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Thermogravimetric and Thermomechanical Analysis of Abrasive Elaborated with *Vitalleria Paradoxa* Tannin Adhesive and *Balanites aegyptiaca* hulls Particules

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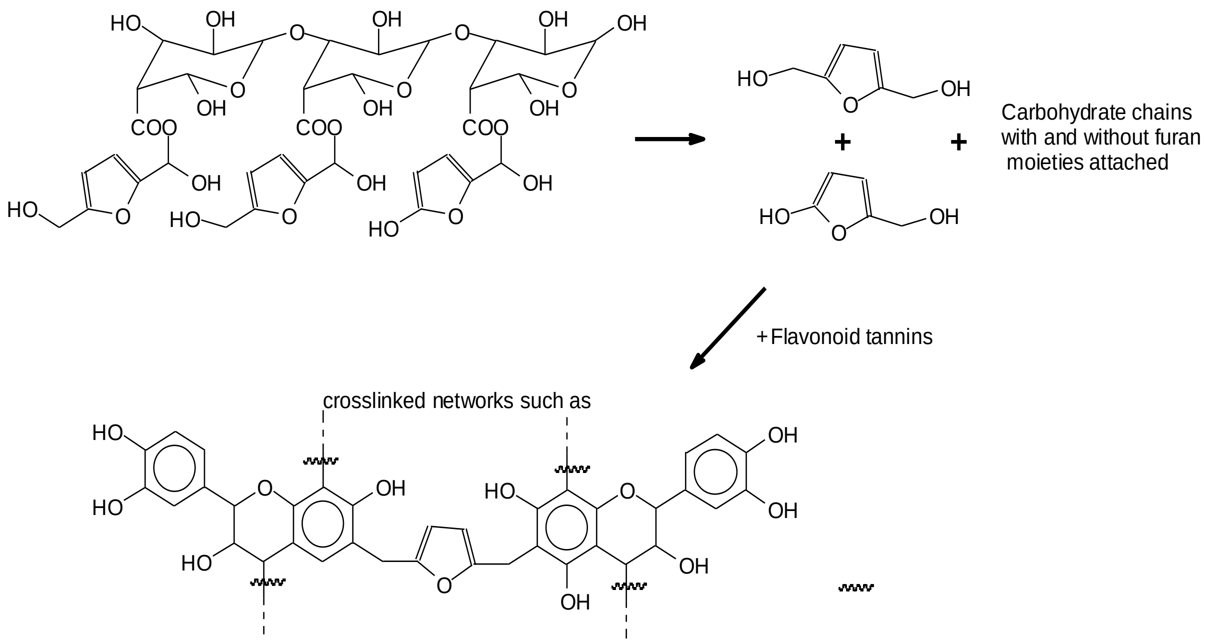
Abstract Performant abrasive was developed with African resources. It was elaborated using an adhesive developed using *Vitalleria paradoxa* tannin bark extract; *Balanites aegyptiaca* hulls particles and quartz particles with a support of banana fibers. Two samples A2 and A1 were elaborated using respectively the *Vitalleria paradoxa* trunk exudate and hexamethylenetetramine (hexamine) as hardeners. The tannin used is a procyanidin with furan residues. The Modulus Elasticity of the A2 adhesive determined by a thermomechanical analysis is higher than that of A1. Their values are respectively 20652 and 12818 MPa. The *Vitalleria paradoxa* trunk exudate used as bio hardener improves the characteristics of abrasives. The HB values of abrasives E1 and E2 are respectively 22, 28 and 27,32 daN/mm^2; their abrasiveness coefficient values are respectively 5,12 .10^{-1} and 7, 28.10^{-1} %. The degradation modes of abrasives E1 and E2 are identically.

Keywords Abrasive, Adhesive, bio hardener, performant

1. Introduction

In the world where oil resources are running out and where there is a very strong demand for plastics and resins, the polymerization and use of natural resources constitute an interesting avenue of research. Wood, which occupies a very important place in the African economy, is considered as a rich resource for the manufacture of biomaterials. Its constituents such as cellulose, hemicelluloses, lignin and tannins are already used to develop adhesives and composites materials. It is with the same idea that several research works have been carried out using tannin [1-2-3]. The work of Ndiwe et al. (2019) [4] showed that the reactive species of bio hardeners were furan hydroxymethyl species linked to carbohydrate oligomers. The bio hardeners analyzed gave 2-hydroxy-5-hydroxymethyl furan and especially 2,5-dihydroxymethyl furan, these last one species being the reactive species leading to cross-linking.



The abrasive industry has undergone a great evolution. Since prehistoric times the abrasive properties of some minerals have been used to sharpen, polish tools, weapons, marble and precious stones. In the 19th century, these natural abrasives (quartz, garnet, emery, corundum) were agglomerated on grinding wheels by binding them with rubber. In the last decades synthetic abrasives (mono- and microcrystalline aluminas, with zirconia; silicon carbide, boron) have been manufactured. These allow the machining of mechanical objects with tight geometrical tolerances and high hardness. Conventional abrasives are reserved for non-precision work by material removal (deburring, cutting off large sections or grinding metals and alloys with a hardness less than 55 HRC (Rockwell hardness). Nowadays, these abrasives are increasingly prepared with local materials. Thus, we can cite the work on grinding wheels based on bio resin matrix and aluminum trioxide or glass sand abrasive [5-6-7] etc. It should be noted that some abrasives have not good thermomechanical properties. It is the case of abrasive grinding wheels presenting low oxidation resistance and poor performance at high temperature [8-9-10].

