

## RESEARCH ARTICLE

# Moisture dependent physico-mechanical and aerodynamic properties of roselle calyces (*Hibiscus sabdariffa*)

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## ABSTRACT

The design of compatible harvesting and post-processing equipment as well processing techniques require information on the physical, mechanical and aerodynamic properties of agricultural materials. Therefore, this study focused on determination of some moisture dependent mechanical and aerodynamic properties of a dark red variety of roselle calyces. Experiments were conducted to measure some physico-mechanical and aerodynamic properties of the roselle calyces (such as dimensions, porosity, true and bulk density, peak rupture force, tensile strength, stiffness, modulus of elasticity, toughness, terminal velocity and drag coefficient) at three different moisture contents (14.40, 18.00 and 23.87%, w.b). Results showed that length, width, and thickness of the calyces ranged from 41.10 - 53.10 mm, 20.60 – 24.1mm, and 1.11 – 1.32mm, respectively. Changes in bulk density was negligible as the calyx moisture content was increased from 14.4 to 23.87 % while increment with increased moisture content was recorded for values of surface area, true density, and porosity. The Peak rupture force, tensile strength, stiffness, modulus of elasticity and toughness of the calyces ranged from 5.30 – 8.47 N; 442 – 692kNm<sup>-2</sup>; 4.06 – 4.31kNm<sup>-1</sup>; 13.47 - 14.78 MNm<sup>-2</sup>, 0.0035 - 0.0112 J, respectively. Terminal velocity (TV) of the calyces was between 2.78 – 3.22 ms<sup>-1</sup> and the values for drag coefficient ranged from 1.60 x 10<sup>-3</sup> – 2.9 x 10<sup>-3</sup>.

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## INTRODUCTION

Research into agricultural and biological materials to identify properties and potential applications has been progressive. Much attention is particularly placed on those that find great industrial application and are economically viable. *Hibiscus Sabdariffa* L. (Malvaceae) (fig. 1), commonly known as hibiscus or roselle, grows in many tropical and sub-tropical countries and is one of highest volume specialty botanical products in international commerce (Plotto et al., 2004). It is an annual herbaceous shrub, growing to about three (3) meters in height, with red or green inflated calyces (Babalola et al., 2001; Plotto et al., 2004; Qi et al., 2005). Different parts of the plant, have found variety of use in different fields. The leaves are used extensively for animal fodder and fiber (Dutt et al., 2009; Plotto et al., 2004). The works of (Akinoso and Suleiman, 2011; Ismail et al., 2008) reveals that the seed contain about 20% edible health promoting oils, which are rich in anti-oxidants.

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The calyces of roselle are the part of commercial interest. Many literature have reported that the calyces could be used for production of fruit drinks, juices, wines, jellies, jams, beverages, and ice-creams (Akinoso and Suleiman, 2011; Babalola et al., 2001; Juliani et al., 2009; Plotto et al., 2004). The extract of the calyces has also been reported for its health benefits in several medical applications (Ali et al., 2005; Juliani et al., 2009). There has been an increasing demand for roselle calyces over the last decade with about 15,000 metric tons entering international trade each year; however the quality of roselle from different countries are markedly different (Plotto et al., 2004). While the production of high quality hibiscus calyces in developing countries is becoming an important for value addition and income generation activities for the benefit of rural communities (Juliani et al., 2009), most calyces produced suffer severe mechanical damage during harvesting and post-processing resulting in significant revenue loss due to lack of compatible equipment and procedures for handling this crop. If the quality of calyces required for international, regional and local markets are to be enhanced, there is the need for information on the mechanical and aerodynamic properties of this herbal product. Therefore, the objective of this study was to determine some physical, mechanical and aerodynamic properties of a dark red variety of roselle calyces at three different moisture levels.



Figure 1. Typical dry roselle calyces

## MATERIALS AND METHODS

### Material preparation

A dried, dark red variety of roselle calyces was used for this study. The sample used was obtained from a local market in Ile-Ife, Osun State, Nigeria. The sample was divided into three equal portions by weight, 50g each. The initial moisture content (14.40% w.b.) of the calyces was determined and then they were conditioned to three different moisture content levels

(14.40%, 18.00% and 23.87% w.b.) respectively by adding calculated amounts of distilled water, sealed hermetically and stored in a refrigerator  $4 \pm 1$  °C for 24h prior to use.

