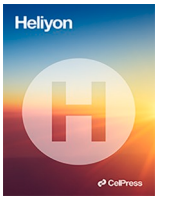
Heliyon 10 (2024) e25582

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon



Research article

Characterization of a composite based on *Cissus dinklagei* tannin resin

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A R T I C L E I N F O

Keywords:
Cissus dinklagei tannin
Adhesive
Particleboard
Thermomechanics
Resins
Composite materials

A B S T R A C T

The tannin extract of *Cissus dinklagei* was used in the preparation of a 3 % paraformaldehyde resin for the manufacture of particleboard. This tannin is of the procyanidin type associated with furan residues. The modulus of elasticity of the resin obtained after the thermomechanical analysis is 3825 MPa. The TGA performed on the panels obtained shows three degradation zones with a thermal stability zone between 74 and 210 °C. These panels have good thermomechanical properties. The values of the best density, internal bond, modulus of elasticity in flexion (MOE) and resistance to flexion (MOR) are respectively 658 kg/m^3; 0.52 MPa; 2035.4 MPa; 16.3 MPa. These results classify this panel for generalinterior construction and furniture uses according to the NF EN 312 standard.

1. Introduction

Wood, a very rich and abundant resource in Central Africa in general and in Cameroon in particular, is composed of polymeric materials such as cellulose, hemicelluloses and lignin. Forest products such as *Sapelli*, *Iroko*, *Ayous*, *Tali* and *Azobe* are transformed into semi-finished products in industrial sawmills. These products generate large volumes of waste, leading to a real problem of environmental pollution and waste management. However, in Europe, this waste is recovered in the production of panels used in the furniture and building industry. However, panels used for interior and industrial applications are bonded with synthetic resins, such as

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https://doi.org/10.1016/j.heliyon.2024.e25582
Received 7 May 2023; Received in revised form 27 January 2024; Accepted 30 January 2024
Available online 4 February 2024

urea formaldehyde, or melamine-urea-formaldehyde [1,2]. Despite their good mechanical properties and ease of processing, these resins have for some time been classified as carcinogenic and hazardous to humans and their environment [3,4]. In view of the above, many researchers have been racing to develop new technologies using forest biomass, namely tannins and particles, in value-added products [5–9]. Tannins are found in bark (or wood) [2,10–12], fruits, leaves [13] and in some lianas. This is the case of the *Cissus dinklagei* liana [14,15] from which tannin has been extracted [16]. *Cissus dinklagei* is a woody vine with simple tendrils opposite the leaves in the Vitaceae family. Its cylindrical stem can reach up to 5 cm in length, and is between 25 and 30 m long [15]. It grows best near wetlands and watercourses. It is found in Cameroon, the Democratic Republic of Congo (D.R.C.), Gabon and Angola. Its leaves are eaten as a vegetable in Gabon, and the cut stems exude a copious, clear, watery sap that is drunk in Gabon and Cameroon. The fibers are used as rope and bath and household sponges. In Equatorial Guinea, it is given to infants to stimulate growth. It is found in abundance in all Cameroonian forests, with a strong predominance in the center and south of the country. It has several recognized pharmacological effects: antispasmodic, anti-inflammatory and anti-carcinogenic [14,15]. In most cases, the bark from these trees is thrown away as waste. So, valuing this waste will enable us to densify the source of tannin extracts. Tannin-based resins have gained popularity in recent decades because of their good properties as binders, although their properties are not superior to those of synthetic resins [6, 17–22]. Investigations by Ref. [23] on the mechanical performance of particleboards based on natural hardeners and tannin showed that particleboards made with these bio-hardeners, especially *Vachellia nilotica* exudates, had better mechanical and physical properties than those made with paraformaldehyde. Similarly [24], evaluated the thermal conductivity and internal cohesion of two panels developed from *Azadiracha* indica tannin resin, with *Vachelia nilotica* exudate and glyoxal as bio-hardeners. It was found that panels containing *Vachellia nilotica* have better internal cohesion and better thermal bio-insulators than those using glyoxal. The work of [5, 22,25–27] show good mechanical properties of high-density bio-composites developed from natural fibers and tannin resin. This work is a first opportunity to valorize local biomass in the densification of the supply of the production of high value-added products that can be beneficial to society from an economic and ecological point of view.

The aims of this article are firstly to extract and characterize tannin from the bark of *Cissus dinklagei using* MALDI-ToF FT-MIR and 13CNMR. On other hand, to formulate a resin with this paraformaldhyde-cured tannin and evaluate its properties, and finally to manufacture particle-based composite panels bonded with this tannin resin and evaluate their performance.

2. Materials and methods

2.1. Cissus dinklagei

Cissus dinklagei bark used for tannin extraction is harvested in the forest of Kelle Mpeck village, Messondo district, Nyong et Kelle department, Centre Cameroon region, from November to December 2022. 10-year-old *Cissus dinklagei* (CD) stems, cut into pieces and still moist, are separated from the bark by light beating. The bark is then sun-dried for two days, crushed and preserved in plastic bags.

2.2. Extraction of tannin

Approximately 300 g of previously ground bark of the *Cissus dinklagei* liana is poured into a cellulose cartridge and introduced into a soxhlet. A prepared water/acetone solution (1:2 v/v) is introduced into a 2000 ml flask to which 1 g of sodium bisulfite is added. The whole is heated to a temperature of 70 °C for 4 h with a frequency of seven (7) siphonings per hour. The resulting substrate was concentrated using a Heidolph steam roaster at a temperature of 60 °C and then lyophilized [28]. Chemical reagents, notably acetone and paraformaldehyde, are supplied by Aldrich France, and sodium bisulfite by Labkem France.

