Scholars Academic Journal of Pharmacy (SAJP)
Sch. Acad. J. Pharm., 2015; 4(4): 258-264
©Scholars Academic and Scientific Publisher
(An International Publisher for Academic and Scientific Resources)
www.saspublisher.com

ISSN 2320-4206 (Online)
ISSN 2347-9531 (Print)

Research Article

Effect of Preservative in the Physicochemical Stability of Cosmetic Products Based on Natural Resources from Costa Rican Flora

Rolando Vargas Zúñiga[1], German Madrigal Redondo[2*]

[1]LaboratorioBiofarmacia y Farmacocinética (LABIOFAR) del Instituto de Investigaciones Farmacéuticas (INIFAR), y del Laboratorio de Fisicoquímica Farmacéutica de la Facultad de Farmacia Universidad de Costa Rica.

[2]LaboratorioBiofarmacia y Farmacocinética (LABIOFAR) del Instituto de Investigaciones Farmacéuticas (INIFAR), y del Laboratorio de Fisicoquímica Farmacéutica de la Facultad de Farmacia Universidad de Costa Rica.

***Corresponding author**
German Madrigal Redondo
Email: generacionlcr96@gmail.com

Abstract: A problem of rural women in Latin America is the poor access to economic resources; a solution we propose is to implement a collaborative model, where are used natural resources in the area for production of value added products such as cosmetics products, to improve their living conditions. Objective: This paper concerns the evaluation of the physicochemical stability of hair cosmetics based on natural resources of the Costa Rican flora. Methodology: cosmetic products were developed based on *Carica papaya*, *Ananascomosus*, and others from the area, equally developed stability testing at 25 ° C and 40 ° C for six months following the guidelines of ANVISA, and quality control which were adapted for reproduction on the production site. Results: Formulations designed contain at least 80% of natural excipients, but preservatives exclude parabens derivatives or other similar substances, also were quantified their physicochemical properties such as viscosity, specific gravity and pH, in different storage conditions during the time of the present study. Using ANOVA, was analyzed the data. Conclusions: the study revealed that the formulations are stable for at least one year under the conditions of no more than 30°C and protected from light packaging, and achieved the respective health registry that allowed the marketing of the product.

Keywords: physicochemical stability, *Carica papaya*, *Ananascomosus*, viscosity, specific gravity.

INTRODUCTION

Humanity has used plants for thousands of years and currently has rediscovered its usefulness, so to use herbal extracts as part of cosmetic products has been growing dramatically[1]. This has led, in recent years, to the fact that consumer preferences have turned significantly towards using natural cosmetic substances (mainly vegetable) as ingredients[2].

Carica papaya is a fruit, extremely accessible in the Caribbean region of Costa Rica, which encourages their use in cosmetic products. It is an important source of vitamins A, B and C and, among others, has been reported antimicrobial activity from aqueous extract[3,4]. Pineapple, *Ananascomosus*, is another large fruit crops in Costa Rica, which contains many nutrients, polyphenols being perhaps one of the most striking, for its antioxidant activity[5].

Stability of cosmetic formulations is necessary to ensure a quality product with potential for commercial success. According to Costa Rica government regulations, cosmetics must submit for registration, a number of specifications of the finished product[6]. Regularly, the tests used to assess physical and microbiological stability of cosmetic products are not so many or very complex. Include determination of pH, rheological behavior and specific gravity[7,8,9]. Additionally, can be performed tests as extensibility, interfacial tension and foam test (in the case of shampoos).

This research grew out of the problems found in some shampoos made by the Association of Women Cariari Hills Pococí, who are self-employed women who wanted to develop a product to be sold and distributed to nearby shops and thereby attract resources to support their families. This group produced shampoos with a variety of plant extracts, among others, papaya, lemon, aloe vera and rosemary, with physical and microbiological instability. Furthermore, the need to replace certain ingredients as parabens raised, by others as sodium benzoate or sorbate, allowed in natural cosmetics[10].

METHODS

Nine different shampoo formulations were prepared with three extracts available -papaya, pineapple and lemon-varying combination of preservatives. Each formulation was subjected to a stability study under conditions at 25°C and 40°C, with the sampling time in both cases, as follows: 0, 3 and 6 months. Viscosity, pH, specific gravity and microbiological limit was determined.

