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Commercial Practice of Roselle (*Hibiscus sabdariffa* L.) Beverage Production: Optimization of Hot Water Extraction and Sweetness Level

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Abstract: This study was aimed at establishing optimized conditions for hot water extraction of roselle beverage (*soborodo*) from its calyces as well as the degree of beverage sweetness in the commercial practice. The dried calyces/water ratios involved in the commercial practice ranged between 1:57 and 1:71 (w/v) while the sweetness level of the beverage ranged between 11.2 and 13.3 °Brix. The indices for assessing the quality of *soborodo* were found to be colour intensity, sweetness level and pleasant taste. The appropriate dried calyces/water ratio for commercial *soborodo* production was found to be 1:62 (w/v) while the extraction period was 30 minutes at a constant temperature of 100°±2°C. The appropriate sweetness level was found to be 13% sugar inclusion.

Key words: *Soborodo* % Roselle % Extraction % Sweetness % Commercial

INTRODUCTION

Roselle (*Hibiscus sabdariffa* L.) is a plant that is widely grown in the tropics and its cultivation in Nigeria is highly concentrated in the North Eastern and Middle-belt regions of the country [1]. The utilization of the plant however goes beyond its area of gross cultivation while the parts of the plant that have been highly valuable to human race are the leaves and the calyces (flowers). The various uses to which roselle plant parts have been put include their uses in traditional medicine as a digestive agent, purgative and diuretic, among others [2]. The roselle plant parts have also been reported to be folk remedy for cancer, obesity, diabetes and hypertension [3-5]. Other uses of the plant parts (particularly calyces) are in food production such as local non-alcoholic beverage, industrial wine, jam, marmalade and tea production [6-7].

In Nigeria, the production of a non- alcoholic beverage (*soborodo*) from dried red roselle (*Hibiscus sabdariffa* L.) calyces is very popular. The drink serves as a cheaper alternative to the industrially-produced carbonated soft drinks also available in every nook and cranny of the country. The preparation procedures for *soborodo* essentially involves soaking of dried red calyces of roselle in hot water for few minutes, filtration, sweetening, flavouring and packaging to obtain the final non-alcoholic beverage called *soborodo*. The principal quality attributes usually used to assess the acceptability of the drink are colour intensity, degree of sweetness and overall pleasant taste.

The preparation procedures for soborodo, however, vary from one locality to another thereby leading to variations in the quality attributes (i.e. colour intensity and taste) of the product. This study therefore sought to determine the optimum levels of these conditions (colour intensity and degree of sweetness) that have overbearing influence on the overall acceptability of *soborodo* which was aimed at establishing quality consistency for the non-alcoholic beverage.

MATERIALS AND METHODS

Materials: The dried red roselle calyces were purchased from a local market (Erekesan) in Akure, Ondo State, Nigeria.

Survey of commercial processing centres of roselle beverage (*soborodo*): The different commercial processing centres of roselle beverage (*soborodo*) were visited at three different localities (Akure, Ibadan and Osogbo; all in Southwestern Nigeria). The information sought for from each of the centres include the processing technique in terms of dried calyces/hot

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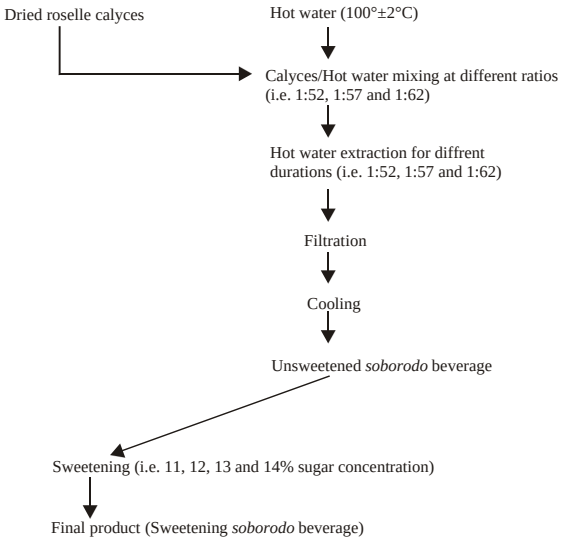

Fig. 1: Steps involved in the optimization process of roselle beverage (*soborodo*) production

water ratio, colour intensity of the beverage, sweetness level of the drink and the quality factors for the assessment of product acceptability.

