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Full Length Research Paper

Studies in concentration and preservation of sorrel extract

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The popular sorrel (zobo) juices drink which is about 99% water is known to be easily susceptible to microbial spoilage. The need thus arises to standardize its production to give quality product of compact packing and adequate shelf life. In this work attempts were made to study thermal concentration of the juice extract and its chemical preservation. Concentrating the juice to about 17% solid was found to improve its life on open shelf from one -and-a-half to three days while reducing the ascorbic acid content by 50%. Addition of sodium benzoate was found to improve the storage of the exposed juice substantially without noticeable change in vitamin C content. The 15% reduction in mass achieved with thermal concentration consumed 20.05, 15.91 and 14.14 MJ per kg juice treated at 60, 80 and 90°C respectively. These correspond respectively to 5.571, 4.420 and 3.929 kWh of energy expenditure per kg of the extract.

Key words: Sorrel drinks, *Hibiscus sabdariffa*, concentrate, preservative, sterilization, heat of vaporization.

INTRODUCTION

Sorrel drink, commonly called 'zobo' in Nigeria, is a red coloured juice drink which is leached from the calyx of *Hibiscus sabdariffa*. This drink contains anthocyanins, sugar and vitamin C among others and is used in curing minor stomach ailments, sore throat and strengthening the heart among other uses (www.magdalin.com, 2004; www.nijb.com, 2004). The calyces are used to produce herbal teas and other food products (Akanya et al, 1997). The juice drink, which is usually obtained by extraction of the calyx of *Hibiscus sabdariffa* with hot water, contains about 1% solid. The drink contains some micro-organisms which can cause food spoilage (Omemu et al., 2006). At present, the production process is neither mechanised nor standardised. Consequently the shelf life of the drink is less than two days (Samy, 1980). Furthermore, the mode of packaging of the juice makes it too bulky for easy transportation and very unhygienic. Due to the popularity of this refreshing drink in Nigeria, improvement of its shelf-life and possibility of more compact packaging need to be investigated. This is to protect consumers and ease the distribution of the product and thus increase its commercial value.

Complete sterilization, pasteurization, carbonation and chemical sterilization are commonly used to retard the onset of microbial spoilage of drink juices and other food products (Casolari et al., 1957; Wikipedia, 2009). However, whereas thermal sterilization (including pasteurization) and carbonation prevent spoilage of capped/canned juices and other food products, only chemical sterilization can prevent spoilage of exposed juice drinks and other food products. Reducing the water content of the extract would lower the transportation cost and making the drink easier to handle. Thermal concentration would also serve as sterilization and thus increase the storage (shelf) life of the product. Thus availability of sorrel drink concentrate would go a long way in enhancing not only its popularity, but also its overall usability. Despite the very large consumption of the drink by Nigerian populace and the awareness of microbial loading and poor keeping quality of the drink, no work appear to have been reported in the literature on preservation of sorrel drink.

Therefore in this work attempt was made to develop sorrel drink concentrate and study its effectiveness in delaying the onset of microbial spoilage of the drink. In addition, the performance of sodium benzoate as a preservative for sorrel drinks which is exposed to the

