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Physio-Chemical Characterization of African Elemi, Black Velvet Tamarind and Date palm Seeds for Possible Application in Infrastructural Development

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Abstract
Effect of elemental contents, morphology and bonding on the hardness of African Elemi (AE), Black Velvet Tamarind (BVT) and Date Palm (DP) were investigated. The hardness of the seeds was studied through various tests which include: rupture machine, Atomic Absorption Spectroscopy (AAS), Scanning Electron Microscopy (SEM) and Fourier Transform Infrared Spectroscopy (FTIRS). The universal testing results showed that BVT seed is the hardest (81.90Pa). DP seed have the hardness of 18.02 Pa and AE have the hardness of 6.74 Pa. BVT and AE required a load of 1.60 kN for a complete rupture, DP required a load of 2.7 kN for a complete rupture. The presence of deformation in DP at 1kN before a final rupture at 2.7 kN and deformation of BVT at 1.30 kN before a final rupture at 1.6 kN shows that both DP and BVT are malleable. AE did not show any form of deformation before a final rupture at 1.6 kN, this shows that the seeds are brittle. The AAS results showed the presence of magnesium, manganese, lead, copper, iron and zinc in all the seeds samples. The negative sign on the concentrations of cadmium, nickel and cobalt in the entire seeds sample shows that the elements concentrations were below the level of detection by the AAS. The presence of magnesium, Copper and Iron in AE, BVT, DP, could be one of the reasons for the hardness of the seeds. The high concentration of magnesium (859.18 mg/kg) and copper (487.25 mg/kg) in BVT contributed to the hardness, ductility and malleability. The SEM results showed that that AE had a refined surface which was responsible for its brittleness. BVT and DP had agglomerated flaky structures which encouraged mechanical interlocking of the seed fibres with the help of protein adhesives which resulted in their hardness, ductility and malleability. The FTIR analysis spectrum showed that the hard shell was composed of compounds with oxygen functional groups related to lignin, cellulose, hemicelluloses structures. BVT and DP were malleable and had similar surface morphology. BVT and DP can find application as additives which require hardness and malleability.
Keywords: Tensile stress, Rupture, Malleability, Fiber, Brittleness.

Introduction

The physical and chemical properties of agricultural seeds are wide knowledge that can be useful in farming, storage, drying, processing etc. The knowledge is important in designing of the machinery to harvest and in preparation of the processing chain. It is important to have an accurate estimation of the size, shape, hardness and other mechanical characteristics which may be considered as designing parameters for food production [1].

There are many types of mechanical loading which are; puncture compression, shearing, twisting, extrusion, crushing, tension, bending, vibration and impact. Mechanical properties include basics parameters such as; hardness, cohesiveness, elasticity and adhesiveness while secondary parameters are; brittleness, fracture, and chewiness. There are also four (4) basic values that can be obtained from mechanical properties test; force, deformation, slope and energy [2].

Quality differences in fruit are often detected by differences in density, mass, volume and projected area is important in determining sizing systems. When the fruits are transported hydraulically, the design fluid velocities are related to both density and shape. Physical characteristics of crops are essential parameters in utilization, development of processing methods and design of equipment [1]. The survey of a country's natural resources is an important prerequisite for proper utilization of the raw materials. The availability, physical and chemical properties of some agricultural products seeds are important in determining its commercial value and other possible uses.

African Elemi (*Canariumchweinfurthii)* belongs to the family of *Burseraceae* (which is also known as torch wood, Frankincense or incense tree family). For the purpose of this research, African Elemi is called AE. The endocarp is a hard-stone shell which consist of crude fibre, protein, carbohydrate and other elements such as sodium, magnesium, zinc, iron, manganese and copper [3]. The Black Velvet Tamarind (D*ialiumguineanse)* is in the family of *Leguminosae*(*Fabaceae*). Its common names are Black Velvet, Velvet tamarind, Yoyi, Icheku (Igbo), TsamiyanKum (Hausa), Awin (Yoruba). The epicarp is the hard-outermost shell that covers the mesocarp and the endocarp. The endocarp is the hard seed of the fruit which contains protein, carbohydrate and crude fibre [4]. The endocarp contains high concentration of magnesium, manganese, chlorine, sodium and calcium [5]. For the purpose of this research, Black Velvet Tamarind is called BVT. Date palm (*Phoenix dactylifera)* is a member of the family of *Aracaceae*. For the purpose of this research, Date Palm is called DP. It is a key plantation crop of many countries of the regions of West Africa and North Africa. In 2009 about 7.4 million tons of Date palm fruits were produced worldwide. About 70% of the total production of Dates is from the Arab world. The top ten producing countries are; Egypt, Saudi Arabia, Iran, United Arab Emirates, Pakistan, Algeria, Sudan, Oman, Libya, and Tunisia [6]. The elemental composition of the pulp, seed, and fruit coat of black velvet tamarind was determined by [5] using activation analysis. The fruit pulp contained Manganese, magnesium Chlorine, Aluminium, Vanadium, Calcium, Sodium and Potassium. The seed and fruit coat contained the minerals mentioned above except Potassium and Sodium respectively.

