.20 \pm 0.60)\cdot10^{-1\ j}$
	05.0	$(1.05 \pm 0.09)\cdot10^{3\ d}$	$(2.40 \pm 0.16)\cdot10^{2\ d}$	$(1.01 \pm 0.08)\cdot10^{1\ f}$
	10.0	$(1.02 \pm 0.04)\cdot10^{4\ a}$	$(2.01 \pm 0.06)\cdot10^{3\ a}$	$(1.04 \pm 0.04)\cdot10^{2\ d}$
LHF	02.5	$(2.74 \pm 0.78)\cdot10^{1\ i}$	$(7.44 \pm 1.39)\cdot10^{0\ h}$	$(7.01 \pm 0.4)\cdot10^{-2\ l}$
	05.0	$(4.23 \pm 0.66)\cdot10^{2f}$	$(1.02 \pm 0.13)\cdot10^{2\ f}$	$(2.89 \pm 0.37)\cdot10^{1\ e}$
	10.0	$(1.14 \pm 0.42)\cdot10^{3\ d}$	$(2.61 \pm 0.12)\cdot10^{2\ d}$	$(1.35 \pm 0.02)\cdot10^{2\ c}$
SHF	02.5	$(1.21 \pm 0.55)\cdot10^{1\ j}$	$(2.17 \pm 0.62)\cdot10^{0\ i}$	$(2.04 \pm 0.31)\cdot10^{-2\ l}$
	05.0	$(7.17 \pm 0.97)\cdot10^{1\ h}$	$(1.12 \pm 0.09)\cdot10^{1\ g}$	$(5.02 \pm 1.02)\cdot10^{-2\ l}$
	10.0	$(3.83 \pm 0.54)\cdot10^{1\ i}$	$(1.30 \pm 0.03)\cdot10^{1\ g}$	$(1.33 \pm 0.34)\cdot10^{-1\ k}$
CLF	02.5	$(1.81 \pm 0.09)\cdot10^{2\ g}$	$(2.83 \pm 0.24)\cdot10^{1\ f}$	$(2.70 \pm 0.10)\cdot10^{-1\ j}$
	05.0	$(7.60 \pm 0.81)\cdot10^{2\ e}$	$(1.26 \pm 0.13)\cdot10^{2\ e}$	$(1.33 \pm 0.39)\cdot10^{0\ i}$
	10.0	$(4.63 \pm 0.02)\cdot10^{3\ b}$	$(7.40 \pm 0.31)\cdot10^{2\ c}$	$(3.85 \pm 0.62)\cdot10^{0\ h}$
Guar Gum	01.0	$(2.12 \pm 0.04)\cdot10^{1\ i}$	$(1.58 \pm 0.02)\cdot10^{1\ g}$	$(1.19 \pm 0.09)\cdot10^{0\ i}$
Xantham Gum	01.0	$(2.66 \pm 0.02)\cdot10^{1\ i}$	$(7.64 \pm 0.12)\cdot10^{0\ h}$	$(8.37 \pm 0.16)\cdot10^{0\ f}$

TDF: total dietary fiber content; LVR: Linear viscoelastic region. Values followed by different superscript letters in the same column are significantly different ($p < 0.05$) according to Tukey's test.

Table 5
Relaxation time and slope of storage and loss moduli from frequency sweeps of macauba pulp polysaccharide fractions obtained after sequential fractionation into water-soluble fraction (WSF), chelator-soluble fraction (CSF), loosely-bound hemicellulose fraction (LHF), strongly-bound hemicellulose fraction (SHF), cellulose-rich fraction (CLF), and commercial hydrocolloids ($n = 3$).

