Studies on moisture sorption isotherm and nutritional properties of dried Roselle calyces

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Abstract: Fruits and vegetables are important in human nutrition and commerce; however, they are seasonal and highly perishable and need to be processed into more stable forms such as so as to derive their maximum benefits. The moisture sorption isotherm and nutritional properties of dried roselle calyces was evaluated. Freshly harvested roselle calyces of dark and light red varieties were sundried and oven dried and evaluated for proximate composition and moisture sorption studies. pH of sundried roselle calyces were higher than oven dried roselle calyces. Also, oven dried dark red Roselle calyces was significantly higher in crude protein, fat, dry matter, moisture and ash Higher values potassium, phosphorus, sodium, magnesium, iron, zinc and calcium were also found in red Roselle calyces. Vitamin C content of oven dried light red Roselle calyces was significantly lower than Vitamin C of other Roselle calyces at $p<0.05$. Roselle calyces exhibited the typical three stage sigmoidal curve found in most foods Braunaeur-Emett-Teller (BET Type II). There was also a concomitant increase in the equilibrium moisture content (EMC) as relative humidity increased in all roselle calyces irrespective of the temperature regimes. Sun dried roselle calyces gave the highest rate of water absorption unlike oven dried roselle calyces. In conclusion oven drying is the best method to dry freshly harvested roselle calyces, oven dried dark red roselle calyces was high in nutrient composition and all roselle calyces exhibited the typical three stage sigmoidal curve found in most foods Braunaeur-Emett-Teller (BET Type II).

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Introduction

Roselle (Hibiscus sabdariffa.L) is a member of the family Malvaceae to which okra, cotton and kenaf belong. The flowers of Roselle are generally small. Both the leaves and the fleshy base of the flower (the calyx) are employed in the preparation of soups and sauces. The vegetable is a popular diet during the raining season. Roselle calyx is a cheap source of vegetable protein, fat and minerals. Regular consumption of roselle may reduce nutritional deficiency problems such as night blindness, scurvy and rickets (Ashaye and Adeleke 2009, Babalola et al., 2001)

Hygroscopy of foods affect storage, handling and processing. Valuable information of hygroscopy of foods can be obtained from moisture sorption characteristics of the food.

Moisture sorption isotherm describes the equilibrium moisture content (E.M.C), which is the limit of the moisture that can be attained when food is exposed to air at a given temperature and water activity. (Ashaye and Aina 2008, Alakali et al 2010).

Information on equilibrium moisture content and nutritional properties of dried roselle calyces will stand as a useful guide on its shelf life properties especially for food processors at culinary and industrial levels.

This work evaluated the moisture sorption isotherm and nutritional properties of dried Roselle calyces

Materials and Methods

Raw materials

Roselle calyces (*Hibiscus sabdariffa*) used for this research study were obtained from the experimental farm of Institute of Agricultural Research and Training, I.A.R. & T., Ibadan.

Preparation of samples

Fresh samples of red and light red roselle calyces were sun dried for three days, oven dried for 24hrs at 50^0C and then packed for analysis

Determination of Moisture Content

One (1) gram of each sample was weighed using mettle pc 4410 balance into dry pre-weighed crucibles.

The samples were dried in the oven at 50°C overnight and were cooled. The percentage of dry matter was calculated by using formula below:

Dry matter % = Dry matter weight x 100/ Weight of sample before oven

Also, the percentage of moisture content was determined by using the formula below:

% Moisture content = 100 - % Dry matter.

Determination of ascorbic acid:

Ascorbic acid was determined using the procedure described by Kirk and Sawyer (1991). Standard indophenol's solution was prepared by dissolving 0.05g 2,6-dichloro Indophenol in water diluted to 100ml and filtered. To standardize, 0.053g of ascorbic acid was dissolved in 90ml of 20% metaphosphoric acid and diluted with water to 100ml. 10ml of this solution was pipette into a small conical flask and titrated with indophenol's solution until a faint pink colour persists for 15seconds. 2ml of the extracted juice from the calyces was pipette into a conical flask and 5ml of 20% metaphosphoric acid (as stabilizing agent) was added and made up to 10ml mark with water. It was titrated with the indophenols solution a faint pink colour persists for 15seconds. The vitamin content in the calyces was calculated

Vitamin C in mg/100g = Titre value x 0.212 x 100 / Wt of sample

pH determination:

The pH meter (model BA 350 EDT instruments) was standardized with standard buffer solution 4. 0.and 7.0 The pH was measured by inserting directly the electrodes into 10ml beaker containing the sample.