2.3. Characterization of tannin

2.3.1. Attenuated total reflectance Fourier transform middle infrared analysis (ATR-FT MIR)

A mass of 2 mg of fine powder of tannin extract is introduced at the diamond crystal/ZnSe of the BRUKER IR spectrophotometer analyzer. A manual force of about 150 N is exerted on the sample to ensure contact. The sample was scanned 5 times, and each spectrum obtained after 32 scans with a resolution between 4000 and 600 cm^{-1} [6,10,17,29].

2.3.2. Analysis by matrix assisted laser desorption ionization time of fligth mass spectrometry (MALDI-TOF MS)

Two solutions of acetone were prepared. The first consisted in dissolving the tannin extract in a solution of acetone in the proportion of (5 mg/ml). The second was to introduce the matrix (2, 5-dihydroxybenzoic acid) in a solution of acetone in the proportion (10 mg/ml). Both solutions were mixed in a 50/50 ratio to form a mixture (A). NaCl was added subsequently to accelerate the formation of the ions. 0.5–1 µl of the mixture (A) was taken and placed on a plate (around a spot). After evaporation of the solvent for a few minutes in the free area, the wafer was introduced into the spectrometer for treatment. The MALDI-TOF spectra were obtained using COMPACT MALDI AXIMA PERFORMANE TOF2 software [6,17,20,23,29–33].

2.3.3. Solid state cross-polarization magic angle spinning carbon 13 nuclear magnetic resonance (CP MAS ^{13}CNMR)

Tannin extract from *Cissus dinklagei* was prepared in solid form: Cross-Polarization Andle Spinning Magic (CP-MAS). Carbon Nuclear Magnetic Resonance spectra (^{13}C NMR) were carried out using a Bruker 400 MHz MSL spectrometer (BRUKER Biospin, Wissembourgfrace). Chemical changes were calculated relative to TMS. The spectra were recorded at a rotor rotation speed of 12 KHZ on a 4 mm double movement controlled by the Bruker probe. The spectrum was acquired with a recycled delay of 5s at 90° pulsation of

4.2μs with a contact time of 1 ms. The transition number was 3000. The spectrum was started with the deletion of one side of the wire band [10,17,20,23].

2.4. Formulation and characterization of the tannin resin

A resin composed of 60 % water, 37 % tannin and 3 % paraformaldehyde were formulated. The principle consisted in mixing these elements in a beaker and then adjusting the pH of the mixture to 7, by adding a 33 % sodium hydroxide (NaOH) solution. The viscosity of the resin was measured with a Brookfield RV viscometer at 25 °C.

2.4.1. Thermomechanical analysis (TMA)

The thermomechanical analysis of the tannin resin of *Cissus dinklagei* consists of applying 25 mg of the previously prepared sticky mixture to two beech wood plates of dimensions 21 × 6 × 1.1 mm^3 each. After applying the sticky mixture to the two plates, they are directly joined together and introduced into the oven of the thermomechanical analyzer. The humidity of the wood plates is between 8 and 12 %. The temperature of the analysis varied between 25 °C and 250 °C with a maximum gradient of 10 °C/min with a Mettler Toledo 40 TMA equipment (Mettler-Toledo, Zurich, Switzerland). They were tested in three-point flexion over a range of 18 mm by applying a force cycle of 0.1/0.5 N on the specimens, each force cycle of 12 s (6 s/6 s). The test was duplicated five times and the mean value was reported [6,18].

2.4.2. Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) of the resin and particleboard was carried out using a NETZSCH STA 449F3 instrument. 10 mg of the sticky mixture, previously oven dried and finely ground, was placed in an aluminium crucible, and then introduced into the instrument under a temperature rise from 20 to 900 °C, with a rate of rise of 5 °C/min, and an isotherm at 900 °C for 30 min under air. The results are reading using Platinun Evaluation V1.0.182 software [34].

2.5. Manufacture and characterization of particle boards

2.5.1. Particle size distribution

Particles of triplochiton scleroxylon commonly known as *Ayous*, from the Société Industrielle de MBANG (SIM) Yaoundé (Cameroon), were used for the manufacture of particleboard. The average density of *Ayous* is 0.38 kg/m^3, for a moisture content of 12 % (CIRAD forêt 2012). Its mechanical properties are low with a compressive and bending stress at break of 30 and 52 MPa respectively and a longitudinal modulus of elasticity of 7260 MPa [35]. Particle sizes of 0.5 mm and 0.25 mm obtained using a set of EN 210 standard sieves were selected. The particles were then dedusted and dried for 72 h at 200 °C in a MEMMENT hot air oven. The moisture content of the particles after drying was 6 %. The tannin resin was first manufactured and then a previously calculated mass of the particles and the resin were introduced into a sizing machine for 10 min.

2.5.2. Thermo-pressing

The particleboards were manufactured with a sizing rate of 10 % by dry mass of the particles [6,20]. An EMMANUEL manual hydraulic press of up to 100 tons equipped with a heated mould of dimensions 350 × 295 × 75 mm^3 was used to form the particleboards. The forming time was 10 min, with a pressure cycle of 32 kg/cm^2, 15 kg/cm^2 and 5 kg/cm^2 for 5 min, 3 min and 2 min respectively. The compressed panels were cooled in the open air for 30 min, then weighed and cut for further testing. Table 1 shows the pressing parameters.

