Journal of Scientific and Innovative Research 2020; 9(1): 1-6
Available online at: www.jsirjournal.com

Research Article

ISSN 2320-4818
JSIR 2020; 9(1): 1-6

Received: 23-10-2019
Accepted: 23-03-2020

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Quality improvement of soaps perfumed with some selected essential oils

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Abstract

This work concerned the valorization of shea butter, palm, coconut and kolo oils and, essential oils from Eucalyptus citriodora, Ocimum gratissimum, Cymbopogan citratus and Plectranthus glandulosus by applying a few stages of quality management procedures for soap making. For this purpose, the consumers' needs regarding the soap quality characteristics have been identified. It emerged that the soap smell was of more concern to them. The product was then designed by mixture design of experiment and, 50 g soap samples produced were subject to consumer assessment. Results showed that samples made with coconut (25 g) and kolo (25 g) oils gave the most popular soap according to the rates they received. Among the data of the four quality characteristics, only those from physical measurements of foam height suited the quadratic model. This lead to optimize this characteristic and the values of variables found were coco (17.171 g) and kolo (32.829 g) for a 9 cm maximum and 8 cm minimum foam height. There was no significant discrimination between both the odors of lemongrass and waya essential oils tested. The soaps perfumed with essential oils from Eucalyptus citriodora, Ocimum basilicum and Plectranthus glandulosus exerted an antimicrobial activity against Bacillus subtilis, Pseudomonas aeruginosa and Staphylococcus aureus.

Keywords: Soaps; Raphia; coco, palm; shea; essentials oils.

INTRODUCTION

The successful development of local products requires the technology transfer and the continuous improvement of the quality of products manufactured by extensive processes involving consumers. In this work concerning cosmetics, the procedure adopted includes innovation which is one of the quality improvements stages, especially the design of prototypes [1]. This stage is recommended in procedures such as Plan-Do-Check-Act (PDCA) and Six Sigma. For this purpose, design procedures including experimental designs are often used [2]. In these approaches, the needs of consumers are the target of the business activity.

A lot of information is available in the literature about the soap quality optimization and manufacturing [2-7]. Soaps are still relevant [8]. They are almost exclusively imported in the Republic of Congo; although, the potentialities exist to sufficiently produce the raw materials. Palm, coconut, eucalyptus, lemongrass and waya oils can be produced in most of the Congolese departments.

Palm oil is still produced mainly traditionally; the large part coming from Asian countries [9]. It is transformed into household soap by some factories. Kolo oil is known in northern Congo and other parts of Central African countries [10-14]. Lemongrass is already used traditionally for various purposes; food, medicine and flagrance. It seems to relieve digestive disorders by its spasmodic effect. It is also attributed anti-inflammatory property and, also a pest repellent and an antimicrobial used to treat flu. Like other essential oils, lemongrass is used in the agro-food industry as an antiseptic or aroma, perfume and pharmacy. Waya is part of the Lamiaceae family, several species of which have medicinal properties. It is an herbal tea consumed to relieve postnatal pains in the department of Lékoumou, in southwestern of Republic of Congo. Its oil is an insect repellent and insecticidal, as well as antimicrobial [15-20].

MATERIALS AND METHODS

Materials

Refined vegetable oils were provided by the following local suppliers: Eco Oil Congo (palm), Pack and

and Shop (coconut). Kolo oil and shea butter were purchased at Poto-Poto Market, Brazzaville. Essential oils of lemongrass (*Cymbopogan citratus*), eucalyptus, *O. gratissimum* and Waya (*Plectranthus glandulosus*) were produced by a rural local NGO called ADM from Douakani village (Congolese South-West).

Laboratory equipment used consisted mainly of various utensils, protective equipment, pH-meter and incubators. Culture medium were used for antimicrobial tests.

