



Physicochemical properties of *Hibiscus sabdariffa* drinks in different packaging materials during ambient and refrigeration storage

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Abstract

Evaluated was the effect of packaging materials and storage temperatures on the physicochemical properties of *Hibiscus sabdariffa* drinks selected after sensory analysis. Sensory analysis was on *Hibiscus sabdariffa* drinks produced with 23, 28, 33 and 38% of *Phoenix dactylifera* slurry and 2% of pineapple peel extract representing sample A-D respectively and a control with artificial pineapple flavour. Degree of likeness for aroma, appearance, colour, flavour, sweetness, sourness, smoothness and overall acceptability respectively, ranged from 6.05 - 7.80, 6.25 - 7.60 and 6.64 - 7.45 5.45 - 7.75, 4.90 - 7.80, 5.35 - 6.60 5.05 - 7.55 and 5.66 - 7.51. Sample D had significantly ($P \leq 0.05$) higher degrees of likeness amongst the test samples and was selected for storage studies. Sets of pasteurized drinks packaged in polyethylene sachet, plastic and glass bottles were stored at refrigeration temperature ($4.4 \pm 2^\circ\text{C}$) for 4 weeks and at ambient temperature ($25 \pm 2^\circ\text{C}$) for 3 weeks. Packaging materials had no significant ($P > 0.05$) influence on the physicochemical properties. pH for HAPF and HPPE decreased respectively, from ≤ 4.60 to ≥ 2.50 and ≤ 4.57 to ≥ 2.76 at $4.4 \pm 2^\circ\text{C}$ and; ≥ 4.10 to ≤ 2.47 and ≥ 4.51 to ≤ 2.54 at $25 \pm 2^\circ\text{C}$. Total soluble solid (TSS) in °Brix decreased from ≤ 2.25 to ≥ 1.95 and ≥ 9.05 to ≤ 7.90 at $4.4 \pm 2^\circ\text{C}$ and; ≤ 2.15 to ≥ 1.85 and ≤ 9.15 to ≥ 7.05 at $25 \pm 2^\circ\text{C}$ respectively, for HAPF and HPPE. HPPE drinks had significantly ($P \leq 0.05$) the highest TSS. Viscosity (Pa.s) increased for HAPF and HPPE respectively, from ≥ 0.22 to ≤ 0.49 and; ≥ 0.19 to ≤ 0.51 at $4.4 \pm 2^\circ\text{C}$ and ≥ 0.20 to ≤ 0.37 and ≥ 0.23 to ≤ 0.46 at $25 \pm 2^\circ\text{C}$. Any of the packaging can be used with storage for not more than 3 weeks at refrigeration temperature.

Keywords: packaging, hibiscus sabdariffa drink, *Phoenix dactylifera*, pineapple peel extract, pineapple flavour, physicochemical properties, storage temperature

Introduction

Roselle (*Hibiscus sabdariffa* Linn) is a shrub belonging to the family Malvaceae. The plant is widely grown in tropics like Caribbean, Central America, India, Africa, Brazil, Australia, Hawaii, Florida and Philippines, Saudi Arabia, Malaysia, Indonesia, Thailand, Philippines, Vietnam, Sudan, Egypt and Mexico (Zaman *et al.*, 2017) [32]. In Nigeria, *Hibiscus sabdariffa* is widely grown in the North Eastern and middle belt regions for its calyx and it is commonly known as *Zoborodo* in the North (Hausa), *Isapa* in the West (Yoruba) and Sorrel in English (Adebayo-tayo and Samuel, 2008) [2]. The calyx is a very important part of the plant containing valuable components such as anthocyanin and organic acids which determine the colour, flavour and aroma of the product. It is also a good source of nutrients (Abdulazeez *et al.* 2017) [1], rich in malic acid, anthocyanins, ascorbic acid and minerals (Calcium, Magnesium, Phosphorus) (Balarabe *et al.* 2019; Javadzadeh *et al.*, 2017) [6, 15]. The fleshy red calyxes are commonly used for the production of soft drinks and tonic without alcohol like wine, juice, jam, jelly, syrup and also dried and brewed into tea and spice (Mady *et al.*, 2009) [17].

Phoenix dactylifera L. popularly called date palm is a delicious perennial monocotyledon tree crop belonging to the family Aracaceae (Uba *et al.*, 2015) [29]. The fruit has great importance in human nutrition due to their rich content of essential nutrients such as carbohydrate mostly inverted sugars (glucose and fructose) (Alghamdi *et al.*, 2018) [3]. Apart from sugars, dates also contain other important components such as proteins, fat, vitamins, dietary fiber, fatty acids, polyphenols, antioxidant and amino acids (Chandrasekaran and Bahkali 2013) [9]. The fruit has been reported to be of great benefit in the control of glycemic and lipid indexes of diabetic patients (Khalid *et al.*, 2017) [16], reducing blood cholesterol levels (Ali *et al.*, 2012) [4]. Date fruit extract is marked by its high sugar content (64% fresh weight basis) and are considered a natural sweetener alternative to sucrose in the production of liquid extract and many food formulations (Noui *et al.*, 2019) [18]. Many derivatives from dates could be employed as ingredients in food sectors such as the baked products, beverage, confectionery, dairy and sugar industries (Tang *et al.*, 2013; Ghazal *et al.*, 2016) [28, 12]. The drink from the red calyx of *Hibiscus sabdariffa* is characterized by a reddish colour from high concentration anthocyanin (Singh *et al.*, 2017) [26] and sour taste from the organic acid component and is commonly called *zobo* in Nigeria. It is a very popular drink among people of various socioeconomic classes in West Africa and Nigeria in particular. Like the raw material it is made from, the drink is rich in Vitamin C, phytochemicals: anthraquinones, glycosides, alkaloids, tannins,