2.5.3. Mechanical characterization of particleboard

A CONTROLAB universal testing machine from LABOGENIE Yaoundé (Cameroon) with data acquisition was used to perform the three-point bending tests according to EN 310 [36] and the dry internal Bond (IB) in accordance with EN 312-2 [6] [37]. The flexural modulus of elasticity (MOE) and flexural strength (MOR) are calculated according to the following Eqs. (1) and (2):

$$MOE = \frac{L^3}{4 \times b \times e^3} \times \frac{F_2 - F_1}{a_2 - a_1} \quad (1)$$

where *Fi* is the force measured for a displacement *ai*; with F_1 taken at 10 % of the breaking force and F_2 at 40 % of the breaking force; *L* is the distance between the support points; *b* is the width of the specimen and *e* thickness of the specimen in (mm).

$$MOR = \frac{3FL}{2 \times b \times e^2} \quad (2)$$

Table 1
Pressing parameters pressing pressure and time.

Sample	Pressure/kg/cm^2	Time/min
PPFAY0.5	32, 15 and 5	5, 3 and 2
PPFAY0.25	32, 15 and 5	5, 3 and 2

The panels, after a light sanding of the surface, were subjected to an internal bond strength test (IB) and calculated according to the following Eq. (3).

$$\sigma_{IB} = \frac{F}{S}\ (MPa) \tag{3}$$

where F is the breaking force in (N) and S is the stressed surface in (mm).

2.6. Physical characterization

2.6.1. Apparent density

The apparent density of particleboard is determined according to the requirements of standard NF EN 323, on specimens of dimensions 50 × 50 mm^2. The apparent density is calculated by Formula (4).

$$\rho_p = \frac{m_p}{V_p} \tag{4}$$

Panel bulk density where m_p is the mass of the panel and V_p is the panel volume.

3. Results and discussion

3.1. Attenuated total reflactance Fourier transform middle infrared (ATR-FT MIR) analysis

Examination of the ATR-FT spectrum of tannin from *Cissus dinklagei* in Fig. 1, in the 1800-600 cm^{-1} wavelength range has shown the presence of condensed and hydrolysable tannins. This was marked by the peaks at 1700 cm^{-1}; 1608 cm^{-1}; 1559 cm^{-1}; 1518 cm^{-1}; 1443 cm^{-1}; 1368 cm^{-1}; 1284 cm^{-1}; 1201 cm^{-1}; 1110 cm^{-1}; 1065 cm^{-1}; 817 cm^{-1}; 768 cm^{-1}; 668 cm^{-1}; 635 cm^{-1}; 618 cm^{-1}. The 1700 cm^{-1} peak was attributed to the C=O of flavanols. The 1608 cm^{-1} and 1559 cm^{-1} peaks represent the vibrations of the C=C bonds of the aromatic rings [10] or the aromatic structure of the OH group which keeps it in its orthogonal position. The vibrations of the C=C bonds of the aromatic rings, the presence of the catechin, the asymmetric and symmetric stretching of the aromatic C-O, as well as the OH groups of the hydrolysable tannin were associated with the peak 1518 cm^{-1}. The 1443 cm^{-1} peak was assigned to the vibrations of the C=C bonds of the aromatic rings. The peak1368 cm^{-1} was assigned to the aromatic ring and deformation of the C-H bonds of methylene and methyl group [10,38].

Symmetric elongation of the C-OH group of the pyran ring of condensed tannins was represented by the 1284 cm^{-1} peak. The 1201 cm^{-1} peak is occupied by aromatic C-H bending and aliphatic C-OH stretching specific to proanthocyanidins and prodelphinins [10]. The 1110 cm^{-1} peak was assigned to the asymmetric and symmetric stretching of the aromatic C-O bonds characteristic of condensed tannins. The stretching of the C=O bonds of condensed tannins was characterized by the 1065 cm^{-1} peak. The peaks at 817 cm^{-1} and 768 cm^{-1} represent the out-of-plane bending of the aromatic C-H bonds and the out-of-plane bending of the flavonoid B ring, respectively. The peaks at 668 cm^{-1} and 635 cm^{-1} are specific to the OH group of hydrolysable tannin. The 618 cm^{-1} peak is suggestive of phenol C-O bond grouping and C-C bond stretching of benzene rings or aromatic torsion, out-of-plane C-H bending [29]. The presence of hydrolysable tannins was also remarked by the presence of the 1518 cm^{-1} peak.

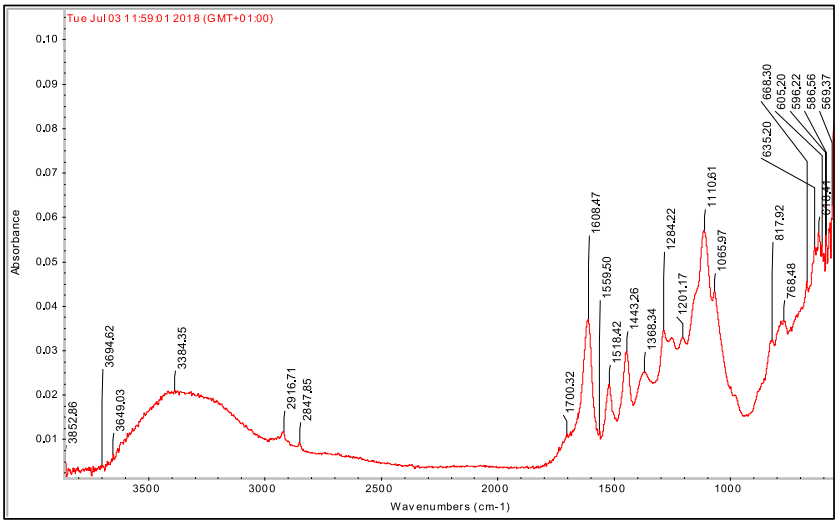

Fig. 1. ATR-FTIR spectrum of soxhlet extracted tannin from *Cissus dinklagei*.