Fraction	Concentration (% TDF)	Relaxation time (s)	G′ slope	G″ slope
WSF	02.5	$(1.24 \pm 0.02)\cdot10^{-2\ d}$	$(1.11 \pm 0.05)\cdot10^{0\ a}$	$(6.75 \pm 0.01)\cdot10^{-1\ a}$
	05.0	$(2.27 \pm 0.07)\ 10^{-1\ c}$	$(5.59 \pm 0.03)\cdot10^{-1\ b}$	$(3.68 \pm 0.02)\cdot10^{-1\ b}$
	10.0	$(1.17 \pm 0.07)\ 10^{0\ a}$	$(4.22 \pm 0.02)\cdot10^{-1\ d}$	$(2.82 \pm 0.03)\cdot10^{-1\ d}$
CSF	02.5	–	$(3.61 \pm 0.23)\cdot10^{-1\ e}$	$(2.86 \pm 0.35)\cdot10^{-1\ d}$
	05.0	–	$(9.48 \pm 0.20)\cdot10^{-2\ m}$	$(2.31 \pm 0.01)\cdot10^{-1\ e}$
	10.0	–	$(7.19 \pm 0.18)\cdot10^{-2\ jkl}$	$(1.28 \pm 0.04)\cdot10^{-1\ g}$
LHF	02.5	–	$(4.76 \pm 0.05)\cdot10^{-1\ c}$	$(3.33 \pm 0.02)\cdot10^{-1\ c}$
	05.0	–	$(1.19 \pm 0.02)\cdot10^{-1\ h}$	$(1.68 \pm 0.03)\cdot10^{-1\ f}$
	10.0	–	$(0.95 \pm 0.02)\cdot10^{-1\ i}$	$(1.22 \pm 0.08)\cdot10^{-1\ g}$
SHF	02.5	–	$(1.66 \pm 0.01)\cdot10^{-1\ f}$	$(5.21 \pm 0.02)\cdot10^{-2\ i}$
	05.0	–	$(1.24 \pm 0.09)\cdot10^{-1\ h}$	$(5.23 \pm 0.19)\cdot10^{-2\ i}$
	10.0	–	$(1.27 \pm 0.05)\cdot10^{-1\ h}$	$(6.15 \pm 0.18)\cdot10^{-2\ h}$
CLF	02.5	–	$(7.09 \pm 0.08)\cdot10^{-2\ kj}$	$(1.64 \pm 0.07)\cdot10^{-1\ f}$
	05.0	–	$(7.60 \pm 0.63)\cdot10^{-2\ j}$	$(1.50 \pm 0.17)\cdot10^{-1\ fg}$
	10.0	–	$(7.48 \pm 0.05)\cdot10^{-2\ jk}$	$(7.69 \pm 0.27)\cdot10^{-2\ j}$
Guar Gum	01.0	$(2.88 \pm 0.19)\ 10^{-1\ b}$	$(5.55 \pm 0.10)\ 10^{-1\ b}$	$(2.99 \pm 0.07)\ 10^{-1\ d}$
Xantham Gum	01.0	–	$(2.74 \pm 0.06)\ 10^{-1\ f}$	$(1.74 \pm 0.08)\ 10^{-1\ f}$

TDF: total dietary fiber content. Values followed by different superscript letters in the same column are significantly different ($p < 0.05$) according to Tukey's test.

These properties allow for desired mechanical behavior at consumption conditions (e.g., mastication and swallowing) while most likely preventing settling and phase separation during storage. Therefore, the results of the present study highlight their potential as stabilizers for applications like salad dressings, emulsions, and frozen desserts.

4. Conclusion

The diverse composition of macauba cell wall polysaccharide fractions comprising galactoglucomannans, pectins, xyloglucans, xylans, and cellulose is reported for the first time. The results highlight the potential of macauba galactoglucomannans as a new food hydrocolloid for their thickening properties, relatively high content, and easy extractability. Therefore, future research can focus on optimizing the extraction of macauba galactoglucomannans, targeting the production of sustainable food ingredients. Unlike the galactoglucomannans, weak gels were obtained with macauba pectins, xyloglucans, xylans, and cellulose, underlining the ability of those polysaccharides to interact and build networks. These findings highlight the potential of macauba polysaccharides as promising food thickeners and stabilizers. Based on the conditions of our measurements, macauba polysaccharides show high potential for applications with neutral pH and low salt content such as frozen desserts, whipped creams, and milk alternatives. Future research addressing the stability of macauba polysaccharides across different pH values and their interaction and compatibility with other food macromolecules can leverage their applicability in diverse food systems. Despite demonstrating the diverse composition and potential of Macauba polysaccharides, our methodology yielded fractions with limited purity, which precluded the assessment of molecular properties like molecular weight distribution, linkage pattern analysis, substitution with functional groups, and degree of branching. In this sense, our work provides the starting point for further purification of macauba cell wall polysaccharides, evaluation of their molecular properties, and elucidation of their structure-function relationship.

CRediT authorship contribution statement

Sérgio Henrique Toledo e Silva: Writing – original draft, Visualization, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Stephanie Bader-Mittermaier:** Writing – review & editing, Methodology, Data curation, Conceptualization. **Lidiane Bataglia Silva:** Writing – review & editing, Investigation. **Carlos**

Augusto Colombo: Writing – review & editing, Visualization, Resources. **Roseli Aparecida Ferrari:** Writing – review & editing. **Peter Eisner:** Writing – review & editing, Supervision, Methodology, Conceptualization.

Declaration of Generative AI and AI-assisted technologies in the writing process

During the preparation of this work, the authors did not use Generative Artificial Intelligence (AI) or AI-assisted technologies in the writing process. The authors take full responsibility for the content of the publication.

Funding sources

This research was financially supported by the German Federal Ministry of Education and Research (BMBF) under the program Bioeconomy International [Grant No. 031B0282].

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.