Initially formulations contained a mixture of methyl paraben and propyl, following the known synergistic effect of these substances in the preservation of cosmetics and natural extracts. Chemically similar substances were selected, such as sodium benzoate and potassium sorbate, knowing that they have not been related to breast cancer and possess the same preservative effect. Moreover, the latter are common in the food field, such as in drinks and fruit pulps, which also ensures protection of the extracts and the cosmetic formulation.

Considering the synergistic effect of the parabens, stabilizer and preservative effect of the combination of sodium benzoate and potassium sorbate, was evaluated the sodium benzoate as a reference. Thus, three types of shampoo were prepared, in the first were combined sodium benzoate and parabens; in the second sodium benzoate and potassium sorbate were used. The third had only sodium benzoate as preservative.

Preservative-free formulation was not an option because it was determined that the extracts were fermented within a few days of preparation and formulations became microbiologically contaminated; hence, it was not useful as a comparison because it included other variables such as substances produced by fermentation or degradation of the formulation.

Table-1: Basic shampoo formulations, indicating the proportions of preservatives used

Substance	Formula 1	Formula 2	Formula 3
Anionic surfactant	7%*	7%	7%
Coconut fatty acids surfactant	5%	5%	5%
Cationic surfactant	1%	1%	|%
Wetting agent	5%	5%	5%
pHbuffer	1%	1%	1%
Viscosizing agent	1%	1%	1%
Sodium benzoate	0,24%	0,24%	0,24%
Sodium sorbate	0,24%	-----	-----
Aqueous extract(80% pulp – 20% water)	25%	25%	25%
Methylparabene	-----	-----	0,24%
Propylparabene	-----	-----	0,24%
Distilled waterq.s.	100%	100%	100%

*Percentages by mass

For viscosity, a Parmer® Cole viscometer model 98936-10/15 was used, with R2 spindle at 5 rpm and 600 mL flask to contain the sample.

The pH determination was performed using a UB-10 Ultra Basic Denver Instruments® pH-meter using a glass electrode.

Specific gravity was determined with Cole Parmer® specific gravity cup (8.32 mL) using distilled water as a reference at 25°C.

To quantify microbiological limit 10 g or 10 mL of sample were used in 90 ml of nutrient media containing preservatives inhibitors. The sample was cultured in nutrient agar plate and incubated under appropriate conditions of time and temperature to bacteria, fungi and yeasts. The number of colony forming units of each group of microorganisms was counted.

The absence of pathogenic strains such as *E. coli, S. aureus* and *P. aeruginosa* was determined by growth on selective media and bacterial identification means

Statistical analysis of data was performed with JMP4® software using a bivariate ANOVA model for variables time and temperature, with the multiple comparison test Tukey; the time variable with three levels and temperature with two levels; three replicates per sample for each of the evaluated parameters: pH, viscosity and density, with a significance level of 90%.

RESULTS

In order to evaluate the stability of the different formulations, activities described above were executed. The results indicated the range in which the evaluated parameters must be maintained to ensure reproducibility from batch to batch.

Figures 1 and 2 show that by using a mixture of sodium benzoate and parabens as preservatives, the product viscosity is very low. According to Figure 1, the final product, at a temperature of 25°C displays a viscosity between 2500 and 9000 cp, with reference to the range of results of formulations that do not contain methyl and propylparaben.