3.2. MALDI-TOF analysis

MALDI-TOF analysis enables us to distinguish between monomers and oligomers present in tannin, and to determine the nature of the tannin. The spectra from the MALDI-TOF analysis of the tannin extract of *Cissus dinklagei* from Fig. 2 show the presence of 8 regular monomers with some traces of furan. These are: catechin, gallocatechin, catechin gallate, fisetinidin, radicinin, chalcone, quercetin and apigenin. Their normal molecular weights are respectively (Fig. 2a): 290.3 Da, 306 Da, 442.4 Da, 274.3 Da, 236.22 Da, 208.25 Da, 270.3 Da. Some are present with their molecular weights increased by 23 Da, due to the use of Na + as an enhancer. Other monomers (Fig. 2b) have lost one or more hydrogen atoms. These are: 579 Da; 607 Da; 611 Da; 645 Da; 782 Da; 880 Da; 960Da; 1076 Da; 1156 Da; others gained one or more hydrogen atoms:430 Da; 456 Da; 502 Da; 523 Da; 527 Da; 529 Da; 551Da; 559 Da; 601 Da; 605 Da; 607 Da; 677 Da; 717 Da; 736 Da; 751 Da; 764 Da; 816 Da; 1043 Da.

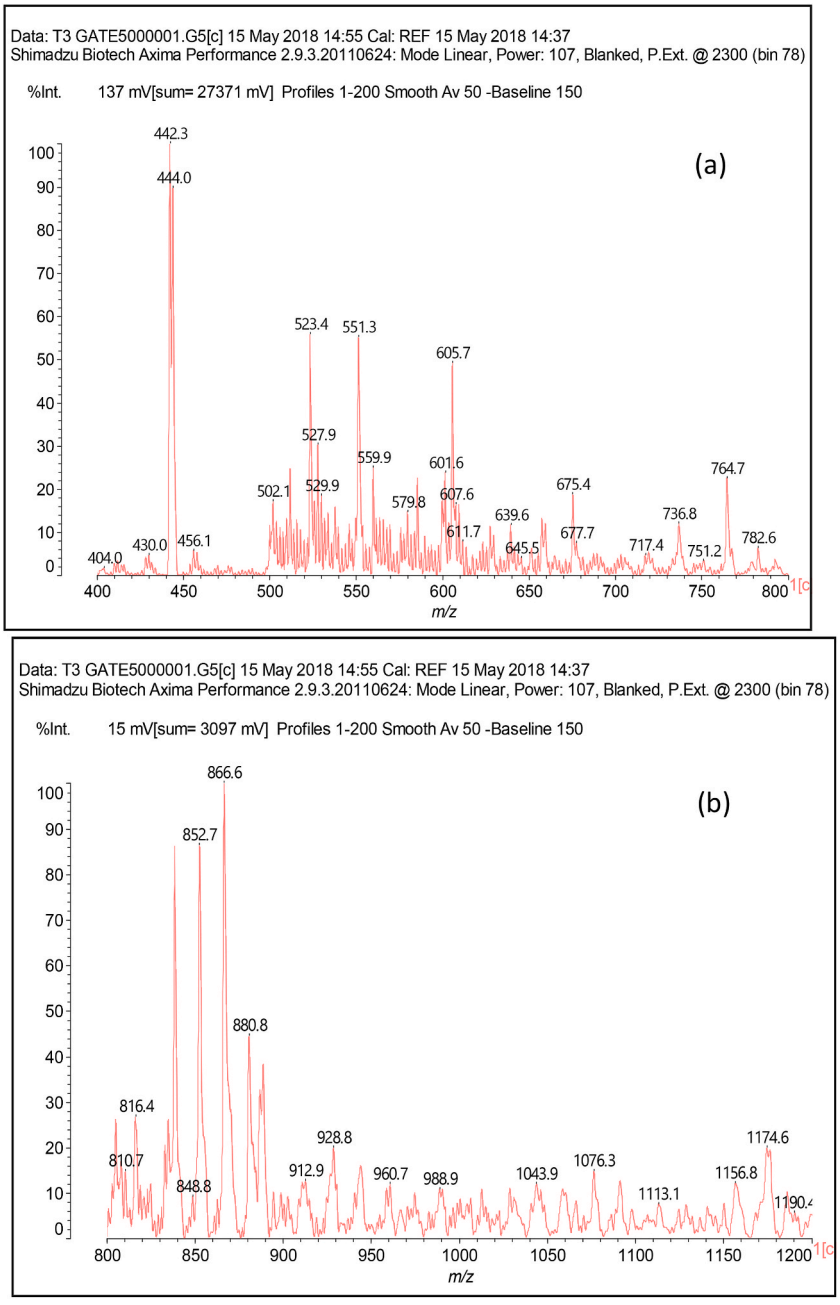

Fig. 2. MALDI-TOF spectrum of *Cissus dinklagei* tannin: (a) 400–800 Da range, (b) 800–1200 Da range.

3.3. Carbon-13 nuclear magnetic resonance spectroscopy (^{13}C NMR)

The ^{13}C-NMR spectrum of *Cissus dinklagei* tannin is shown in Fig. 3. Table 2 summarises the functional shifts of the monomers present in the tannin extract.