Thus, the aim of this paper is to obtain performant abrasives developed with African resources and particularly using bio adhesives developed with *Vitalleria paradoxa* tannin and a bio hardener from *Vitalleria paradoxa* exudates, with a mixture of *Balanites aegyptiaca* hulls particles and quartz particles as well as a support made with the *banana* stem fibers. These plants are widespread in central Africa.

The *Balanite aegyptiaca* plant is used in Africa in the wood industry and traditional pharmacopeia, the liquid from its seeds is traditionally used to stimulate the milk production of nursing mothers, to treat stomach pain, against typhoid and to treat skin problems. Its wood is extensively exploited for exterior carpentry. The *Vitalleria paradoxa* is popular for its antioxidant power and gum arabic, its almond nuts are used to make shea butter and as traditional medicine.

2. Materials and Methods

2.1. *Vitalleria paradoxa* tannin extraction

The *Vitalleria paradoxa* crushed tree bark has been placed in an aqueous solution containing powdered bark and with added 2% sodium bisulfite and 0.5% sodium bicarbonate. The volume of water used is six times more than that of the powdered bark. This mixture was continuously stirred at 70 ° C during 4 hours. After that, it was proceeded to the separation and filtration and a reddish liquid was obtained. This liquid was then placed in an oven at 60°C for evaporation during 24 hours to obtain powdered tannin [11-12].

2.2. MALDI-TOF analysis

The sample dissolved in acetone (4 mg/ml) were mixed with an acetone solution (10 mg/ml acetone) of the matrix. 2.5-dihydroxybenzoic acid was used as matrix. The matrix was mixed using NaCl for the enhancement

of ion formation. The samples solutions and the matrix were mixed (50:50) and 0.5–1 l of the resulting solution was placed on the MALDI target. The MALDI target was introduced into the spectrometer after evaporation of the solvent. The analysis was done with MALDI AXIMA PERFORMANCE TOF 2 instruments [12].
Maldi TOF: Matrix Assisted Laser Desorption Ionization Time of Flight

2.3. *Vitalleria paradoxa* hardener extraction

The *Vitalleria paradoxa* tree trunk was incised at several places at 1 cm depth and after a few minutes, an organic liquid of high viscosity appeared. This liquid was protected with a plastic sheet and was left run and dry during two days. It was then collected and dried at ambient temperature during three weeks. Finally, we obtained a dried exudates whose was powdered for easy stocking and usable as a hardener [4]. The *Vitalleria paradoxa* trees used originated from the forest of Zidim in Cameroon.

2.4. Adhesive development

Two adhesive formulations A1 and A2 have been developed (table 2) using respectively the hexamine and the exudate of *Vitellaria paradoxa* as hardener. The principle consisted to mix 35% of tannin extract with 10% of hardener (bio hardener) powdered in 55% of water by adjusting the pH to 12 adding a sodium hydroxide solution (NaOH). It was at this pH that the gluing was good Each formulation was mixed mechanically until the viscosity reached 650 mPa s. It was measured with a Brookfield RV viscometer [2].

2.5. Thermomechanical Analysis of the adhesives

The test was done using a METTLER TOLEDO TMA 40 device (Viroflay, France) linked to a processor TC11 and a computer. About 25 mg of adhesive were placed on two beech wood veneers of dimensions 17×5×1.1 mm, then assembled with the adhesive in the middle to form a beech-adhesive-beech sandwich. This was then placed in the Thermomechanical Analyzer furnace to undergo tests. The samples were then tested in non – isothermal mode between 25°C and 250°C at a heating rate of 10°C/min, according to a procedure already reported [12].

2.6. Abrasive elaboration

The produced abrasive is composed of grains, support, binder and filler. The grains used are those of *Balanite aegyptiaca* hulls and quartz grains cut into triangular right prism of 3 mm side and 4 mm high. The adhesive is elaborated with the *Vitellaria paradoxa* bark tannin. The support is a woven tissue support of *Banana* stem fibers. The fine particles of *Balanite Aegyptiaca* hulls constitute the fillers. The principle is to pour a small quantity of adhesive at the bottom of a mold of 150 mm x 150 mm x 5 mm and then pour the mixture of grains, particles of *Balanite Aegyptiaca* hulls and the quartz, and adhesive mixed in a beaker, on the support placed at the bottom of the mold. The whole mold was then placed in an oven at 60 ° C during 48 hours. After demolding, the abrasive obtained was cut into 50 mm x 50 mm x 5 mm samples ready to use. The compositions of the E1 and E2 samples elaborated respectively with A1 and A2 adhesives are synthesized in Table 1