Date seeds are reported by [7, 8] to contain many minerals such as Sodium, Potassium, Magnesium, Calcium, Phosphorus, Iron, Manganese, Zinc, Copper, Nickel, Cobalt, Chromium, Lead, and Cadmium. Potassium, Phosphorus, Magnesium, Calcium and Sodium are in higher concentration in Date seeds [9]. Iron, Manganese, Zinc and Copper are also found in higher concentrations among the microelements [10].

[11, 12] focused on the Physio-chemical and thermal properties of *Canarium Schweinfurthii* England (CS) core shells due to their hardness with a view to improve the state of knowledge to use them for the reinforcement of composites. The results revealed that the CS shells had a water content of 6.26%, a holocellulose content of 53.07% and lignin content of 35.79%. Maryam et al. [13] Extracted and Characterized cellulose from date seeds and observed that the seeds contain fibre fraction at a range of 39.6-57.5% for acid detergent fiber (cellulose + lignin), 51.6-70% for neutral detergent (hemicellulose + cellulose + lignin), 12 to 17.50% for hemicelluloses, 26.10 to 42.50% for cellulose and 7.2 to 11% for lignin.

The seeds of African Elemi, Black Velvet Tamarind and Date Palm are known to be very hard in nature. These shells abandoned after consumption pose environmental problems and their hardness inspires the study of their properties for other possible applications like reinforcement of composites. This study therefore hopes to achieve this through physical and chemical characterization of the seeds for the properties responsible for their hardness and suggest possible application(s) as well.

Materials and Method

Materials

The materials used for this research include three different agricultural fruits (African elemi (AE), Black velvet tamarind (BVT) and Date palm(DP), laboratory grinding machine, electronic beam balance, wash bowl, air tight bottles, filter papers, beakers, spatula, pipette and burner.

Method

Preparation of the Samples

i. The African Elemi fruits were soaked in warm water at 50°C for 20 minutes to separate the seed from the pulp. The seeds were washed with clean water and air dried. The dried seeds were grinded into powder using a laboratory grinding machine. The powder was stored in an air tight bottle for AAS, SEM and FTIRS analyses.

ii. The black velvet tamarind seeds were manually separated from the coat and pulp. The seeds were washed with clean water and air dried. The dried seeds were grinded into powder using a laboratory grinding machine. The powder was stored in an air tight bottle for AAS, SEM and FTIRS analyses.

iii. The Date palm seeds were manually separated from the flesh and washed to free them from any adhering date flesh. The seeds were air dried and grinded into powder using a laboratory grinding machine. The powder was stored in an air tight bottle for AAS, SEM and FTIRS analyses.

iv. Five seeds from AE, BVT and DP each were preserved for rupture tests.

Characterization of the Samples

i. Rupture

A SM1000d universal compression testing machine was used to determine the hardness of AE, BVT and DP seeds respectively. Each seed was loaded between two (2) parallel plates of the universal testing machine and compressed along the thickness until rupture occurs. The hardness of the seed (H) is expressed in terms of tensile stress (σ) given by equation 1 [14];

$$\sigma = \frac{H}{2.73} \qquad (1)$$

$$\therefore H = 2.73\sigma \qquad (2)$$

Ii. Atomic Absorption Spectroscopy (AAS)

Atomic absorption spectrometer 240FS AA was used for the analysis of 0.53 g, 0.51 g and 0.52 g of AE, BVT and DP seeds powders respectively to determine the elemental concentrations of the samples. The concentration of each element is determined using equation 3 [15];

$$ACS = \frac{IC \times DF \times FV}{WS} \qquad (3)$$

Where ACS is the actual concentration in the sample in mg/kg, IC is the instrument concentration in mg/L, DF is the dilution factor, FV is the final volume of the filtrate in mL and WS is the weight of the sample in grams (g).