References

[1] A.S. César, F.A. Almeida, R.P. de Souza, G.C. Silva, A.E. Atabani, The prospects of using *Acrocomia aculeata* (macaúba) a non-edible biodiesel feedstock in Brazil, Renew. Sustain. Energy Rev. 49 (2015) 1213–1220, https://doi.org/10.1016/j.rser.2015.04.125.
[2] C.A. Colombo, L.H. Chorfi Berton, B.G. Diaz, R.A. Ferrari, Macauba: a promising tropical palm for the production of vegetable oil, OCL 25 (2018) D108, https://doi.org/10.1051/ocl/2017038.
[3] OECD/FAO, OECD-FAO Agricultural Outlook 2021–2030, OECD Publishing, 2021.
[4] R. Corley, How much palm oil do we need? Environ. Sci. & Policy 12 (2009) 134–139, https://doi.org/10.1016/j.envsci.2008.10.011.
[5] S.A. Vianna, L.H.C. Berton, A. Pott, S.M.C. Guerreiro, C.A. Colombo, Biometric characterization of fruits and morphoanatomy of the mesocarp of *Acrocomia* species (Arecaceae), Int. J. Biol. 9 (2017) 78, https://doi.org/10.5539/ijb.v9n3p78.
[6] M.C. Coimbra, N. Jorge, Proximate composition of guariroba (*Syagrus oleracea*), jerivá (*Syagrus romanzoffiana*) and macaúba (*Acrocomia aculeata*) palm fruits, Food Res. Int. 44 (2011) 2139–2142, https://doi.org/10.1016/j.foodres.2011.03.032.
[7] C.H. Lescano, I.P. Oliveira, L.R. Silva, D.S. Baldivia, E.J. Sanjinez-Argandoña, E. J. Arruda, I.C.F. Moraes, F.F. Lima, Nutrients content, characterization and oil extraction from *Acrocomia aculeata* (Jacq.), Lodd. fruits, Afr. J. Food Sci. 9 (2015) 113–119, https://doi.org/10.5897/AJFS2014.1212.
[8] R. Schex, V.M. Lieb, V.M. Jiménez, P. Esquivel, R.M. Schweiggert, R. Carle, C. B. Steingass, HPLC-DAD-APCI/ESI-MSn analysis of carotenoids and α-tocopherol in Costa Rican *Acrocomia aculeata* fruits of varying maturity stages, Food Res. Int. 105 (2018) 645–653, https://doi.org/10.1016/j.foodres.2017.11.041.
[9] K. Kotecka-Majchrzak, A. Sumara, E. Fornal, M. Montowska, Oilseed proteins – properties and application as a food ingredient, Trends Food Sci. Technol. 106 (2020) 160–170, https://doi.org/10.1016/j.tifs.2020.10.004.
[10] M.C. Coimbra, N. Jorge, Fatty acids and bioactive compounds of the pulps and kernels of Brazilian palm species, guariroba (*Syagrus oleraces*), jerivá (*Syagrus romanzoffiana*) and macaúba (*Acrocomia aculeata*), J. Sci. Food Agric. 92 (2012) 679–684, https://doi.org/10.1002/jsfa.4630.
[11] A.C. Andrade, J.F.U. Marinho, A.C. Souza, T.S. Tavares, D.R. Dias, R.F. Schwan, C. A. Nunes, S.C. Bastos, Prebiotic potential of pulp and kernel cake from Jerivá (*Syagrus romanzoffiana*) and Macaúba palm fruits (*Acrocomia aculeata*), Food Res. Int. 136 (2020) 109595, https://doi.org/10.1016/j.foodres.2020.109595.
[12] D. Mudgil, S. Barak, Composition, properties and health benefits of indigestible carbohydrate polymers as dietary fiber: a review, Int. J. Biol. Macromol. 61 (2013) 1–6, https://doi.org/10.1016/j.ijbiomac.2013.06.044.
[13] J. van Audenhove, T. Bernaerts, N.I. Putri, A.M. van Loey, M.E. Hendrickx, The functionalisation of fruit and vegetable cell wall material as texturizing agent: the role of pectin depletion and particle size reduction techniques, Food Hydrocoll. 142 (2023) 108814, https://doi.org/10.1016/j.foodhyd.2023.108814.
[14] T.J. Foster, Natural structuring with cell wall materials, Food Hydrocoll. 25 (2011) 1828–1832, https://doi.org/10.1016/j.foodhyd.2011.05.016.
[15] A. Yemenicioğlu, S. Farris, M. Turkyilmaz, S. Gulec, A review of current and future food applications of natural hydrocolloids, Int. J. Food Sci. Technol. 55 (2020) 1389–1406, https://doi.org/10.1111/ijfs.14363.
[16] D. Saha, S. Bhattacharya, Hydrocolloids as thickening and gelling agents in food: a critical review, J. Food Sci. Technol. 47 (2010) 587–597, https://doi.org/10.1007/s13197-010-0162-6.
[17] R.J. Redgwell, M. Fischer, Dietary fiber as a versatile food component: an industrial perspective, Mol. Nutr. Food Res. 49 (2005) 521–535, https://doi.org/10.1002/mnfr.200500028.
[18] B.P. Silva, G.M. Silva, J.P. Parente, Chemical properties and adjuvant activity of a galactoglucomannan from *Acrocomia aculeata*, Carbohydr. Polym. 75 (2009) 380–384, https://doi.org/10.1016/j.carbpol.2008.09.031.
[19] W. Denagbe, R. Covis, J.-P. Guegan, J.-C. Robinson, D. Bereau, T. Benvegnu, Structure and emulsifying properties of unprecedent glucomannan oligo- and polysaccharides from Amazonia Acrocomia aculeata palm fruit, Carbohydr. Polym. 324 (2024) 121510, https://doi.org/10.1016/j.carbpol.2023.121510.
[20] V. Krstonošić, J. Jovičić-Bata, N. Maravić, I. Nikolić, L. Dokić, Rheology, Structure, and Sensory Perception of Hydrocolloids, Food Structure and Functionality, Elsevier, in, 2021, pp. 23–47.