Table 1: E1 and E2 abrasive formulations

Sample	Adhesive(g)	Grain		Support	Fillers (g)
		Quartz (g)	*Balanites aegyptiaca* hulls (g)	Banana stem Fibers web (g)	(g)
E1	40	10	10	0	11
E2	45	12	12	20	15

2.7. Brinell Hardness (HB)

The Brinell Hardness was determined using an INSTRON device (Norwood, Massachusetts, USA), consisting of a hardened steel ball with a diameter of 10 ± 0.01 mm, a 1/10 mm precision caliper and a comparator to measure the indentation length with an accuracy of ± 0.01 Apply a load on the face of the specimen. After applying the force, measure the depth of penetration (P) of the punch and the residual depth. The HB value is calculated using the formula below:

$$HB = \frac{3.18}{1000}\frac{F}{P}$$

HB: Brinell Hardness; **F**: Necessary strength to determinate the penetration depth (N); P: Penetration depth of the ball/balk (mm)

2.8. Abrasiveness coefficient determination

The test consists to rotate a steel blade in a metal container filled with the grains described above at 4500 revolutions per minute during 5 minutes. The abrasiveness coefficient (Abr) (wear index) is the mass loss of the steel blade at the end of this test. It is the ratio of the blade mass loss to the mass of aggregates that caused it, affected by a factor 1000 (NFP 18-579) [13].

$$Abr = \frac{1000(M_{PO} - M_t)}{M_{GO}}$$

M_{p0}: Abrasive weight before abrasion (mg); M_t : Particle board weight after abrasion (mg) ; M_{G0} :Cycles Number of abrasion record;

2.9. Thermogravimetric analysis

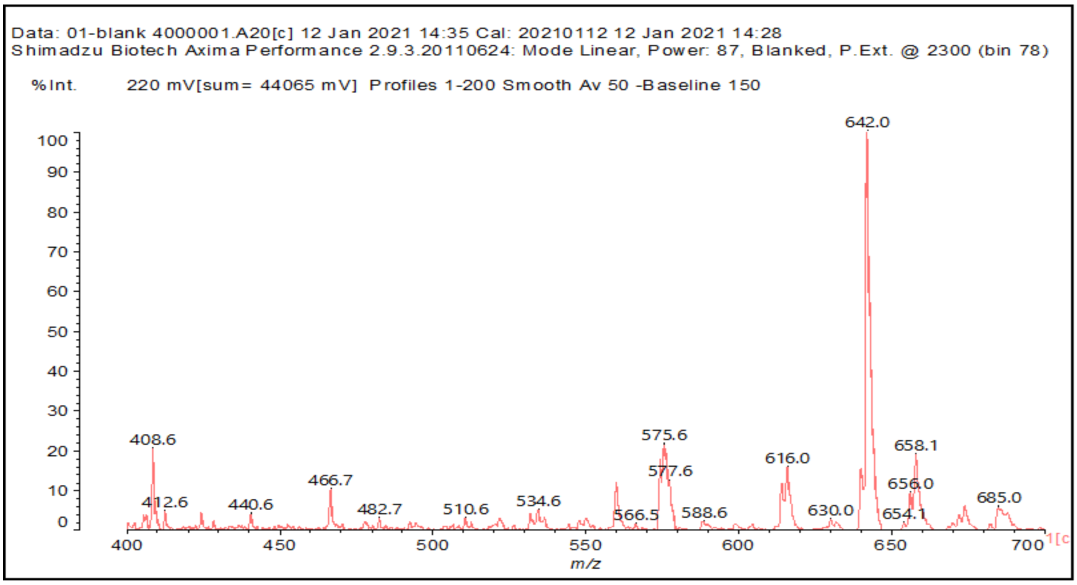
The TGA was done using NETZSCH STA 449F3 Jupiter (Selb, Germany) analyzer. 100 mg of powdered abrasive was placed on a balance located in the furnace of the device and heat was sent over the temperature range between 20 and 900°C during 30 min at a heating rate of 5°C/min in argon gas.