Iii. Scanning Electron Microscopy (SEM)

Morphological examination of the samples was determined using a Phenom proX scanning electron microscope (with a magnification of X1000). The seed powders of the seeds were placed in the sample pallet of the microscope separately and the micrographs were obtained.

Iv. Fourier Transform Infrared Spectroscopy (FTIR)

The grinded samples were placed on the surface of a KBr plate of a Cary 630 FTIR. There was a back and forth rubbing motion of the two windows to evenly distribute the mixture between the plates. The sandwiched plates were placed in the spectrometer and their spectra were obtained.

Results and Discussion

Rupture Analysis

Table 1 shows the results of the rupture analysis for African elemi, Black velvet tamarind and Date palm. From the Table 1, it can be observed that Black velvet tamarind seed sustained applied load within deformable range of 1.3 kN to 1.6 kN (0.3 kN). The tensile stress on the seed at rupture was $30 Nmm^{-2}$. The seed is therefore ductile, malleable and possess engineering application characteristics. From equation 2, the hardness of the seed is 81.90 Pa.

Date palm seed showed a form of deformation from the load of 1 kN before a final rupture at 2.7 kN (1.7 kN). The tensile stress on the seed at rupture is 6.60 Nmm^{-2}. The seed is therefore more ductile and malleable but can withstand fewer loads than the BVT seed. From equation 2, the hardness of the seed is 18.02 Pa.

African elemi showed no form of deformation before the final rupture at a load of 1.6 kN with a tensile stress of 2.47 Nmm^{-2}. The seed is therefore brittle. The hardness is 6.74 Pa.

BVT seed is therefore the hardest while AE seed has the least strength. From literature, the hardness of AE seed is from the endocarp, for the BVT, it is from the epicarp and endocarp while for DP, it is from the seed itself [4, 3 and 16]

Table 1: Mechanical Properties of African Elemi (AE), Black Velvet Tamarind (BVT) and Date Palm (DP)

Seeds	Length (m)	Diameter (m)	Deformation (kN)	Rupture point (kN)	Tensile Stress (Nmm^{-2})	Hardness (Pa)
AE	0.1136	0.2872	0.00	1.60	2.47	6.74
BVT	0.0312	0.0824	1.30	1.60	30.00	81.90
DP	0.0677	0.2283	1.00	2.70	6.60	18.02

Atomic Absorption Spectroscopy (AAS) Analysis

The chemical composition and concentration analysis of the three seed samples investigated are summarised in Table 2. All the samples contained Magnesium (Mg), Manganese (Mn), Lead (Pb), Copper (Cu), Iron (Fe) and Zinc (Zn). The negative signs on the concentrations of Cadmium (Cd), Nickel (Ni) and Cobalt (Co) show that the elements are below the level of detection by the AAS.

From Table 2, the high concentrations of magnesium (462.95 mg/kg) and iron (648.46 mg/kg) in DP is responsible for DP's hardness. The high concentrations of magnesium in BVT (859.18 mg/kg) and Copper (487.25 mg/kg) is the reason for BVT's hardness, higher ductility and malleability. The low concentration of all the concerned elements could be one of the reasons for AE's brittleness.

Table 2: Element Concentration of African Elemi, Black Velvet Tamarind and Date fruit

Atomic Element Number	Symbol	Element	Conc. in AE mg/kg	Conc. in BVT mg/kg	Conc.in DP mg/kg
12	Mg	Magnesium	127.59	859.18	462.95
25	Mn	Manganese	26.51	125.69	30.58
82	Pb	Lead	41.89	47.25	53.85
29	Cu	Copper	8.30	487.25	8.85
26	Fe	Iron	215.94	95.69	648.46
30	Zn	Zinc	5.08	15.43	16.56
48	Cd	Cadmium	-15.94	-18.53	-17.60
28	Ni	Nickel	-11.98	-28.43	-37.70
27	Co	Cobalt	-27.36	-33.33	-20.19

Scanning Electron Microscopy (SEM) Analysis

Figure 1 shows the SEM micrograph of African elemi which has a mass of aggregates that have less surface area and a refined (smooth) surface which does not encourage interlocking of the aggregates compared to other samples which is one of the reasons for the brittle characteristics of the seed. Figures 2 and 3 show the SEM micrograph of Black velvet tamarind and Date palm, both Black velvet tamarind and Date palm contain a porous network of agglomerate flaky structures which contain solid clusters. The Date palm shows a form of fractures between the solid clusters. The agglomerated flaky structure of Black velvet tamarind and Date palm encourages mechanical interlocking of seed fibers with the help of protein which is a natural adhesive that binds all the constituents of seed kernel and increase the surface roughness that results in their hardness, ductility and malleability.