polyphenols and saponins and is of high medicinal value, used as antihypertensive, astringent, diuretic and purgative agents, which translates to its numerous health benefits (Nwachukwu *et al.*, 2015) ^[19]. The drink is often flavoured and sweetened with sucrose, fruits such as pineapples, apples, oranges etc. and with other artificial sweeteners and flavour agents. However, the consumers' health concerns over the use of additives calls for an alternative that will not only enhance taste and flavour but the nutrient component of the drinks. The addition of *Phoenix dactylifera* to the reddish sour *Hibiscus sabdariffa* extract could be that good alternative to a quality *Hibiscus sabdariffa* drink.

The traditional way by which the drink is produced predisposes to microbial contamination and a short shelf-life (2-3 days) manifested as losses in physicochemical and organoleptic quality under room temperature (Damisa *et al.*, 2007; Bello *et al.*, 2014) ^[11, 8]. The preservation of the drink will depend not only on the processing and storage conditions which must be hygienic but also on the suitability of the packaging materials. Various packaging materials including glass bottles, plastic bottles and polythene sachets are currently used for packaging and storage of *zobo* drink in Nigeria. In most cases, there is no consideration on their suitability. The use of plastic bottles which may not be adequately sterilized renders the product susceptible to microbial contamination thereby reducing the shelf life of the product (Obinna-Echem *et al.*, 2019) ^[22]. Numerous complex factors, including the protection provided by the packaging material, affect nutrient loss in juice and beverages. The permeation through them and the degradation of color and nutrients by oxygen transmission through packages have been an increasing area of research (Sharma and Dinesh, 2010) ^[25]. This study therefore evaluated the sensory properties of *Hibiscus sabdariffa* drink sweetened and flavoured with *Phoenix dactylifera* and pineapple peel extracts, against its counterpart with artificial pineapple flavour; and the effect of packaging materials on the physicochemical properties drinks during ambient and refrigeration storage.

Materials and Methods

Roselle calyces, Date and Pineapple

Dried calyces of roselle (*Hibiscus sabdariffa*), pineapple, date (*Phoenix dactylifera*) were purchased from fruit garden market, Port Harcourt, Rivers State Nigeria. Chemicals and equipment of analytical grade were obtained from the analytical laboratory, Department of Food Science and Technology, Rivers State University, Port Harcourt.

Preparation of Pineapple peel oil extract

Cold extraction technique was applied for the extraction of the pineapple peel oil. Peels were sun dried for 3 days, oven dried for 24 h at 80°C, ground to powder and soaked in n-hexane for 3 days. The mixture was decanted and the oil was obtained after evaporation in a water bath to remove the excess solvent from the oil.

Laboratory production of drink from *Hibiscus sabdariffa* calyces

Roselle drink was prepared using the method of Chibueze *et al.*, (2019) ^[10]. Six hundred grams (600 g) of the dried calyces of *Hibiscus sabdariffa* was sorted, washed with portable water to remove dirt's and impurities and boiled for 30-45 min in a pot containing 10 liters of water. After boiling, it was set aside to cool to room temperature and liquid extracts filtered using a clean sterile muslin cloth.

Preparation of date slurry

The dried dates were sorted, manually pitted by cutting open one side of the fruit and removing the pit, washed, conditioned by soaking in water for 5 h and then wet milled into slurry using blender.

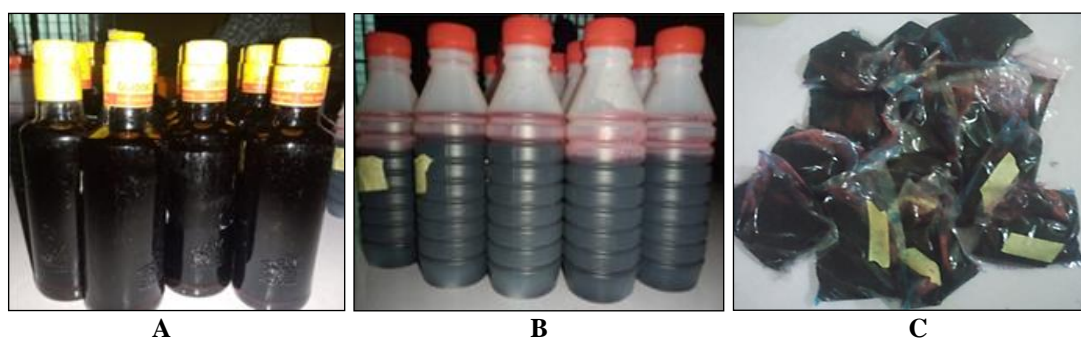


Fig 1: Samples of *Hibiscus sabdariffa* drinks in A. Glass bottles, B. Plastic bottles and C. Polyethylene sachets

