Engineering properties of Roselle (Hibiscus sabdariffa) calyxes

Omotayo Adeteju Aregbesola*, Micheal Olademeji Faborode,
Obinna Ikenna Ezeokoli

(Department of Agricultural and Environmental Engineering, Obafemi Awolowo University, Ile Ife, Osun State, Nigeria)

Abstract: Physical, mechanical and thermal properties of dried and freshly harvested Roselle calyxes were determined using standard procedures based on literature. The obtained results showed that in terms of shape and size, the red variety was more slender with a pointed apex when compared to the dark red variety (Hibiscus sabdariffa. L). Porosity values were close for the dried calyxes and were 53.85% for fresh calyxes. A high percent (72.21%) of whole calyxes was obtained for the fresh calyxes when compared to that for the two varieties of dried calyxes studied. The value of angle of repose for the fresh calyxes was 51.530 while the dry calyxes had lower values (23.10 and 22.40o) for red and dark red calyxes, respectively. Results also showed that the coefficient of static friction on all surfaces used was highest for fresh Roselle calyxes while results obtained for the dry calyxes were similar. Thermal properties of the calyxes varied and were a function of the moisture content of the calyxes.
The results of this study are useful for further studies relating to processing, handling and storage of Roselle calyxes.

Keywords: Roselle calyxes, physical properties, mechanical properties, thermal properties, dry calyxes, fresh calyxes

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1 Introduction

Hibiscus is one of the most common flower plants grown worldwide. More than 300 species of *Hibiscus* can be found around the world, growing in both tropical and subtropical regions (Yadong et al., 2005). Most of the varieties of hibiscus plant are used as ornamental plants; and partly for fibre and food but only *Hibiscus sabdariffa* (Roselle) provides swollen red and green calyxes which are beneficial for both food and non-food applications.

As part of the plant’s flower system, the calyx consists of the group of sepals which surround and protect the flower petals (Meza-Jimenez et al., 2008). These calyxes are usually harvested and separated from the seed capsules once the flowers drop and used for various applications. In Nigeria and the most parts of Africa, two botanical varieties are recognized as the red calyx varieties used mainly for juice production and green calyx varieties used to cook soup, stew and sauces (Ojokoh et al., 2002). Babalola et al. (2001) and Schippers (2000) also classified calyxes grown in Nigeria by three different colour groups: green, red and dark red. It has, however, been reported that there are significant differences in the Roselle plant structure including plant height, leaf lobe, petal colour, shape (long, short, conical or not conical) and colour of calyx (green to red and even purple) (Schippers, 2000). Based on this, classification/characterisation of calyxes on the basis of visual colour alone seems inadequate and hence the need to characterize calyxes extensively to obtain data important for the purposes of design of processes, machinery and methods for processing these calyxes.

Review of literature shows some work on the properties of Roselle seed (Omobuwajo et al., 2000; Ismail et al., 2008; Bamgboye and Adejumo, 2009, 2010, 2011). There is also some information on nutritional composition of Roselle calyxes (Babalola et al., 2001) but there seems to be dearth of information on other

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***Corresponding author: Omotayo Adeteju Aregbesola,** Senior Lecturer, Department of Agricultural and Environmental Engineering, Obafemi Awolowo University, Ile-Ife, Osun State, Nigeria. Email: moregbe@gmail.com; soaregbs@oauife.edu.ng

properties of Roselle calyxes necessary to provide baseline data for engineering and other scientific applications. The aim of this study, therefore, is to determine some physical (calyx dimensions, densities, porosity, percent whole calyx, colour), mechanical (angle of repose, coefficient of friction) and thermal properties (specific heat, thermal conductivity and diffusivity) of varieties of Roselle calyxes grown in Nigeria.

2 Materials and methods

2.1 Materials

Two different types (red and dark red coloured) of dried (low moisture) Roselle calyxes (*Hibiscus sabdariffa*. L) were purchased from a local market in South West, Nigeria while fresh (high moisture), red coloured calyxes were obtained from the Roselle plant cultivated at the Teaching and Research Farm, Obafemi Awolowo University, Ile-Ife, Nigeria. At maturity (after 4 months), about 9 kg of calyxes with capsules containing seeds were harvested and prepared for experiments by separating harvested calyxes (about 5 kg) from capsules. Fresh calyxes were then kept in a freezer and brought out to thaw 24 h before use.