Methods

Classification of soap quality characteristics by consumers

Before designing the soaps, a survey was conducted in which 50 randomly selected male and female subjects were asked to rank, on a ordinal scale, four quality characteristics of soaps (cleaning and foaming powers, odor and color) from the largest (score 4) to the least important (score 1) through intermediate levels. The features were randomly listed using a random number table.

Soap formulations

The basic soap formulations were designed using Minitab. A centered four-component mixture design was realized. The maximum and minimum levels were fixed at 50 g and 0 g respectively. The components used were palm(A), coconut(B) and kolo(C) oils and shea butter(D). The total was fixed at 50 g without points on axes. This made it possible to design 15 mixtures in 3 replicates shown in Table 1, giving a total of 45 trials. To each formulation was added a fixed amount of honey (1 ml). The amounts of sodium hydroxide needed for saponification were calculated according to the saponification indices of the oils in each formulation by the following formula:

$$m_{NaOH1} = m_{oil} \times I_s$$

where I_s is the saponification index in NaOH.

The bars were prepared in accordance with the super fatted honey-enriched soap method described by Oden Bella [21].

Table 1 : Soap formulations

Ingredients	Samples														
	E1	E2	E3	E4	E5	E6	E7	E8	E9	E10	E11	E12	E13	E14	E15
Palme (g)	0	25	0	25	16,6	16,6	0	25	50	0	0	0	0	12,5	16,6
Coco (g)	25	25	50	0	16,6	16,6	0	0	0	0	16,6	0	25	12,5	0
Shea butter (g)	0	0	0	25	0,0	16,6	50	0	0	0	16,6	25	25	12,5	16,6
Kolo (g)	25	0	0	0	16,6	0	0	25	0	50	16,6	25	0	12,5	16,6
Honey bee (mL)	1	1	1	1	1	1	1	1	1	1	1	1	1	1	1

Odor optimization of the most preferred soap

The most preferred formulation was used to prepare three further samples including two test samples named E1 and E2 in which 2 ml of lemongrass and 2 ml of Waya essential oils were added in each sample and, the third control sample.

Soap characterization

Soaps were characterized by color, odor, consistency, pH, and foam height. These parameters were evaluated by sensory tests except the foam height that was measured physically. We performed two types of sensory tests: an affective test and a grading test. The affective hedonic rating test was applied to determine the degree of appreciation of the product quality characteristics; namely color, odor and hardness [22]. Fifteen inexperienced assessors were recruited among the students to evaluate the soap bars. So, each sample was evaluated 15 times, making a total of 225 trials.

Samples coded with 5-digit random numbers were presented simultaneously to the assessors in the same order. Assessors were asked to rate their appreciation degree of the product characteristics on a 9-point hedonic scale ranging from "extremely hateful" (level 1) to "love extremely" (level 9) with intermediate levels. The classification test concerned soaps scented with lemongrass and waya essential oils. For this purpose, the assessors were asked to evaluate the samples in the order provided and to rank them in order of preference of the characteristics studied, on a hedonic scale by assigning the highest score (3) to the sample having the most pleasant odor and the lower (1) to the least pleasant smell via intermediate score (2).

The foam height was measured as follows. Two grams (2 g) of different soap samples were added to 100 ml conical labelled flasks containing 50 ml of distilled water each. The mixture was warmed to dissolve the soap and get a clear solution. One milliliter of the soap solution from each flask was introduced in a corresponding test tube. The tests tube was vortexed for 1 min by covering their mouths by the thumb. The foam heights were measured after 5 min. The test was replicated three times, making a total of 45 trials.

Antimicrobial activities of the soaps perfumed with essential oils

Four soap samples made with coco (17.171 g) and kolo (32.829 g) oils were perfumed with three essential oils according to the formulation shown in table 3. They were subject to antimicrobial tests against *Staphylococcus aureus*, *Pseudomonas aeruginosa* and *Bacillus subtilis* following the method described in Chaudari [23].