Preparation of *Hibiscus sabdariffa* drink with *Phoenix dactylifera* and pineapple peel extract

A set of the extract from *Hibiscus sabdariffa* calyce was sweetened with an artificial pineapple flavour which served as control and another set had the addition of 23, 28, 33 and 38% of *Phoenix dactylifera* slurry and 2% of pineapple extract representing sample A-D respectively. The mixture was homogenized and sieved using a muslin cloth and thereafter pasteurized at 72°C for 10 min, allowed to cool and chilled in the refrigerator for sensory analysis. Sample D (Table 3) with significantly ($P < 0.05$) the highest degree of likeness from the sensory analysis was selected for storage stability studies. More quantity of sample D drink and the drink with artificial

pineapple flavour was prepared, pasteurized and aseptically dispensed in 100 ml quantity into plastic bottles, glass bottles and polythene sachet (Figure 1). A set of the drinks was stored at refrigeration temperature ($4.4\pm 2^{\circ}\text{C}$) for 4 weeks and another set at ambient temperature ($25\pm 2^{\circ}\text{C}$) for 3 weeks.

Sampling for Physicochemical Analysis

Samples were aseptically withdrawn each week from the packaging materials for the duration of storage under the two temperature conditions for physicochemical analysis

Physicochemical Analysis

Determination of pH

Standard analytical method of AOAC, (2012) [5] was used. pH of the drink was determined using a pH meter (TS 625, USA). Twenty (20 ml) of the drink was measured into a beaker and the pH was determined after the meter was calibrated using standard buffer of pH 4.0 and 7.0, sufficient time was allowed for stabilization before readings were taken.

Determination of Total Soluble Solids as Sugar

The hand-held sugar refractometer (Shanghai Tianlei Instrument, Shanghai, China) was used to determine the total soluble solid content as sugars in the *Hibiscus sabdarifa* drinks. The prism of the refractometer was cleaned and a drop of the drink sample was placed on the prism and closed. The sugar content percentage (soluble sugar) was read from the scale of the refractometer when held close to the eye and expressed in °Brix.

Determination of Viscosity

Viscosity was determined with the aid of a Rotary digital viscometer (NDJ.8S, China) using spindle number 2 at 12 rpm. About 25 ml of the sample was transferred into a 250 ml beaker. The content of the beaker was introduced directly unto the rotating spindle and the value of the viscosity displayed on the LCD screen in Pa.s was recorded.

Sensory analysis

Sensory attributes: aroma, appearance, colour, flavour, sweetness, sourness, smoothness and overall acceptability of the drink samples were analyzed using a 9 - point hedonic scale, with the degree of likeness of the product attribute expressed as: 1 - dislike extremely, 2 - dislike very much, 3 - dislike moderately, 4 - dislike slightly, 5 - neither like nor dislike, 6 - like slightly, 7 - like moderately, 8 - like very much and 9 - like extremely (Obinna-Echem and Torporo, 2018)[21]. The coded drinks were served chilled in 20 ml disposable plastic cups to a panel of 20 assessors consisting of staff and students of the Department of Food Science and Technology, River State University, Port Harcourt who are regular consumers of *Hibiscus sabdarifa* drinks.

Statistical Analysis

Data collected were statistically analyzed using Minitab (Release 18.1) Statistical Software English (Minitab Ltd. Coventry, UK). Statistical differences among variables were evaluated by analysis of variance (ANOVA) under general linear model and Tukey pairwise comparisons at 95% confidence level. The non-parametric Friedman test was employed in determining the statistical differences among the product sensory attributes.

Results and Discussion

pH of *Hibiscus sabdarifa* drinks in different packaging materials during refrigeration ($4.4\pm 2^{\circ}\text{C}$) and ambient ($25\pm 2^{\circ}\text{C}$) storage.