Optimization of hot water extraction in the production of roselle beverage (*soborodo*): Roselle beverage (*soborodo*) was prepared using the procedures illustrated in Fig. 1. Well water was used for the preparation of the beverage. The ratios of calyces/hot water used for the extraction were 35 g of dried calyces to 1.82 litres of hot water (1:52, w/v), 35 g of dried calyces to 2.0 litres of hot water (1:57, w/v) and 35 g of dried calyces to 2.17 litres of hot water (1:62, w/v) respectively. The extraction time was varied (i.e. 20, 25 and 30 min respectively) while the extraction temperature was kept constant (i.e. 100° ±2°C). At the end of each hot extraction, the extract was filtered using a clean muslin cloth after which the extract was cooled to ambient temperature (30° ±2°C).

Roselle beverage (*soborodo*) was also prepared using the commonest traditional processing technique. The dried calyces were boiled in hot water for 5 minutes after which it was allowed to stand for another 30 minutes for proper extraction. The mixture was then filtered using a muslin cloth to obtain the unsweetened *soborodo*.

Analysis of well water used for the preparation of roselle beverage (*soborodo*): Some physical and chemical characteristics of well water used for the preparation of *soborodo* were analyzed using Standard Methods [8]. The parameters evaluated were appearance through visual inspection, pH, total solids, total chlorine, total hardness ($CaCO_3$), sulphate (SO_4^{2-}), phosphate (PO_4^{3-}) and chloride (Cl^-).

Optimization of sweetness level of roselle beverage (*soborodo*): The roselle extract identified as the best (in terms of colour intensity) from the various dried calyces/hot water ratios was eventually used to determine the optimum level of sweetness in the beverage. The percentage of sugar in the beverage was varied (i.e. 11, 12, 13 and 14%) and the beverage subjected to sensory evaluation.

Chemical analysis of *soborodo*: The colour intensity of roselle beverage (*soborodo*) was determined using an absorption spectrophotometer (model SP9, Pye Unicam, UK) set at 480 nm wavelength [9]. The optical density was taken as an index of colour intensity of the beverage. The sweetness level in roselle beverage (*soborodo*) was determined using hand refractometer (model N1, Atago, Japan). The refractometer was used to measure °Brix of the beverage and the value was taken as an index of the sweetness level. Total solids in roselle beverage was determined according to the method of Lees [10]. Ascorbic acid was determined using titrimetric method as described by James [11]. The concentration of ascorbic acid in the sample was expressed as mg ascorbic acid /100 ml or 100 g of sample. The pH of roselle calyx and beverage was measured using a digital pH meter (model EA513-055, ELE, England) standardized with buffer solutions of 4.0 and 7.0. The total titratable acidity of *soborodo* was determined using a method as described by Egan *et al.* [12]. The result was expressed as percentage of malic acid equivalent present in the beverage.

Sensory evaluation of roselle beverage (*soborodo*): Roselle beverage sample sweetened with sugar at 11, 12, 13 and 14% concentration were evaluated for their sensory qualities and general acceptability. A scoring test was used which was designed to determined which of the samples was most preferred. A 21-member taste panel (members were familiar with the beverage) was requested to rate the sample using a nine- point hedonic scale (i.e 9 = like extremely; 5= neither like nor dislike; 1= dislike extremely). The scores from the rating were subsequently subjected to analysis of variance (ANOVA) and means separated using Duncan Multiple Range test [13-14].

Statistical Analyses: All the analyses reported in this study were carried out in triplicates. In each case, a mean value and standard deviation were calculated. Analysis of

variance (ANOVA) was also performed and separation of the mean values was carried out using Duncan Multiple Range Test at $p<0.05$ [15].