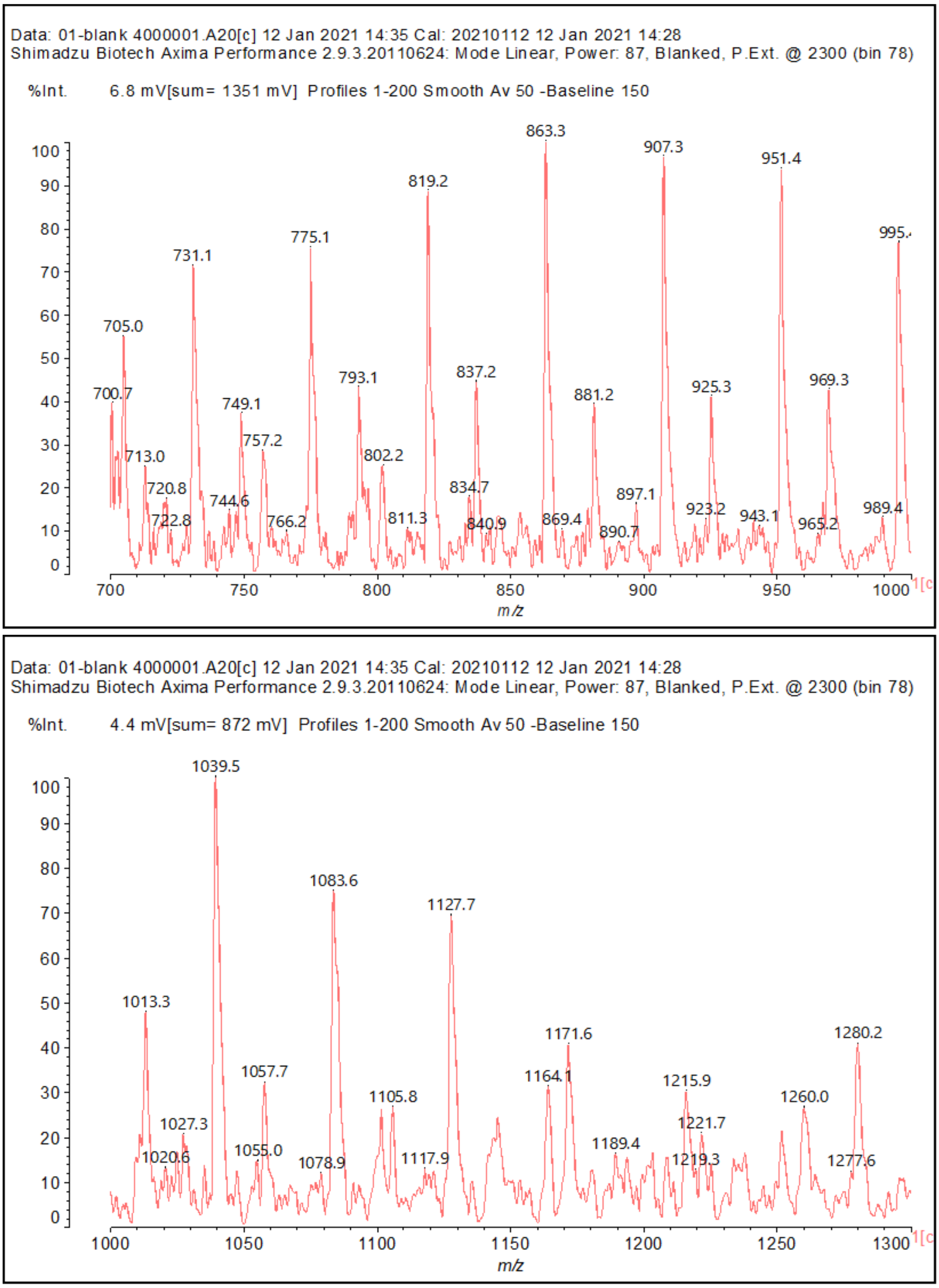
3. Results and discussions

3.1. Tannin extraction and Maldi Tof Analysis

The examination of the Maldi ToF Analysis Spectra (Fig.1) in the range 400 to 2000 Da denoted the presence of several oligomers in the *Vitelllaria Paradoxa* tannin extract. They are consistent with some monomers such as catechin gallate, myricetin, gallocatechin, quercetin, cyanidin, fisetinidin, apigenin chalcone and some furan residues. The molecular weight of these monomers above are summarized in table 1 below and are respectively 442 Da; 318 Da; 306 Da; 302 Da; 287 Da; 274 Da; 270 Da, 208 Da and 68 Da (for furan). Some of these oligomers are protonated and others are linked or not with the Na^+ used to enhance the MALDI spectra. The presence of Na^+ (23 Da) is due to the NaCl used in the matrix. The normal values of different molecular weights are obtained by deducting 23 Da from the value of the different peaks. The structures summarized in fig. 2 can be attributed to the peaks (with or without Na^+) 482 Da ; 616 Da ; 654-656 Da ; 711-713 Da; 802 Da ; 812 Da ; 862 Da ; 880 Da ; 880 Da

From the different monomers present in this tannin extract (table 4), it is clear that this extract is of the condensed type with a predominance of quercetin, chalcon and some furan residues. Thus, it can be used as an adhesive for wood [1].

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%Int. 6.8 mV[sum= 1351 mV] Profiles 1-200 Smooth Av 50 -Baseline 150
m/z
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Shimadzu Biotech Axima Performance 2.9.3.20110624: Mode Linear, Power: 87, Blanked, P.Ext. @ 2300 (bin 78)
%Int. 4.4 mV[sum= 872 mV] Profiles 1-200 Smooth Av 50 -Baseline 150
m/z