pH of *Hibiscus sabdarifa* drinks packagings under storage is presented in Table 1. pH of the drink varied significantly ($P\leq 0.05$) with storage time at both temperatures. The rate of decrease at ambient temperature was significantly ($P\leq 0.05$) higher than at refrigeration temperature. At refrigeration temperature, there was no significant ($P>0.05$) difference between the artificially flavoured drink (HAPF) and the *Phoenix dactylifera* and pineapple extract drink (HPPE) samples. There was significant ($P\leq 0.05$) decrease in pH of the drinks with increase in storage time from $\leq 4.60 - \geq 2.50$ for HAPF drinks and $\leq 4.57 - \geq 2.76$ for HPPE samples. Packaging materials had no significant ($P>0.05$) effect on the pH of the drinks irrespective of the composition in week 1 and 4 but there were significant ($P\leq 0.05$) variations in week 2 and 3. The values ranged from 4.24 - 4.60, 3.04 - 3.68, 3.01 - 2.81 and 2.83 - 2.50 in week 1, 2, 3 and 4 respectively. The drinks packaged in polyethylene sachets had significantly ($P\leq 0.05$) the highest pH values in week 2 while the pH of the other samples did not differ significantly ($P>0.05$). In week 3, the drinks packaged in sachet had the highest pH that did not vary significantly ($P>0.05$) from that of pineapple extract drink in glass bottle. At ambient temperature, there was significant ($P\leq 0.05$) decrease in pH of the drinks with storage time. In HAPF samples the values ranged from $\geq 4.10 - \leq 2.47$. There was no significant ($P>0.05$) difference between the packing materials on week 1 and week 3. pH of the drink packaged in sachet was significantly ($P\leq 0.05$) higher than glass and plastic samples in week 2. In HPPE samples pH decreased from $\geq 4.51 - \leq 2.54$. There was no significant ($P>0.05$) difference between the packaging materials in week 1 and 2, while in week 3 drinks packaged in glass had the least pH. Between the two drinks, the decrease in pH of HPPE samples were significantly ($P\leq 0.05$) higher than HAPF samples.

pH of the drinks confirmed the report that *Hibiscus sabdarifa* drinks is acidic due to the presence of organic acids mainly citric, hibiscus, malic, tartaric and ascorbic acids (Izquierdo-Vega *et al.*, 2020) [14]. The final pH of

the drinks at the end of the storage period were within the range of pH (2.49 – 2.73) reported by Bamishaiye *et al.*, (2011) [17]. The drinks beyond the storage duration at the different temperatures may become more acidic and unpleasant for consumption. The high acidity is a good quality for extension of the shelf-life of the drink due to its effect on microbial growth. However, for people with stomach ulceration it will be preferable to consume the drink within the first week of storage when the pH (4.10 – 4.60) is not highly acidic. Decrease in pH of the drinks with the increase in storage time could be attributed to microbial or other biochemical activities. The composition of the drink, had no significant ($P \leq 0.05$) different on the change in pH but the storage temperature affected the rate of decrease in pH which was higher at ambient temperature. This implies that the activities that led to decrease in pH is not dependent on the drink composition but on temperature of storage and partly on the packaging materials. This is obvious as microbial and chemical activities are slowed down at refrigeration temperature. The higher pH of the samples in the sachet indicated a slowed down activity in the sachet although there was no difference in final pH on week 4 at refrigeration temperature. At ambient temperature, there was a faster utilization of other content of the drink producing organic acids that led to higher decrease in pH in HAPF samples.

Table 1: pH of *Hibiscus sabdarifa* drinks in different packaging materials during storage at refrigeration and ambient temperatures

Samples	Packaging	Refrigeration Temperature (4.4±2°C)				Ambient Temperature (25±2°C)		
		Time (Weeks)						
		1	2	3	4	1	2	3
HAPF	Glass	4.50±0.01 ^{al}	3.10±0.03 ^{b2}	2.89±0.04 ^{bc3}	2.71±0.04 ^{a3}	4.25±0.01 ^{bc1}	3.26±0.07 ^{ab2}	2.47±0.01 ^{bc3}
	Plastic	4.54±0.02 ^{al}	3.11±0.01 ^{b2}	2.88±0.01 ^{bc23}	2.50±0.43 ^{a3}	4.38±0.35 ^{abc1}	3.17±0.21 ^{b2}	2.41±0.00 ^{de3}
	Sachet	4.60±0.12 ^{al}	3.65±0.04 ^{a2}	2.93±0.02 ^{ab3}	2.68±0.07 ^{a3}	4.10±0.09 ^{c1}	3.52±0.14 ^{a2}	2.46±0.01 ^{cd3}
HPPE	Glass	4.45±0.06 ^{al}	3.13±0.02 ^{b2}	2.97±0.04 ^{ab3}	2.81±0.04 ^{a4}	4.53±0.01 ^{ab1}	3.08±0.06 ^{b2}	2.37±0.04 ^{e3}
	Plastic	4.42±0.14 ^{al}	3.04±0.04 ^{b2}	2.81±0.01 ^{c2}	2.76±0.05 ^{a2}	4.51±0.03 ^{ab1}	3.13±0.07 ^{b2}	2.53±0.02 ^{ab3}
	Sachet	4.57±0.01 ^{al}	3.68±0.06 ^{a2}	3.01±0.01 ^{a3}	2.83±0.04 ^{a4}	4.63±0.03 ^{al}	3.19±0.04 ^{b2}	2.54±0.04 ^{a3}

Values are mean ± Standard deviation of duplicate samples.