2.2 Moisture content determination

The moisture content of calyxes was obtained by adopting the procedure used for medicine & herbal plants (Soysal and Oztekin 2001; Park et al., 2002). This involved weighing samples, drying in a Gallenkamp oven at a temperature of 105°C for 24 h, cooling and weighing again to determine the amount of water in the calyxes. Moisture content (wet basis) was calculated as the ratio of the weight of water in the calyxes to the total weight of the calyxes before drying. Samples were analyzed in triplicate.

2.3 Determination of physical properties

Thirty calyxes were used to determine physical dimensions of the calyxes. Length and breadth were obtained by measuring the length (from base to the mid apex of the calyx) and width (measured around the base of whole calyxes fully opened up) with a digital vernier caliper. Calyx thickness was measured using a micrometer screw gauge. The sphericity and geometric mean diameter were calculated from the length, width and thickness obtained using the following Equation 1 and Equation 2 (according to Mohsenin, 1978; Keramat et al., 2008 and Kibar and Öztük, 2008)

$$Sphericity = \frac{(L \times W \times T)^{1/3}}{L} \qquad (1)$$

$$Dg = (L \times W \times T)^{1/3} \qquad (2)$$

where;

Dg = geometric mean diameter (mm)

The surface area was obtained by tracing the calyxes on graph paper and calculating the area of squares covered by the calyxes.

A sample size (about 30 g) of calyxes was selected and separated into complete, whole calyxes (i.e. those with 5 complete sepals) and incomplete calyxes. The mass of the two separated fractions (i.e. mass of complete and incomplete calyxes) was obtained using an electronic balance and the percent whole calyxes was obtained as ratio of mass of complete, whole calyxes to total calyxes (30 g) multiplied by 100. This procedure was repeated five times. The unit mass of a single calyx was also determined using the electronic balance. 30 g of calyxes were sorted. The mass of impurities (dry leaves, stones, stalks etc.) was determined and expressed as a percentage. This procedure was also replicated five times.

To determine bulk density of calyxes, an empty container of known volume was loosely filled from a height of 1500 mm with the calyxes. The mass of the container and its contents were then determined using an electronic balance. The bulk density was calculated as ratio of mass of calyxes to volume of the container. This procedure was repeated five times. True density was obtained by liquid displacement method using toulene as liquid while porosity was determined using the Equation 3 (Bagherpour et al., 2010; Emadi and Saiedirad, 2011; Kibar et al., 2014).

$$\text{Porosity} = 1 - \left[{}^{bulk\ density}/_{true\ density} \right] \qquad (3)$$

The colour of calyxes was measured by using a Macbet Munsell colorimeter (Munsell, 1976).

2.4 Determination of mechanical properties

Mechanical properties determined for the calyxes were the coefficient of static friction on different surfaces and angle of repose. The coefficient of static friction of the calyxes was obtained on three surfaces (plastic, galvanized sheet of gauge 18 and black sheet) by using an inclined plane apparatus. The table with calyx sample was gently raised and the angle of inclination to the horizontal at which the sample starts sliding was read off the protractor attached to the apparatus. The tangent of the angle read was also taken as coefficient of friction. The procedure was replicated five times.

The dynamic angle of repose or emptying angle was determined by the method described by Davis and El-Okene, 2009 and Patel et al., 2011. A cylinder opened at both ends, filled with the calyxes was gently raised on a circular plane until the calyxes formed a cone with the surface. The dynamic angle of repose was calculated from the height and base radius of the cone formed. Readings were taken five times.