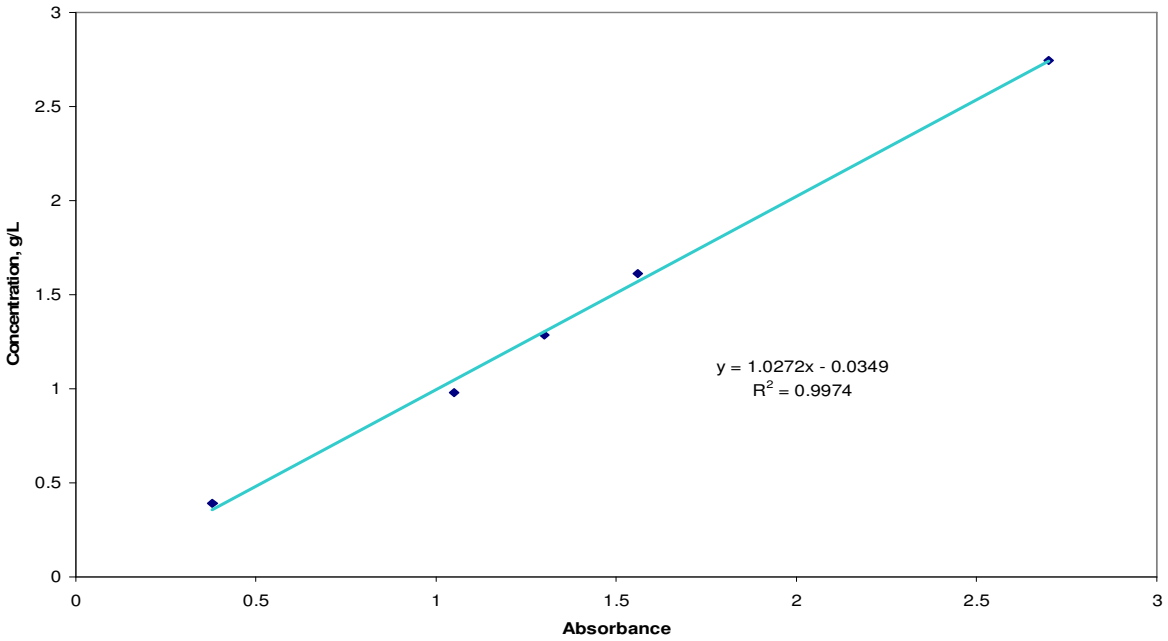

Figure 1. Variation of concentration with absorbance of sorrel extract.

atmosphere is evaluated.

Materials and Methods

Leaching of calyces and calibration of extract concentration

Calyces of *H. sabdariffa* were obtained from a market in Zaria, Nigeria. After removing dirt and other foreign matters from the dry calyces, the cleansed bulk was reduced in size using a mortar and pistil and thereafter leached with water (using a solute to solvent ratio of 1:20) for 20 min at about 90°C. The leachate was recovered with the aid of muslin cloth; this extract was subsequently used in the remaining part of this work.

Exactly 10, 20, 30, 40 and 50 ml of the extract obtained was diluted to 500 ml in a volumetric flask to obtain five different solutions. The absorbance of each of the solution was measured with a Cecil Instrument UV-spectrophotometer (Model CE 202) at 520 nm. At the same time the mass of 50 ml of each of the five prepared solutions were determined gravimetrically by drying each to a constant weight as reported by Aboki (2004). The results obtained were then used to plot the calibration curve given in Figure 1.

Concentration of the extract

100 ml of the extract was put in a pre-weighed round bottom flask which was connected through a condenser to a condensate receiving flask, vapour traps and an Edward High vacuum pump (Model ES100C) having a rated current of 2.85 A. The mass of the solid in the fresh extract (ME) was determined by taking its absorbance and then read off the corresponding concentration from Figure 1. The content of the flask was heated to 60°C on a water bath (fitted with a Haake Fisons thermo-stated heater (Model DC 3) having a rated current input of 10 A) and then the vacuum pump was turned on and adjusted to 150 mmHg (0.1974 bar). The extract was then concentrated at this operating condition to three solid content levels and the resulting masses of the concentrates (MC) determined as the mass difference between the heated flask and the empty flask. The extent of concentration or solid content (SC) of the concentrates achieved over different periods of time were obtained as the ratio of the mass of the solid in the extract (ME) and mass of the concentrate (MC) as given in Equation 1.

$$SC = (ME/MC) \times 100 \quad (1)$$

Where SC is solid content of concentrate (%), ME is mass of solid in the original 100 ml (ca. 100g.) extract (g), and MC is mass of the concentrate (g).

Furthermore the amount of energy consumed (E) to concentrate 1 kg of the extract to the indicated concentrate levels were calculated as given in Equation 2.

$$E = 10\left[Pt + \left(VIyt + 418 \times \Delta T\right)\right] \quad (2)$$

Where E is energy consumed per kg extract juice (kJ/kg), t is time (s), P is power input of the vacuum pump (P = 0.001 VIye; kW), V is rated voltage (ca. 200 V), I is the rated current (A), y is power factor, dimensionless, and ΔT is temperature difference between ambient (27°C) and operating states.

The above procedure was repeated at 80 and 90°C corresponding to 355 mm Hg (0.4671 bar) and 525 mm Hg (0.6908 bar), respec-tively. Thus a total of nine samples were obtained; these samples were cooled to room temperature and stored in the refrigerator for further analysis. The change in concentration with time was then plotted for the nine samples as given in Figures 2a and 2b. Also plotted were the rate of heating and the variation of solid content of the concentrate with energy consumed and these are given in Figures 3 and 4, respectively.