Figure 1: SEM of African Elemi

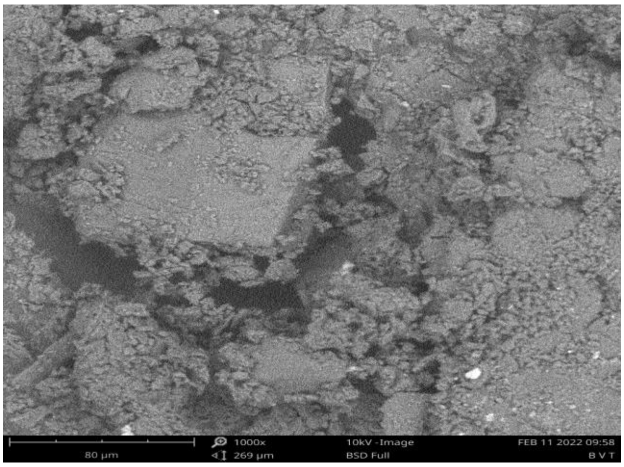

Figure 2: SEM of Black Velvet Tamarind

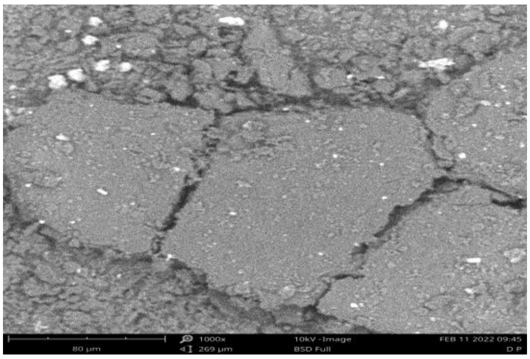

Figure 3: SEM of Date Palm Seed

Fourier Transform Infrared Spectroscopy (FTIRS) Analysis

Table 3 shows the FTIR spectrum of AE. The spectrum contains multiple absorption bands referred to different functional groups of AE. The presence of OH hydroxyl bond of cellulose and hemicelluloses is strongly present in the bond at 3347.10 cm^{-1}. The intense peak at 2922.20 cm^{-1} is caused by the symmetric of the C-H bond of cellulose and hemicelluloses. The 2855.10 cm^{-1} peak correspond to the symmetrical elongation of CH_2 bonds of fats. The peak at 1744.10 cm^{-1} represents the symmetric elongation of carboxyl and acetyl groups of hemicelluloses. The 1654.90 cm^{-1} peak represent the OH bond of free water. The 1461.10 cm^{-1} peak indicates the in-phase deformation of C-H bond and aromatic vibration of hemicelluloses and lignin. The peak 1375.00 cm^{-1} represents the C-H bending vibration associated to the aliphatic bonds of cellulose. The peak at 1230.10cm^{-1} represents the deformation of acetyl groups. The strongest peak of 1028.00 cm^{-1} corresponds to the vibration of C-O-C bond of cellulose. The 838.70 cm^{-1} peak indicates the vibration of C-O-C glycosidic bonds of aromatic compounds of polysaccharides.

The FTIR spectrum of BVT is presented in Table 4. The peak at 3267.50 cm^{-1} represents the strong broad absorption band characteristic of OH hydroxyl group of cellulose. The sharp peak found at 2907.30 cm^{-1} represents the stretch of C-H bonds of cellulose and hemicelluloses. The sharp peak at 1636.30 cm^{-1} indicates the presence of carboxylic group C=O stretch of lignin. The peaks at 1438.80 cm^{-1} and 1349.30 cm^{-1} are due to symmetric and asymmetric bonding vibration of secondary OH hydroxyl group of cellulose.