2.5 Determination of thermal properties

The method of mixtures (Aviara et al., 2008) was adopted to determine the specific heat of the calyxes. Thermal conductivity of Roselle calyxes was determined from the food composition of calyxes (proximate analysis) using the empirical Equation4 developed by Sweat (1986) as given in

$$k = 0.25\ m_c + 0.155\ m_p + 0.16\ m_f + 0.135\ m_a + 0.58\ m_m \qquad (4)$$

where $m_c$is the mass fraction of carbohydrate, m_p, is the mass fraction of protein, m_f, is the mass fraction of fat; m_a, is the mass fraction of ash and m_m is the mass fraction of water in the product.

Proximate analysis of the Roselle calyxes was determined using AOAC standards (AOAC, 1990) and procedures outlined by Babalola et al., 2001.

Thermal diffusivity of the calyxes were obtained from the relationship between density, specific heat and thermal conductivity using Equation 5

$$K= \alpha\ Cp\rho \qquad (5)$$

where K= thermal conductivity, α = thermal diffusivity, Cp = specific heat and ρ = density.

3 Results and discussion

3.1 Physical properties

Results for some physical properties of Roselle calyxes used for the study are presented in Table 1. Dimensions of the length, width and thickness for the red variety of Roselle were in the range of 41.3 – 62.3 mm, 19.5 – 35.0 mm and 3.2 – 9.3 mm, respectively, while the dark red variety had values in the range of 34.5 – 50.3 mm for length, 18.5 – 29.4 mm for width and 5.4 – 9.5 mm for thickness. The red variety appeared more slender with a pointed apex when compared to the dark red variety, hence varietal differences were observed between the two varieties of dried calyxes used for this study. Factors such as soil characteristics and climatic situations during growth could also affect the sizes of the calyxes. Dimensions obtained for the fresh calyxes were within the range of 28.8 – 53.2 mm, 12.6 – 22.6 mm and 14.2 – 24.5 mm for length, width and thickness, respectively. The physical dimensions of the fresh calyxes were, however, within the range of 3.2 - 5.7 cm for length and 1.25 - 2 cm for thickness for wet calyxes given by Morton (1987). It was noticed that similar mean values were obtained for unit mass, volume and surface area of the two varieties of dried calyxes studied. This implied that similar equipment (design based on mass, volume and area of calyxes) could be used for processing, storage and handling of these two varieties.

Table 1 Physical properties of Roselle calyxes

Physical property	Red calyx	Dark red calyx	Fresh calyx
Length, mm	53.7 ±4.8	42.6 ±4.6	42.4 ±5.4
Width, mm	25.3 ±4.6	24.8 ±3.0	18.0 ±2.5
Thickness, mm	5.8 ±0.9	7.4 ±0.9	21.4 ±3.2
Geometric mean diameter, Dg mm	19.8±2.7	19.7 ±2.3	25.1 ±3.5
Surface area,mm^2	1227±27	1220±23	1982 ±35
Sphericity,%	36.87±0.56	46.24±0.51	59.20±0.66
Unit mass of calyx,g	0.98 ±0.01	0.84 ±0.06	5.53 ±1.16
True density ,kg/m^3	0.84±0.05	0.81±0.08	0.78 ±0.02
Bulk density,kg/m^3	0.10 ±0.07	0.11 ±0.04	0.36 ±0.01
Porosity ,%	88.09±0.02	86.42±0.05	53.85±0.01
% impurity	0.83	0.66	negligible
Colour	1.9YR1.15/2.59	5YR7.26/11	3.4YR4.25/6.21

Note: *YR means yellow-red in Munsell notation; ± standard deviation

Bulk and true density values for the two dried red varieties studied were about the same although the free flow bulk densities for calyxes were below the minimum bulk density value of 0.45 g/cm^3 required by importers interviewed by A-SNAPP (2000). As expected, higher density values were obtained for the wet calyxes.

The red calyx had the highest porosity value (88.09%), followed by the dark red calyxes (86.42%) while fresh calyxes had the lowest value of 53.85%. Bulk and true densities for fresh Roselle calyxes obtained in this study were also lower than that given by (Patel et al., 2011) for fresh Mahua flowers but close to values reported for Saffron flower in literature (Emadi and Saiedirad, 2011). Porosity values (49.06 and 52.42) of the fresh Mahua flowers were also close to the value obtained for the high moisture calyxes in this study.