Values with different alphabetic superscript (^{abc}) along the column for each storage condition are significantly ($P \leq 0.05$) different

Values with different numeric superscript (¹²³) along the row for each storage condition are significantly ($P \leq 0.05$) different

HAPF - *Hibiscus sabdarifa* drink + artificial pineapple flavour

HPPE - *Hibiscus sabdarifa* drink + *Phoenix dactylifera* + pineapple peel extract

Total soluble solid (TSS) content of *Hibiscus sabdarifa* drinks in different packaging materials during refrigeration (4.4±2°C) and ambient (25±2°C) storage.

As shown in Table 2, total soluble solid (TSS) content of the drinks decreased with storage time and some of the packaging materials showed variations. Temperature of storage also had significant ($P \leq 0.05$) effect on the decrease in the TSS content of the drinks; the decrease was significantly ($P \leq 0.05$) greater at ambient temperature. At refrigeration temperature, TSS decreased from ≤ 2.25 - ≥ 1.95 in HAPF and ≥ 9.05 - ≤ 7.90 in HPPE drink samples. Packaging materials had no significant ($P > 0.05$) effect on the TSS of HAPF samples at any given time while HPPE drink samples in plastic bottle had significantly ($P \leq 0.05$) the least in week 3 and 4. At ambient temperature, TSS in HAPF samples decreased from ≤ 2.15 - ≥ 1.85 and there was no significant ($P > 0.05$) difference between the packaging materials. In HPPE samples, TSS decreased from ≤ 9.15 - ≥ 7.05 . There was no significant ($P > 0.05$) difference between the packaging materials on week 1, but on week 2 and 3 samples packaged in plastic had significantly ($P \leq 0.05$) the least TSS content and samples packaged in glass had the highest.

Total soluble solid content of the HPPE samples on week were comparable with the report by Nwafor and Ikenebome, (2009) [20]. Addition of *Phoenix dactylifera* and pineapple extract were responsible for the significantly ($P \leq 0.05$) higher TSS content in HPPE drink samples. The decrease in TSS content of the drinks with storage is indicative of biochemical reaction which is significantly ($P \leq 0.05$) higher at ambient temperature as molecular reactions are speed up by temperature (Paquet *et al.*, 2014) [24]. These reactions could be said to be higher in plastic bottles as drinks in them had the least TSS content in week 2 and 3.

Table 2: Total Soluble Solid ($^{\circ}$ Brix) of *Hibiscus sabdarifa* drinks in different packaging materials during storage at refrigeration and ambient temperatures

Samples	Packaging	Refrigeration Temperature (4.4±2 $^{\circ}$ C)				Ambient Temperature (25±2 $^{\circ}$ C)		
		Time (Weeks)						
		1	2	3	4	1	2	3
HAPF	Glass	2.30±0.00 ^{b1}	2.05±0.00 ^{b2}	2.00±0.00 ^{c2}	2.00±0.00 ^{c2}	2.05±0.07 ^{b1}	1.85±0.07 ^{c2}	1.80±0.00 ^{c2}
	Plastic	2.40±0.14 ^{b1}	2.00±0.07 ^{b2}	2.00±0.07 ^{c2}	2.00±0.00 ^{c2}	2.15±0.07 ^{b1}	1.85±0.14 ^{c2}	1.85±0.07 ^{c2}
	Sachet	2.25±0.07 ^{b1}	2.10±0.07 ^{b12}	2.00±0.07 ^{c2}	1.95±0.07 ^{c2}	2.05±0.07 ^{b1}	1.85±0.14 ^{c2}	1.75±0.07 ^{c2}
HPPE	Glass	9.40±0.14 ^{a1}	9.00±0.64 ^{a1}	7.90±0.21 ^{a2}	7.70±0.28 ^{a2}	9.00±0.00 ^{a1}	7.95±0.00 ^{a2}	7.55±0.00 ^{a3}
	Plastic	9.35±0.07 ^{a1}	8.45±0.07 ^{a12}	7.35±0.14 ^{b2}	7.10±0.14 ^{b2}	9.15±0.07 ^{a1}	7.20±0.00 ^{b2}	7.05±0.14 ^{ab3}
	Sachet	9.05±0.07 ^{a1}	8.70±0.07 ^{a1}	7.95±0.07 ^{a2}	7.90±0.14 ^{a2}	9.10±0.14 ^{a1}	7.75±0.07 ^{a2}	7.35±0.07 ^{b3}

Values are mean ± Standard deviation of duplicate samples.