The FTIR spectrum of DP is presented in Table 5. The largest absorption peak of 3330.00 cm^{-1} represents the OH hydroxyl stretch of cellulose. The peak at 2922.20 cm^{-1} represents CH_2 stretch of cellulose and hemicelluloses. The peak at 1736.90 cm^{-1} is attributed to the C=O stretch vibration of acetyl of hemicelluloses. The peak at 1517.00 cm^{-1} indicates the presence of C=C aromatic skeletal vibration. The peak 1606.50 cm^{-1} indicates C=O stretch of carboxylic acids of lignin. The peaks at 1372.70 cm^{-1} and 1438.80 cm^{-1} correspond to the C-H deformation vibration and CH_2 bending of cellulose. The peak at 1241.20 cm^{-1} corresponds to the C=O stretch of hemicelluloses. The peak 1036.20 cm^{-1} corresponds to the presence of C-O stretching ring of polysaccharide in cellulose. The peak 872.20cm^{-1} indicates the presence of C-O-C stretching of the glycosidic linkage of cellulose [17].

The high concentration of fiber in DP is one of the reasons why DP is a hard seeds. The seed protein is a natural adhesive which binds the fiber and other particles of the seed together. Lignin is an organic polymer that forms key structure materials in support of plant tissue. It gives rigidity to woods and seeds and does not rot. The high percentage of lignin in AE is the reason for its brittleness due to high rigidity.