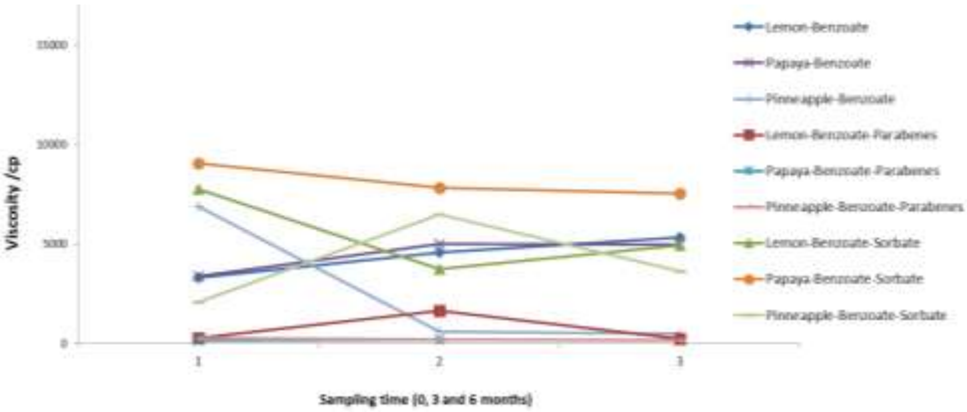

Fig-1: Shampoos viscosity at 25 ^{o}C

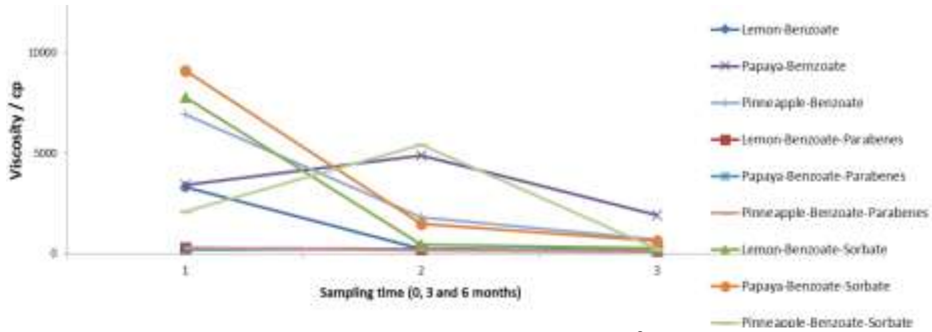

Fig-2: Shampoos viscosity at 40 ^{o}C

Regarding the pH of the final product, it was found that the buffer system used is functioning properly in any of the two conditions tested, since, as shown in Figures 3 and 4, the pH of all the formulations are kept, practically, within a range appropriate for such preparations, of 5 to 6.5.