Table 2: Formulations of essential oils in the soaps

Soap sample	Essential oil (ml		
	Eucalyptus citriodora	*Ocimum Gratissimum*	*Plectranthus glandulosus*
1	0.67	0.67	0.67
2	0.000	2.000	0.000
3	2.000	0.000	0.000
4	0.000	0.000	2.000

Data analysis

The ranking test data were analyzed by the Friedman's method to show that the difference existed between attributes [22] (Kemp et al., 2009). The identity of the different attributes was determined by the Fisher's smallest significant difference multiple comparison test for ranks at 5% significance level. The statistic T was calculated by the following formula:

$$T = \left(\frac{12\Sigma R^2}{bt(t+1)}\right) - (3b(t+1))$$

where *t* is the number of samples, *b* the number of assessors and *R* the sum of ranks. The value of *T* was compared to its threshold value for *n-1* degrees of freedom and $\alpha = 0.05$. When the value of calculated *T* was greater than that of the table, the least significant difference multiple comparison test for ranks (LSRD) was used. Then, the Fishers LSRD (α= 0.05) was calculated as follows.

$$LSRD = 1.96\sqrt{(bt(t+1)/6)}$$

Where, $t_{\alpha/2}$ was read in tables for Student's t distribution. When the difference between two ranks exceeded the value of the LSRD, the samples were declared significantly different.

Other data were analyzed by ANOVA at the 5% significance level using Minitab software. The null hypothesis of equality of means was accepted for *P*-values > 0.05, otherwise it was rejected. The goodness-of-fit statistics (*S* and *R*) were used to determine how well the model fits the data.

RESULTS AND DISCUSSION

Ranking of the quality characteristics of soaps expected by consumers

The results of the survey on consumer expectations, concerning the quality characteristics of soaps, are shown in Table 3. The *T*-value is 44.52 (α = 5%, n-1 = 49) greater than that of the table which is 7.81. The value of the LSRD is 12.65. Differences between samples, two-by-two comparison, are all greater than the LSRD value. Then, there are significant differences between the ranks. The odor is the most rated characteristic (score 154), followed by cleaning (141) and foaming capacities (score 112) and then color (score 83).

Assessment of soap sensory characteristics and foam height

The mean scores of soap quality characteristics assigned by assessors with their corresponding standard deviations are shown in Table 4. Analysis of crude data gave results shown in Table 5. These results show that there are statistically significant associations only between: two terms (palm*kolo and coco*kolo) and color, between three terms (coco*kolo, coco*shea butter kolo*shea butter) and odor, palm*kolo and hardness, three terms (palm*coco, coco*kolo and palm*shea butter) and foam height. The kolo variable appears in six terms out of nine with a significant effect and, the other variables four times. There are eight component blends with negative coefficients that act antagonistically for which the corresponding terms contain coco (5 times), shea butter (5), palm (4) and kolo (1). Shea butter appears in the two significant associations for which coefficients are negative and, coco and palm once. However, coco is involved more times (6) than palm (4) in the nine negative associations.

The regression statistics values along with residual plot checks show that only data of foam height fit the quadratic model (R -sq = 60.9%). However, this model has small predicted R-sq (pred) values; so, it doesn't have better ability to predict the response for new observations. The trend of the foam height vs the type of oil is illustrated in the surface plot shown in Figure 1 for the configuration palm, coco and kolo. Shea butter was eliminated for the reasons mentioned above. The plot shows that variable coco tends to pull down the response. Plots of the surface show that the response variable evolution is similar for coco and kolo oils. The response increases and reaches the summit, then it deceases. This is explained by the estimated regression coefficients for foam height (component quantities) that is too low (-9,67560E-05). If the terms with palm are ignored, the model equation is written as follows.

Foam height = 0.0877115*coco+0.153865*kolo+0.00436569*coco*kolo.

If the project aim is to maximize the response at 9 cm, for example, with a minimum at 8 cm, it results in the following values: 17,171 g for coco and 32,829 g for kolo.

R-sq values obtained from subjective evaluations were about 10%. These do not fit the models available in Minitab.