[21] A. Montoya-Arroyo, R.M. Schweiggert, M.-L. Pineda-Castro, M. Sramek, R. Kohlus, R. Carle, P. Esquivel, Characterization of cell wall polysaccharides of purple pitaya (*Hylocereus* sp.) pericarp, Food Hydrocoll. 35 (2014) 557–564, https://doi.org/10.1016/j.foodhyd.2013.07.010.
[22] A. Schieber, R. Fügel, M. Henke, R. Carle, Determination of the fruit content of strawberry fruit preparations by gravimetric quantification of hemicellulose, Food Chem. 91 (2005) 365–371, https://doi.org/10.1016/j.foodchem.2004.09.003.
[23] Q. Li, J. Li, H. Li, R. Xu, Y. Yuan, J. Cao, Physicochemical properties and functional bioactivities of different bonding state polysaccharides extracted from tomato fruit, Carbohydr. Polym. 219 (2019) 181–190, https://doi.org/10.1016/j.carbpol.2019.05.020.
[24] German Food Act, Amtliche Sammlung von Untersuchungsverfahren nach × 64 LFGB, × 35 Vorlaeufiges Tabakgesetz, × 28b GenTG - I - Lebensmittel Band I (L) Verfahren zur Probenahme und Untersuchung von Lebensmitteln, Berlin, Germany, 1980.
[25] AOAC International, Method 968.06 Protein (crude) in animal feed. Dumas Method, 15th ed., Arlington, VA, USA, 1990.
[26] AOAC International, Method 991.43 Total Dietary fiber. Enzymatic-Gravimetric Method, 17th ed, 2000 (Gaitherburg, MD, USA).
[27] S. Naumann, U. Schweiggert-Weisz, D. Haller, P. Eisner, Retention of primary bile acids by lupin cell wall polysaccharides under *in vitro* digestion conditions, Nutr 11 (2019), https://doi.org/10.3390/nu11092117.
[28] M. Tabarsa, M. Anvari, H.S. Joyner, S. Behnam, A. Tabarsa, Rheological behavior and antioxidant activity of a highly acidic gum from *Althaea* officinalis flower, Food Hydrocoll. 69 (2017) 432–439, https://doi.org/10.1016/j.foodhyd.2017.02.009.
[29] M. Manzoor, J. Singh, J.D. Bandral, A. Gani, R. Shams, Food hydrocolloids: functional, nutraceutical and novel applications for delivery of bioactive compounds, Int. J. Food Sci. Technol. 165 (2020) 554–567, https://doi.org/10.1016/j.ijbiomac.2020.09.182.
[30] Z. Fan, P. Cheng, P. Zhang, G. Zhang, J. Han, Rheological insight of polysaccharide/protein based hydrogels in recent food and biomedical fields: a review, Int. J. Food Sci. Technol. 222 (2022) 1642–1664, https://doi.org/10.1016/j.ijbiomac.2022.10.082.
[31] I. Prabasari, F. Pettolino, M.-L. Liao, A. Bacic, Pectic polysaccharides from mature orange (*Citrus sinensis*) fruit albedo cell walls: sequential extraction and chemical characterization, Carbohydr. Polym. 84 (2011) 484–494, https://doi.org/10.1016/j.carbpol.2010.12.012.
[32] E. Meersman, N. Struyf, C. Kyomugasho, Z. Jamsazzadeh Kermani, J.S. Santiago, E. Baert, S. Hemdane, G. Vrancken, K.J. Verstrepen, C.M. Courtin, M. Hendrickx, J. Steensels, Characterization and degradation of pectic polysaccharides in cocoa pulp, J. Agric. Food Chem. 65 (2017) 9726–9734, https://doi.org/10.1021/acs.jafc.7b03854.
[33] R. Fügel, R. Carle, A. Schieber, A novel approach to quality and authenticity control of fruit products using fractionation and characterisation of cell wall polysaccharides, Food Chem. 87 (2004) 141–150, https://doi.org/10.1016/j.foodchem.2003.10.031.
[34] A. Ebringerová, Z. Hromádková, T. Heinze, Hemicellulose, in: T. Heinze (Ed.), Polysaccharides I, Springer-Verlag, Berlin/Heidelberg, 2005, pp. 1–67.
[35] S.C. Fry, Cell wall polysaccharide composition and covalent crosslinking, in: J. A. Roberts (Ed.), Annual Plant Reviews Online, Wiley, 2018, pp. 1–42.
[36] T.M. Cantu-Jungles, M. Iacomini, T.R. Cipriani, L.M.C. Cordeiro, Structural diversity of alkali-soluble polysaccharides from the fruit cell walls of tucumã (*Astrocaryum aculeatum*), a commelinid monocotyledon from the family Arecaceae, Plant physiology and biochemistry PPB 118 (2017) 356–361, https://doi.org/10.1016/j.plaphy.2017.07.002.
[37] L.M.C. Cordeiro, C.P. de Almeida, M. Iacomini, Unusual linear polysaccharides: (1→5)-α-L-arabinan, (1→3)-(1→4)-α-D-glucan and (1→4)-β-D-xylan from pulp of Buriti (*Mauritia flexuosa*), an edible palm fruit from the Amazon region, Food Chem. 173 (2015) 141–146, https://doi.org/10.1016/j.foodchem.2014.10.020.
[38] M.F. Basanta, M.F.E. Plá, C.A. Stortz, A.M. Rojas, Chemical and functional properties of cell wall polymers from two cherry varieties at two developmental stages, Carbohydr. Polym. 92 (2013) 830–841, https://doi.org/10.1016/j.carbpol.2012.09.091.
[39] K. Houben, R.P. Jolie, I. Fraeye, A.M. van Loey, M.E. Hendrickx, Comparative study of the cell wall composition of broccoli, carrot, and tomato: structural

