INNSPUB

International Journal of Biosciences | IJB |
ISSN: 2220-6655 (Print), 2222-5234 (Online)
http://www.innspub.net
Vol. 23, No. 3, p. 24-32, 2023

RESEARCH PAPER OPEN ACCESS

Formulation, Development, and Characterization of Herbal Soap infused with KaPaMa *(Euphorbia neriifolia, Diplazium esculentum, and Coleus blumei)* extracts

Jinky Marie T. Chua*

Cagayan State University, Andrews Campus, Tuguegarao City, Cagayan North, Northern Philippines

Key words: *C. blumei, D. esculentum, E. Neriifolia*, KaPaMa soap, Medicinal plants

http://dx.doi.org/10.12692/ijb/23.3.24-32 Article published on September 03, 2023

Abstract

Herbal soap production promotes the use of natural and sustainable personal care products; and non-wood forest products, which can provide economic opportunities for local communities while contributing to biodiversity conservation and promoting the use of traditional medicinal plants. This study aimed to formulate, develop, and characterize an herbal soap infused with KaPaMa (Karimbuaya, Pako, Mayana) leaf extracts and evaluate its physicochemical properties. Six different soap base formulations were prepared, and the best soap formulation was selected, which did not contain propylene glycol. KaPaMa extracts were incorporated at three different concentrations (0.2%, 0.4%, 0.8%), and the developed KaPaMa soaps were evaluated for their physicochemical properties. The extracts of *Euphorbia neriifolia* (Karimbuaya), *Diplazium esculentum* (Pako), and *Coleus blumei* (Mayana) contain phytochemicals that exhibit antimicrobial, antioxidant, and anti-inflammatory properties, making them suitable for use in soap formulations. The KaPaMa soaps were found to have acceptable pH and low total alkali content, indicating that these are not corrosive to human skin. However, the soaps had low foaming ability, high moisture content, and total fatty matter. Further optimization of the formulation is recommended to improve foaming ability and reduce moisture content.

* **Corresponding Author:** Jinky Marie T. Chua ✉ jchua@csu.edu.ph

Introduction

The use of ingredients in products has a rich history spanning traditional medicine, perfumery, and cosmetics (Carvalho *et al.,* 2016). There is a growing demand for cosmetics that incorporate ingredients as they are seen as organic and environmentally friendly options. Recently, herbal soaps have gained popularity due to their perceived health benefits and sustainable nature. These soaps are crafted from plant extracts, essential oils, and natural colorants, which are believed to offer advantages like moisturizing effects, anti-aging properties, and anti-inflammatory benefits (Razal and Palijon, 2008). Moreover, these soaps are considered more environmentally friendly than conventional ones because they are made from renewable resources without the addition of synthetic chemicals. Additionally, natural herbal soaps tend to have a level of phenolic content and antioxidant activities compared to commercial varieties, which could potentially enhance their shelf life (Chirani *et al.,* 2021).

The region of Cagayan Valley in the Philippines is famous for its range of plant and forest resources. These resources include plants and aromatic plants that have been traditionally utilized for a variety of purposes, including personal care. Formulating soaps using these plants presents an excellent opportunity to promote sustainable development and biodiversity conservation (Razal and Palijon, 2008). However, further studies are needed to ensure the safety and effectiveness of soap ingredients as well as their potential interactions with other components (Nadeeshani Dilhara Gamage *et al.,* 2022). Moreover, more research is necessary to determine the formulation and processing methods for soaps to guarantee their quality and efficacy. Additionally, it is crucial to educate consumers about the advantages of using soaps while also raising awareness about their impact on the environment. The significance of producing herbal soaps extends beyond care and environmental sustainability in the Philippines. The production of these soaps can also create opportunities for communities, particularly those involved in harvesting and processing non-wood forest products. By promoting the use of natural ingredients and sustainable practices, herbal soap production can support livelihoods within these communities while contributing to the development of the Philippine economy (Eastin, 2001).

Karimbuaya (*Euphorbia neriifolia*), Pako (*Diplazium esculentum*), and Mayana (*Coleus blumei*) have been traditionally used in the Philippines for their properties. Ayurvedic medicine has long recognized the healing properties of *E. neriifolia*, which can be used to treat skin ailments, wounds, and respiratory issues (Rajčević *et al.*, 2022). Similarly, *D. esculentum* and *C. blumei* are known for their bioactive compounds, such, as flavonoids, phenolics, and terpenoids that contribute to their benefits (Rajčević *et al.*, 2022). These research findings suggest that incorporating these plants into soap formulations could offer effectiveness and safety.