Regarding the results of data analysis shown in Table 4 again, the formulation coco*kolo was selected for subsequent tests because it meets two of the three first consumer needs cited, odor and foamability with strong coefficients, 4.025 and 10.914 respectively. The formulation retained was the one estimated by optimization of the foam height: 17.171g coco and 82.829 g kolo.

The preferred smell

The results of the soap classification test are shown in Table 6. Friedman's treatment of the data obtained gives a T-value of 11.45 higher than that from the T table which is 6.2 for 3 samples, 10 degrees of freedom and P = 0.05, what leads to the Fisher's LSRD calculation whose value is 9.10. Differences between sample scores give the following values: 3 between E1 and E2, 15 between E1 and E3, and 12 between E2 and E3. It appears that the value of the difference between the scores of the test samples E1 and E2 is lower than that of the LSRD. This difference is not significant between those two samples; contrary to what it is between those and the control. It can be said that the lemongrass and waya smells are so much appreciated by the assessors than that of the control.

Table 3: Scores of soap quality characteristics rated by 50 respondents, and resulting overall rank sums

Respondent	Foaming Capacity	Cleaning capacity	Color	Odor	Respondent	Foaming Capacity	Cleaning capacity	Color	Odor
1	1	4	2	3	26	2	4	1	3
2	3	1	2	4	27	3	4	2	1
3	3	4	1	2	28	2	4	1	3
4	3	4	1	2	29	2	4	1	3
5	3	1	2	4	30	3	2	1	4
6	2	3	1	4	31	2	3	1	4
7	1	2	3	4	32	3	4	1	2
8	3	2	1	4	33	2	1	3	4
9	2	3	1	4	34	3	4	2	1
10	4	3	1	2	35	2	1	3	4
11	1	3	2	4	36	2	3	1	4
12	3	2	1	4	37	2	3	1	4
13	3	2	1	4	38	2	3	1	4

14	2	3	1	4	39	2	3	1	4
15	3	4	1	2	40	2	3	1	4
16	3	1	2	4	41	1	3	2	4
17	2	1	4	3	42	1	4	2	3
18	1	3	4	2	43	3	4	1	2
19	3	2	1	4	44	2	4	1	3
20	3	2	1	4	45	1	4	3	2
21	3	4	1	2	46	2	3	1	4
22	2	1	3	4	47	3	4	1	2
23	2	4	1	3	48	1	2	3	4
24	1	2	3	4	49	1	2	3	4
25	2	1	3	4	50	4	3	1	2
Total (R)						112	141	83	154
T-Value						44.52			

Table 4: Means of soap color, odor and hardness scores and foam height with their corresponding standard deviations (s)

Sample	Color		Odor		Hardness		Foam height(cm)	
	Mean	s	Mean	S	Mean	s	Mean	S
1	6,1	2,1	7,1	1,3	7,5	1,6	9,0	0,8
2	6,1	1,2	6,3	1,4	5,3	1,4	8,6	0,7
3	6,5	2,2	5,9	1,7	7,1	1,4	3,4	1,6
4	6,3	1,4	6,0	1,3	6,5	2,0	5,5	1,4
5	6,9	0,8	5,5	1,7	7,1	1,3	8,6	0,6
6	6,3	1,2	4,9	1,3	6,5	2,0	6,4	0,5
7	5,3	1,9	5,5	1,9	6,1	1,6	7,5	1,8
8	7,1	1,4	6,0	1,6	7,3	1,7	8,0	0,5
9	5,9	1,8	5,5	1,2	5,3	1,5	8,0	0,5
10	5,8	1,6	5,1	1,7	7,1	1,7	8,0	1,1
11	5,6	1,7	5,5	1,5	6,4	2,1	7,5	0,9
12	7,0	1,8	6,3	1,3	7,3	1,6	7,3	0,9
13	5,1	1,6	5,1	1,7	6,3	1,5	6,1	2,4
14	6,5	1,7	6,5	0,8	6,5	1,5	7,8	1,6
15	6,9	1,8	6,2	1,7	7,0	1,4	6,2	1,1

Table 5: Estimated regression coefficients and good-of-fit statistics for soap color, odor, and hardness and foam height (components proportions).