Moisture content was determined by the method, AACC Method 44-15A (Moisture – Air-Oven Methods) (AACC, 2000; Akharume et al., 2016). 10g weighted sample of the calyxes were oven-dry at  $105^{\circ}\text{C}$  for 24hrs. The moisture content (w.b) was calculated according to the equation.

$$MC = \frac{W_i - W_f}{W_i} \times 100 \quad (1)$$

Where  $W_i$  and  $W_f$  are initial and final moisture content respectively.

### Physical properties

The physical properties determined for the roselle calyxes include dimensions, surface area, bulk and true densities, and porosity. The dimensions of the calyxes (fig. 2) was obtained by randomly selecting thirty (30) calyxes and measuring their length (from mid apex to the base of the calyx), breadth (the length of the base of the calyx) and thickness using a Vernier caliper and micro meter screw gauge (Mitutoyo, Japan) with accuracy of 0.001 mm. Surface area of the calyxes was determined according to (Nnebue et al., 2015). Calyxes were carefully spread and traced out on a graph paper and counting the number of squares. This method was used because of the irregular shape of the calyxes. The true density ( $\rho_t$ ) of the roselle calyxes were determined by displacement method (Emadi and Saiedirad, 2011; Omobuwajo, Sanni, and Balami, 2000) using Toluene as liquid to avoid any possible absorption of moisture. The bulk density ( $\rho_b$ ) was determined by using the mass/volume relationship (Omobuwajo et al., 2000). The porosity ( $\epsilon$ ) was calculated by the equation (Emadi and Saiedirad, 2011)

$$\epsilon = \frac{\rho_t - \rho_b}{\rho_t} \times 100 \quad (2)$$

Where:

$\epsilon$  = Porosity (dimensionless)

$\rho_t$  = True density ( $\text{g}/\text{cm}^3$ )

$\rho_b$  = True density ( $\text{g}/\text{cm}^3$ )

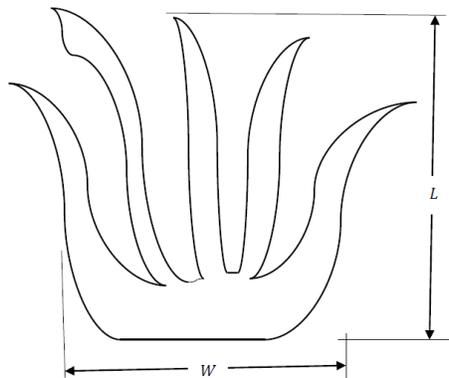


Figure 2. Schematic diagram of roselle calyx showing the axial dimension:  $L$  – length,  $W$  – width / breadth

## Mechanical properties

An Instron universal testing machine (model 3369 UTM, M500 – 50KN, USA) was used to perform tensile tests on the calyces. The instrument (Fig 3) consist of a fixed and adjustable clamps. Owing to the texture of the sample material, the loading speed was set as 2 mm/min for each of the tests. The calyces were cut into rectangular strips and the dimension (thickness and width) were measured. The specimen was held longitudinally in the clamp and pulled axially automatically by the Instron machine until rupture. A computer data acquisition system recorded the magnitude of the applied load and the resulting elongation of the specimen. The test was replicated five (5) times for each sample and average obtained. A force – deformation curve was then generated from these data. The obtained data were used to determine the following tensile properties of roselle calyces;

1. Rupture force ( $F_r$ ) was taken as the maximum peak force required to rupture the calyx in accordance with (Singh and Reddy, 2006).
2. Tensile strength was calculated according to (Singh and Reddy, 2006) as

$$\text{Tensile strength} = \frac{\text{peak rupture force}}{\text{cross-sectional area (thickness x width) of the initial specimen.}} \quad (3)$$

3. Stiffness modulus (S) which is the slope of the force – deformation curve, in the apparent elastic region measured in kN/m (Koya et al., 2011).
4. Modulus of elasticity (E) was calculated as the slope of the initial linear portion of the stress and strain curve (Singh and Reddy, 2006).
5. Toughness which is the energy absorbed per unit volume. This was estimated by the area under the curve up to the rupture point (Koya et al., 2011)