Determination of Ash

The sample (2g) was weighed into a porcelain crucible. This was transferred into the muffle furnace set at 550°C and left for about 4 hours. About this time it had turned to white ash. The crucible and its content were cooled to about 100°C in air, then room temperature in a desiccator and weighed (AOAC, 1990).

The percentage ash was calculated from the formula below:

% Ash content = Weight of ash x 100/ Original weight of sample

Determination of Crude Protein

The micro-Kjeldahl method for protein determination is employed for protein determination. This is based on three principles:

Digestion: $RNH_2 + 2H_2SO_4 \longrightarrow (NH_4)_2 + SO_4 + CO_2 + H_2O$

Distillation: $(NH_4)_2\ SO_4 + 2NaOH \longrightarrow (NH_3 + H_2O + Na_2SO_4$

Absorption: $3NH_3 + H_3\ BO_3 \longrightarrow (NH_4)_3\ BO_3$

Titration: $(NH_4)_3\ BO_3 + HCl \longrightarrow H_3BO_3 + 3NH_4Cl$

Procedure

The finely ground dried sample (0.5g) was weighed into the micro-Kjeldahl flask. To this were added 1 Kjeldahl catalyst tablet and 10ml of conc. H_2SO_4. These were set in the appropriate hole of the digestion block heaters in a fume cupboard. The digestion was left on for 4 hours after which a clear colourless solution was left in the tube. The digest was carefully transferred into 100ml volumetric flask, thoroughly rinsing the digestion tube with distilled water and the volume of the flask made up to the mark with distilled water. 5ml portion of the digest was then pipetted to Kjeldahl apparatus and 5ml of 40% ($^{w}/_{v}$) NaOH added.

The mixture was then steam distilled and the liberated ammonia collected into a 50ml conical flask containing 10ml of 2% boric acid plus mixed indicator solution. The green colour lsolution was then titrated against 0.01 NHCl solution. At the end point, the green colour turns to wine colour, which indicates that, all the nitrogen trapped as ammonium borate have been removed as ammonium chloride. The percentage nitrogen was calculated by using the formula:

% N = Titre value x atomic mass of nitrogen x normality of HCl used x 4

The crude protein is determined by multiplying percentage nitrogen by a constant factor of 6.25 (AOAC, 1990).

Crude Fat Determination

The dried sample (1g) was weighed into fat free extraction thimble and plug lightly with cotton wool. The thimble was placed in the extractor and fitted up with reflux condenser and a 250ml soxhlet flask which has been previously dried in the oven, cooled in the dessicator and weighed. The soxhlet flask is then filled to ¾ of it volume with petroleum either (b.pt. 40 – 60°C) and the soxhlet flask extractor plus condenser set was placed on the heater. The heater was put on for six hours with constant running water from the tap for condensation of ether vapour. The set is constantly watched for ether leaks and the heat sources is adjusted appropriately for the either to bril gently. The ether is left to siphon over several times at least 10 – 12 times until it is short of siphoning. It is after this is noticed that any ether content of the extractor is carefully drained into the ether stock bottle. The thimble-containing sample is then removed and dried on a clock glass on the bench top. The extractor flask with condenser is replaced and the distillation continues until the flask is practically dried. The flask which now contains the fat or oil is detached, its exterior cleaned and dried to a constant weight in the oven (AOAC, 1990). If the initial weight of dry soxhlet flask is Wo and the final weight of oven dried flask + oil/fat is W_1, percentage fat/oil is obtained by the formula:

$(W_1 – Wo) \times 100$/Weight of sample taken

Crude Fibre Determination

The sample (2g) was accurately weighed into the fibre flask and 100ml of $0.25NH_2SO_4$ added. The mixture was heated under reflux for 1 hour with the heating mantle. The hot mixture was filtered through a fibre sieve cloth. The filterate obtained was thrown off and the residue was returned to the fibre flask to

which 100ml of (0.31NNaOH) was added and heated under reflex for another 1 hour.