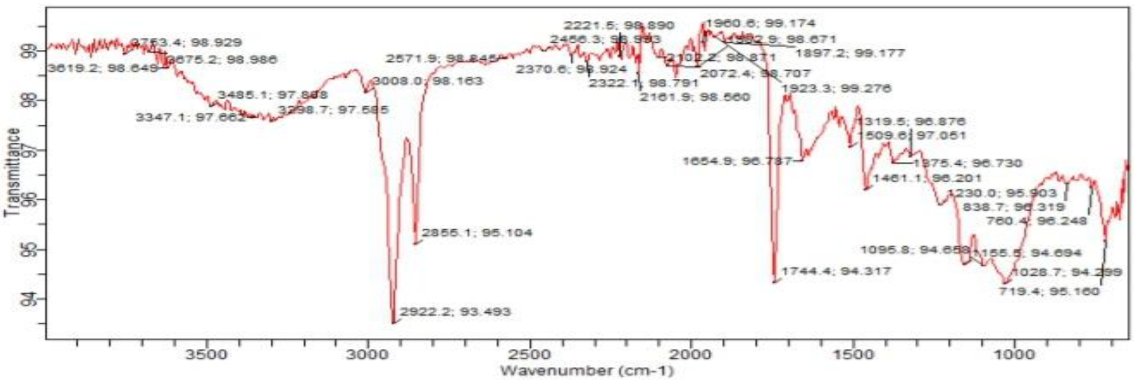

Figure 4: FTIRS of African Elemi

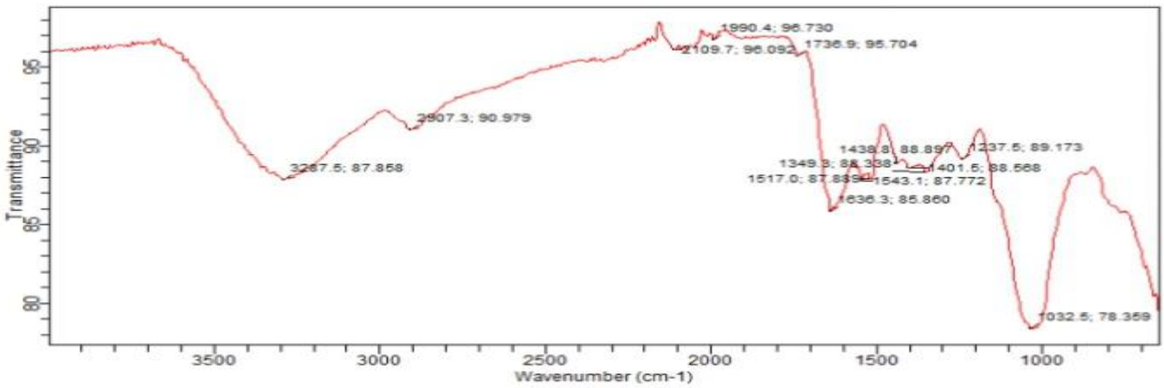

Figure 5: FTIRS of Black Velvet Tamarind

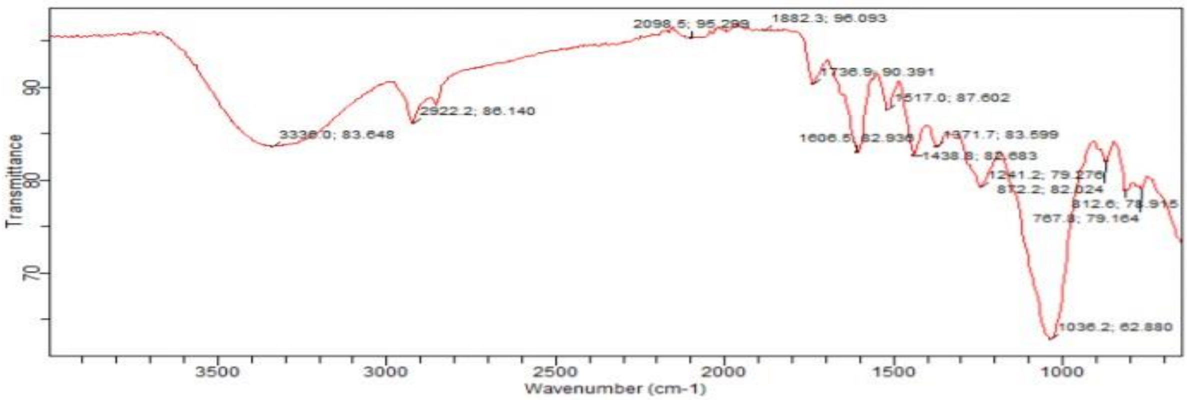

Figure 6: FTIRS of Date Palm

Table 3: Peak Data Values for African Elemi

Wavenumber (cm^{-1})	Bond	Components
3347.10	O-H vibration	Hemicellulose and cellulose
2922.20	C-H bond stretch	Polysaccharide
2855.10	CH_2 symmetric	Fats
1744.40	C=O stretch	Hemicelluloses
1654.90	O-H vibration	Water
1461.10	C-H carboxylic	Lignin and hemicelluloses
1375.00	C-O aliphatic ring	Cellulose
1230.00	C-O acetyl vibration	Lignin
1028.70	C-O-C glycosidic bond	Cellulose
838.70	C – O stretch	Polysaccharide

Table 4: Peak Data Values for Black Velvet Tamarind

Wavenumber (cm^{-1})	**Bond**	**Components**
3267.50	O – H stretch	Cellulose
2907.30	C – H vibration	Hemicelluloses and cellulose
1636.30	C=O carboxylic stretch	Lignin
1438.80	O-H secondary symmetric vibration	Cellulose
1349.30	O-H secondary asymmetric	Cellulose
1032.50	C – O stretch	Cellulose

Table 5: Peak Data Values for Date Seeds

Wavenumber (cm^{-1})	Bond	Components
3336.00	O – H stretch	Cellulose
2922.20	C – H stretch	Celluloseand hemicelluloses
1736.90	C =O acetyl stretch	Hemicelluloses
1606.50	C = O carboxylic stretch	Lignin
1517.00	C=C aromatic skeletal Vibration	Lignin
1438.80	CH_2 bond	Cellulose
1371.70	C–H deformation vibration	Cellulose
1241.20	C=O stretch	Hemicelluloses
1036.20	C-O stretch ring	Cellulose
872.20	C-O-C stretch	Cellulose

Conclusion

The hardness of African elemi (AE), Black velvet tamarind (BVT) and Date palm (DP) seeds were investigated using Rupture, Atomic absorption spectrometry (AAS), Scanning electron microscopy (SEM) and Fourier transform infrared spectroscopy (FTIRS) analysis.

1. Rupture analysis showed that black velvet tamarind seed is the hardest of the three seeds. Date palm is the second hardest seed. African elemi is the least hard among the seeds.
2. BVT and DP are ductile and malleable. However, AE is brittle.
3. High concentrations of magnesium (462.95 mg/kg) and iron (648.46 mg/kg) in DP is one of the reasons for its Hardness
4. High concentrations of magnesium (859.18 mg/kg) and Copper (487.25 mg/kg) in BVT is one of the reasons for its ductility and malleability.
5. The agglomerated flaky structure of Black velvet tamarind and Date palm encourages mechanical interlocking of seed fibres with the help of protein which is a natural adhesive that binds all the constituents of seed kernel and increase the surface roughness that results in their hardness, ductility and malleability. The hard shell is composed of compounds with oxygen

functional groups related to lignin, cellulose and hemicelluloses structures.

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Competing Interests

Authors have declared that no competing interests exist.

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