**Figure 3. The Instron Universal Testing Machine (Model 3369) showing a rectangular strip of roselle calyx being pulled axially for determination of tensile properties**

## Aerodynamic properties

The terminal velocity of each part was experimentally measured using the floating method. A vertical air duct (Fig. 4) which was designed by the department of Agricultural and Environmental engineering, OAU, Ile – Ife was used for the test. The apparatus consists of a voltage regulator, an electric motor, a blower and a transparent wind tunnel. The method provides blowing air in a vertical duct for calyces to be floated, the voltage was regulated intermittently to obtain the required air flux speed to suspend the calyx. The airflow velocity at time of suspension called terminal velocity (TV) was measured using a hot wire anemometer (Dwyer 471B-1, MA, USA) with an accuracy of 0.1% accuracy (Emadi, 2008; Emadi and Saiedirad, 2011). Five calyces each were randomly selected from each of the moisture levels and used for the experiment with average values recorded for the various parameters determined.

The drag coefficient of the calyces for each of the sample portion was calculated from the equation

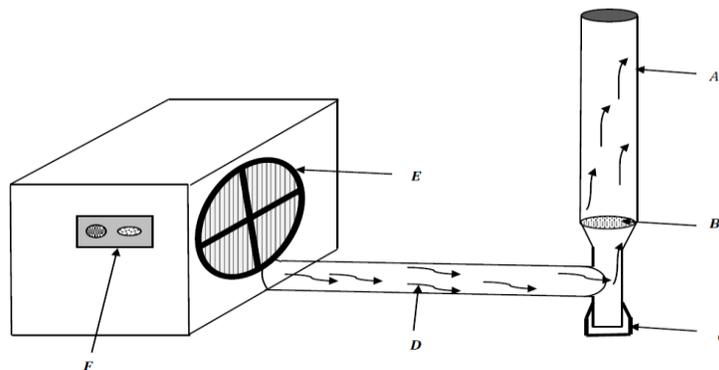
$$C = \frac{2m_t g}{\rho_a (v_t)^2 A_t} \quad (4)$$

Where  $C$  is the drag coefficient,  $m_t$  is the mass of calyx in kg,  $v_t$  is terminal velocity experimentally measured in m/s,  $A_t$  is projected area of the calyx in  $m^2$ ,  $g$  = gravitational acceleration in  $m/s^2$ ,  $\rho_a$  is true density of air in  $kg/m^3$ .

While the projected area,  $A_t$  was calculated using the expression (Helmy, Derbala, Badr, and Shohyieb, 2009)

$$A_t = \frac{\pi}{4} LW \quad (5)$$

Where  $L$  is the length in meter and  $W$  is the width in meter.



**Figure 4. The Experimental set-up for determination of terminal velocity of the roselle calyces: A – transparent wind tunnel; B – wire screen; C – base of the tunnel; D – air current; E – blower and F – voltage regulator**

## Experimental design and Statistical analysis

A completely randomized design was applied for the experiment in which there were three treatment samples (calyces at moisture contents 14.40, 18.00 and 23.87%, w.b), thirty replications for dimensions (Length, width, thickness and surface area) and five replications for true density, bulk density, terminal velocity and the mechanical properties experiments. The

analysis of variance (ANOVA) for the results was performed using the SAS Statistical Software 10.0 (StatSoft, 2012) (at 95% confidence interval).

## RESULTS AND DISCUSSION

### Physical Properties

The physical characteristic, dimensions, surface area, true density, bulk density and porosity of roselle calyces are presented in Table 1. The values presented are within the ranges reported in literature for biological materials (Kaleta and Górnicki, 2011). Emadi and Saiedirad (2011) reported the principal dimension of saffron petals at 87.7% w.b. moisture content as  $38.28 \pm 3.28$ ,  $17.15 \pm 2.40$  and  $0.15 \pm 0.02$  mm for length, width, and thickness respectively (Emadi and Saiedirad, 2011). The results from physical properties measurements show an increase in the thickness, true density and porosity with moisture content from 14.40 – 23.87% w.b. However, the difference in this properties with moisture content, is statistically significant at ( $p < 0.05$ ). The increase could be as a result of rehydration of the cell wall of the calyces which is accompanied by a higher change in mass relative to volume. This was confirmed by the works of (Chavoshgoli et al., 2014; Emadi and Saiedirad, 2011).