The 155.07 ppm peak in the spectrum of *Cissus dinklagei* tannin was assigned to the -OHs carrying C5, C7 and C8 of procyanidins, while the 144.28 ppm peak corresponds to the C3' and C4' resonances of the B-ring of procyanidins [16,17]. The 131.07 ppm peak was assigned to either the C1' of procyanidins or the C4' of prodelphindin [39]. The peak at 116.50 ppm represents C5' and C2' of the B-ring of procyanidins [40]. In contrast, the 106.52 ppm signal was attributed to C6' and C2' or to the C4-C8 interflavonoid bond. The 98.97 ppm shift [40] was associated with C6, C8 and C10, while the carbohydrate residues and stereochemistry of the procyanidin C-ring are represented by the 76.32 ppm peak. The peak located around 72.27 ppm corresponds to C3 of the B ring of flavonoids on the one hand, and to OH associated with flavonoids on the other hand [41]. This peak was also assigned to trace carbohydrates. The 55.82 ppm signal can be attributed to three groups: firstly, to carbohydrates and CH_2-O-CH_2 bond displacements, secondly to the presence of the methoxyl group -OCH_3 and thirdly to flavonoid-associated glucoses [17,42]. The 36.40 ppm peak was assigned to the C1, C5, C7 and C8 of flavonoids, and to the C4-C6 or C4-C8 inter-flavonoid bonds [43], while C3 associated with the OH and C4 bond of catechin are represented by the 33.17 ppm peak [16].

Fig. 4, some structure of the monomers and oligomers found in *Cissus Dinklagei* tannin following MALDI-TOF (Table 2) and ^{13}C-NMR (Table 3).

3.4. Characterization of the tannin resin

3.4.1. Thermomechanical analysis

Fig. 5 Shows the behavior of the MOE modulus of elasticity and the variation of the DMOE of the tannin resin elaborated with 3 % paraformaldehyde as hardener. This resin was analysed by three-point bending and the value of the MOE is calculated by Eq. (5). The maximum value obtained for this resin is 3825 MPa. This value is higher than that obtained for *Aningré* tannin with 5.5 % paraformaldehyde [6].

$$E = \frac{L^3}{4bh^3} \times \frac{F}{(f_{wood} - f_{adhesive})} \tag{5}$$

Each peak in the above curves has a specific meaning. The 43 °C peak of the DMOE indicates the presence of a pseudo gelation reaction, characterized by either cross-linking or most likely physical entanglement due to water loss in the resin. The peak around 102 °C is the glass transition temperature.

3.4.2. Thermogravimetric analysis of resin RTPF40/60

Fig. 6 shows the TG, DTG and DSC curves from the thermogravimetric analysis of the tannin resin of *Cissus dinklagei* RTPF60/40. These curves show remarkable peaks corresponding to the precise degradation of the elements contained in the processed resin.

The 82 °C peak reflects the melting point of the furan residues in the tannin. The peak at 102 °C corresponds to the melting point of paraformaldehyde. The variation in the peak at 300 °C is due to the auto-ignition of paraformaldehyde releasing the following gases: CO_2, CO and CH_4. The peaks that occur between 300 and 700 °C reflect the degradation of the rigid elements contained in the tannin. At the end of the pyrolysis, about 3.91 % of the initial mass remains, corresponding to the mass of the remaining rigid elements

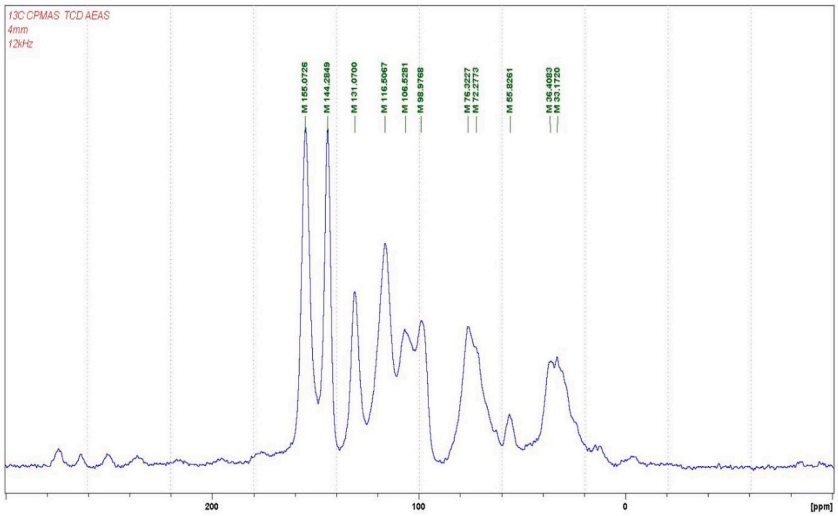

Fig. 3. ^{13}C-NMR spectrum of tannin from *Cissus dinklagei* extracted with Soxhlet.

Table 2
Oligomers present in the tannin of *Cissus dinklagei*.

