EXTENDED ABSTRACT

FORMULATION OF A NATURAL LIQUID SOAP ENRICHED WITH CAROTENOIDS FROM PALMYRAH FRUIT PULP AND ROCK SALT

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Abstract

The free alkali present in the toilet soaps disrupt the natural pH of the skin, leading to skin dryness. Hence this study was conducted to produce a natural liquid soap with lesser free alkali content using soapberry (*Sapindus mukorossi*) drupes, rock salt, cold pressed virgin coconut oil (*Cocos nucifera*) and palmyrah (*Borassus flabellifer*) fruit pulp which contains oil-soluble carotenoids capable of rejuvenating our skin. Carotenoids from palmyrah fruit pulp were extracted using 80 %(v/v) aqueous glycerol and cold pressed virgin coconut oil in 2:1 (v/v) ratio. Three different formulations were established. The soap formulation containing rock salt to replace one third of KOH contained significantly less free alkali (g/L) (0.75± 0.05) than the formulation containing no rock salt (1.34 ± 0.28). Meanwhile the soap without carotenoids had significantly lower fatty matter (%) and higher free alkali than the formulation containing carotenoids. Carotenoids and rock salt reduce the drying effect of soaps.

Keywords: Free alkali content, liquid soap; oil-soluble carotenoids, palmyrah fruit pulp, rock salt

1. Introduction

Palmyrah (*Borassus flabellifer*) is a widely distributed well-grown plant species especially in the northern parts in Sri Lanka which has the potential of producing several value added products. Palmyrah fruit pulp is used for the production of several food items, cosmetics and alcohol based products as well (Sobini et al., 2018). Mesocarp of palmyrah fruit is yellow and orange in colour as it is rich in carotenoids, a source with proven antioxidant properties (Janz & Wickramasekara., 2002). The properties of palmyrah fruit pulp are useful in making soap enriched with antioxidants, and have already been used to formulate a facewash (Vinoja et al., 2020). However, no effort was made in this study to reduce the total free alkali content which may disrupt the natural pH of the skin that leads to skin dryness and related problems. Free caustic alkali is one of the parameters that determine the abrasiveness of any soap (Onyekwere, 1996). Higher free alkali content in soaps affect the skin in negative ways. In the present study the feasibility of making a natural liquid soap using the oil-soluble carotenoids extracted from palmyrah which is capable of rejuvenating our skin with lesser free alkali content was investigated.

2. Materials and Methods

2.1 Materials

Fresh palmyrah fruits were collected from Jaffna, Sri Lanka. Rock salt, soapberry (*Sapindus mukorossi*) drupes, jasmine (*Jasminum grandiflorum*) oil and cinnamon (*Cinnamomum verum*) oil were purchased from the Ayurvedic shop in Jaffna. Glycerol and cold-pressed virgin coconut oil were purchased from Pettah Essence and Cargills Food City, Jaffna respectively. Potassium hydroxide was purchased from Sigma Aldrich, USA.

2.2 Preparation of plant extracts

Palmyrah fruit pulp was extracted by squeezing and scraping portions of the peeled fruits in a tray containing distilled water (50 ml of water per seed) and filtered using muslin cloth. Soapberry extract was prepared by boiling the soapberry drupes with distilled water in the ratio of 1:20 drupes: water (w/w). All the prepared extracts were filtered using filter paper.

2.3 Pigment oil extraction

Carotenoids from palmyrah fruit pulp was extracted by macerating the pulp with 80% aqueous glycerol and cold pressed virgin coconut oil in 2:1 (v/v) ratio, using a mortar and pestle and then shaking the mixture at 250 rpm for 3 hours using an orbital shaker, followed by heating in a water bath at 35 °C for 30 minutes in which the solid: solvent ratio was maintained at 1:10 ratio (dw:v). Then the oil fraction was separated via centrifuging at 2000g for 10 minutes at room temperature (29 °C). The mixture was then separated using a separating funnel. The oil layer containing the pigments was subsequently used for the preparation of soap.

2.4 Formulation of the liquid soap

The saponification value of cold-pressed virgin coconut oil was determined to calculate the required amount of KOH per g oil. Three different formulations of soap were prepared and tested (Table 1). Except for soap formulation C, one third of the KOH required was replaced by adding rock salt. Pigment oil/coconut oil without pigments was heated at 35 °C for 10 minutes by using a magnetic stirrer. Then KOH and rock salt solution was added to the heated oil and heated at same temperature for 30 minutes and allowed to cool to room temperature. Then the mixture was homogenized for 15 minutes followed by pouring into molds. After being set, the soap was dissolved in distilled water in 2:3 soap: water (w/w) ratio and heated at 60-70 °C for 10 minutes. After leaving the mixture to settle overnight, glycerol was added to the mixture in 1:5 soap mixture: glycerol (w/w) ratio and mixed well.