The mixture was filtered through a fibre sieve cloth and 10ml of acetone added to dissolve any organic constituent. The residue was washed with about 50ml hot water twice on the sieve cloth before it was finally transferred into the crucible. The crucible and the residue was oven-dried at 105°C overnight to drive off moisture. The oven-dried crucible containing the residue was cooled in a dessicator and later weighed to obtain the weight W_1. The crucible with weight W_1 was transferred to the muffle furnace for ashing at 550°C for 4 hours. The crucible containing white or grey ash (free of carbonaceous material) was cooled in the dessicator and weighed to obtain W_2. The difference $W_1 - W_2$ gives the weight of fibre (AOAC, 1990). The percentage fibre was obtained by the formula:

% Fibre = $(W_1 - W_2)$x 100/Weight of sample

Phosphorus determination

The ash of each sample obtained was treated with 2MHCL solution 10ml of the filtrate solution was pipetted into 50ml standard flask and 10ml of vanadate-molybdrate yellow solution was added and the flask was made up to mark with distilled water, stoppered and left for 10 minutes for full yellow colour development. The concentration of phosphorus was obtained by measuring the optical density (OD) or absorbance of the solution in a Spectronic 20 spectrophotometer or calorimeter at a wave length of 470mm.

The percentage phosphorus was calculated from a standard graph by appropriate mathematical relationship applicable to such determination (AOAC, 1990).

Potassium and Sodium

Potassium and Sodium were estimated using a Jenway digital flame photometer spectronic 20. (AOAC,1990).

Calcium, magnesium, zinc and Iron

Calcium, magnesium, zinc and iron were determined spectrophotometrically using Bulk 200 atomic absorption spectrophotometer (Buck Scientific, Norwalk) and the absorption compared with absorption of standards of these minerals (Osundahunsi and Aworh, 2003).

Determination of equilibrium moisture content (EMC)

Samples were conditioned to constant weights over either 90% concentrated sulphuric acid (drying) before the EMC was determined. A static gravimetric method (Spiess and Wolf, 1983) was used.

Duplicate sample, 1g each were placed in the upper section of each glass desicator on wire mesh, while the lower section contained standard salt solution over excess salt. After inscertation by the samples and salts, the desicators were seated with silicone grease and placed in a constant temperature environment of 18°C. The samples were weighed at interval of 24 hours until equilibrium was reached when four consecutive measurements are the same. This book between 20 – 25 days. The EMC was determined by Labuza (1984) and average values were used for all calculations. The salt used and their corresponding water activity (aw).

EMC values were obtained using equation below:

EMC = WF – Wi (Wt of sample x moisture content)/ (Sample Wt – MC)

Wf = Final weight of sample + crucible

Wi = Initial weight of sample + crucible.

Statistical Analysis

Data was subjected to analysis of variance and their means were separated by Duncan Multiple range test Duncan 1955.

Results and discussion

Proximate composition of oven and sun dried roselle calyces

In Table 1, it can be depicted that the pH of sundried roselle calyces were higher than oven dried roselle calyces. This may be due to the increased activities of the microorganisms resulting in production of organic acids from available nutrients as described by (Okafor 1978). Also, oven dried dark red Roselle calyces was significantly higher in crude protein, fat, dry matter, moisture and ash This slight increase may be due to chance fermentation by microorganisms as a result of increase in the extracellular enzymatic secretion (Ashaye et al 2008, Bolade et al 2009). Higher values potassium, phosphorus, sodium, magnesium, iron, zinc and calcium were also found in red Roselle calyces. This observation agreed with the findings of Ho-Hsien et al.,2005 who highlighted that the higher values found in the calyces may be as a result of varietal characteristics, soil nutrient and climatic condition.

Vitamin C content of oven dried light red Roselle calyces was significantly lower than Vitamin C of other Roselle calyces at $p<0.05$ due to instability of Vitamin C at higher temperatures (Ashaye et al 2008).

Water adsorption isotherm of sun dried and oven dried roselle calyces

Figures (1, 2 and 3) show the rate of water adsorption of sun dried and oven dried roselle calyces at 27°C, 35°C, and 37°C. The roselle calyces exhibited the typical three stage sigmoidal curve found in most foods Braunaeur-Emett-Teller (BET Type II).

There was also a concomitant increase in the equilibrium moisture content (EMC) as relative

humidity increased in all roselle calyces irrespective of the temperature regimes. Sun dried roselle calyces gave the highest rate of water absorption unlike oven dried roselle calyces. From this observation; it is not advisable to store these roselle calyces at relative humidity's greater than 60% because of greater rate of water uptake. Maintaining relative humidity between 60% and 62% will be safe for storage purposes. This trend agrees with published studies of (Ajibola *et al* 2003, Denloye and Adejohn 1983).