Determination of the metallic contents and vitamin C

The original extract, as well as some of the concentrates obtained above were analyzed for metallic contaminants and vitamin C using

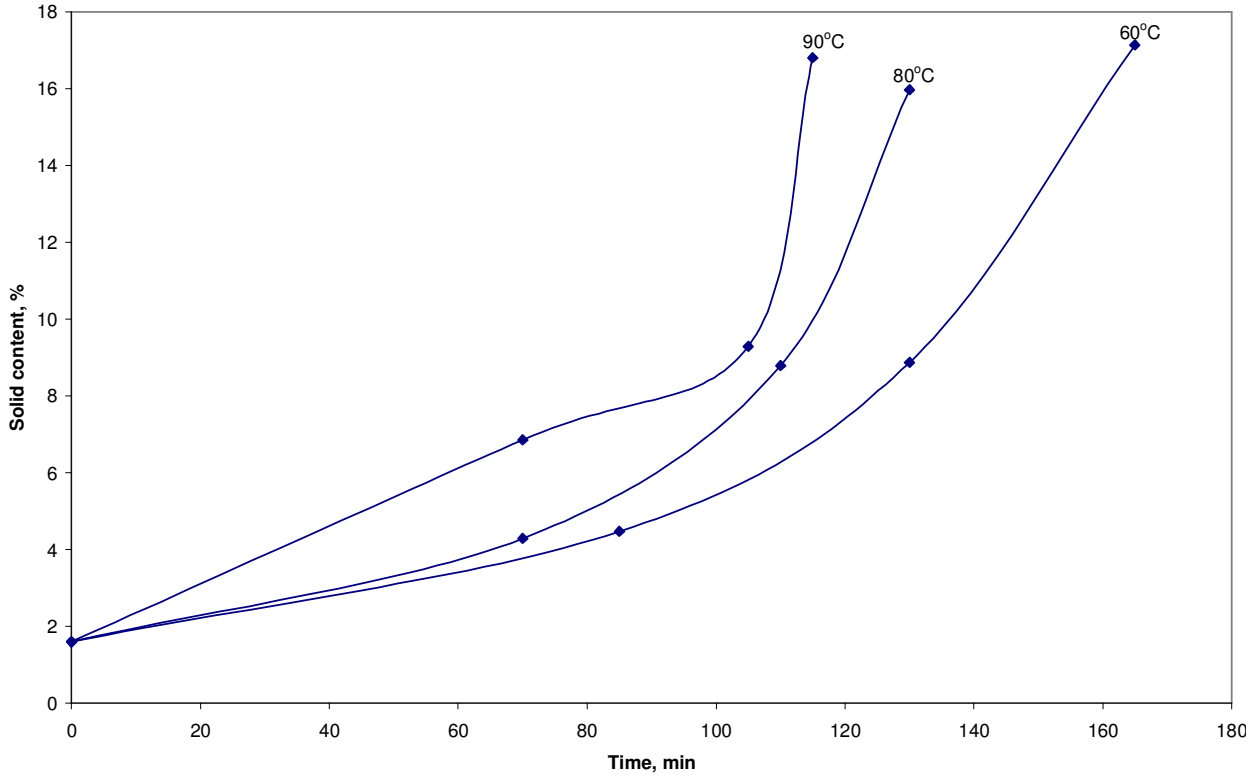

Figure 2a. Variation of solid content of juice extract with time and temperature.