Da	Peak Intensity (%)	Species	Remarks
404	2	Chalcon + 2 furan (-2H)	Flavonoid monomer
430	4	Apigenin protonated + 2 furan + Na	
442	50	Catechin gallate	Flavonoid monomer
444	90	Radicinin + chalcone	Flavonoid
456	8	Radicinin tetraprotonated + chalcone	Dimer
502	18	Cathechin triprotonated + chalcone triprotonated	
523	58	Cathechin tetraprotonated + chalcone deprotonated + Na^+	
527	30	Myricetin protonated + chalcone	
529	20	Myricetin protonated + chalcon deprotonated	
551	58	Myricetin diprotonated + radicinine + Na^+	
559	28	Quercetin protonated + chalcon + furan	Flavonoid dimer
579	14	Gallocatechine + fisetinidin (-H)	Flavonoid dimer
601	20	Galocatechin + apigenin diprotonated+ Na^+	
605	50	Quercetin dimer protonated	
607	16	Quercetin + gallocatechin (-H)	
611	10	Galocatechin dimer (-H)	
639	12	Quercetin dimer + Na^+ [+OH]	
645	4	chalcone trimer + Na^+ (-2H)	
675	20	Radicinin + chalcon dimer+ Na^+	
677	6	Radicinin diprotonted + chalcon dimer+ Na^+	Flavonoid trimer
717	4	Catechin gallate protonated + fisetinidin	Flavonoid Dimer
736	14	Chalcon dimer + myricetin deprotonated	Flavonoid trimer
751.	4	Catechin gallate pentaprotonated + gallo catechin	Flavonoid dimer
764.	24	Radicinin dimer pentaprotonated + catechin	Flavonoid trimer
782.	8	Radicinin dimer + catechin + Na^+ (-H)	
810	16	Fisetinidin + radicinedin + quercetine	
816	28	Fisetinidin triprotonated + radicinin + gallocatechine	
848	10	Fisetinidin dimer + quercetin	
852	88	Fisetinidin dimer + gallocatechine	Flavonoid trimer
866	100	Catechin + apigenin + gallocatechin	Flavonoid trimer
880	46	Catechin gallate + chalcon dimer + Na+ (-H)	Flavonoid dimer
912	12	Myricetin dimer + fisetinidin deprotonated	Flavonoid trimer
928	20	Myricetin dimer + apigenin+ Na^+	Flavonoid tetramer
960	14	Myricetin dimer + radicinin + furan + Na^+ (-2H)	Flavonoid dimer
988	12	Catechin gallate + chalcone + $2Na^+$(+6H)	
1043	12	Chalcone pentaprotonated + fisetinidin + catechin + apigenin	Flavonoid tetramer
1076	16	Catechin gallate + myricetin + gallocatechin + Na^+ (-2H)	Flavonoid trimer
1113	8	Galocatechin + quercetin + chalcon + fisetinidin+ Na^+	Flavonoid trimer
1156	12	Myricetin + apigenin dimer + chalcon + furan+ Na^+(-H)	Flavonoid trimer
1174	20	Catechin gallate dimer + catechin	
1190	10	Catechin gallate dimer + gallocatechin	

contained in the resin.

3.5. Mechanical and physical properties of particleboard

Density of the boards made from the particle sizes 0.25 mm (PPFAY0.25) and 0.5 mm (PPFAY0.5) are 620 kg/m^3 and 658 kg/m^3 respectively (Table 4). The density of PPFAY0.25 panels does not comply with EN323 standard, while the density of PPFAY0.5 complies with EN312 (2010) standard.

The required internal bond (IB) strength according to the EN319 [44] standard is 0.35 MPa. The panels satisfying this standard were used in an indoor environment. The PPFAY0.25 panels have an internal bond of 0.38 MPa lower than that of the PPFAY0.5 panels which is 0.52 MPa. This IB value is lower than that found by Refs. [6,23]. This difference may be due to the sizing rate in relation to the particle size, the percentage of paraformaldehyde or the forming pressure. However, the IBs of both panels are above the standard and can be used as structural panels. Table 3 shows the MOE and MOR values obtained during the three-point bending test of the different particleboards. These values are in accordance with EN 312 (2010)[37], categories P2 (Panels for interior fittings, including furniture, used in dry conditions) and P4 (Working panels used in dry conditions), at 1900.6 and 2035.4 MPa respectively (Table 4).

The results of 2 h and 24 h cold water thickness swelling in Table 2 show that particleboards made from *Ayous* particles absorb much more water than other wood species, a swelling 5 to 3 times their thickness respectively for 0.25 mm and 0.5 mm size particles. Such behavior was observed in the work of [45]. This high-water absorption capacity of *Ayous* particles is due to the presence of a high proportion of hydroxyl groups [46]. It is also observed that PPFAY0.25 particleboard is soluble in hot water at 2 h compared to PPFAY0.5 which is not. All these values are far from the normative value of the EN 317 standard [47].

TG, DTG and DSC curves from the thermogravimetric analysis of *Cissus dinklagei* tannin resin-based particleboards are shown in Fig. 7. The thermogram shows the pyrolysis of the PPFAY0.5 panels with the best mechanical properties. It shows four ranges of degradation. The first range of mass loss is between 40 and 200 °C. The peak around 74 °C is attributed to the evaporation of the