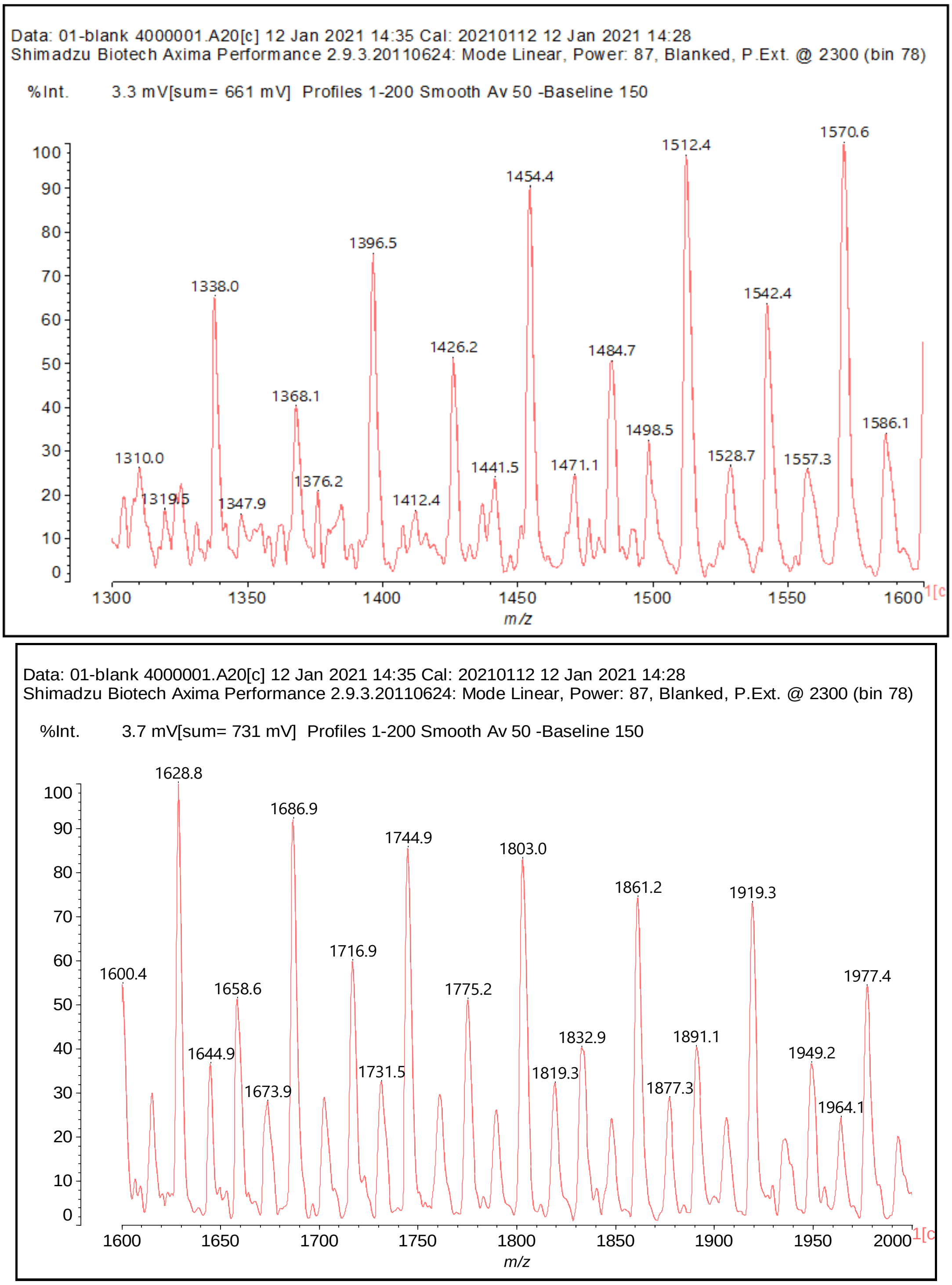
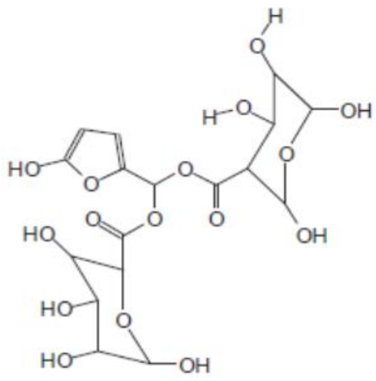
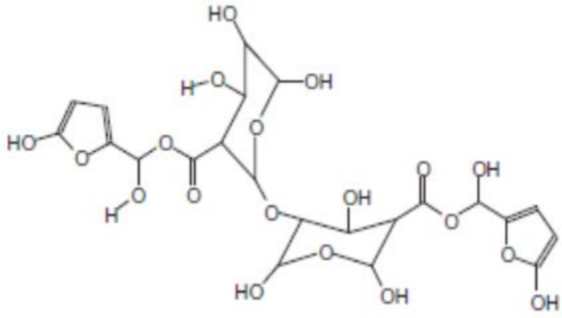