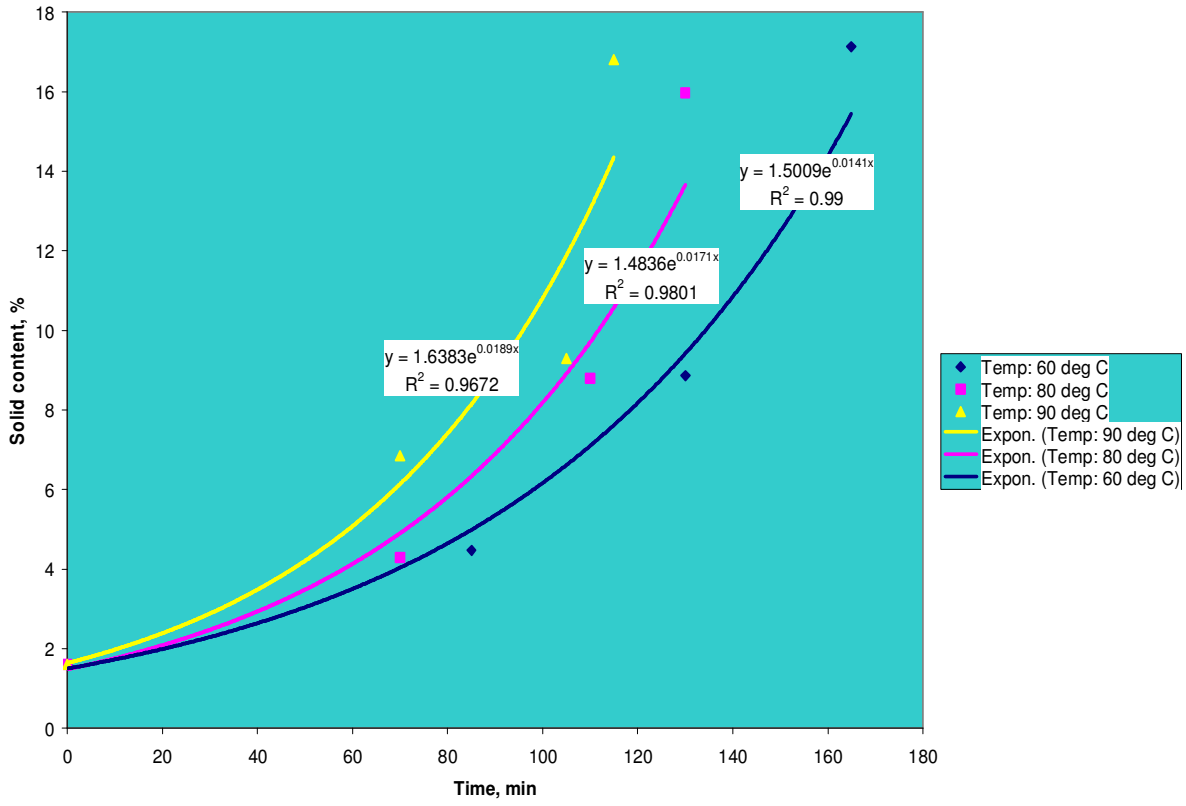

Figure 2b. Fitted functions of solid content of juice extract with time and temperature.