RESULTS AND DISCUSSION

The characteristics of commercial roselle beverage (*soborodo*) production are presented in Table 1. The ratios of dried calyces to water being used for *soborodo* production ranged between 1:57 and 1:71 (w/v) across different processing centres. The colour intensity (optical density) of *soborodo* from different processing centres also ranged between 0.082 and 0.098. The colour intensity in the commercial *soborodo* is essentially a function of quantity and temperature of water involved in the extraction [16].The processing techniques adopted by the commercial producers of *soborodo* also varied. Initial boiling of dried calyces was carried out by some of the producers while initial soaking in hot water was adopted by some. Sweetness levels (11.2-13.3 °Brix) was observed to be a common denominator among the producers while flavouring materials involved in *soborodo* production include pineapple, apple and orange flavours [17]. The significance of flavouring in *soborodo* production is to enhance the overall taste of the drink. The quality indices being used by the consumers of *soborodo* for assessing the drink's acceptability were observed by all the commercial producers to be colour intensity, sweetness level and pleasant taste.

Table 1: Characteristics of roselle beverage (*soborodo*) production in the commercial practice

Location of commercial processing of *soborodo*		Dried calyces/ water ratio (w/v)	Processing technique adopted	Colour Intensity of the beverage (Optical Density)	Sweetness level (°Brix)	Quality indices for assessing product's acceptability
Akure, Nigeria	1A	1 'Kongo' (350 g): 20L (1:57)	Boiling of dried calyces in hot water for 10 min6 Allowed to stand for additional 20 min6 Filteration6 Dilution6 Sweetening and flavouring6 Packaging	0.096	11.5	Colour intensity, sweetness level and pleasant taste
	1B	1 'Kongo' (350 g): 25L (1:71)	Boiling in hot water for 5 min6 Allowed to stand for additional 30 min6 Filteration6 Dilution6 Sweetening and flavouring6 Packaging	0.094	12.1	-ditto-
	1C	1 'Kongo' (350 g): 22L (1:63)	Soaking in hot water for 30 min6 Filteration6 Dilution6 Sweetening and flavouring6 Packaging	0.082	12.7	-ditto- -ditto-
Ibadan, Nigeria	2A	1 'Kongo' (350 g): 23L (1:66)	Boiling in hot water for 10 min6 Allowed to stand for additional 30 min6 Filteration6 Dilution6 Sweetening and flavouring6 Packaging	0.095	11.2	-ditto-
	2B	1 'Kongo' (350 g): 25L (1:71)	Boiling in hot water for 5 min6 Allowed to stand for additional 30 min6 Filteration6 Dilution6 Sweetening6 Packaging	0.092	12.9	-ditto-
	2C	1 'Kongo' (350 g): 20L (1:57)	Boiling in hot water for 10 min6 Allowed to stand for additional 25 min6 Filteration6 Dilution6 Sweetening and flavouring6 Packaging	0.098	13.2	-ditto-
Osogbo, Nigeria	3A	1 'Kongo' (350 g): 21L (1:60)	Boiling in hot water for 5 min6 Allowed to stand for additional 30 min6 Filteration6 Dilution6 Sweetening 6 Packaging	0.091	12.4	-ditto-
	3B	1 'Kongo' (350 g): 20L (1:57)	Soaking in hot water for 30 min6 Filteration6 Dilution Sweetening and flavouring6 Packaging	0.088	13.3	-ditto-
	3C	1 'Kongo' (350 g): 25L (1:71)	Soaking of calyces in hot water for 30 min6 Filteration6 Dilution6 Sweetening6 Packaging	0.086	11.8	-ditto-

Table 2: Characteristics of roselle beverage (*soborodo*) obtained from different dried calyces/water ratios and boiling durations

Sample source	Dried calyces/Water ratio (w/v) and boiling duration at 100°±2°C	Total volume of *soborodo* recovered w.r.t water volume (%)[1]	Colour intensity (Optical Density)	pH	Ascorbic acid (mg/100 ml)	Total solids (%)	Total titratable acidity (%)
A_1	1:52, 20 min	91.1	0.102	3.25	30.7	3.3	0.18
A_2	1:52, 25 min	89.7	0.163	3.28	28.7	3.8	0.21
A_3	1:52, 30 min	90.2	0.106	3.21	25.3	3.6	0.15
A_T	1:52, Traditional method	90.8	0.099	3.15	26.3	3.4	0.22
B_1	1:57, 20 min	89.4	0.095	3.21	20.6	3.1	0.19
B_2	1:57, 25 min	89.1	0.108	3.21	21.9	3.5	0.18
B_3	1:57, 30 min	90.2	0.103	3.21	20.3	3.3	0.23
B_T	1:57, Traditional method	90.1	0.088	3.27	23.7	2.6	0.16
C_1	1:62, 20 min	89.2	0.156	3.17	16.7	3.7	0.19
C_2	1:62, 25 min	89.7	0.144	3.13	14.8	3.4	0.17
C_3	1:62, 30 min	90.3	0.218	3.16	15.5	3.9	0.21
C_T	1:62, Traditional method	90.1	0.086	3.13	17.2	2.4	0.18
Dried calyx	--	--	--	2.69	69.3 mg/100 g	--	--