The significance of herbal combinations lies in their potential to provide enhanced therapeutic benefits compared to individual herbs or drugs. By harnessing the synergistic effects of different components, herbal combinations can offer a more comprehensive and effective approach to treating various health problems. This has implications for traditional medicine practices, as well as for the development of new herbal-based therapies in modern medicine. The utilization of herbal combinations also promotes the sustainable use of medicinal plants and contributes to the preservation of biodiversity. Hence, this study focuses on the formulation and development, and characterization of herbal soap infused with KaPaMa (*E. neriifolia*- Karimbuaya, *D. esculentum*- Pako, and *C. blumei*- Mayana) leaf extracts.

Methodology

Sample collection and extraction

Fresh leaves/ferns were collected within the vicinity of Cagayan Province. These are sorted, washed, air-dried for 24 hours, and oven-dried until brittle. The dried samples were ground and mixed with 95% ethanol. The extracts were then obtained using a rotary evaporator.

Preliminary phytochemical screening analyses

Preliminary phytochemical screening was carried out to determine the presence of bioactive constituents such as alkaloids, anthocyanins, carbohydrates, carotenoids, coumarins, flavonoids, phenols, quinones, saponins, steroids, tannins and terpenoids of the three aqueous extracts by using qualitative phytochemical screening using standard methods (Guevara, 2015; Munoz *et al.,* 2021).

Soap base formulation

Six different soap formulations (SF) were done to determine the best soap base before the addition of the plant extracts. Coconut oil and lye solution were mixed and stirred. With continuous stirring, propylene glycol was added to SF2 to SF6, and the solutions were allowed to cool down. The resulting solution is then poured into a soap molding container. The SFs were evaluated in terms of color, odor, texture, phase separation, homogeneity, stiffness, greasiness, and foam formation.

Development of KaPaMa soap

The development of the herbal soap using the best soap formulation involved incorporating KaPaMa extracts at three different concentrations (0.2%, 0.4%, and 0.8% w/w) and creating a control group without any KaPaMa extract. These soaps were designed to create variations in the active ingredient's concentration to study its effects on the physicochemical properties of the soap products. The KaPaMa extracts were carefully added to the best SF in specific ratios to achieve the desired concentrations. To ensure the reliability and consistency of the results, 20 products for each soap concentration were produced. This would help account for any variations that might occur due to factors like manufacturing variability.

Physicochemical evaluation of KaPaMa soap

1. Organoleptic parameters like color, odor, texture, phase separation, homogeneity, stiffness, and greasiness were evaluated manually or physically.

2. The pH was determined using a pH meter using 2 g of the finished soap, which was dissolved in 10 mL of distilled water.

3. Foaming ability was determined by measuring the height of the foam. Two grams of each soap was dissolved in 50 mL of distilled water in a 100 mL flask and shaken using an orbital shaker for 2 minutes. These were allowed to stand for 10 minutes, after which the height of the foam was measured. This procedure was repeated thrice.

4. The total fatty matter test was carried out by reacting the soap with acid in the presence of hot water and measuring the fatty acids obtained. About 10 g of the finished soaps were weighed, and 150 mL of distilled water was added and then heated. Each soap was dissolved in 20 ml of 15% sulfuric acid while heating until a clear solution was obtained. Fatty acids on the surface of the resulting solution were solidified by adding 7 g of beeswax and reheating. The setup was allowed to cool to form a cake. The cake was removed, blotted to dry, and weighed to obtain the total fatty matter using a formula: $\%TFM = (A - X)/W \times 100$ Where A= weight of wax+ oil, X= weight of wax, W= weight of soap.

5. Moisture content was determined by weighing 10 g of each soap sample, transferring it to a porcelain dish of known weight, and keeping it in a hot air oven at 100 – 105°C for an hour. The sample was then weighed to deduct the actual weight of the porcelain dish. The percentage moisture content was computed using the formula: % Moisture content = (Difference in weight/initial weight) × 100

6. The total alkali was determined by titrating excess acid contained in the aqueous phase with a standard volumetric NaOH solution. Each gram of the finished soap was weighed, and 5 mL of ethanol and 0.5 mL of 1M sulfuric acid solution were added to the mixture and heated until the soap sample dissolved. The test solution was titrated against 1.0M NaOH using phenol phenolphthalein as an indicator. The total alkali was obtained using the formula: % alkali =

[(VA-VB)/W] × 3.1.