Term	Color		Odor		Hardness		Foam height	
	Coeff	P	Coeff	P	Coeff	P	Coeff	P
Palm	6.090	*	5.817	*	5.552	*	7.602	*
Coco(B)	6.245	*	6.075	*	7.058	*	4.386	*
Kolo	5.833	*	5.146	*	6.852	*	7.693	*
Shea butter	5.361	*	5.450	*	6.108	*	7.517	*
Palm*Coco	0.601	0.737	-0.592	0.730	-2.997	0.103	9.886	0.001
Palm*Kolo	4.824	0.008	1.192	0.487	3.647	0.047	-0.242	0.924
Palm*Shea butter	2.394	0.182	1.050	0.540	2.918	0.112	-6.473	0.024
Coco*Kolo	0.166	0.926	4.025	0.020	1.603	0.382	10.914	0.000
Coco*Sche butter	-3.053	0.094	-3.907	0.025	-1.754	0.346	-2.038	0.441
Kolo* Sche butter	4.359	0.016	4.067	0.019	1.918	0.296	1.700	0.504
S	1.62		1.55		1.663		1.02	
R sq (%)	10.63		8.13		10.34		60.91	

R sq (prev) (%)	1.54		0.00		1.77		27.10	
R sq (ajust) (%)	6.87		4.26		6.56		50.25	
Regression		0.004		0.031		0.005		0.000
Linear		0.461		0.375		0.051		0.003
Quadratic		0.011		0.010		0.068		0.000

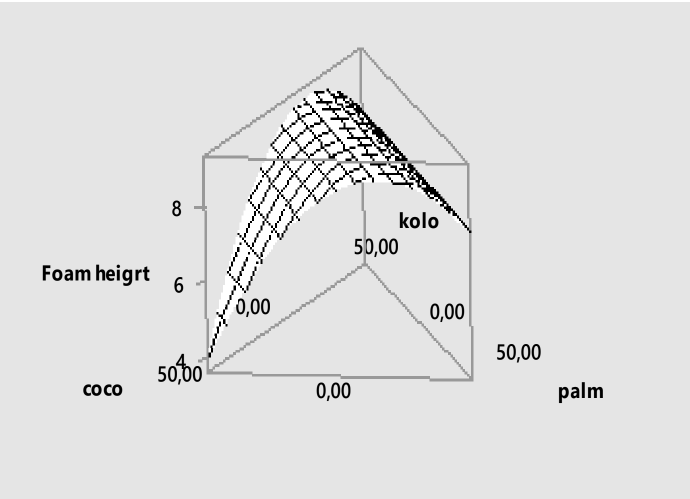

Figure 1: Foam height blend surface chart (Quantities of components)

Table 6: Classification of soaps made of coco (17.117 g)+kolo (32.829 g) and perfumed with lemongrass and waya essential oils.

Assessors	Samples		
	Lemongrass (E1)	Waya(E2)	Control(E3)
1	2	3	1
2	3	2	1
3	1	3	2
4	3	1	2
5	3	2	1
6	3	2	1
7	3	2	1
8	2	3	1
9	3	2	1
10	2	3	1
11	3	2	1
Total (R)	28	25	13
T –value	11.45		

Antimicrobial activity of the soaps prepared

Results of inhibition tests are shown in Tables 7 and 8 and, Figures 2. According to these results the minimum and maximum diameters of inhibition were 6 and 11 mm respectively. The main effects of the soap, strain and concentration were significant (P<0.05). Soap 1 was the most efficient and soap 4 the least one. *Bacillus subtilis* was the least sensible pathogen strain unlike *Pseudomonas aeruginosa* and *Staphylococcus aureus*. Effect of concentration was also significant. This means that the essential oils studied inhibit those three human pathogens.

