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An Instrumented Macro-Indentation Method for Determining the Mechanical Properties of Coconut Shell (Coco Nucifera of Cameroon)

E. Njeugna^{1,2}, M.B.K. Ganou^{1,3a}, D. Ndapeu^{4,5}, J.N.T. Foba^{6,7}, N.R.T. Sikame^{4,5}, P.W.M. Huisken^{1,8}

1 – Laboratory of Mechanics and Production (LMP)-UFD, University of Douala-Cameroon

2 – Professor, Senior researcher, Head of LAMMA, Laboratory of Mechanics and Appropriate Materials (LAMMA)-ENSET, University of Douala-Cameroon

3 – Master's Degree Student, researcher in LAMMA, Laboratory of Mechanics and Appropriate Materials (LAMMA)-ENSET, University of Douala-Cameroon

4 – PhD, Senior researcher in LAMMA, Head of LAMMA, Laboratory of Mechanics and Appropriate Materials (LAMMA)-ENSET, University of Douala-Cameroon

5 – Laboratory of Mechanics and Modelling of Physical System (L2MSP)-University of Dschang, Cameroon

6 – Professor, Senior researcher in LAMMA, Laboratory of Mechanics and Appropriate Materials (LAMMA)-ENSET, University of Douala-Cameroon

7 – Laboratory of Chemistry-University of Buea-Cameroon

8 – PhD Student, Senior researcher in LAMMA, Laboratory of Mechanics and Appropriate Materials (LAMMA)-ENSET, University of Douala-Cameroon

a – morinoganou@yahoo.fr



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ABSTRACT. An instrumented macro-indentation test was used to determine the viscoelastic parameters and hardness of the shell of *Coco Nucifera* from Cameroon in order to promote their use in the manufacture of abrasives. Samples measuring 10 mm x 10 mm x 3 mm were cut out from the bottom of the fruit, close to the natural indentations (the eye) of an approximately round-shaped fruit. The indentation load ranged from 50 to 500 N with an increment of 50 N. A comparator with digital display and a camera attached to the machine measured the total penetration and rate of penetration. The Oliver and Pharr indentation model and Hertz contact theory were used to determine the Young's modulus, hardness and creep ratio of coconut shells. Young's modulus determined in the polar zone was 3.52×10^3 MPa and 1.45×10^3 MPa in the equatorial zone. The hardness was 1.16×10^2 MPa in the polar zone and 0.7×10^2 MPa in the equatorial zone. The creep ratio was 16 in the polar region and 8 in the equatorial zone. Validation of the procedure and results will be done with indentation tests on a kind of wood: Azobé (*Lophira alata*).

Introduction. Coconut Shell of *Coco Nucifera* (CSCN) is a product of the coconut tree (*Coco nucifera*) which is exploited for the valuable flesh (copra) enclosed in the hard shell. Important quantities of CSCN are generated during the production of copra [1]. The CSCN alone occupies about 25% by weight of the nuts and with about 54 million tons of coconuts produced annually worldwide [2], an estimated 13.5 million tonnes of CSCNs is generated. A small part of these shells is used in the production of activated carbon. [3]. However, most of CSCN is not used, presenting a challenge for solid waste management [4]. Usually the CSCN is simply discarded in nature with negative environmental impact (occupying land that could otherwise be used for agriculture, breeding sites for mosquitos). In recent years, the instrumented indentation test has been used as an alternative method for determining mechanical properties of materials [5]. The hard nature and the abrasiveness of the shell makes it a potential food processing material. A good knowledge of the properties of this hull allows it to be used as a green alternative in agro-processing tools to plump goods such as beans, maize and groundnuts, otherwise like abrasives for woods. Preliminary characterisation of this shell

by Njeugna et al. [5] was bend tests and beam theory to determine Young's modulus. The rectangular test pieces used in their study showed an unavoidable curvature due to shape of the coconut shell. Young's modulus calculated by this method is expected to be strongly influenced by the curved shape of the test pieces. We propose in this work to use an instrumented macro-indentation testing as Oliver and Pharr method to determine some mechanical properties of the shell. The validation of the procedure and results will be done with indentation tests on a kind of wood: Azobé (*Lophira alata*) part of the harder and dense wood species.