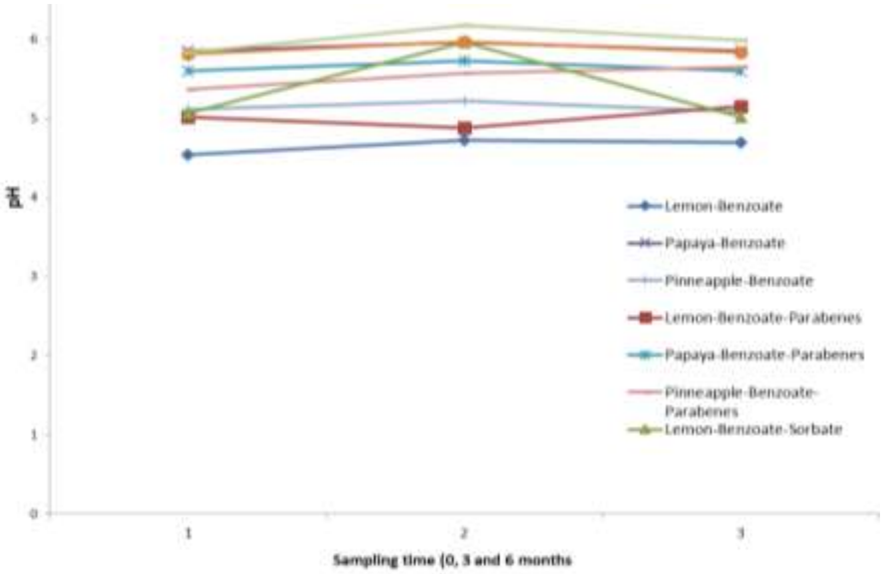

Fig-3: Shampoos pH at 25 ^{o}C

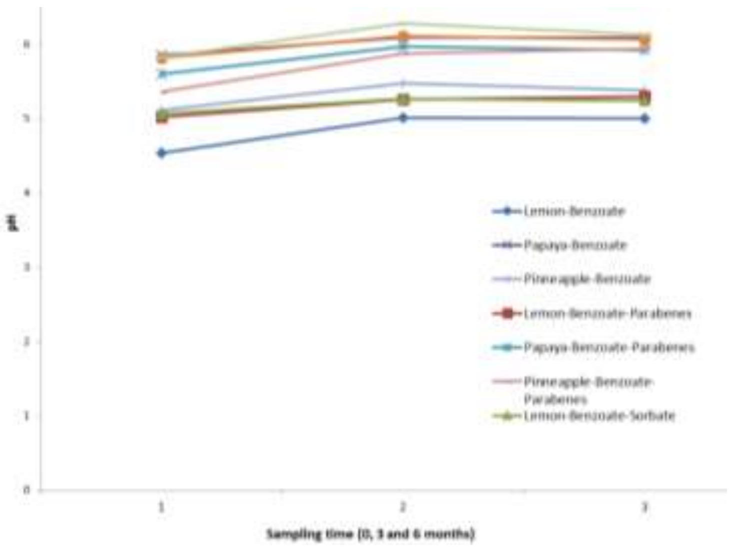

Fig-4: Shampoos pH at 40 °C

It was found that the specific gravity values range from 1.02 to 1.045, with very little variation when considering the change of temperature (25°C to 40°C).

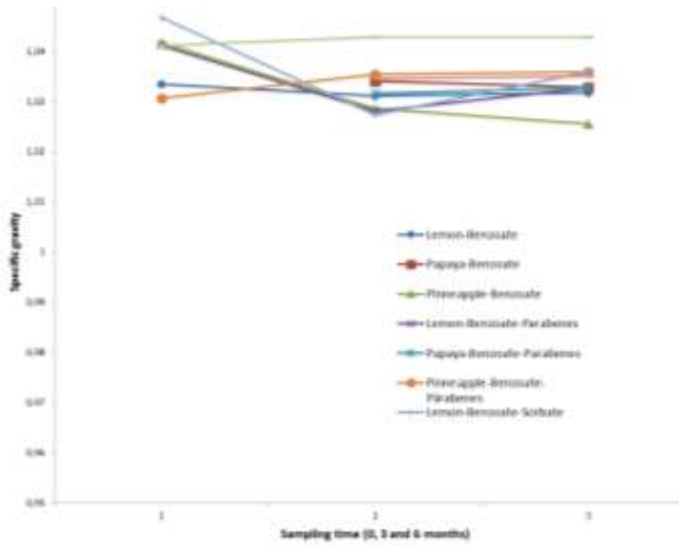

Fig-5: Shampoos specific gravity at 25 °C

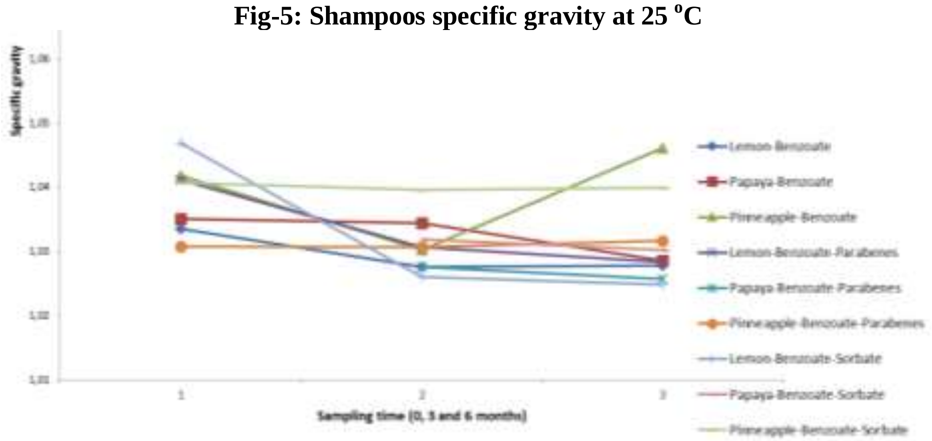

Fig-6: Shampoos specific gravity at 40 °C

In all formulations, microbiological testing was satisfactory, the total bacterial count as for fungi and yeast was less than 10 CFU/g and non-pathogenic organisms were found.

DISCUSSION

Only standard deviations with a significant importance below 0.1 were used to determine the limits allowed in shampoo formulations. In practical terms, there are no significant differences between the several formulations, and, all of them present a stability that cannot be differentiated statistically. These quality limits are arbitrary, only established as an internal control and correspond as follows: pH ± 5%, specific gravity ± 10% and viscosity ±20% the average value.

As observed in the graphs of viscosity, varying only the system preservative, it was found that the effect of these is critical for shampoo viscosity, being that they modify the electrical double layer on the micelle and therefore, the size of the same, which causes a change in the flow resistance of the particles, affecting the viscosity. Especially, the electrolyte sodium benzoate and potassium sorbate, become part of the ionic double layer, cooperating with sodium chloride (used as viscosizing), to stabilize the bilayer and the micelle size. As parabens are nonionic substances, this effect is absent.