The percent of whole calyxes (i.e. calyxes with five complete sepals) was quite low for the two varieties of dried calyxes studied. The red variety of calyx had a particularly low amount of whole calyxes. This could be attributed to how the calyxes were handled especially during harvest, separation of the calyx from their seed capsule and drying. A high percent of whole calyxes (72.21%) was however obtained for the fresh calyxes which were handled with care during harvest and separation of calyx from seed capsule. According to Plotto (2001), producers are also expected to sell whole calyxes for easy pre-processing cleaning operations, as cleaning becomes almost impossible after processing the calyxes. It is, therefore, important that the calyxes be handled in such a way as to preserve them in whole form as broken calyxes are difficult to sell (Schippers, 2000).

Percent impurities (by mass) in the fresh calyxes were negligible. This could probably be due to the closely monitored handling of the calyxes during harvest. Impurities seen amongst the mass of dried calyxes included Roselle stalk, debris and seed capsules, jute bag parts and sand particles. Though the percentage impurity by weight was not high, there is however the tendency that these impurities could affect the quality of the downstream sector i.e. secondary products (e.g. juice) obtained from the calyxes. Plotto (2001) reported that only Thailand was able to process calyxes to tea because of the superior clean calyxes from this country. Hence, it is important to produce calyxes with reliable qualities, which are also clean.

Scientifically, the colours of the Roselle calyxes were represented by three different values on the colour chart. The dark red had the highest hue value followed by the wet calyxes and then the red variety. Schippers (2000) reported that Roselle varieties differ in colours which ranges from green to red and even purple; even among these colours there are different shades such as purple, dark red, light red, bright red and deep red as given by Obadina and Oyewole (2007). In order to obtain and identify varieties with good quality, it might be advisable to describe colour of calyxes using scientific means instead of the use of mere visual characterization.

3.2 Mechanical properties

Figure 1 shows the angle of repose (θ), in degrees, for dry dark red and red calyxes; and also, for the fresh calyxes while Figure 2 shows the coefficient of static friction on three different surfaces for the Roselle calyxes.