Materials and methods.

Materials. Coconut shell used in this work came from the Littoral region of Cameroon. To prepare prismatic test pieces, we chose mature round hulls because of ease of cutting out specimens 10 mm x 10 mm x 3 mm by grinding of the faces. Sampling was done in two zones: Equatorial and Polar zones as shown in Fig. 1. In a test rig, we performed 5 indentation tests after conditioning the shells at ambient temperature in the laboratory for 2 months.

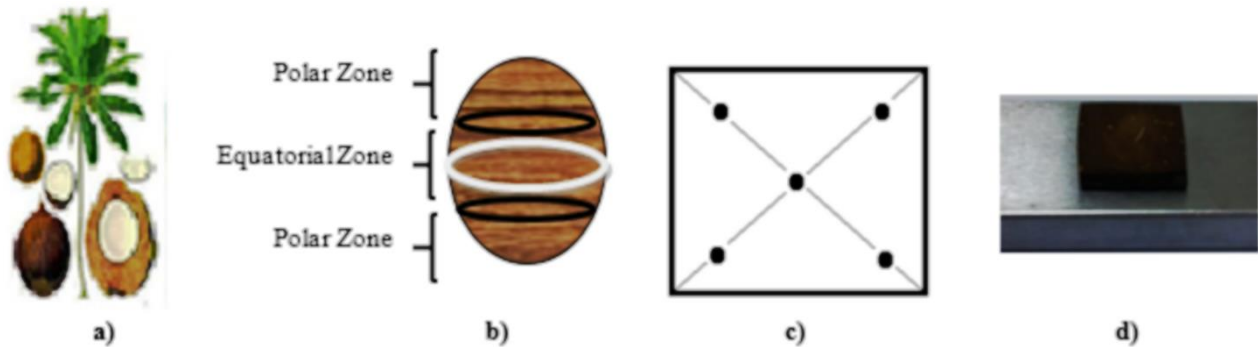
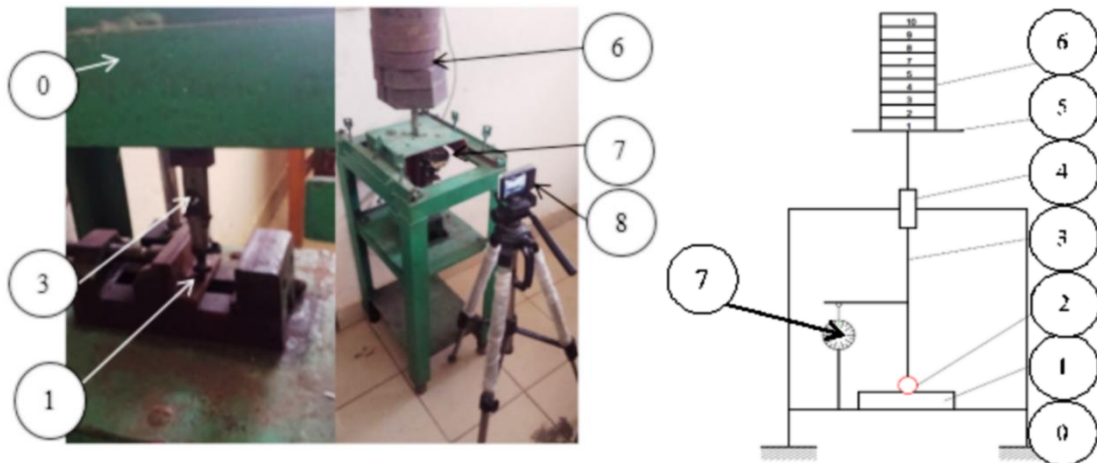


Fig. 1. Localisation of indentation points: a) Coconut tree b) and Sampling from a shell c) indication of indentation d) plates samples indented.