characterization of the extractable pectins and hemicelluloses, Carbohydr. Res. 346 (2011) 1105–1111, https://doi.org/10.1016/j.carres.2011.04.014.
[40] L.C. Vriesmann, C.L.O. Petkowicz, Polysaccharides from the pulp of cupuassu (*Theobroma grandiflorum*): structural characterization of a pectic fraction, Carbohydr. Polym. 77 (2009) 72–79, https://doi.org/10.1016/j.carbpol.2008.12.007.
[41] I. Mafra, B. Lanza, A. Reis, V. Marsilio, C. Campestre, M. Angelis, M.A. Coimbra, Effect of ripening on texture, microstructure and cell wall polysaccharide composition of olive fruit (*Olea europaea*), Physiol. Plant. 111 (2001) 439–447, https://doi.org/10.1034/j.1399-3054.2001.1110403.x.
[42] E. Vierhuis, H. Schols, G. Beldman, A. Voragen, Isolation and characterisation of cell wall material from olive fruit (*Olea europaea* cv koroneiki) at different ripening stages, Carbohydr. Polym. 43 (2000) 11–21, https://doi.org/10.1016/S0144-8617(99)00204-0.
[43] D. Watt, D. Brasch, D. Larsen, L. Melton, Isolation, characterisation, and NMR study of xyloglucan from enzymatically depectinised and non-depectinised apple pomace, Carbohydr. Polym. 39 (1999) 165–180, https://doi.org/10.1016/S0144-8617(99)00002-8.
[44] T.M. Cantu-Jungles, M. Iacomini, T.R. Cipriani, L.M.C. Cordeiro, Extraction and characterization of pectins from primary cell walls of edible açaí (*Euterpe oleraceae*) berries, fruits of a monocotyledon palm, Carbohydr. Polym. 158 (2017) 37–43, https://doi.org/10.1016/j.carbpol.2016.11.090.
[45] A. Kamal-Eldin, N. George, B. Sobti, N. AlRashidi, S. Ghnimi, A.A. Ali, A.A. M. Andersson, R. Andersson, A. Antony, F. Hamed, Dietary fiber components, microstructure, and texture of date fruits (*Phoenix dactylifera*, L.), Sci. Rep. 10 (2020) 21767, https://doi.org/10.1038/s41598-020-78713-4.
[46] L. Medina-Torres, E.B. Fuente, B. Torrestiana-Sanchez, R. Katthain, Rheological properties of the mucilage gum (*Opuntia ficus* indica), Food Hydrocoll. 14 (2000) 417–424, https://doi.org/10.1016/S0268-005X(00)00015-1.
[47] C. Silva, M.D. Torres, F. Chenlo, R. Moreira, Rheology of aqueous mixtures of tragacanth and guar gums: effects of temperature and polymer ratio, Food Hydrocoll. 69 (2017) 293–300, https://doi.org/10.1016/j.foodhyd.2017.02.018.
[48] V. Kontogiorgos, I. Margelou, N. Georgiadis, C. Ritzoulis, Rheological characterization of okra pectins, Food Hydrocoll. 29 (2012) 356–362, https://doi.org/10.1016/j.foodhyd.2012.04.003.
[49] X. Zhang, J. Lin, F. Pi, T. Zhang, C. Ai, S. Yu, Rheological characterization of RG-I chicory root pectin extracted by hot alkali and chelators, Int. J. Biol. Macromol. 164 (2020) 759–770, https://doi.org/10.1016/j.ijbiomac.2020.07.020.
[50] I.M. Sims, A.M. Gane, D. Dunstan, G.C. Allan, D.V. Boger, L.D. Melton, A. Bacic, Rheological properties of xyloglucans from different plant species, Carbohydr. Polym. 37 (1998) 61–69, https://doi.org/10.1016/S0144-8617(97)00105-7.
[51] H. Zhang, J. Chen, J. Li, L. Yan, S. Li, X. Ye, D. Liu, T. Ding, R.J. Linhardt, C. Orfila, S. Chen, Extraction and characterization of RG-I enriched pectic polysaccharides from mandarin citrus peel, Food Hydrocoll. 79 (2018) 579–586, https://doi.org/10.1016/j.foodhyd.2017.12.002.
[52] L.P. Martínez-Padilla, Rheology of liquid foods under shear flow conditions: recently used models, J. Texture Studies 55 (2024) e12802, https://doi.org/10.1111/jtxs.12802.
[53] C. Gamonpilas, A. Kongjaroen, P. Methacanon, The importance of shear and extensional rheology and tribology as the design tools for developing food thickeners for dysphagia management, Food Hydrocoll. 140 (2023) 108603, https://doi.org/10.1016/j.foodhyd.2023.108603.
[54] Y. Cheng, K. Liang, Y. Chen, W. Gao, X. Kang, T. Li, B. Cui, Effect of molecular structure changes during starch gelatinization on its rheological and 3D printing properties, Food Hydrocoll. 137 (2023) 108364, https://doi.org/10.1016/j.foodhyd.2022.108364.