**Table 1. Axial dimensions, surface area, true density, bulk density and porosity of roselle calyces at different moisture contents.**

Physical properties	Calyxes moisture content (% w.b)		
	14.40	18.00	23.87
<i>Length (mm)</i>	41.10 (3.7)	42.60 (4.40)	53.10 (4.40)
<i>Width (mm)</i>	20.60 (2.8)	21.60 (1.80)	24.10 (2.70)
<i>Thickness (mm)</i>	1.23 (0.13)	1.27 (0.11)	1.32 (0.12)
<i>Surface Area (mm<sup>2</sup>)</i>	11.74 (1.70)	11.69 (2.30)	15.48 (0.93)
<i>True density (g/cm<sup>3</sup>)</i>	0.74 (0.04)	0.75 (0.006)	0.83 (0.08)
<i>Bulk density (g/cm<sup>3</sup>)</i>	0.11 (0.002)	0.11 (0.002)	0.11 (0.004)
<i>Porosity (%)</i>	85.14	85.64	86.42

Values in parentheses are standard deviation

### Mechanical properties

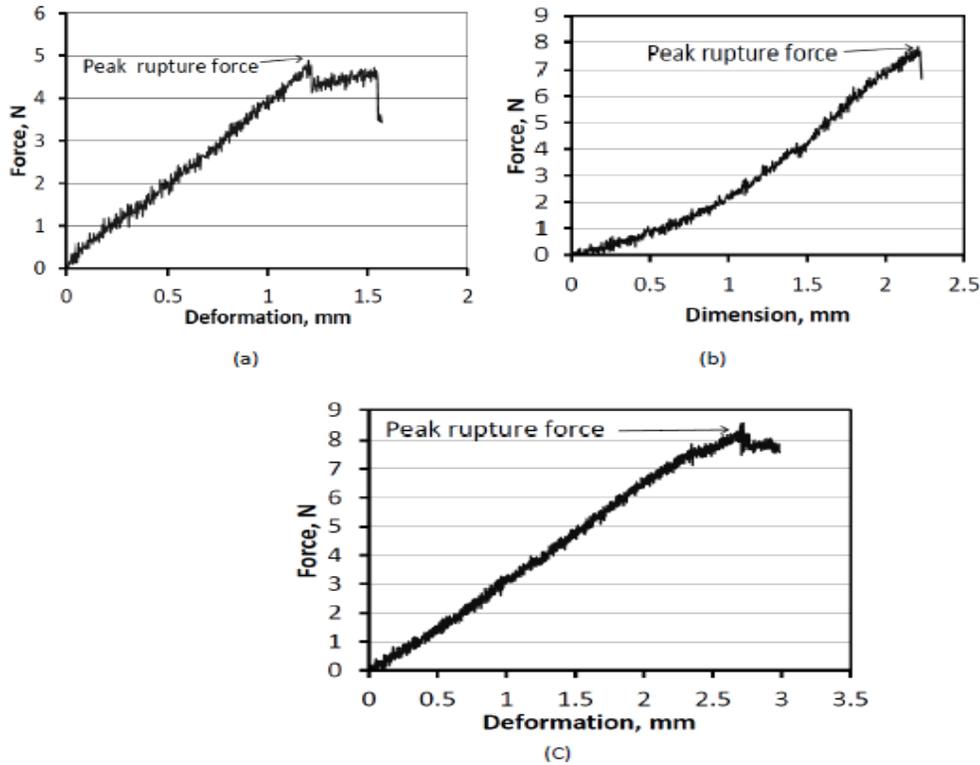
The ranges of peak rupture force; tensile strength; stiffness; modulus of elasticity and toughness of roselle calyces assayed at different moisture content are presented in Table 2. While Figure 5 presents the force-deformation curves of roselle calyces under tensile loading at 2.00 mm/min at the three-moisture content (14.40, 18.00 and 23.87% w.b.). The values obtained for the calyx mechanical properties (peak rupture force, tensile strength, stiffness and toughness) increase with increasing moisture content, except for the modulus of elasticity. peak rupture force ranged from 5.30 – 8.47 N; tensile strength 442 – 692kNm<sup>-2</sup>; stiffness 4.06 – 4.31kNm<sup>-1</sup>; modulus of elasticity 13.47 - 14.78 MNm<sup>-2</sup>and toughness 0.0035 - 0.0112 J. The increase in mechanical properties could be attributed to increase rigidity of the calyx cell wall from hydration by water molecules. Calyx cell wall like every other plant contains a lot of cellulosic material which upon absorption of water becomes more stable due to formation of hydrogen bonding with the water molecules, making it more tensile and less brittle. The decrease in the modulus of elasticity could be explained by the significant change in length of the sample due to an increase in