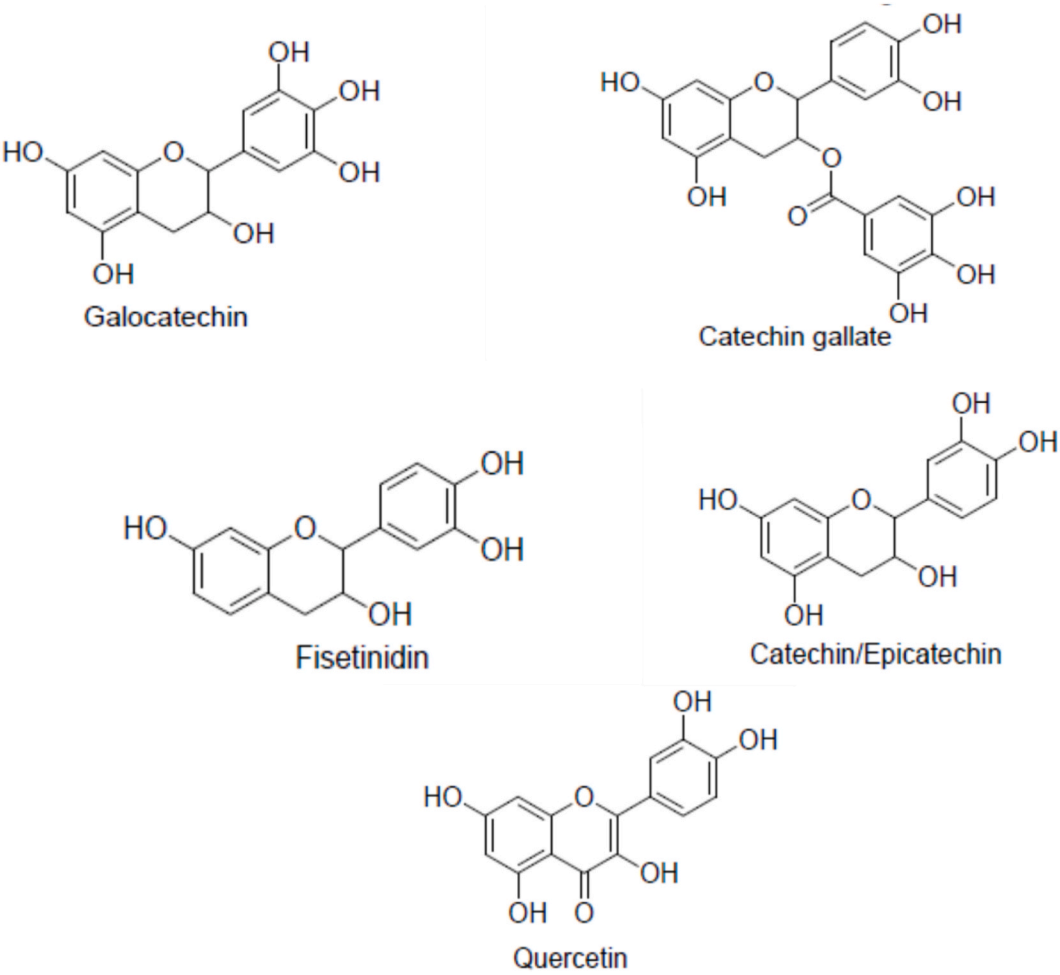

Fig. 4. Structure of the monomers and oligomers found in the tannin of *Cissus dinklagei* following MALDI-TOF and ^{13}C NMR analyses.

Table 3
Summary of the functional group components of the monomers present in the tannin extract of *Cissus dinklagei*.

Peaks (ppm)	Functional components or groupings	Refs.
155,07	OH associated with C5, C7 and C8a of procyanidines	[16,17]
144,28	C3' and C4' resonances of the B-ring of procyanidines (catechin/epicatechin)	[16,17]
131,07	C1' of procyanidins/C4' prodelphinidin	[39]
116,50	C5' and C2' of the B-ring of procyanidins	[40]
106,52	C6' and C2' of catechin or at the C4-C8 flavonoid bonds	[39]
98,97	C6, C8 and C10	[40]
76,32	Carbohydrate residues and the stereochemistry of the C-ring of procyanidins	[40]
72,27	C3 of flavonoid ring B, OH associated with flavonoids and trace carbohydrates	[41]
55,82	Carbohydrates and CH_2-O-CH_2 or presence of the methoxyl group (OCH_3) or glucoses associated with flavonoids	[17,42]
36,40	C1, C5, C7 and C8 flavonoids and C4-C6 or C4-C8 inter-flavonoid bonds	[43]
33,17	C3 associated with the OH bond and C4 of catechin	[16]

volatile organic compounds released by the resin and the wood particles, namely carbon dioxide CO_2 and water H_2O. The second range, between 220 °C and 300 °C, also reflects another loss of mass, with the 289 °C peak corresponding to the degradation of the tannin contained in the resin. This variation also participates in the decomposition of the cellulose from the wood particles, and of the hemicellulose by emitting the following volatile gases: CO, CO_2, CH_4 and CH_3OH. The third range of mass loss is between 300° and 380 °C. This mass loss results in the splitting of the C-O bonds, followed by the maximum decomposition of cellulose and hemicellulose. The peak at 309 °C reveals the total decomposition of tannin and part of the lignin as observed for *Aninigé altissima* tannin [48].

The mass variation between the temperatures 380–480 °C, reflects a total decomposition of the lignin around the 400 °C peak. From 480 °C onwards, we notice an elimination of CO, CO_2, CH_4 gases and coal. In short, a total decomposition of the elements that started

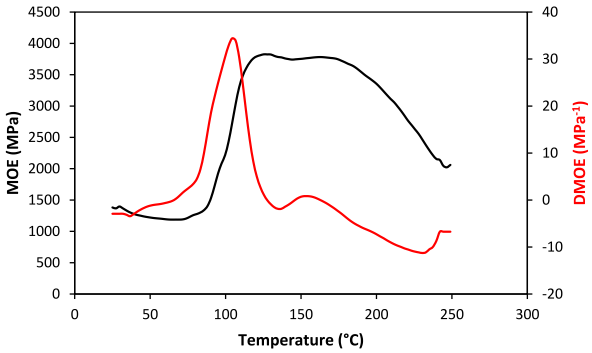

Fig. 5. Variation in MOE and DMOE of RTPF40/60 resin.