2.5 Sensory evaluation

Sensory evaluation was conducted using 30 untrained panelists for the parameters of texture, colour, aroma, touch, formability, washing ability; feel after washing, dirt removing ability and overall acceptability.

2.6 Evaluation of physico-chemical parameters

Physico-chemical parameters namely such as pH, lather volume, total fatty matter (TFM) and total free alkali were determined according to the methods described in Sri Lanka Standard (SLS) 1390: 2009 developed for liquid soap.

Table 1: Composition of the three different formulations of liquid soap

Ingredient	Quantity (%) (W/W)		
	Formulation A	**Formulation B**	**Formulation C**
Pigment oil	46.0	0.0	46.0
Coconut oil without pigments	0.0	46.0	0.0
Rock salt	3.5	3.5	0.0
KOH	7.0	7.0	10.5
Distilled water	20.0	20.0	20.0
Soapberry extract	20.0	20.0	20.0
Essential oil	3.5	3.5	3.5

2.6.1 Determination of pH

pH of the 0.5g of homogenized sample dissolved in 45 ml of distilled water was measured using a digital pH meter at room temperature.

2.6.2 Determination of lather volume

Five (5) g of soap sample was taken in a clean beaker and dissolved in 100ml hard water. It was then homogenized for 60 seconds at low speed and was transferred to a 500ml graduated cylinder. Lather volume for the sample was obtained directly by reading the calibrated scale.

2.6.3 Determination of total fatty matter (TFM)

Five (5) g of sample was taken into a 250 ml beaker and 100 ml of hot distilled water was added to completely dissolve the soap. Then 40 ml of 0.5N HNO_3 was added until the contents turned slightly acidic. The mixture was heated over a water bath until fatty acids were floating as a layer above the solution. Then it was cooled suddenly in ice water in order to solidify the fatty acids and the layer of fatty acids was separated and taken in a weighed porcelain dish. The contents were evaporated and the residue was weighed. The % of fatty matter in the analyzed soap samples was calculated from differences in weight, using the following equation:

$$\text{Fatty matter (\%)} = \frac{B - A}{C}(100) \qquad (1)$$

Where:
A – Weight of the porcelain dish (g);
B – Weight of the porcelain dish + soap after drying (g);
C – Weight of the initial sample of soap (g)

2.6.4 Determination of total free alkali

Two (2) g of soap sample was taken and mixed with 10 ml of distilled water. Then (10) ml of the soap solution was taken into the titration flask and titrated against 0.1 M HCl, using phenolphthalein as the indicator. Total free alkali was determined using the titre volume.

2.6.5 Statistical analysis

Statistical analysis for physico-chemical parameters was performed using one-way Anova in Minitab17. Data for sensory evaluation were analyzed using Friedman non parametric test.

3. Results and Discussion

Oil-soluble carotenoids were successfully extracted (Fig.1a & b) in oil and the oil containing carotenoids was yellow in colour (Fig. 1 c), in comparison to the colour less cold-pressed virgin coconut oil. Between the liquid soaps produced with either pigment oil or colour less virgin