1 mean volume of *soborodo* recovered w.r.t water volume = 90.0%

The characteristics of roselle beverage (*soborodo*) obtained from different dried calyces/water ratios and boiling durations are presented in Table 2. The total quantity of *soborodo* that could be recovered with respect to the initial volume of water involved in the extraction fell within a range of 89.1 and 91.1%; with a mean value being about 90%. Some quantity of water was obviously lost to the calyces which became softer and water absorbent after the extraction. The colour intensity (optical density) of the extract ranged between 0.086 and 0.218 with C_3 (dried calyces/water ratio of 1:62 [w/v] and 30 min boiling duration) giving the highest colour intensity while C_T (dried calyces/water ratio of 1:62 [w/v] and traditionally- processed) gave the lowest colour intensity. The colouring component in roselle beverage (*soborodo*) has been observed to be anthocyanins [18] and the stability or degradation of the brilliant red colour of the beverage is dependent on such factors as pH, light, temperature and oxygen [19]. Therefore, the variation in the colour intensity of *soborodo* is most probably related to the calyces/water ratios (i.e. dilution that affects the pH) and the extent of boiling at 100±2°C. The pH of roselle beverage (soborodo) obtained from different dried calyces/water ratios and boiling durations ranged between 3.13 and 3.28 while that of the calyces was 2.69. It has been observed that the colour intensity in roselle beverage is also favoured by the low pH value as anthocyanins have little colour above pH 3.5 [19]. The ascorbic acid of *soborodo* ranged between 14.8 and 30.7 mg/100 ml while that of the calyces was 69.3 mg/100 g. The presence of ascorbic acid in the beverage essentially confirms the nutritional benefit of the drink to the consumers. Samples A_1, (dried calyces/water ratio of 1:52 [w/v] and 20-min boiling duration) had the highest ascorbic acid content (30.7 mg/100 ml) while sample C_2 (dried calyces/water ratio of 1:62 [w/v] and 25-min boiling duration) had the lowest value (14.8 mg/100 ml). The lower volume of water for extraction seemed to favour greater concentration of ascorbic acid in the beverage. The total solids in the beverage ranged between 2.4 and 3.9% with C_3 giving the highest value while C_T gave the lowest value. The colour intensity seems to be related to the total solid content of the beverage as higher colour intensity led to higher total solid content. Total solid content in beverage has been observed to have a contributory effect on the overall mouth-feel of the beverage [17]. The total titratable acidity (TTA) of *soborodo* from different dried calyces/water ratios and boiling durations ranged between 0.15 and 0.23%. The general low values of TTA are a reflection of low pH values of the beverage as well as signifying *soborodo* as a non-fermented drink. Therefore, since colour plays a principal role in the acceptability of roselle beverage by the consumers [20], the first two samples with highest colour intensity (A_2 and C_3; dried calyces/water ratio of 1:52 and 1:62 [w/v] for 25-min and 30-min boiling duration respectively) were selected representing the best two optimal levels of hot water extraction. It was these two products that were used for subsequent investigations.