Values with different alphabetic superscript (^{abc}) along the column for each storage condition are significantly ($P \leq 0.05$) different

Values with different numeric superscript (¹²³) along the row for each storage condition are significantly ($P \leq 0.05$) different

HAPF - *Hibiscus sabdarifa* drink + artificial pineapple flavour

HPPE - *Hibiscus sabdarifa* drink + *Phoenix dactylifera* + pineapple peel extract

Table 3: Viscosity (Pa.s) of *Hibiscus sabdarifa* drinks in different packaging materials during storage at refrigeration and ambient temperatures

Samples	Packaging	Refrigeration Temperature (4.4±2 $^{\circ}$ C)				Ambient Temperature (25±2 $^{\circ}$ C)		
		Time (Weeks)						
		1	2	3	4	1	2	3
HAPF	Glass	0.22±0.06 ^{a2}	0.26±0.05 ^{b2}	0.30±0.02 ^{bc2}	0.47±0.04 ^{a1}	0.20±0.05 ^{ab2}	0.26±0.03 ^{c1}	0.28±0.01 ^{c1}
	Plastic	0.32±0.09 ^{a2}	0.41±0.02 ^{a12}	0.44 ±0.03 ^{a12}	0.49±0.01 ^{a1}	0.24±0.06 ^{ab2}	0.31±0.01 ^{ab1}	0.37±0.04 ^{b1}
	Sachet	0.32±0.05 ^{a12}	0.27±0.05 ^{bc2}	0.26±0.02 ^{c2}	0.47±0.03 ^{a1}	0.27±0.03 ^{ab2}	0.30±0.00 ^{bc1}	0.33±0.04 ^{bc1}
HPPE	Glass	0.22±0.08 ^{a2}	0.18±0.00 ^{c2}	0.34±0.01 ^{b12}	0.44±0.04 ^{a1}	0.23±0.00 ^{b2}	0.26±0.02 ^{c2}	0.33±0.03 ^{bc1}
	Plastic	0.21±0.03 ^{a2}	0.24±0.04 ^{bc2}	0.26±0.01 ^{c2}	0.51±0.01 ^{a1}	0.24±0.00 ^{ab3}	0.35±0.03 ^{a2}	0.46±0.06 ^{a1}
	Sachet	0.19±0.02 ^{a3}	0.24±0.04 ^{bc23}	0.29±0.01 ^{bc2}	0.48±0.01 ^{a1}	0.29±0.01 ^{a2}	0.32±0.00 ^{ab1}	0.35±0.01 ^{bc1}

Values are mean ± Standard deviation of duplicate samples.

Values with different alphabetic superscript (^{abc}) along the column for each storage condition are significantly ($P \leq 0.05$) different

Values with different numeric superscript (¹²³) along the row for each storage condition are significantly ($P \leq 0.05$) different

HAPF - *Hibiscus sabdarifa* drink + artificial pineapple flavour

HPPE - *Hibiscus sabdarifa* drink + *Phoenix dactylifera* + pineapple peel extract

Viscosity of *Hibiscus sabdarifa* drinks in different packaging materials during refrigeration (4.4±2 $^{\circ}$ C) and ambient (25±2 $^{\circ}$ C) storage.

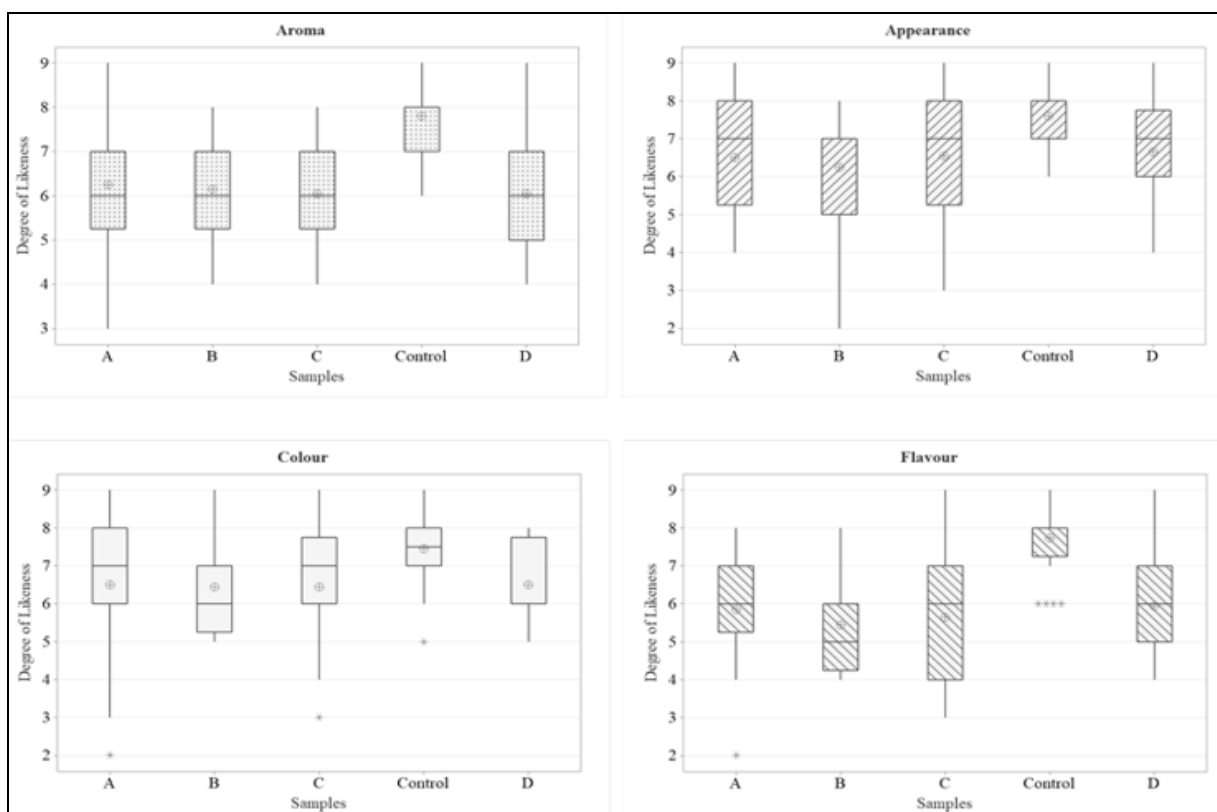
There was significant ($P \leq 0.05$) increase in viscosity of the *Hibiscus sabdarifa* drink samples with storage time as shown in Table 3. At refrigeration temperature, packaging materials had significant ($P \leq 0.05$) effect on the viscosity of the drinks in week 2 and 3. In week 2, HAPF packaged in plastic bottles had significantly ($P \leq 0.05$) the highest viscosity and HPPE in glass bottle had the least. In week 3, HAPF packaged in plastic bottles had significantly ($P \leq 0.05$) the highest viscosity and HPPE in plastic and HAPF in sachet had the least. At ambient temperature, at week 2 and 3 HAPF drinks packaged in glass bottles had significantly ($P \leq 0.05$) the least viscosity and plastic the highest. In HPPE samples, drinks packaged in glass had significantly ($P \leq 0.05$) the least viscosity on week 1, 2 and 3. Viscosity of the drinks at ambient temperature for the first 3 weeks was significantly ($P \leq 0.05$) higher than those at refrigeration temperatures except for HAPF sample in glass and plastic bottles.