A Dial gauge to the thousandth and a Facom brand digital display was used to measure displacement. A Canon camera provided video monitoring of comparator variations throughout the duration of the test. The indentation was done with an adapted macro-indentation device designed and constructed in our laboratory [11] (Figs. 2, 3).



0 – Built, 1– Specimen of CSCN, 2 – Indenter Ball, 3 – indenter rod, 4 – Slide the rod, 5 – Loading platform, 6 – load, 7 – Dial gauge to the thousandth, 8 – Canon camera

Fig. 2. Image Test device.

Fig. 3. Device block diagram.

Methods. The instrumented macro-indentation test takes place in three phases: a charging phase; a hold phase and an unloading phase [10]. Loading is achieved by the masses placed on the tray 5. The loads applied ranged from 50N to 500N with an increment of 50N. For Each load there are equivalent penetration of the steel ball in the sample was measured. The comparator measured and displayed the value of penetration. When the maximum load of 500N was reached, a hold time of 30 minutes was maintained and the flow of the CSCN was observed as the continuous penetration of the indentation ball in the shell sample. The camera recorded the display of comparator over the entire hold time. During the unloading phase, we reduced the load from 500N to 50N in stepwise reduction of 50N. The elastic recovery of the material was displayed by the comparator. The pairs of load vs penetration depth during loading and unloading were plotted to obtain the characteristic curve of the CSCN material covering the three phases as shown in figure 4. 50 samples were tested.

To exploit these curves, we adopted the method of Oliver and Pharr [6, 18, 21], this method describes the upper limit of the discharge curve by the law in Eq. (1):

$$P = P_{max} \left(\frac{h-h_f}{h_{max}-h_f} \right)^m, \tag{1}$$

where P – is the force;

P_{max} – is the maximum applied force;

h –is the depth of indentation;

h_f – is the indentation depth unloading;

h_{max} – is the maximum indentation depth at F_{max} ;

m – is a function of the geometry of the indenter [12-15]. For a spherical indenter $m = 1$.

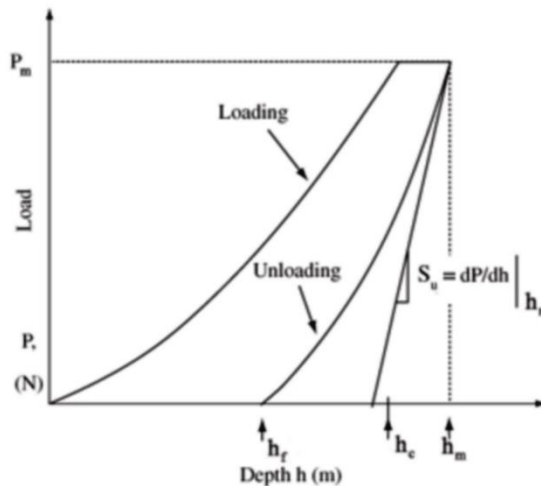


Fig. 4. Load vs depth plot.

Modulus of elasticity (Young's modulus). The determination of Young's modulus is based on the assumption that during the unloading curve the removal of the indenter is accompanied by a spring back due to the elasticity of the material [7]. Thus the slope of the unloading curve provides a measure Young's modulus. The method is based on the recovery theory of Hertz and Bulychov, later developed by Snedon [8, 20]. It provides for the modelling of the unloading slope with a function that relates the contact area to the reduced Young's modulus according to the Eq. (2):

$$S = \left(\frac{dP}{dh} \right)_{h=h_{max}} = \frac{2}{\sqrt{\pi}} E_r \sqrt{A_c}, \quad (2)$$

Where S – is the stiffness of the contact between the indenter and the material when unloading, measured at the maximum penetration (h_{max}) recorded;