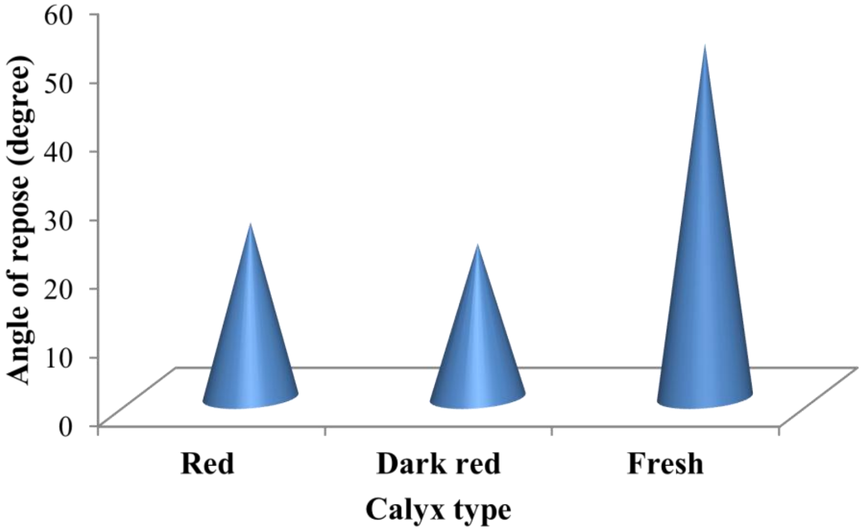

Figure 1 Angle of repose of Roselle calyxes

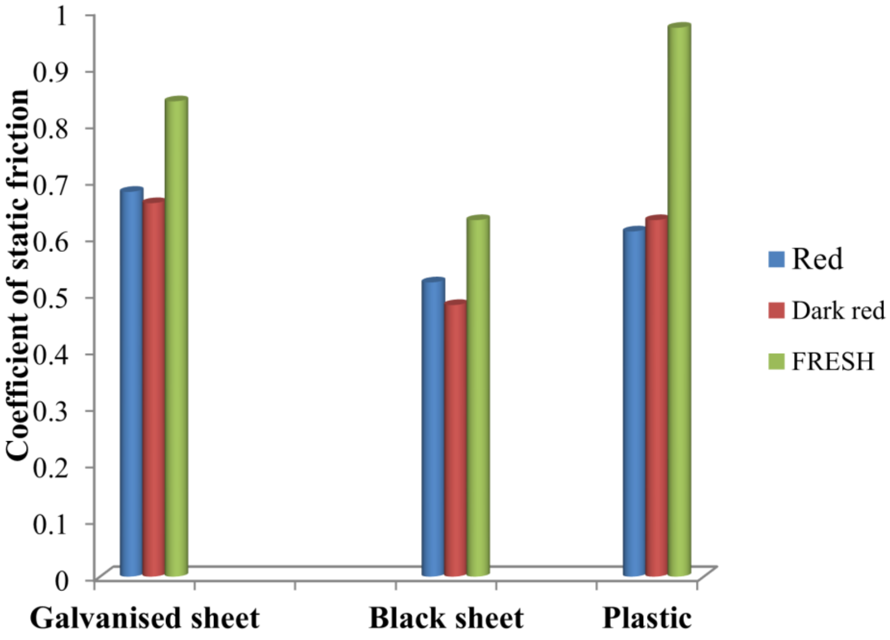

Figure 2 Coefficient of static friction of Roselle calyxes on different surfaces

The highest value of angle of repose (51.53^0) was obtained for the fresh calyxes and dry calyxes had slightly different values (23.10 and 22.40^0) for red and dark red calyxes, respectively). This is expected as the size, shape, moisture content and orientation of the particles influence the angle of repose. The variation of angle of repose with moisture content occurs because the layer of moisture surrounding the particle holds the aggregate of the grain together by surface tension (Sahay and Singh, 2001). The angle of repose obtained for fresh Roselle calyxes was higher when compared to values (45.1 and 47.1) reported for fresh Mahua flowers (Patel et al., 2011).

The coefficient of static friction depends on the type and roughness of the surface horizontal plane, weight and moisture content of the product. Also, like angle of repose, lower values indicate easy flowing material and vice versa (Sahay and Singh, 2001). Results (Figure 2) from this study showed that the coefficient of static friction on all surfaces used was highest for fresh (wet) Roselle calyxes while results obtained for the dry calyxes were similar with little differences among the surfaces studied. For the dry calyxes, the coefficient of static friction was highest on the galvanized sheet surface (0.68 for red and 0.66 dark red calyxes, respectively), followed by plastic (0.61 for red and 0.63 dark red calyxes, respectively) and lowest on black sheet (0.54 for red and 0.48 dark red calyxes, respectively). The lowest value for the fresh calyxes was also on the black sheet surface (0.63) but unlike the dry calyxes, the highest value was obtained for the calyxes on the plastic surface (0.97). This implies that Roselle calyxes flow more easily on black sheet when comparison is made with galvanized sheet and plastic. Therefore, if there is no other design considerations, black sheet is preferred for construction of processing and preservation equipment for Roselle calyxes.

3.3 Proximate composition of calyxes

Table 2 gives the proximate composition of dry and fresh Roselle calyxes used for the study.

Table 2 Percent proximate composition of Roselle calyxes

	Moisture	*Ash*	*Ether extract*	*Crude fibre*	*Carbohydrate*	**Crude protein*
Red calyx	15.28	6.92	1.05	7.90	64.26	4.59
Dark red calyx	16.38	7.59	0.45	7.50	61.65	6.43
Fresh calyx	85.84	1.04	0.94	3.71	5.16	3.31

Note: **Crude protein= total protein× 6.25*

The dry, dark red coloured calyxes had higher protein, ash and moisture values while crude fibre, ether and carbohydrate contents in the dry, red calyxes were greater than those for the dark red calyxes. Variations in proximate composition of Roselle calyxes was also reported by Babalola et al., 2001

3.4 Thermal properties

Thermal properties (specific heat, thermal conductivity and diffusivity) of dry and fresh *Hibiscus sabdariffa* calyxes are shown in Table 3.