rupture force. A similar trend was reported by (Singh and Reddy, 2006) who worked on the post-harvest physico - mechanical properties of orange peel. Their results show that the rupture force decrease from  $15.60 \pm 2.5 - 10.80 \pm 2.5$  N, tensile strength decreased from  $0.173 \pm 0.031 - 0.125 \pm 0.03$  Mpa and modulus of elasticity decreased from  $1.57 \pm 0.54 - 1.11 \pm 0.19$  Mpa for orange peels with initial moisture content of 292% db, stored in ambient condition for 1, 3, 7, and 10 days reaching a final moisture content of 252.8%.db. The difference in peak ruptures, tensile strength, stiffness, and modulus of elasticity, with moisture content except toughness were insignificant at ( $P > 0.05$ ).

**Table 2. Peak rupture force; tensile strength; stiffness; modulus of elasticity and toughness of roselle calyces at different moisture contents.**

Mechanical properties	Calyces moisture content (% w.b)		
	14.40	18.00	23.87
Peak rupture force $F_r$ (N)	5.30 (1.16)	7.63 (0.7)	8.47 (1.48)
Tensile strength, $\sigma$ ( $kNm^{-2}$ )	442 (134.71)	605 (39.97)	692 (141.84)
Stiffness ( $kNm^{-1}$ )	4.06 (0.46)	4.31 (1.08)	3.19 (1.14)
Modulus of elasticity, $E$ ( $MNm^{-1}$ )	14.78 (1.71)	13.47 (1.34)	3.81 (2.36)
Toughness (J)	0.0035 (0.0011)	0.0078 (0.0010)	0.0112 (0.0018)

Values in parentheses are standard deviation



**Figure 5. Average\* Force-Deformation Curve of roselle calyces under tensile loading at 2.00 mm/min at different moisture content (a) 14.40 % w.b (b) 18.00% w.b and (c) 23.87% w.b**

## Aerodynamic properties

Table 3 presents the average mass, terminal velocity and drag coefficient of roselle calyces at different moisture contents. The calyces exhibited aerodynamic instability during the experiments due to their asymmetrical and non-uniform shape. Results showed that the terminal velocity of calyces ranges between 2.78 – 3.22 ms<sup>-1</sup>, and the drag coefficient ranges from 1.60 x 10<sup>-3</sup> – 2.9 x 10<sup>-3</sup>, this is within the range reported for sunflower petals and other similar materials (Emadi and Saiedirad, 2011). Although the result did not show a linear increase in terminal velocity as moisture content increases, the drag coefficient decreases with increasing moisture content. The variation could be due to inconsistencies in mass and surface area of sampled calyces.

**Table 3. Average mass, terminal velocity and drag coefficient of roselle calyces at different moisture content**

Aerodynamic properties	Calyces moisture content (% w.b)		
	14.40	18.00	23.87
<i>Average mass (kg)</i>	0.56 (0.047)	0.62 (0.050)	0.64 (0.054)
<i>Terminal velocity (ms<sup>-1</sup>)</i>	2.78 (0.27)	3.22 (0.43)	3.07 (0.36)
<i>Drag coefficient</i>	0.0029	0.0022	0.0016

Values in parentheses are standard deviation

## CONCLUSION

The physical, mechanical and aerodynamic properties of roselle calyces determined in relation to different moisture contents shows that most of the parameters measured increases significantly with moisture content, with the exception of the length, width, bulk density, modulus of elasticity and drag coefficient. The results provide preliminary data that will find application in the design of harvesting and post-processing handling equipment and techniques (e.g. separators, cleaners) for processing and handling roselle calyces.

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