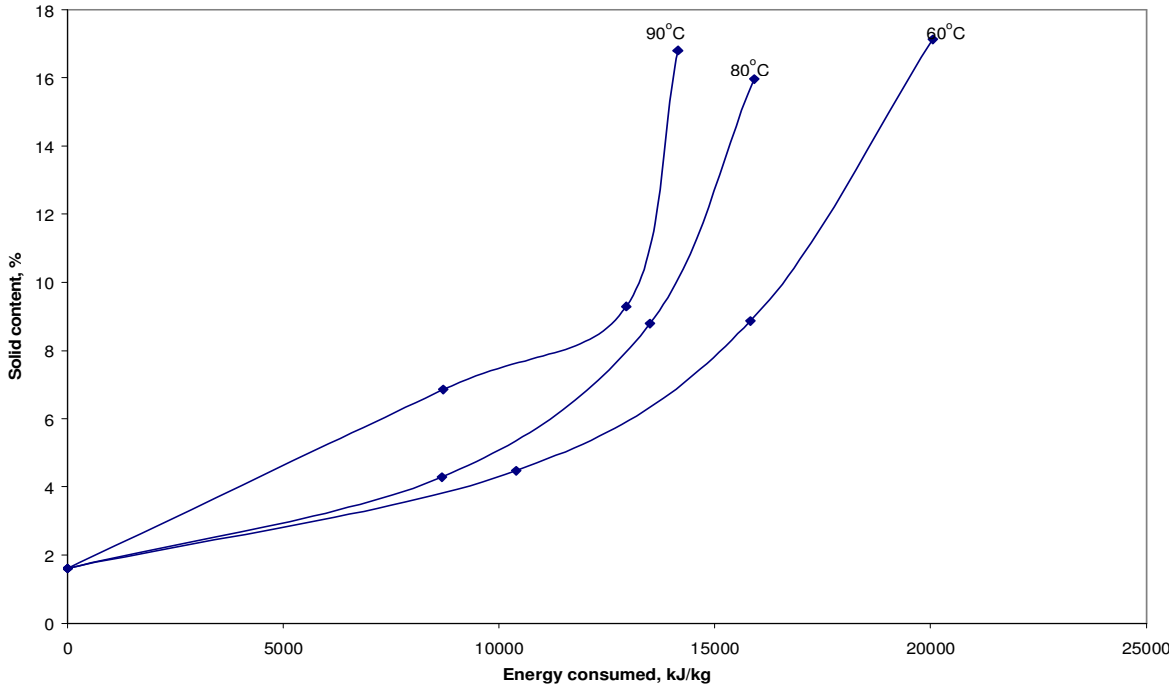

Figure 3. Variation of solid content of juice extract with energy consumed at different temperatures.

atomic absorption spectrophotometer (Model UNICAM 969) and the dichlorophenol-indolephenol method (Adams and Moss, 1999), respectively. Result of vitamin C determination is given in Figure 5 and those of the rest analyses are presented in Table 1.

Preservation of the juice drink and microbiological analysis

Each of the nine concentrate samples and the original extract were divided into two parts. To the first part, 0.03% sodium benzoate was added while the other part had no preservative added. The resulting twenty samples were subjected to microbial analysis. The bacteria and fungi count over a period of about two weeks were determined using the Pour plate method (Aboki, 2004; Adams and Moss, 1999). Results of these analyses are given in Tables 2 to 4. Further more, graphs were plotted to determine the shelf life as presented by Aboki (2004).

RESULTS AND DISCUSSION

Calibration curve preparation

The calibration curve which relates the solid content (concentration) of the fresh sorrel juice extract to its absorbance is presented in Figure 1. As can be seen from the graph, the resulting linear plot was correlated as:

$$y = 1.0272x - 0.0349 \quad (3)$$

Where y is solid content (concentration) of the extract (g/L), and x is absorbance of the liquid extract, dimensionless. A coefficient of determination value of 0.9974 indicates a high goodness of fit.

Concentration of the extract

The concentration profile of the extract is presented in Figure 2a. It shows that as the concentration temperature (with its corresponding vapour pressure) increased, the time for achieving the same level of solid content for the concentrates reduced. This trend is expected since rate of vaporization of water is expected to increase with temperature as heat of vaporization normally reduces with temperature.

The rate of increase in solid content at different temperature is given as linear functions up to 70 min for 60°C and 110 min for 90°C. Thereafter sharper increments were observed, with the highest occurring with 90°C. Figure 2b shows that the concentration profiles approximates exponential functions with the profile obtained at 60°C giving the best fit (R^2 = 0.99).

The energy consumed to obtain the concentrates (E) includes work done by the compressor to produce sub-atmospheric condition and the thermal energy that provide sensible and vaporization heat. While the later heat was evaluated from the power rating of the heater and vaporization time the former was obtained from the heat capacity of water (4.18 J/g) and temperature difference between vaporization and ambient states. From Figure 3, it is seen that the lowest energy was consumed at 90°C and the highest at 60°C. Thus the higher the evaporation temperature, the less the energy consumed