Table 3 Thermal properties of *Hibiscus sabdariffa* calyxes

Calyxes	**Specific heat (kJ/ kg K)**	**Thermal conductivity(W/m K)**	**Thermal diffusivity (×10^{-7} m^2/s)**
Red *(15.28%, 0.10 kg/m^3)	1.70 ±0.10	0.27 ±0.08	1.60 ±0.10
Dark red (16.38%, 0.11 kg/m^3)	1.73 ±0.11	0.27 ±0.10	1.42 ±0.08
Fresh (85.84%, 0.36 kg/m^3)	3.36 ±0.08	0.52 ±0.10	4.30 ±0.08

Note: **Moisture content and density in bracket; ±standard deviation*

Fresh calyxes had higher values of the thermal properties studied when compared to the dry calyxes. Bamgboye and Adejumo (2010) reported a decrease in specific heat and thermal conductivity values with increasing moisture content for Roselle seeds; thermal diffusivity for the Roselle seeds, however, was said to increase with an increase in moisture content. Specific heat, thermal conductivity and diffusivity of dry Roselle calyxes were much lower than values for thermal properties of dry Roselle seeds as reported by Omobuwajo et al. (2000) and Bamgboye and Adejumo (2010).The specific heat of the dry Roselle calyxes was almost 50% less than the value obtained for the fresh (wet) calyxes. This is in agreement with Wilhem et al., (2004) who reported that the specific heat of a product is influenced by the products' components, moisture content, temperature and pressure with specific heat increasing as the moisture content increases. The specific heat obtained for fresh *Hibiscus sabdariffa* calyxes were, however, within the range reported for some vegetables in literature. For instance, specific heat for cabbage (91%, moisture), carrot (88%, moisture) and spinach having 87% moisture was given as 3.89, 3.89 and 3.8 kJ / kg K, respectively, according to Toledo (2000).

Values obtained for thermal conductivity of dry calyxes were similar while thermal conductivity of fresh calyxes was almost twice the value for dry calyxes. The influence of moisture content on thermal conductivity has been found to be a dominant factor (Vazarova, 2005). According to Wilhelm (2004), thermal conductivity of most food materials is relatively in the narrow range of 0.2 and 0.5 W/m K and is strongly influenced by the materials' moisture content and to a lesser degree temperature and pressure with foods having high moisture having thermal conductivity values close to the thermal conductivity of water.

Thermal diffusivity is a measure of how fast heat propagates or diffuses through a material. The influence of moisture content on thermal properties of Roselle calyxes was highest with thermal diffusivity values. Thus, thermal diffusivities of dry red and dark red calyxes were 62.86% and 67.05% lower than the thermal diffusivity of the fresh calyxes. This could be attributed to the dependence of thermal diffusivity on bulk density which in turn affects the porosity and movement of heat within the void spaces of the calyxes. Values of thermal diffusivity of dry calyxes obtained in this study were within the range of 1×10^{-7} to 2×10^{-7} m^2/s given for food products by Wilhem (2004). Information on thermal properties of Roselle calyxes provided by this study is useful for the prediction of heat transfer rate and design of heating and storage/packaging systems for the calyxes.

4 Conclusions

The following conclusions can be drawn from this study on the determination some engineering properties of dry (low moisture) and fresh (high moisture) Roselle calyxes:

1. Varietal differences existed between the varieties of dry and wet calyxes studied.
2. The properties of the calyxes determined varied with moisture content of the calyxes.
3. Percent whole calyxes for the dried calyxes was quite low; based on this, it is necessary that fresh calyxes be carefully processed and handled in such a way to obtain higher percentages of whole calyxes desired in regional and international markets.
4. Description of calyxes using scientific means might be a better way to go in identifying and differentiating different varieties of Roselle calyxes and their possible applications.

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