Standard deviations below will relate the factorial model prediction error, using the analysis of variance technique, for each of the interactions and factors, in order to determine which of the model parameters are significant (10% significance was used), therefore the following is concluded:

Table-2: Diagnosis and interpretation of the criteria obtained for the parameters evaluated in shampoo formulations.

Termfor viscosity	**Standard error**	**Prob>|t|**
Intercept	311,3109	<.0001
40°C	311,3109	0,0027
Time[0]	440,2601	0,0030
Time[1]	440,2601	0,0693
40°C*Time[0]	440,2601	0,0301
40°C*Time[1]	440,2601	0,4990
Term for pH	**Standard error**	**Prob>|t|**
Intercept	0,040683	<.0001
40°C	0,040683	0,3462
Time[0]	0,057534	0,0067
Time[1]	0,057534	0,0182
40°C*Time[0]	0,057534	0,5543
40°C*Time[1]	0,057534	0,4863
Term for specific gravity	**Standard error**	**Prob>|t|**
Intercept	0,001004	<.0001
40°C	0,001004	0,1800
Time[0]	0,001419	<.0001
Time[1]	0,001419	0,0016
40°C*Time[0]	0,001419	0,3408
40°C*Time[1]	0,001419	0,4942

With respect to viscosity, the model predicts, with a 90% confidence level for interception, and the coefficients accelerated temperature (40 ° C), time 0, time 1 and the accelerated temperature (40 ° C) interaction over time 0,compared with shelf temperature and time, are not equal statistically, so that within three months showsa significant difference between the predicted values and those obtained by the model, but not for the shelf temperature. So, the modelshould be based only in relation shelf temperature - time 1.

For pH the model only predicts for interception, accelerated temperature (40°C) time0 and time1, finding no correlation with temperature accelerated time 0 and time1 interactions; this means that the model predicts for independent values, unrelated with variables interactions.

For density model only predicts for interception, accelerated temperature (40 ° C) time 1 and time 0, so there is no correlation with temperature interactions accelerated time 0 and time1, this means that the model predicts independent values, unrelated with variables interactions.

The interaction between the temperatures, and temperatures with time, do not appear to be significant, however observing the graphs which relate each of the above factors with different physicochemical properties studied, it is clear that the model is very constant over time, namely the physicochemical properties do not vary significantly over time or temperature. An exception appears in the case of formulations containing parabens, which lose viscosity; it appears to be that the stabilizing effect is based on two facts: prevent microbiological contamination for which all combinations of preservatives proved effective, but also is a second effect of maintaining the viscosity of the formulation.

The non-significant result between time and temperature indicates that only one time should be taken to predict the behavior of the formulations, in this case, independently analyzing the shelf temperature and accelerated, can be obtained the upper and lower limits for extreme conditions of the formulation as time passes. Also a ratio of zero-order constant for most of physicochemical properties in different formulations is shown, with a differencein viscosity for the formulations containing parabens as shown in Figure 1 and in the case of most of the formulations as shown in Figure 2.

Another important fact is that each physicochemical property can be predicted for their behavior in time to the same temperature, as shown in figures 3 and 4; since the values are constant, and being theintercept significant statistically for the model, and maintaining the value constant in time to study the same temperature, it can come to this conclusion, except for figure 1, where parabens do not show this behavior.Also, the effect of viscosity loss is shown in figure 2 every formulation with parabens, indicating the origin of each extract is not the factor in the loss of viscosity of the formulations, but it is the act of including in the formulation parabens which affects their physicochemical stability, viscosity here.

As noted above, under the statistical significance of the intercept to the statistical model proposed and not that of the other variables that interact, and the constancy of the value of the physicochemical properties studied at each temperature, it may be noted that the formulationsare stable, except for viscosity formulations containing parabens. Similarly, figure 2 shows the significant loss of viscosity for most formulations, while pH and density variables are not significantly affected in any of the study conditions. Significantly, the viscosity and maximum storage temperature are critical factors to consider when maintaining the stability of the formulations in terms of marketing.