[55] T.M.M. Bernaerts, C. Kyomugasho, N. van Looveren, L. Gheysen, I. Foubert, M. E. Hendrickx, A.M. van Loey, Molecular and rheological characterization of different cell wall fractions of Porphyridium cruentum, Carbohydr. Polym. 195 (2018) 542–550, https://doi.org/10.1016/j.carbpol.2018.05.001.
[56] Z. Qin, H.-M. Liu, T.-T. Lv, X.-D. Wang, Structure, rheological, thermal and antioxidant properties of cell wall polysaccharides from Chinese quince fruits, Int. J. Biol. Macromol. 147 (2020) 1146–1155, https://doi.org/10.1016/j.ijbiomac.2019.10.083.
[57] K. Nishinari, Rheological and thickening properties, in: Y. Fang, H. Zhang, K. Nishinari (Eds.), Food Hydrocolloids, Springer Singapore, Singapore, 2021, pp. 75–117.
[58] G.A. Morris, T.R. Patel, D.R. Picout, S.B. Ross-Murphy, A. Ortega, J. La Garcia de Torre, S.E. Harding, Global hydrodynamic analysis of the molecular flexibility of galactomannans, Carbohydr. Polym. 72 (2008) 356–360, https://doi.org/10.1016/j.carbpol.2007.08.017.
[59] G.A. Morris, J.G. Torre, A. Ortega, J. Castile, A. Smith, S.E. Harding, Molecular flexibility of citrus pectins by combined sedimentation and viscosity analysis, Food Hydrocoll. 22 (2008) 1435–1442, https://doi.org/10.1016/j.foodhyd.2007.09.005.
[60] M.S. Izydorczyk, C.G. Biliaderis, Effect of molecular size on physical properties of wheat arabinoxylan, J. Agric. Food Chem. 40 (1992) 561–568, https://doi.org/10.1021/jf00016a006.
[61] P.A. Williams, G.O. Phillips, Introduction to Food Hydrocolloids, Handbook of Hydrocolloids, Elsevier, in, 2021, pp. 3–26.
[62] P. Himashree, A.S. Sengar, C.K. Sunil, Food thickening agents: sources, chemistry, properties and applications - a review, Int. J. Gastron. Food Sci. 27 (2022) 100468, https://doi.org/10.1016/j.ijgfs.2022.100468.
[63] X. Yang, A. Li, X. Li, L. Sun, Y. Guo, An overview of classifications, properties of food polysaccharides and their links to applications in improving food textures, T Trends Food Sci. Technol. 102 (2020) 1–15, https://doi.org/10.1016/j.tifs.2020.05.020.
[64] L. Guo, W. Yokoyama, L. Chen, F. Liu, M. Chen, F. Zhong, Characterization and physicochemical properties analysis of konjac glucomannan: implications for structure-properties relationships, Food Hydrocoll. 120 (2021) 106818, https://doi.org/10.1016/j.foodhyd.2021.106818.
[65] S. Gillet, M. Aguedo, R. Petrut, G. Olive, P. Anastas, C. Blecker, A. Richel, Structure impact of two galactomannan fractions on their viscosity properties in dilute solution, unperturbed state and gel state, Int. J. Biol. Macromol. 96 (2017) 550–559, https://doi.org/10.1016/j.ijbiomac.2016.12.057.
[66] A.G. Sousa, H.L. Nielsen, I. Armagan, J. Larsen, S.O. Sørensen, The impact of rhamnogalacturonan-I side chain monosaccharides on the rheological properties of citrus pectin, Food Hydrocoll. 47 (2015) 130–139, https://doi.org/10.1016/j.foodhyd.2015.01.013.
[67] K. Chalah, A. Benmounah, M. Mahdad, R. Kheribet, Rheological study of sodium carboxymethylcellulose: effect of concentration and molecular weight, Mater. Today: Proc. 53 (2022) 185–190, https://doi.org/10.1016/j.matpr.2021.12.502.
[68] L.C. Ampese, L.S. Buller, Y.M. Monroy, M.P. Garcia, A.R. Ramos-Rodriguez, T. Forster-Carneiro, Macaúba's world scenario: a bibliometric analysis, Biomass Conv. Bioref. 13 (2023) 3329–3347, https://doi.org/10.1007/s13399-021-01376-2.
[69] Y. Yue, B. Wang, W. Xi, X. Liu, S. Tang, X. Tan, G. Li, L. Huang, Y. Liu, J. Bai, Modification methods, biological activities and applications of pectin: a review, Int. J. Biol. Macromol. 253 (2023) 127523, https://doi.org/10.1016/j.ijbiomac.2023.127523.
[70] K. El Bourakadi, F.-Z. Semlali, M. Hammi, M. El Achaby, A review on natural cellulose fiber applications: empowering industry with sustainable solutions, Int. J. Biol. Macromol. 281 (2024) 135773, https://doi.org/10.1016/j.ijbiomac.2024.135773.