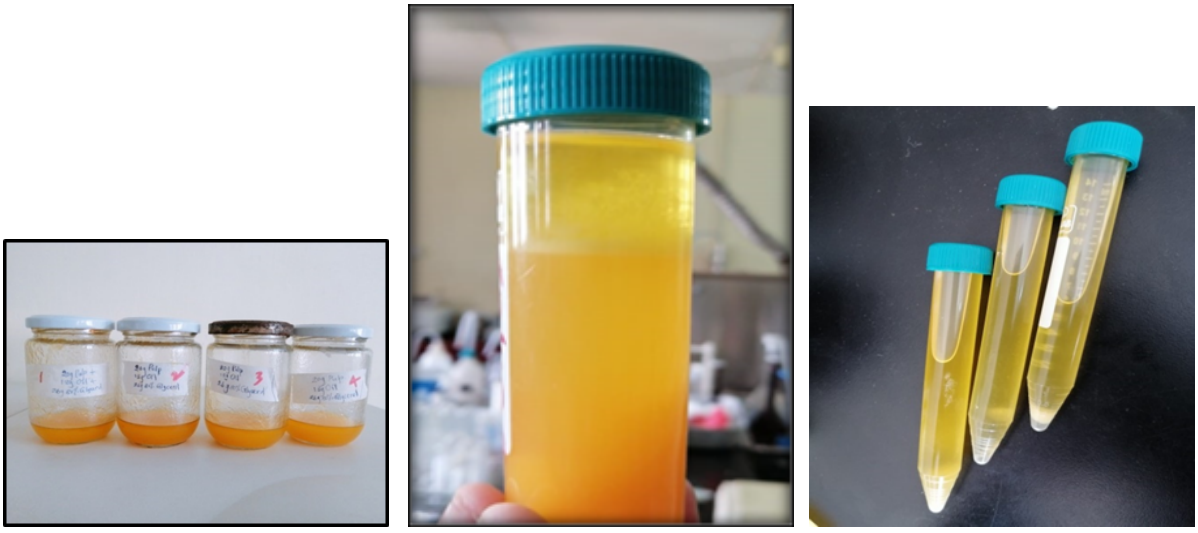

Figure 1. (a: The extraction mixture containing macerated pulp and organic solvents in 1:10 (dw:v) ratio b: The extraction mixture after being centrifuged c: Pigment oil

coconut oil, the formulation A (Table 1) had higher values for the quality parameters, texture, colour, aroma, and foam ability than formulation B, which is clearly seen in the spider web shown in Fig.2. Based on the analysis of data on the sensory attributes through Friedman test, formulation A which showed a significant difference (p value - 0.00) for the overall acceptability of the product, was chosen as the best. In this formulation the addition of pigment oil has significantly contributed towards the natural creamy yellow colour preferred by the panelists.

Excess free caustic alkali causes skin itching and make clothes worn-out. According to SLS requirement for liquid soap (SLS 1390: 2009), the maximum permitted percentage by mass of total free alkali, as KOH, is 0.05%. Soap formulation A contained 67 fold less amount of the maximum permitted percent by mass of total free alkali, whereas formulation B contained 50 fold less and formulation C contained 37 fold less respectively (Table 2). Accordingly, the replacement of a portion of KOH with rock salt as well as incorporation of pigment oil have contributed remarkably to the reduction of free alkali content. The addition of soapberry extract could have also contributed towards lesser free alkali content. Meanwhile there is no significant difference in the pH between the different formulations. Majority of the commercial soaps have a pH within the range of 9-10 (Tarun et al. 2014), and the formulations tested in the present study also had a pH within this range.

The SLS minimum requirement for the lather regarding soaps in ml, is 200. The formulations tested in present study generated about twice the minimum requirement for lather volume. TFM is a

Figure 2. Spider web analysis of the sensory attributes of the liquid soap A – Soap formulation A: B - Soap formulation B

significant characteristic which plays an important role in the grading of the soap. According to the SLS standards on TFM, minimum requirement for TFM of liquid soap percent by mass is 15. The TFM of the liquid soap formulations tested had 2-3 fold higher TFM than the minimum amount required. A high percent of cold pressed virgin coconut oil in the formulations could have resulted in high amount of TFM. Pigment oil soap had significantly (p value - 0.00) higher percent of TFM, due to the presence of lipophilic carotenoids. The high TFM in the soaps will have an advantageous effect such as retention of moisture (Betsy et al., 2013).

Table 2: Physico-chemical parameters of the liquid soap A, B and C

Parameters	A	B	C
pH	9.75± 0.31 ^{a}	9.35± 0.62^{a}	9.703 ± 0.30^{a}
Lather volume(ml)	415.01± 4.11 ^{a}	386.71± 2.11^{a}	476.7 ± 3.20^{a}
Total fatty matter (%)	48.99 ± 0.80 ^{a}	36.98± 0.49^{c}	46.127 ±0.58^{b}
Total free alkali(g/L)	0.749± 0.05^{a} (0.0007%)	1.062±0.01^{ab} (0.0011%)	1.341 ± 0.28^{b} (0.0013%)

The values in the same row with the same letter as the superscript do not have significant difference between them at 0.05% confidence level

4. Conclusions

Incorporation of oil-soluble carotenoids from palmyrah fruit pulp, and cold –pressed virgin coconut oil raise the TFM of liquid soap whereas the oil-soluble carotenoids and replacement of one third of required KOH by rock salt reduce the total free alkali content. Formulation A which contains rock salt and oil-soluble carotenoids was selected as the best in this study.

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