With these results it is concluded that the formulations prepared are stable for marketing purposes, except the one that only contains parabens as preservatives. Of the three base formulas with which we worked, differentiated only by the preservative mixture employed, one containing sodium benzoate and potassium sorbate was selected, as well as being stable, the initial goal was to find a formula that did not use parabens and used a lesser amount of surfactants. It should be noted that, probably, the fact of including a larger amount of nonionic surfactant in formulations containing parabens seeks to correct the problem of viscosity loss due to larger micelle formation; however, this involves an increase in production costs of the formulation.

Moreover, it was possible to resume the execution of tests in a standardized operating protocol that allowed the adaptation of the physicochemical and microbiological tests to conditions of production in rural areas, validating the results obtained instrumentally, compared with the results using a basic portable and economical physicochemical kit, where manufacturers can check quality control tests results are within the limits established in this study for each product.

Thus, in the following table the parameters determined for the selected formulation are summarized, which should serve as a guide for the reproducibility of each lot produced. It is necessary to note, that for such formulations there are no official references to the value required for each parameter, then it corresponds to the manufacturer to establish it, and then fit their own reference.

Table-3: Baseline parameters for selected formulation (with sodium benzoate and potassium sorbate)

Shampoo	Estability	Viscosity/cp	pH	Specific gravity	Foam test	Appearance
Lemon	Room temperature	3700 – 7800	5 – 6	1,02 – 1,05	120 min, 300 cm^3	Clear
Lemon	40°C	300 – 7800	5 – 6	1,02 – 1,05	120 min, 300 cm^3	Clear
Papaya	Room temperature	7000 – 10000	5 – 6	1,03– 1,05	120 min, 250 cm^3	Clear
Papaya	40°C	600 – 10000	5 – 6	1,03 – 1,05	120 min, 350 cm^3	Clear
Pinneapple	Room temperature	2000 – 7000	5 – 6	1,02 – 1,05	120 min, 300 cm^3	Clear
Pinneapple	40°C	300 – 6000	5 – 6	1,02 – 1,05	120 min, 300 cm^3	Clear

In conclusion, Table 3 summarizes the lower and upper limits for the formulations designed, which can stay within this range on storage conditions at 10-30°C and protected from light PET plastic containers with flip-flop lid, allowing not only to reduce production costs, but the packaging and transport of products to more remote areas, ensuring the permanence of the physicochemical and microbiological properties in time.

REFERENCES

1. Shivanand P, Nilam M, Viral D; Herbs Play an Important Role in the Field of. International Journal of Pharm Tech Research, 2010; 2(1):632-639.
2. Houghton P; Biologically active compounds: detection and isolation from plant material. Cosmetic and Toiletry, 1994;109(6):39 - 47.
3. Adekunle A, Adekunle O; Preliminary assessment of antimicrobial properties of aqueous. Biology and Medicine, 2009;1(3): 20-24.
4. Telis V, Telis-Romero J, Sobral P, Gabas A; Freezing point and thermal conductivity of tropical fruit pulps: mango and papaya. International Journal of Food Properties, 2007; 10: 73-84.
5. Haripyaree A, Guneshwor K, Damayanti M; Evaluation of antioxidant properties of phenolics extracted from Ananascomosus L. Notulae Scientia Biologicae, 2010; 2(2): 68-71.
6. Costa Rica Asamblea Legislativa; Reglamento Técnico Centroamericano RTCA 71.01.35:06 Productos Cosméticos. Registro e Inscripción Sanitaria de Productos Cosméticos, 231-2008 (COMIECO-L) (June 26 2008).
7. Bouftira I, Abdelly C, Sfar S; Characterization of cosmetic cream with Mesembryanthemum crystallinum plant extract: influence of formulation composition on physical stability and anti-oxidant activity. International Journal of Cosmetic Science, 2008;30: 443-452.
8. Moulai N, Hadj A, Sabr N, Hadji A; Determination of optimal cream formulation from long-term stability investigation using a surface response modelling. International Journal of Cosmetic Science, 2006; 28: 211-218.
9. The United States Pharmacopeial Convention; Farmacopea de los Estados Unidos. USP 30. 2007. Rockville.
10. COSMOS-standard AISBL. COSMetics Organic Standard; January 2011.Available at: http://www.cosmos-standard.org/docs/COSMOS-standard_v1.1_310111.pdf