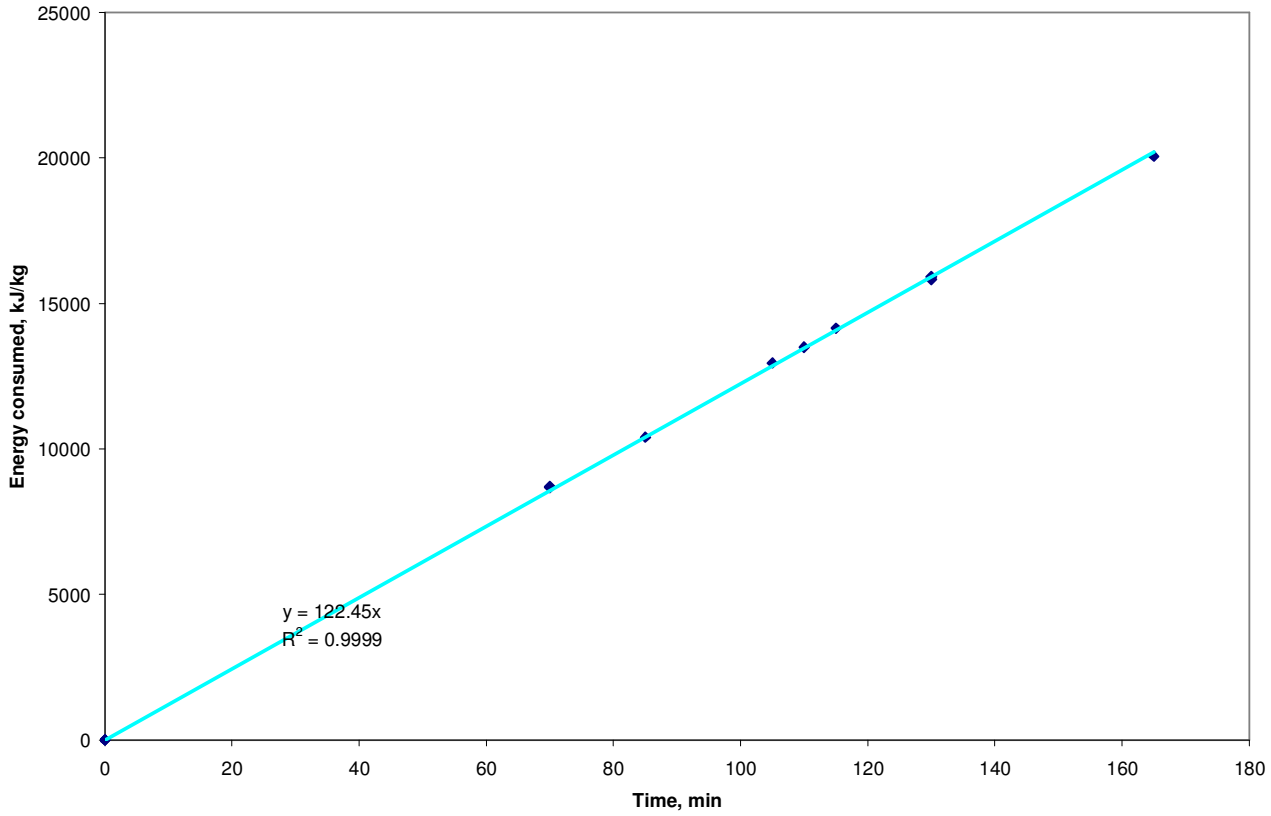

Figure 4. Variation of energy consumed with time between 60 and 90°C.

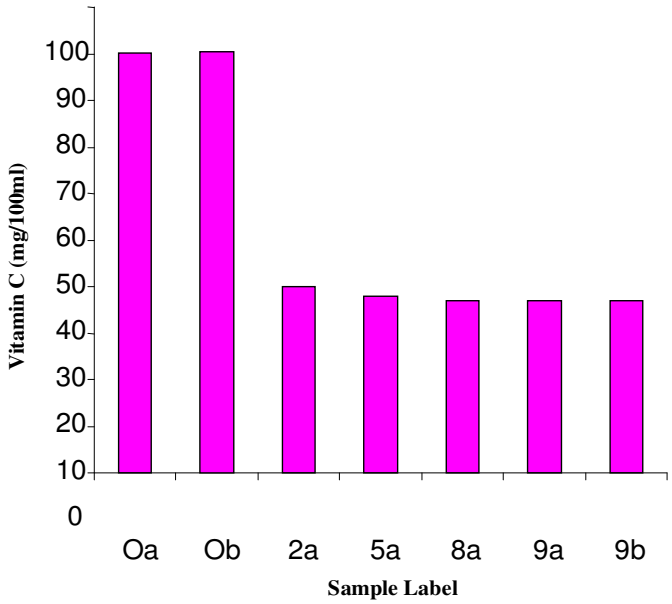

Figure 5. Vitamin C levels of various samples. Label description: 0 is original extract, 2 is extract concentrated at 60°C for 130 min, 5 is extract concentrated at 80°C for 110 min, 8 is extract concentrated at 90°C for 105 min, and 9 is extract concentrated at 90°C for 115 min. a is Extract treated at indicated conditions with no preservative added, and b is extract treated at indicated conditions with preservative added.

to achieve the same degree of evaporation of extract.The total energy used to increase the solid content of the 1 kg sorrel juice to 17.13% at 60°C was 20.05 MJ (5.571 kWh) while at 90°C, 14.14MJ (3.929 kWh) was con-sumed to give 16.80% solid content. The heat heater's duty was observed to be far more than the pump's work in the concentration process at each temperature.