Some physical and chemical characteristics of well water used in the preparation of roselle beverage (*soborodo*) are presented in Table 3. The interaction of

Table 3: Some physical and chemical characteristics of well water used for the preparation of roselle beverage (*soborodo*)

Parameter	Measurement
Appearance	Clear
pH at 20°C	6.9
Total solids (ppm)	852
Total chlorine (ppm)	0
Total hardness, $CaCO_3$ (ppm)	47
Sulphate, SO_4^{2-} (ppm)	315
Phosphate, PO_4^{3-} (ppm)	0.01
Chloride, Cl^- (ppm)	383

Table 4: Sensory quality rating of selected *soborodo* beverage

Sample source[1]	Sensory quality rating[2]			
	Colour	Taste	Aroma	Overall acceptability
A_2A	6.3^{b}	5.8^{c}	6.9^{a}	6.4^{b}
A_2B	6.5^{ab}	6.3^{bc}	6.8^{a}	7.0^{ab}
A_2C	6.4^{ab}	7.1^{ab}	6.7^{a}	7.2^{ab}
A_2D	6.4^{ab}	6.6^{bc}	6.8^{a}	6.9^{ab}
C_3A	7.1^{ab}	5.9^{c}	6.2^{a}	6.5^{b}
C_3B	7.0^{ab}	6.5^{bc}	6.3^{a}	7.1^{ab}
C_3C	6.9^{ab}	7.7^{a}	6.3^{a}	7.6^{a}
C_3D	7.3^{a}	$6.7b^{c}$	6.5^{a}	7.0^{ab}

[1]Sample source:

A_2A= Dried calyces/Hot water ratio of 1:52, 25-min extraction period and 11% sugar inclusion.

A_2B= Dried calyces/Hot water ratio of 1:52, 25-min extraction period and 12% sugar inclusion.

A_2C= Dried calyces/Hot water ratio of 1:52, 25-min extraction period and 13% sugar inclusion.

A_2D= Dried calyces/Hot water ratio of 1:52, 25-min extraction period and 14% sugar inclusion.

C_3A= Dried calyces/Hot water ratio of 1:62, 30-min extraction period and 11% sugar inclusion.

C_3B= Dried calyces/Hot water ratio of 1:62, 30-min extraction period and 12% sugar inclusion.

C_3C= Dried calyces/Hot water ratio of 1:62, 30-min extraction period and 13% sugar inclusion.

C_3D= Dried calyces/Hot water ratio of 1:62, 30-min extraction period and 14% sugar inclusion.

2 mean values followed by the same superscripts in each column are not significantly different at P<0.05.

the chemical constituents of well water with that of anthocyanin (delphinidin 3-sambubioside and cyanidin 3-sambubioside) may not be that pronounced as significant properties of anthocyanin (colour intensity and colour hue) are mostly affected by pH, SO_2, heat, light, metalsand copigmentation [19, 21]. However, high residual chlorine in water and high water quantity may have bleaching and dilution effects on the colour intensity respectively.

The sensory quality rating of selected *soborodo* is presented in Table 4. Sample C_3D (dried calyces/hot water ratio of 1:62 [w/v], 30 min extraction duration and 14% sugar inclusion) was the rated highest in terms of colour though not significantly different from others at P<0.05 except A_2A (dried calyces/hot water ratio of 1:52, 25-min extraction duration and 11% sugar inclusion). The highest concentration of sugar in C_3D might have contributed to the highest colour rating as co-pigmentation of sugar with anthocyanins in roselle beverage (*soborodo*) has been observed as one of the factors influencing colour intensity in the beverage [22]. Sample C_3C (dried calyces/hot water ratio of 1:62, 30-min extraction duration and 13% sugar inclusion) was rated the highest in terms of taste but not significantly different from A_2C (dried calyces/hot water ratio of 1:52, 30-min extraction duration and 13% sugar inclusion) at P <0.05. In the case of aroma, sample A_2A was rated the highest but not significantly different from others at P< 0.05. Lower quantity of water involved in the extraction of sample A_2A might have contributed to greater aroma concentration in the sample. In the case of overall acceptability, sample C_3C was rated highest but not significantly different from others at P<0.05 except A_2A and C_3A (dried calyces/hot water extraction ratio of 1:62, 30-min extraction duration and 11% sugar inclusion). The practical implication of this assessment is that the sweetness level in *soborodo* could be between 12 and 14% sugar inclusion but with greatest preference at 13% level.

It may be concluded that the optimal level of hot water extraction for the beverage should be dried calyces/hot water ratio of 1:62 (w/v) at 100±2°C for 30 minutes while the sweetness level should be 13% of sugar. The practical application of this study is that it can guarantee consistency in the product quality in terms of colour intensity and sweetness level of the beverage at commercial level.

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