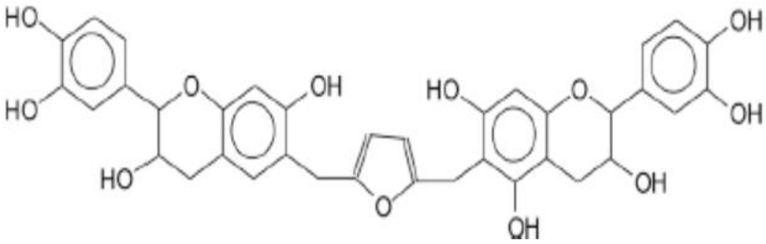
Figure 1: MALDI TOF curve of Vitellaria paradoxa bark tannin extract in the range 400 to 2000 Da



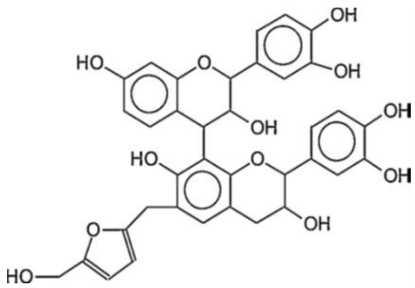
S1=482 Da



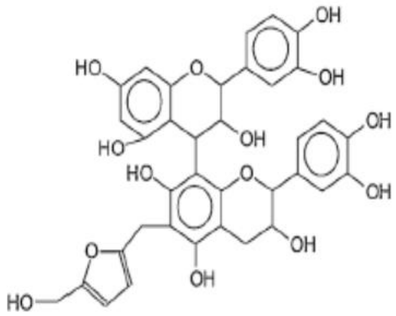
S2=616/617Da



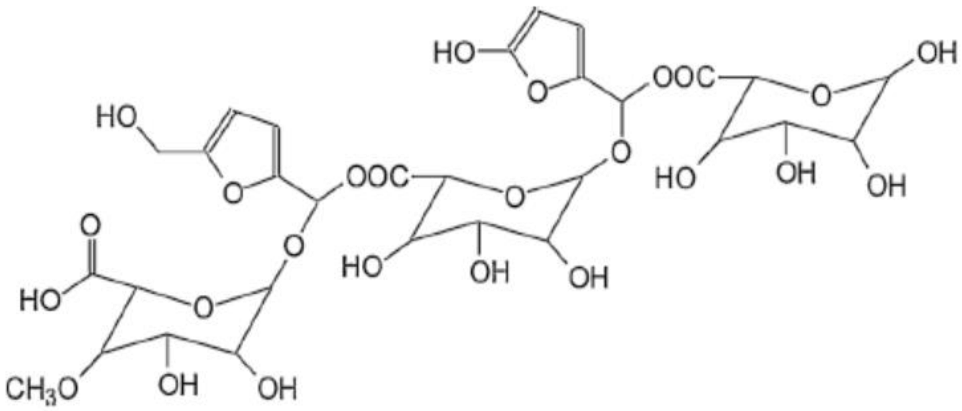
S3=655



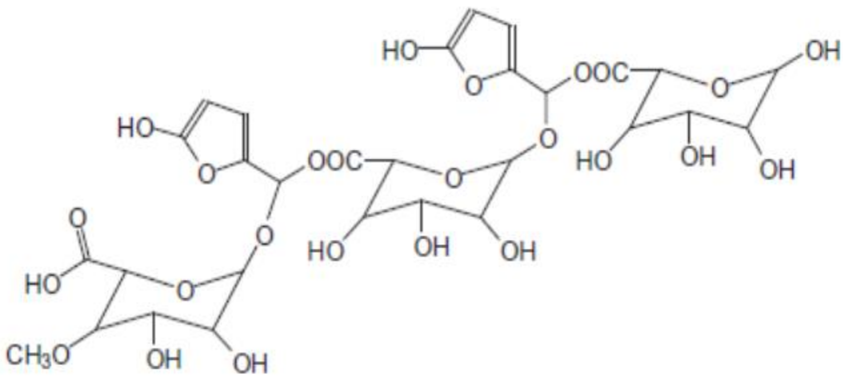
S4=656 Da



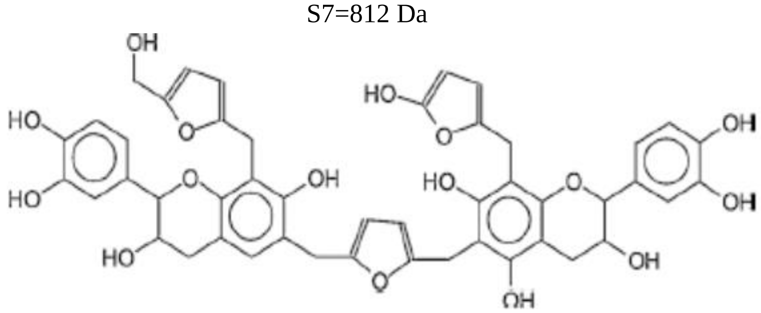
S5=711-713 Da



S6=802 Da



S7=812 Da



S8=862 Da

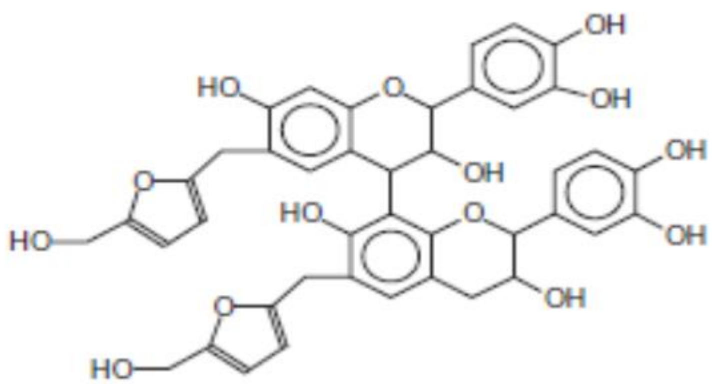


Figure 2: Some structures present in tannin

Table 2: Monomers and oligomers present in tannin extract

A : Monomers ; B : Peaks

A B	442	318	306	302	290	287	274	270	208	68	23	+1	-1
408		1								1	1		1
412							1			2		2	
440									2		1	1	
466	1										1	1	
482							1		1				
510				1					1		1	1	
534				1					1		1	1	
566							1	1			1		
575				1			1						
577				1			1					1	
588					1		1				1	1	
616			1	1							1	1	
630					1		1				1	2	
642		1		1							1		1
654					1		1			1	1		1
656					1		1			1	1	1	
658		2									1		1
685								1	2				
700			1	1						1	1	1	
705						1			2			2	
713							1		2		1	1	
720				1					2			2	
722			1						2				
731					1				2		1	2	
744	1			1									
749	1		1									1	
757	1		1								1	2	
766	1			1							1		1
775							1	1	1		1		
793			1					1	1		1	2	
802				1				1	1		1		1
811				2					1				
819			2						1				
834				2					1		1		1
837				2					1			2	
840			1	1					1		1	1	
863				2	1			1				1	
869				1			1	1			1		
881				1				1	1			1	
890				1	1		1				1	1	
897		1				1		1			1		1
907	2										1		
923			1	1	1								
925			2		1						1		
943			3								1	2	
951				1					3		1		
965		1							3		1		

969				2			1			1	1		
985			3							1			1
995		1	1	1						1			
1013	1						2				1		