A_c – is the projected contact area given by the Eq. (3):

$$A_c = \pi \cdot a^2 = 2 \cdot \pi \cdot R \cdot h_c - \pi \cdot h_c^2 \quad (3)$$

where a – projected contact radius;

R – radius of the indenter;

h_c – penetration depth given by the Eq. (4):

$$h_c = h_{max} - \varepsilon \frac{P_{max}}{S}, \quad (4)$$

where ε – a constant related to the geometry of the indenter, which is 0.75 for a spherical indenter [12, 19];

E_r – Reduced Young's modulus from the theory of elastic contact of Hertz [8], given by Eq. (5).

$$\frac{1}{E_r} = \frac{(1-\nu_m^2)}{E_m} + \frac{(1-\nu_i^2)}{E_i}. \quad (5)$$

In this equation the indices i and m refer to the characteristics of the indenter and the material respectively.

Since the indenter is made of stainless steel, we consider that: $\nu_i = 0.3$ and $E_i = 210.000$ MPa.

Thus, Young's modulus of the CSCN is given by Eq. (6):

$$E_m = (1 - \nu_m^2) \cdot \left[\frac{2}{\sqrt{\pi}} \frac{1}{S} \sqrt{A_c} - \frac{(1-\nu_i^2)}{E_i} \right]^{-1} \quad (6)$$

Hardness. Once the method for the calculation of the contact area is available, it is possible to calculate hardness. [9, 15, 17]. This is done using the formula from Eq. (7) and the contact area A_c is obtained by Eq. (3):

$$H = \frac{P}{A_c}. \quad (7)$$

Creep ratio. C_{IT} denoted indentation creep ratio is defined by the relative change in depth of

corresponding penetration, according to Eq. (8):

$$C_{IT} = \frac{h_2 - h_1}{h_1} \times 100 \% . \quad (8)$$

Results and discussion. Figure 5 is an example of the instrumented macro-indentation curve of CSCNs.

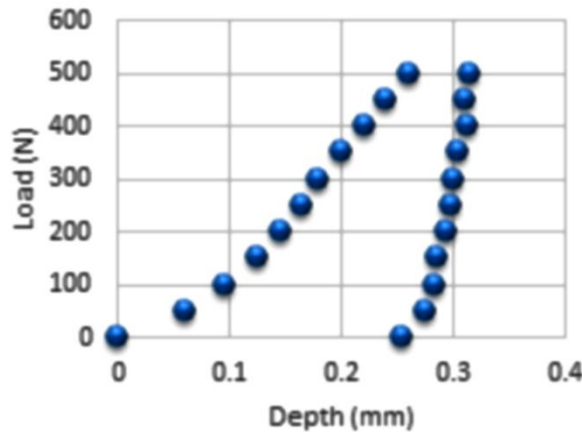


Fig. 5. Sample of macro-indentation curve CSCNs.

The Oliver and Pharr method and Hertz’s theory were applied to curves obtained by testing samples from polar and equatorial zones. The values of Young’s modulus, hardness and creep ratio obtained are presented in Table 1.

Table 1. Features of the CSCN by macro-instrumented indentation

	Young Modulus E	Hardness H	Creep ratio
Equatorial zone	$1.45 (\pm 0.179) \cdot 10^3$ MPa	$0.70 (\pm 0.12) \cdot 10^2$ MPa	8.31 ± 1.35
Polar zone	$3.52 (\pm 0.562) \cdot 10^3$ MPa	$1.16 (\pm 0.10) \cdot 10^2$ MPa	16.2 ± 3.51

Looking at the results shown in Table I, we find that the Young's modulus, hardness and creep coefficient are relatively higher in the polar zone than in the equatorial zone. A possible explanation could be differences in thickness from the equatorial to polar zones. It was observed that the nut is thicker in the polar zone compared to the equator.