Table 7: Diameter of Zone of Inhibition (mm) on, *P. aeruginosa* and *B. subtilis* by four soap samples.

Strains	Concentration (mg/ml)	Soap 1			Soap 2			Soap 3			Soap 4		
S. aureus	250	7	8	7	7	8	8	6	7	6	6	7	7
	500	9	8	9	10	11	10	6	7	8	8	9	9
	250	8	8	9	7	7	7	6	6	6	7	7	8
	500	9	9	10	8	9	9	7	8	7	6	7	6
P. aeruginosa	250	7	7	8	6	6	6	6	6	6	6	6	6
	500	8	8	8	10	11	10	9	10	9	9	9	9
	250	7	7	8	6	6	6	6	6	6	6	6	6
	500	8	8	9	10	10	10	9	9	10	10	11	10
B. subtilis	250	6	7	6	6	6	6	6	6	6	6	6	6
	500	8	9	8	7	7	8	10	10	10	7	7	7
	250	6	7	7	6	6	6	6	6	6	6	6	6
	500	8	8	8	8	8	9	9	9	10	6	6	6

Table 8: Analysis of variance for zone of inhibition

Source	DL	Sum of Squares	Mean Square	F	P
Soap	3	12.556	4.185	4.46	0.005
Strain	2	14.111	7.299	7.77	0.001
Concentration	1	136.111	136.111	144.97	0.000
Error	137	128.625	0.939		
Total	143	291.889			

S = 0,968952 R squared = 55,93% R squared (ajust) = 54,00%

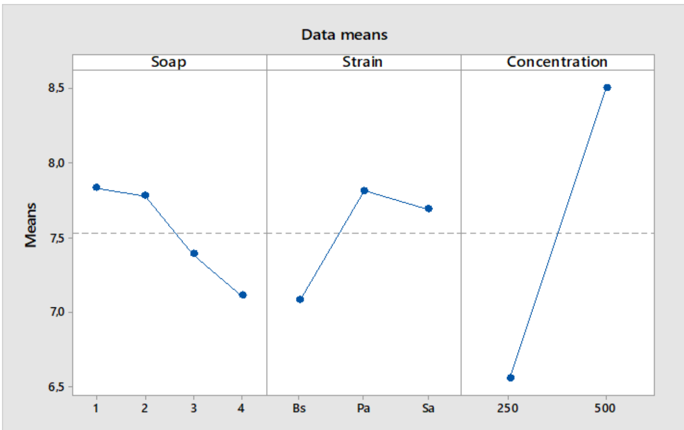

Figure 2: Graphs of main effects for the diameter of Zone of Inhibition (mm) (Bs: *B. subtilis*, Pa: *P. aeruginosa* and Sa: *S. aureus*).

CONCLUSION

From the public contacted judgment, it appears that the optimization of soap passes first by the smell. The smell that seduces consumers is the first quality characteristic they need, followed by cleansing and foaming capacities. Among the oils tested kolo was the one that contributed to significant associations between variables and all characteristics, following by coco oil. This study showed that the most popular soap was that made of coconut (25 g) and kolo (25 g) oils mixture, according to data statistical analysis. The results obtained also showed that physical measurement data can be easier to be modelized in contrast to those from subjective assessment tests, particularly for complex mixtures.

It is also noted that the odors of the essential oils of lemongrass and waya have received almost equal scores. The assessors did not express a significant preference for the smell of either oil.

The soap perfumed with essential oils of *E. citrodora*, *O. gratissimum* and *P. glandulosus* gave the zones of inhibition varying from 6 to 11 mm on *B. subtilis*, *P. aeruginosa* and *S. aureus.* Those results are close to those reported by Chaudari [23], on herbal soaps. The results also showed that soap 1 perfumed with the four essential oil mixture and soap 4 with essential oil of *P. gladulosus* were the most and the efficient respectively. *P. aeruginosa* and *S. aureus* where much sensible than *B. subtilis*.

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