1020			1				1		2		1		
1027	1				1			1			1	2	
1039	1				1								
1055	1				2							1	
1057	1			1	1						1		
1078	1				2						1	1	
1083				2				1	2				
1105				2				1	1		1		
1117				2	1				1				
1127	2	1									1	2	
1164				2	1			1					
1171	2					1							
1189				2	1			1			1	2	
1215	2		1								1	2	
1219				2	1						1		
1221			1	3							1	2	
1260		3	1										
1280		3	1								1		2
1277	2			1						1	1		
1310	1	1			1			1			1		
1319	1		1	1				1					1
1338		1		2				2					
1347	1			3									
1368	1	1	1	1									
1376	1		1	2							1	1	
1396				2	1			1	1			1	
1412			1	2				1	1		1	1	
1426				3	1				1		1		
1441				4					1		1	2	
1454									7				
1471	2	1						1					
1484	1			2					2		1		
1498			1	2	1		1				1	1	
1512	2			2							1	1	
1528			2	2	1						1		1
1542			2	3							1	1	
1557			1	2					3		1		
1570			1	3	1					1			
1586		3	1	1							1	1	
1600	3						1						
1628	1			2	1			1			1		1
1644	1		1	2				1			1		1
1658	1		2	2									
1673	1			4							1		
1686				4				1	1				
1716				5					1				
1731			1	3					2			2	

1744		1	4				1	1		1
1775			4		1	1		1		
1803			5				1	1		
1819		2	4							1
1832			4				3			
1861		1	3				3	1	2	
1877										
1891			3		1	1	2	1	2	
1919			4			1	2	1	2	
1949	1		4		1			1	2	
1964	1		4	1				1	1	
1977	1		5					1	2	

3.2. Extraction of bio hardener and adhesives development

The Elasticity Modulus of adhesives A1 and A2 are determined from the maximum values of curves of fig.3. Their values are respectively 12818 and 20652 MPa. The transition temperatures of these two adhesives A1 and A2 are respectively 153 ° C and 160 ° C. The Elasticity Modulus of the adhesive A2 is better than that of A1. It means that the tannin used has a good reactivity with the bio hardener [14].