These results are significantly different from results obtained by Njeugna et al. [5]. The difference could be attributed to difference in test method. The values of Young’s modulus obtained in this study are closer to values for polymers and gypsum (Table 2), an indication that the instrumented macro-indentation test may be more suitable for characterising the mechanical properties of this material.

Applying the same indentation tests (Fig. 6) on wood named Azobé with a moisture content of 12% in the same conditions and protocols, we got Young modulus $E(az) = 12.304$ GPa. This value falls within the values gotten from literature review [21]. We found an indentation hardness $H(az) = 41.5398$ MPa and with this hardness $H(az)$, it is clear the CSCNs can be used for sanding wood.

Table 2. Comparison of specific rigidities

Materials		Young modulus in GPa	References
Polyethylene		0.20	[5]
Polycarbonate		2.40	[5]
Epoxy		2.40	[5]
Polyester		5.00	[5]
EDP (Gypsum A)		9.90	[16]
EDP (Gypsum B)		2.58	[16]
Azobé		12.30	[21]

		Instrumented indentation test	3 points flexural test	
Coconuts shells	Polar zone	3.52	19,80	[5]
	Equatorial zone	1.45	12,70	[5]

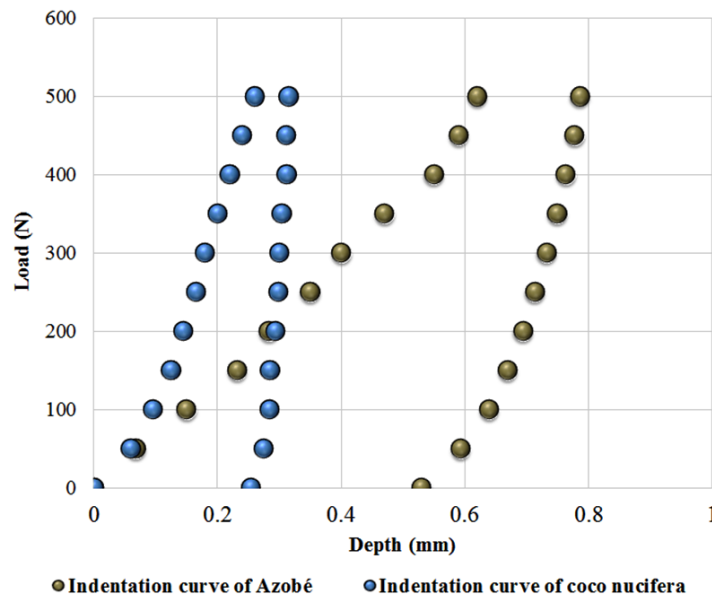


Fig. 6. Comparison between the indentation of CSCNs and Azobé.

Summary. Our work focused on determining the mechanical properties of coconut shell from *Coco nucifera* by an instrumented macro-indentation test. A series of tests were conducted on prismatic samples extracted from 10 mm x 10 mm x 3 mm elongated CSCNs. Sampling was done in two areas: the equatorial zone and polar zone and 50 samples were tested. The method of Oliver and Pharr was applied to analyse the behaviour of the material at the upper limit of loading and the Hertz contact theory was used to calculate fundamental mechanical properties of CSCN. Thus, the Young's modulus, hardness and the creep ratio were determined. The Young's modulus determined is of the order of $3.52 \times 10^3 \pm 562.81$ MPa in the polar zone and $1.45 \times 10^3 \pm 179.29$ MPa in the equatorial zone. The hardness is $1.16 \times 10^2 \pm 10.69$ MPa in the polar zone and $0.70 \times 10^2 \pm 12.01$ MPa in the equatorial zone. The creep ratio is 16.2 ± 3.51 in the polar region and 8.31 ± 1.35 in the equatorial zone. Currently, we are using these shells after grinding in the manufacturing of abrasives and composites. Therefore, it is imperative to know its mechanical properties for more valorisation.

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