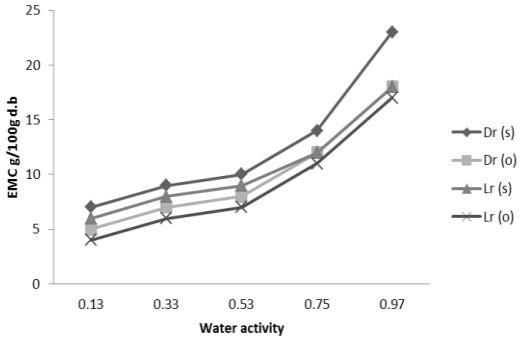

Fig 1 Water absorption property of dried roselle calyces at 27^0C
Dr (s): Sun dried dark red roselle calyce
Dr (o): Oven dried dark red roselle calyce
Lr (s): Sun dried light red roselle calyce
Lr (o): Oven dried light red roselle calyce

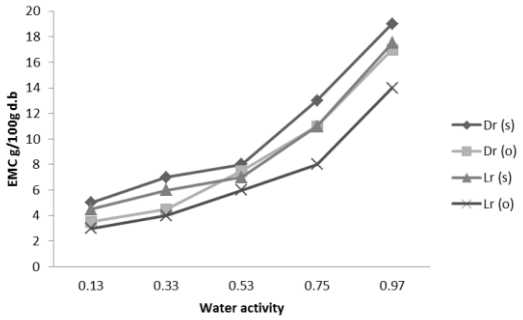

Fig 2 Water absorption property of dried roselle calyces at 35^0C
Dr (s): Sun dried dark red roselle calyce
Dr (o): Oven dried dark red roselle calyce
Lr (s): Sun dried light red roselle calyce
Lr (o): Oven dried light red roselle calyce

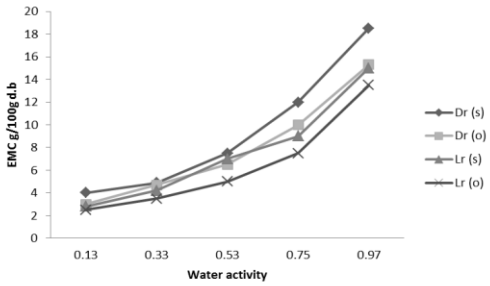

Fig 3 Water absorption property of dried roselle calyces at 37^0C
Dr (s): Sun dried dark red roselle calyce
Dr (o): Oven dried dark red roselle calyce
Lr (s): Sun dried light red roselle calyce
Lr (o): Oven dried light red roselle calyce

Table 1: Chemical Composition of Oven and Sun-dried Roselle Calyces.

	pH	Crude Protein (%)	Fat (%)	Dry Matter (%)	Moisture (%)	Crude Fibre (%)	Ash (%)	K (%)	P (%)	Na (%)	Mg (%)	Fe (%)	Zn (%)	Ca (%)	Vit. C Mg/100g
Sun dried Dark Red Roselle	2.56d	1.575b	0.57b	84.42c	15.58b	2.48b	8.79b	0.078c	0.059b	0.0113	0.029c	0.0038	0.0014	0.168c	14.84a
Sun dried Light Red Roselle	2.68c	0.875d	0.19d	83.26d	16.74a	1.61d	8.14d	0.072d	0.017d	0.0108d	0.021d	0.0034d	0.0009d	0.155d	11.66a
Oven dried Dark Red Roselle	3.48b	2.10a	0.61a	93.75a	6.25d	2.69a	8.97a	0.092a	0.072a	0.0134a	0.043a	0.0057a	0.0021a	0.23a	11.66b
Oven dried Light Red Roselle	3.62a	1.225c	0.23c	92.69b	7.31c	1.74c	8.32c	0.083b	0.026c	0.0119b	0.031b	0.0045b	0.0016b	0.18b	8.48c

Means in the same column followed by the same letter are not significantly different from each other at P<0.05.

Conclusion.

It can be concluded thatoven drying is the best method to dry freshly harvested roselle calyces, oven dried dark red roselle calyces was high in nutrient composition and all roselle calyces exhibited the typical three stage sigmoidal curve found in most foods Braunaeur-Emett-Teller (BET Type II).

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