Figure 4 shows that a uniform rate of energy consumption (22.45kJ/kg.min) applied at all temperatures between 60 and 90°C. This observation appears to support the findings that the least energy was expended on concentration at 90°C (Figure 3).

Analyses of the extract and the concentrates

The analyses carried out on the products were the determination of pH, vitamin C content and metallic contaminants content and microbiological analysis. The tastes of some of the samples were also determined using a taste panel. The results are discussed below.

pH values

The pH of the extract was found to be 2.50 and those of the concentrates produced have values between 2.35

Table 1. Permitted levels for metallic contaminants in juice drink.

Substance	Maximum level (mg/kg)
Arsenic	0.2
Lead	0.3
Copper	5
Zinc	5
Iron	5
Tin	250

Source: Standard Organization of Nigeria (1997).

Table 2. Bacteria growth in samples with no preservatives added.

Sample label	Bacteria count x 10^3 CFU/ml			
	Day 0	Day 4	Day 8	Day 13
0	2.0	45	130	420
1	1.6	40	108	345
2	1.2	28	76	240
3	0.8	20	54	175
4	1.5	38	102	330
5	1.2	32	96	280
6	0.8	19	51	160
7	1.2	34	98	273
8	1.0	25	69	182
9	0.6	16	47	151

Label description: 0 is original extract, 1 is extract concentrated at 60°C for 85 min, 2 is extract concentrated at 60°C for 130 min, 3 is extract concentrated at 60°C for 165 min, 4 is extract concentrated at 80°C for 70 min, 5 is extract concentrated at 80°C for 110 min, 6 is extract concentrated at 80°C for 130 min, 7 is extract concentrated at 90°C for 70 min, 8 is extract concentrated at 90°C for 105 min, and 9 is extract concentrated at 90°C for 115 min.

and 2.40. The results indicated that concentration decreased the pH of sorrel drink. This might be because water removal rate was more than the corresponding reduction in acidic constituents (ascorbic acid for example) in the solution.

Vitamin C content

The amount of vitamin C in the original extract was 90.25 and 90 mg/100 ml when preservative was added. The values were 40mg/100 ml for sample 2a (heated at 60°C for 130 min with no preservative), 38 mg/100 ml for sample 5a (heated at 80°C for 110 min with no preservative) and 37 mg/100 ml for samples 8a (heated at 90°C for 105 min with no preservative), 9a (heated at 90°C for 115 min with no preservative) and 9b (heated at 90°C for 115 min with preservative). These results show that vitamin C content in the drink reduced significantly when concentrated thermally at the conditions indicated. This would be due to the fact that vitamin C is readily destroyed by heat as already established in previous works (www. any vitamins.net, 2004).

It was also observed that all the concentrates produced had about the same amounts of vitamin C content. This tends to suggest that the temperatures used have nearlythe same effect on vitamin C degradation. The use

Table 3. Bacteria growth in samples with preservatives added.

Sample label	Bacteria count x 10^3 CFU/ml			
	Day 0	Day 4	Day 8	Day 13
0	0	1.5	1.5	2.0
1	0	1.0	1.0	1.5
2	0	1.0	1.0	1.5
3	0	0.5	0.5	1.0
4	0	1.0	1.0	1.5
5	0	1.0	1.0	1.5
6	0	0.5	0.5	1.0
7	0	0.8	1.1	1.4
8	0	0.5	0.6	1.0
9	0	0	0.4	0.9

Label description: 0 is original extract, 1 is extract concentrated at 60°C for 85 min, 2 is extract concentrated at 60°C for 130 min, 3 is extract concentrated at 60°C for 165 min, 4 is extract concentrated at 80°C for 70 min, 5 is extract concentrated at 80°C for 110 min, 6 is extract concentrated at 80°C for 130 min, 7 is extract concentrated at 90°C for 70 min, 8 is extract concentrated at 90°C for 105 min, and 9 is extract concentrated at 90°C for 115 min.

Table 4. Fungi growth in samples with no preservatives added.