[71] M. Peydayesh, M. Bagnani, W.L. Soon, R. Mezzenga, Turning food protein waste into sustainable technologies, Chem. Rev. 123 (2023) 2112–2154, https://doi.org/10.1021/acs.chemrev.2c00236.
[72] A.Y. Malkin, S.R. Derkach, V.G. Kulichikhin, Rheology of gels and yielding liquids, Gels 9 (2023), https://doi.org/10.3390/gels9090715.
[73] G. Stojkov, Z. Niyazov, F. Picchioni, R.K. Bose, Relationship between structure and rheology of hydrogels for various applications, Gels 7 (2021), https://doi.org/10.3390/gels7040255.
[74] C. Sandolo, P. Matricardi, F. Alhaique, T. Coviello, Effect of temperature and cross-linking density on rheology of chemical cross-linked guar gum at the gel point, Food Hydrocoll. 23 (2009) 210–220, https://doi.org/10.1016/j.foodhyd.2008.01.001.
[75] H.M. Moreno, F. Domínguez-Timón, M.T. Díaz, M.M. Pedrosa, A.J. Borderías, C. A. Tovar, Evaluation of gels made with different commercial pea protein isolate: rheological, structural and functional properties, Food Hydrocoll. 99 (2020) 105375, https://doi.org/10.1016/j.foodhyd.2019.105375.
[76] P.V. Gadkari, M.J.T. Reaney, S. Ghosh, Assessment of gelation behaviour of fenugreek gum and other galactomannans by dynamic viscoelasticity, fractal analysis and temperature cycle, Int. J. Biol. Macromol. 126 (2019) 337–344, https://doi.org/10.1016/j.ijbiomac.2018.12.132.
[77] W. Sittikijyothin, D. Torres, M.P. Gonçalves, Modelling the rheological behaviour of galactomannan aqueous solutions, Carbohydr. Polym. 59 (2005) 339–350, https://doi.org/10.1016/j.carbpol.2004.10.005.
[78] A. Alghooneh, S.M.A. Razavi, F. Behrouzian, Rheological characterization of hydrocolloids interaction: a case study on sage seed gum-xanthan blends, Food Hydrocoll. 66 (2017) 206–215, https://doi.org/10.1016/j.foodhyd.2016.11.022.
[79] L. Feng, Y. Zhou, T.J. Ashaolu, F. Ye, G. Zhao, Physicochemical and rheological characterization of pectin-rich fraction from blueberry (*Vaccinium ashei*) wine pomace, Int. J. Biol. Macromol. 128 (2019) 629–637, https://doi.org/10.1016/j.ijbiomac.2019.01.166.
[80] P. Shao, M. Qin, L. Han, P. Sun, Rheology and characteristics of sulfated polysaccharides from chlorophytan seaweeds *Ulva fasciata*, Carbohydr. Polym. 113 (2014) 365–372, https://doi.org/10.1016/j.carbpol.2014.07.008.
[81] G. Sworn, Xanthan gum, in: Handbook of Hydrocolloids, Elsevier, 2021, pp. 833–853.
[82] L.-Y. Li, Y.-X. Wang, T. Zhang, J.-F. Zhang, M. Pan, X.-J. Huang, J.-Y. Yin, S.-P. Nie, Structural characteristics and rheological properties of alkali-extracted arabinoxylan from dehulled barley kernel, Carbohydr. Polym. 249 (2020) 116813, https://doi.org/10.1016/j.carbpol.2020.116813.
[83] L. Wang, L. Zhang, S. Qiu, C. Liu, P. Zhang, L. Yin, F. Chen, Rheological properties and structural characteristics of arabinoxylan hydrogels prepared from three wheat bran sources, J. Cereal Sci. 88 (2019) 79–86, https://doi.org/10.1016/j.jcs.2019.05.003.
[84] L. Liu, M. Chen, T.E. Coldea, H. Yang, H. Zhao, Emulsifying properties of arabinoxylans derived from brewers' spent grain by ultrasound-assisted extraction: structural and functional properties correlation, Cellul 30 (2023) 359–372, https://doi.org/10.1007/s10570-022-04912-z.
[85] D. Agarwal, L. Hewson, T.J. Foster, A comparison of the sensory and rheological properties of different cellulosic fibres for food, Food Funct. 9 (2018) 1144–1151, https://doi.org/10.1039/c7fo01495c.
[86] Y. Numata, H. Kono, A. Mori, R. Kishimoto, K. Tajima, Structural and rheological characterization of bacterial cellulose gels obtained from *Gluconacetobacter* genus, Food Hydrocoll. 92 (2019) 233–239, https://doi.org/10.1016/j.foodhyd.2019.01.060.
[87] K. Kristensen, G. Warne, D. Agarwal, T.J. Foster, Effects of different moisture contents on the structural and functional properties of cellulose with cell wall