Viscosity is an important parameter that can affect the quality and consumer acceptance of fluid foods. It changes the flow properties, determines textural properties and influences the appearance and consistency of the drink (Hollowood *et al.*, 2002; Wagoner *et al.*, 2019) [13, 31]. Change in viscosity over time has been attributed to the fiber behaviour in solution as well as molecular interactions between the different polysaccharides (Paquet *et al.*, 2014) [24] and increasing volatility (Sukarminah *et al.*, 2019) [27]. Increase in volatility is due to increase in organic acids. This is evidenced in the decreasing pH of the drinks during storage. The drinks stored at ambient temperature had significantly ($P \leq 0.05$) had lower pH on week 3 which explained the higher viscosity of the drinks stored at ambient temperature. Other factors such as intra and/or intermolecular bonds as well as hydration of some insoluble molecules, increased molecular reactions can also cause increased viscosity at higher temperatures (Paquet *et al.*, 2014) [24]. The higher viscosity of the *Hibiscus sabdarifa* with *Phoenix*

dactylifera and pineapple extract drink (HPPE) over the artificially flavoured drink (HAPF) at ambient temperature could be attributed to the endogenous aggregation of the soluble solid content of the date and pineapple extract in the HPPE samples. Such endogenous self-aggregation effect in the increasing viscosity was reported by Paquet *et al.*, (2014) [24]. The change in viscosity was independent of the packing materials implying that other factors rather than influence of packaging is responsible for increase in viscosity.

Sensory Properties of *Hibiscus sabdarifa* drink with different levels of *Phoenix dactylifera* and pineapple peel extract

Figure 2, presented the mean scores of the assessor's degree of likeness of the sensory attributes of *Hibiscus sabdarifa* drink with *Phoenix dactylifera* and pineapple extract. The mean degree of likeness for aroma, appearance and colour respectively, ranged from 6.05 - 7.80, 6.25 - 7.60 and 6.64 - 7.45 representing like slightly to like moderately. The degree of likeness for flavour, sourness and overall acceptability ranged from 5.45 - 7.75, 5.05 - 7.55 and 5.66 - 7.51 respectively signifying neither like nor dislike to light moderately while the likeness for sweetness and sourness ranged from 4.90 - 7.80 and 5.35 - 6.60 respectively indicating dislike slightly to like moderately and neither like nor dislike to like slightly. Nonparametric Friedman's P-value was less than the set P-value of 0.05 indicative of the significant differences between the samples. The control sample with artificial pineapple flavour (HAPF) had significantly ($P \leq 0.05$) the highest degree of likeness. The lower mean scores of the test samples can be attributed to the addition of natural pineapple extract and date unlike the artificial pineapple flavour that had various other additives. Similar degrees of likeness were reported for blends of roselle calyce and date fruit extract (Ukwo *et al.*, 2019) [30] and for mixed fruit juice with date as a sweetener (Onyekwelu, 2017) [23]. Though the degree of likeness of the test samples (*Hibiscus sabdarifa* drink with *Phoenix dactylifera* and pineapple peel extract: A-D) were very close, sample B with 23% *Phoenix dactylifera* had the least degree of likeness for all attributes except for aroma while sample D with 38% *Phoenix dactylifera* had higher degree of likeness amongst the test samples except for aroma and smoothness. It competed favorably with the control and was chosen for storage studies.