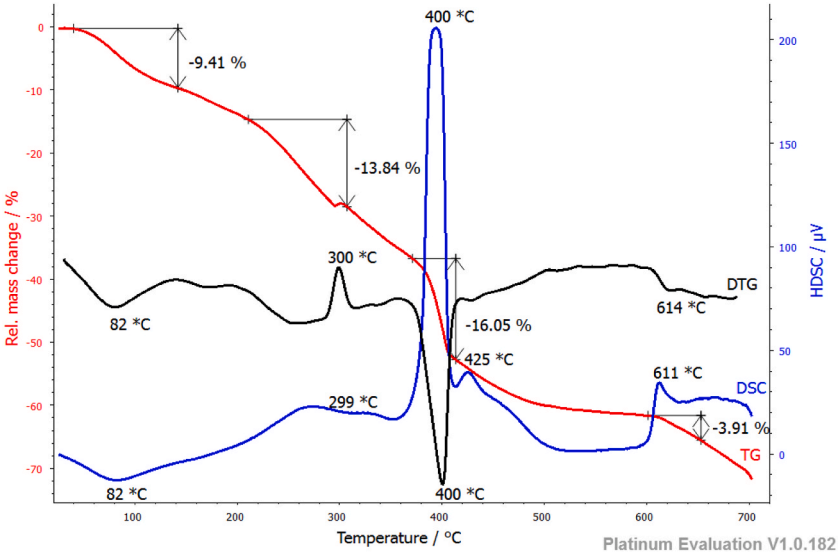

Fig. 6. ATG curve under area of *Cissus dinklagei* RTPF60/40 tannin resin.

Table 4
Mechanical and physical properties of particleboard based from *Cissus dinklagei* tannin and *Ayous* particles.

Particleboard	Density (kg/m^3)	IB (Mpa)	MOE (Mpa)	MOR (Mpa)	Thickness in Swelling (%) 2 h–24 h
PPFAY0.25	620	0.38	1900.6	15.17	63.66–70.92
PPFAY0.5	658	0.52	2035.4	16,3	32.39–48.21
NF EN319	**630–700**	**0.35**	–	–	–
NF EN310	–	–	**1800**	**14**	**16**

their degradation in the previous stages. At the end of the pyrolysis of the panels, about 18 % of the initial mass remains, corresponding to the residues of the solid segments of these panels.

4. Conclusion

The objective of this paper was to evaluate the adhesive potential of tannin from the *Cissus dinklagei* liana and apply it as a binder in particleboard. The soxhlet extraction method used to extract the tannin is not widely used for the purpose, due to the high temperatures that can damage the extract, although the yield is high. The results of the different FT-IR, ^{13}C-NMR and MALDI-TOF analyses allow us to conclude that this tannin is predominantly a condensed tannin. The thermomechanical characteristics of PPFAY0.5 particleboard are superior to those of PPFAY0.25 particleboard made from tannin resin with 3 % paraformaldehyde. PPFAY0.5 panels can be used in dry structural construction and furniture. These results show that this tannin has good adhesive properties.

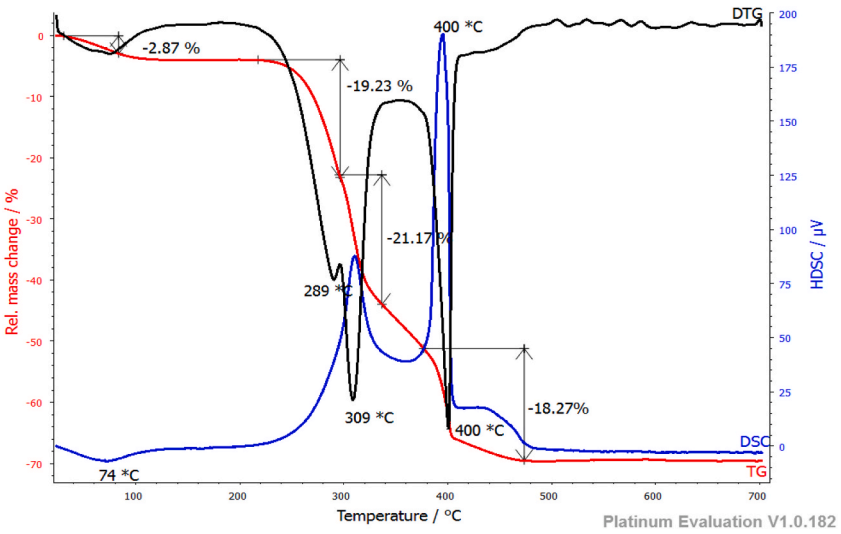

Fig. 7. TGA curve for particleboard based on *Cissus dinklagei* tannin resin.

CRediT authorship contribution statement

Abel Emmanuel Njom: Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Joseph Voufo:** Writing – original draft, Supervision, Software, Investigation. **Cesar Segovia:** Writing – review & editing, Writing – original draft, Visualization, Resources, Data curation, Conceptualization. **Noël Konai:** Writing – review & editing, Writing – original draft, Validation, Supervision, Formal analysis, Conceptualization. **Armel Mewoli:** Writing – review & editing, Writing – original draft, Validation, Software, Formal analysis, Data curation. **Leonnel Karga Tapsia:** Writing – review & editing, Writing – original draft, Visualization, Validation, Methodology, Investigation, Conceptualization. **Jean Raymond Lucien Meva'a:** Writing – review & editing, Writing – original draft, Visualization, Validation, Supervision, Conceptualization. **Antonio Pizzi:** Writing – review & editing, Writing – original draft, Supervision, Formal analysis, Data curation, 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.

Acknowledgements

The authors thank LABOGENIE (Yaoundé, Cameroun), for the mechanical characterization of particleboard products. CETELOR (Centre d'Essais Textile Lorrain) and LERMAB (Laboratoire d'Etudes et de Recherches sur le Matériau Bois) of the University of Lorraine, for the characterization of tannin and tannin resin. Thanks, are also addressed to the authors of the references.

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