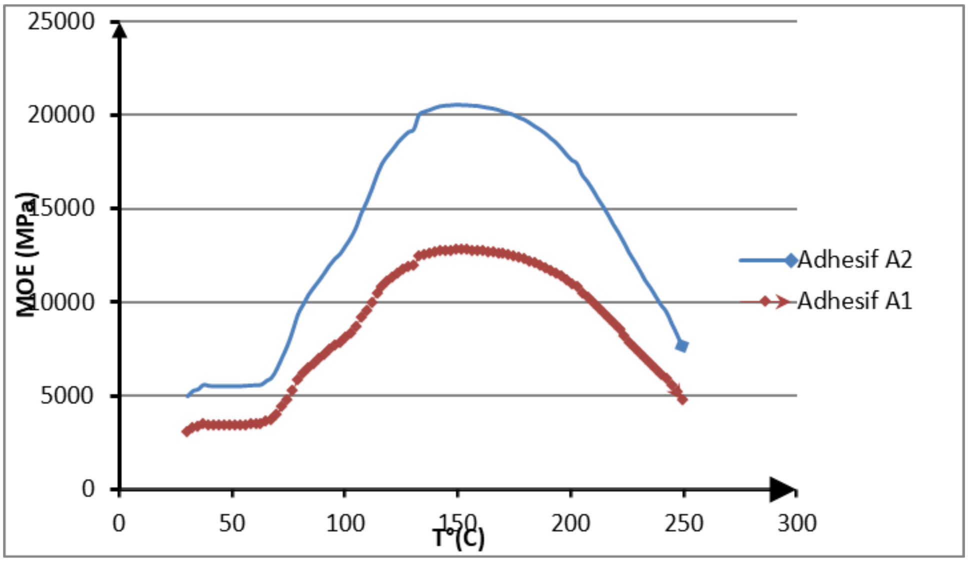


Figure 3: TMA curves of adhesive

3.3. Elaboration and abrasive characterization.

The table 2 show that the abrasive E2 has better characteristics than E1. The Brinell hardness of abrasives E1 and E2 are respectively 22,28 daN/mm^2 and 27,32 daN/mm^2. The coefficient of abrasiveness of E2 is higher than that of E1 (7, 28.10^{-1}> 5,12.10^{-1} %). These values are better than those found by Zhang et al. (2015) [7]. It is mean that the abrasive E2 has a better capacity to cause wear of a tool (crusher, drilling cutter, grinders ...) than the abrasive E1. These both results denote that the bio hardener tested was the most reactive with the tanning used.

Table 3: Mechanical properties of abrasives

Properties	**Abrasive sample**	
	E1	**E2**
HB (daN/mm^2)	22, 28	27,32
Abr (%)	5,12 .10^{-1}	7, 28.10^{-1}

HB: Brinell Hardness (daN/mm^2); **Abr**: Abrasiveness coefficient

3.4. Thermogravimetric analysis (TGA)

The TGA curves of Fig. 4 show several remarkable points. The peaks at 155°C and 170°C for the E2 curve reflect the evaporation of the volatile compounds contained in the abrasive, the peak located around 400°C of curves E1 and E2 reflects the degradation of the hexamine and bio hardener used in the elaboration of adhesives [15]. The peaks located at 575°C and 760°C on the two curves reflect the transformation points of α quartz (low temperature polymorph) transforms into β quartz (high temperature polymorph) [17]. At the end of the different transformations, approximately 20% of the different initial masses remain for the two samples, this 20% rest being due to the quartz and other rigid elements of the abrasive which are not consumed.

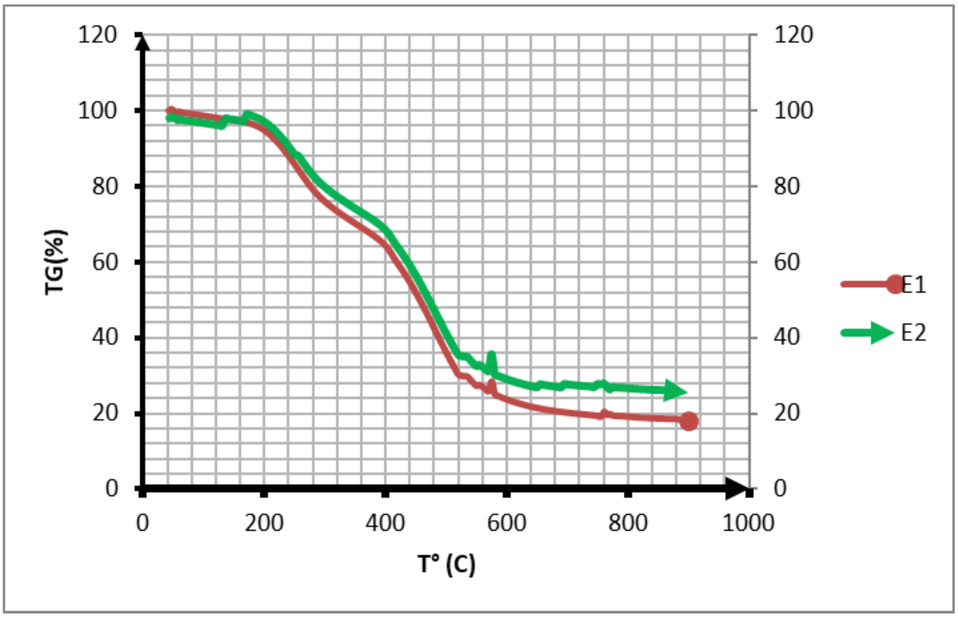


Figure 4: Mass loss depending the temperature

4. Conclusion

The abrasive elaborated using African resources, as it is constituted of adhesive, support and grain is an applied–type development. Its mechanical characteristics are performant. The *Vitellaria paradoxa* exploited as a hardener and used in the development of the adhesive is then of paramount importance for the economic development of Africa.

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