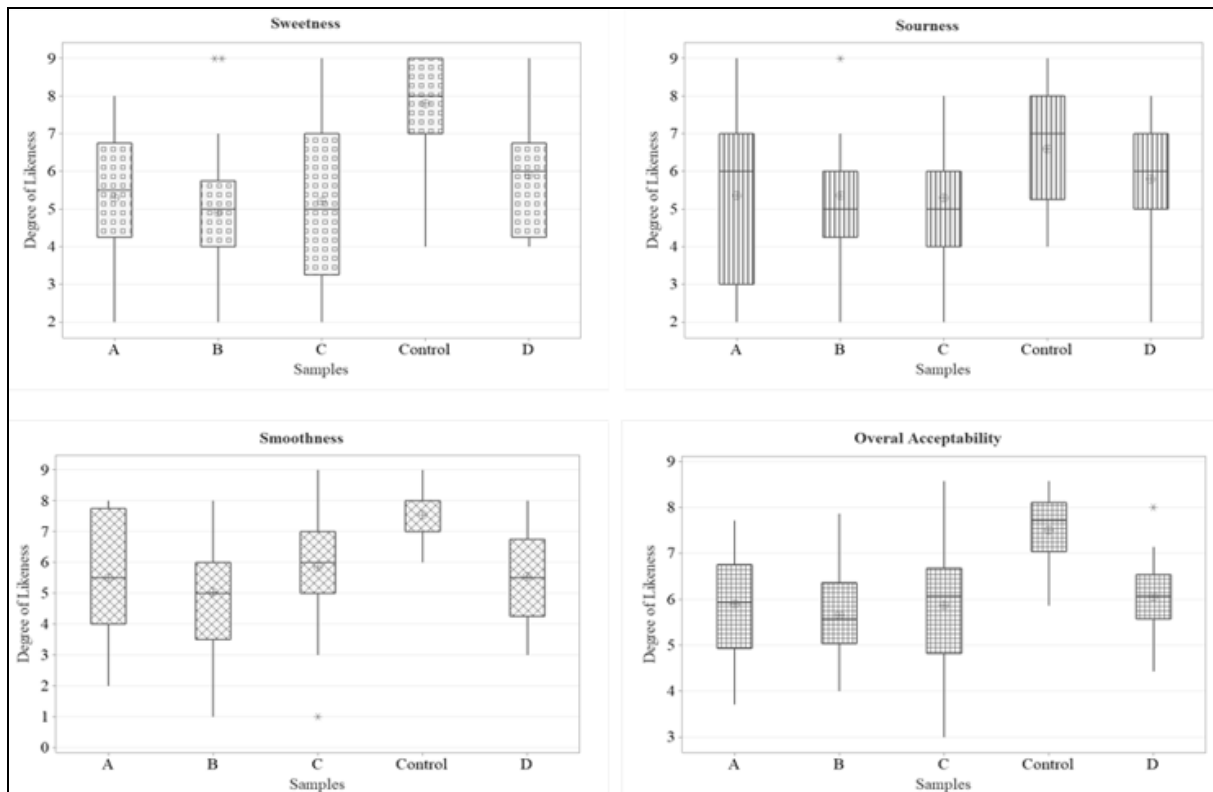


Fig 2: Sensory properties of *Hibiscus sabdariffa* drinks

Control = *Hibiscus sabdariffa* + artificial pineapple flavour

A = *Hibiscus sabdariffa* + 23% *Phoenix dactylifera* + 2% pineapple peel extract

B = *Hibiscus sabdariffa* + 28% *Phoenix dactylifera* + 2% pineapple peel extract

C = *Hibiscus sabdariffa* + 33% *Phoenix dactylifera* + 2% pineapple peel extract

D = *Hibiscus sabdariffa* + 38% *Phoenix dactylifera* + 2% pineapple peel extract

Conclusion

The study revealed that the packaging materials: glass bottles, plastic bottles and polyethylene sachets had no significant ($P > 0.05$) effect on the pH, viscosity and total soluble solid content of the *Hibiscus sabdariffa* drinks. Storage at ambient ($25 \pm 2^\circ\text{C}$) temperature resulted in significant ($P \leq 0.05$) decreased in pH and total soluble solid and significant ($P \leq 0.05$) increase in viscosity of the drinks. The drink type influenced the total solid content. *Hibiscus sabdariffa* drink with the artificial pineapple flavour (HAPF) had significantly ($P \leq 0.05$) the least total soluble solid content. *Phoenix dactylifera* slurry and the pineapple peel extract resulted in significantly ($P \leq 0.05$) higher total soluble solid content in *Hibiscus sabdariffa* drink. The addition of 38% of *Phoenix dactylifera* slurry and 2% of pineapple peel extract gave a product with the degree of likeness of sensory attributes comparable with the artificially sweetened drink commonly sold and consumed. This is informative to the both the consumers and producers of *Hibiscus sabdariffa* drink and any of the packaging materials is good without impairing the physicochemical properties of the drink, however, storage at refrigeration temperature is better.

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