components in different citrus fibres, Food Funct. 13 (2022) 2756–2767, https://doi.org/10.1039/D1FO02808A.
[88] M. Ulbrich, E. Flöter, Impact of high pressure homogenization modification of a cellulose based fiber product on water binding properties, Food Hydrocoll. 41 (2014) 281–289, https://doi.org/10.1016/j.foodhyd.2014.04.020.
[89] S.A. Siddiqui, T. Alvi, A. Biswas, S. Shityakov, T. Gusinskaia, F. Lavrentev, K. Dutta, M.K.I. Khan, J. Stephen, M. Radhakrishnan, Food gels: principles, interaction mechanisms and its microstructure, Crit. Rev. Food Sci. Nutr. 63 (2023) 12530–12551, https://doi.org/10.1080/10408398.2022.2103087.
[90] M. Rinaudo, Main properties and current applications of some polysaccharides as biomaterials, Polym. Int. 57 (2008) 397–430, https://doi.org/10.1002/pi.2378.
[91] K. Nishinari, R. Takahashi, Interaction in polysaccharide solutions and gels, Curr. Opin. Colloid Interface Sci. 8 (2003) 396–400, https://doi.org/10.1016/S1359-0294(03)00099-2.
[92] A. Ström, E. Schuster, S.M. Goh, Rheological characterization of acid pectin samples in the absence and presence of monovalent ions, Carbohydr. Polym. 113 (2014) 336–343, https://doi.org/10.1016/j.carbpol.2014.06.090.
[93] I. Ratcliffe, P.A. Williams, C. Viebke, J. Meadows, Physicochemical characterization of konjac glucomannan, Biomacromolecules 6 (2005) 1977–1986, https://doi.org/10.1021/bm0492226.
[94] A.K.A.S. Brun-Graeppi, C. Richard, M. Bessodes, D. Scherman, T. Narita, G. Ducouret, O.-W. Merten, Study on the sol–gel transition of xyloglucan hydrogels, Carbohydr. Polym. 80 (2010) 555–562, https://doi.org/10.1016/j.carbpol.2009.12.026.