Sample label	Fungi count x 10^3 CFU/ml			
	Day 0	Day 4	Day 8	Day 13
0	1.0	10	2.5	2.0
1	1.0	10	2.5	1.5
2	1.0	10	2.5	1.5
3	0.5	5	2.0	1.0
4	1.0	10	2.5	1.5
5	1.0	10	2.0	1.5
6	0.5	5	1.5	1.0
7	1.0	8	2.0	1.4
8	0.5	5	1.8	1.1
9	0.5	5	1.7	1.0

Label description: 0 is original extract, 1 is extract concentrated at 60°C for 85 min, 2 is extract concentrated at 60°C for 130 min, 3 is extract concentrated at 60°C for 165 min, 4 is extract concentrated at 80°C for 70 min, 5 is extract concentrated at 80°C for 110 min, 6 is extract concentrated at 80°C for 130 min, 7 is extract concentrated at 90°C for 70 min, 8 is extract concentrated at 90°C for 105 min, and 9 is extract concentrated at 90°C for 115 min.

of sodium benzoate as preservative appears to have little or no effect on vitamin C content as can be seen from the results (Figure 2).

Metallic contaminants content

The metallic contaminants determined were iron (Fe) and lead (Pb). Their concentrations were found to be 0.815 mg/l (0.749 mg/kg) and 0.375 mg/l (0.3409 mg/kg), respec-tively, for the original extract (sample 0) and 0.873 mg/l (0.7936 mg/kg) and 0.596 mg/l (0.5418 mg/kg), respectively, for sample 9. This result seems to suggest that concentrating the extract increased the proportion of both Fe and Pb in the drink. By comparing the results obtained with the specifications in Table 1 it is seen that the sorrel drink prepared under the stated conditions meets the expected quality with respect to concentration of iron. However, the values obtained for lead in the concentrates are slightly higher than the standard specification. This therefore suggests that it is not safe to consume the drink in the concentrated form.

Microbiological analysis and shelf life estimation

Results of the microbiological analysis of the extract and the concentrates (that are exposed to the atmosphere) which was carried out for about two weeks and monitored at regular interval are given in Tables 2 to 4. The results are applicable to juices which are neither corked nor refrigerated even when sterilized thermally or by carbonation.

Table 2 shows that bacteria growth rate was very high in the first four days of the analysis, then reduced and became relatively constant from then onwards. This observation is in concurrence with the normal growth cycle of these microorganisms (Aboki, 2004).

The data given in Table 3 show that sodium benzoate greatly reduced the growth of bacteria. This suggests that the use of the preservative would increase the shelf life of both the original sorrel drink extract and the concentrates even when exposed to the atmosphere. The results presented in Table 4 show that fungi growth increased rapidly at first, reached a maximum level and then declined. This is in accordance with the typical growth cycle of such microorganisms, where they pass through several growth phases (Aboki, 2004; Adams and Moss, 1999; www.agr.gouv.qc.ca/qasa, 2004). The use of sodium benzoate as preservative on both the extract and the concentrates completely stopped the growth of fungi. This further signifies its effectiveness as a preservative.

The maximum amount of bacteria population allowed in juice drinks is given as 10^4 CFU/ml (Wikipedia, 2009). This information was used to estimate the shelf life of the extract and the concentrates by plotting a graph of log of bacteria count against time (Aboki, 2004). The shelf life of the original extract without preservative added was 1.8 days. For the concentrates obtained at 60°C, the shelf lives were 1.9, 2.4 and 2.9 days for pro-ducts heated for 85, 130 and 160 min, respectively. For similar products obtained at 80°C, results indicated 1.9, 2.4 and 3.0 days while 2.2, 2.5 and 3.0 days were obtained for concentrates produced at 90°C. The detailed results are presented by Aboki (2004). These results indicate that the shelf life of the concentrates were un-affected significantly by temperature within the range of conditions used in this work.

Taste determination

A taste panel comprising of sixteen students was set up to determine the acceptability of the products. A dilution ratio of 1:30 was used and about 6% sugar was added. Responses of the panel indicated that the taste of the drink was only slightly affected by concentration. All the concentrates were found to be acceptable, but the original extract was the most acceptable.

Conclusion

a) Time required for concentrating sorrel juice to the same solid content level reduced with increase temperature in a non-linear fashion between 60°C and 90°C.
b) Vitamin C level in the drink reduced with concentration while iron and lead level increased under the concentration conditions used.
c) Sodium benzoate was found to be an effective preservative even for zobo juices exposed to the atmosphere.
d) Thermal sterilization of the drink between 60 and 90°C over a period of time improved the shelf life (without capping or refrigerating) by two-fold.
e) Evaporation of the extract under vacuum between 60 and 90°C reduced the volume of the extract substantially and make for cheaper transportation and packaging costs.

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