Results and discussion

Preliminary phytochemical screening analyses

The qualitative phytochemical analysis of *E. neriifolia, D. esculentum,* and *C. blumei* plants revealed the presence of phytochemicals, like alkaloids, flavonoids, phenols, saponins, tannins, and terpenoids (Table 1). These compounds have been known to possess antioxidant and inflammatory properties that can be advantageous for maintaining healthy skin. Research studies indicate that the extracts obtained from these plants have antioxidant properties, which make them suitable for incorporation into soap formulations.

By including these phytochemicals in soap extracts, it is possible to enhance their ability to cleanse and moisturize the skin effectively by removing dirt and oil while simultaneously keeping it hydrated.

Table 1. Phytochemical analysis

Phytochemicals	*E. neriifolia*	*D. esculentum*	*C. blumei*
Alkaloids	+	+	+
Anthocyanins	-	-	-
Carbohydrates	-	-	-
Carotenoids	-	-	-
Coumarins	-	-	-
Flavonoids	+	+	+
Phenols	+	+	+
Quinones	-	-	-
Saponins	+	+	+
Steroids	-	-	-
Tannins	+	+	+
Terpenoids	+	+	+

**generated by the Central Analytical Laboratory of Cagayan State University.*

Soap base formulation

The prepared base soaps are shown in Fig. 1. The parameters for soap formulations SF1, SF2, SF3, SF4, SF5, and SF6, such as color, texture, phase separation, homogeneity, stiffness, grittiness, greasiness, and foam ability, were determined (Table 2). Acceptability of these was scored by chemists from 1 (lowest) to 5 (highest). The formulations have a clear to light white to dirty white color without the addition of any colorant. All formulations have no odor, and the texture of the formulations is smooth and consistent. SF1 and SF5 have a score of 5 in phase separation, which indicates no phase separation, while SF4 and SF6 indicate a non-uniform soap under the homogeneity parameter. For stiffness, only SF1 and SF5 are considered firm and sturdy soaps, but only SF5 has no grittiness. However, SF5, together with SF4, are very greasy. Also, all have excellent foam ability except for SF4.

The selection of ingredients for soap differs through the amount of lye solution used and propylene glycol as the humectant. SF 1 has an average of 4.375 with 5 points for texture, phase separation, homogeneity, and stiffness. In contrast, it scored 4 points for the foam ability. The 2nd, 3rd, 5th, and 6th formulations have an average of 4.125 with 5 points for color and homogeneity for both the 2nd and 3rd protocols while a score of 5 for the texture, phase separation, and homogeneity for the 5th protocol and a score of 5 points also for SF6 for texture, stiffness, and grittiness.

The best formulation based on the optimization result is SF1, which showed that soap formulation is best without the addition of propylene glycol, which serves as a humectant. The other protocols contained an addition of propylene glycol and an increased/decreased lye solution.

Physicochemical evaluation of KaPaMa soaps of different concentrations

Three concentrations and control were produced and characterized (Fig. 2). The physicochemical properties of the soaps include color, odor, texture, phase separation, homogeneity, stiffness, greasiness, pH, foaming ability, total fatty matter, moisture content, and total alkali content (Table 3).

The organoleptic evaluation showed that as the concentration of the KaPaMa extract increases, the green color and leafy odor of the soap also increase.

The texture became rough, and there has been significant grittiness upon the addition of the extracts. However, there is no change in terms of phase separation, homogeneity, stiffness, and greasiness.

Table 2. Soap base formulation evaluation.

Parameters	SF1	SF2	SF3	SF4	SF5	SF6
Color	3	5	5	4	4	4
Odor	5	4	4	4	5	5
Texture	5	4	4	4	5	5
Phase separation	5	4	4	4	5	4
Homogeneity	5	5	5	3	5	4
Stiffness	5	4	4	3	4	5
Grittiness	4	4	4	3	3	5
Greasiness	4	3	3	2	3	2
Foaming ability	4	4	4	3	4	4
Average	4.375	4.125	4.125	3.25	4.125	4.125

The pH of a soap is an important factor as it determines its mildness and compatibility with the skin. Human adult skin has a pH of slightly less than 5, indicating that it is somewhat acidic, and it is believed that long-term use of cleaning chemicals such as soap can modify the pH of the skin (Mendes *et al.*, 2016). The pH of the KaPaMa soap ranges from 9.01 to 9.09, with the control (no extract or base formulation) having 9.12. These lie between the normal pH range of 8-10, as reported by the National Agency for Food and Drug Administration and Control (NAFDAC), and ISO standard range (9.0-10.5), which indicate that KaPaMa herbal soaps are not corrosive to human skin.

Table 3. Physicochemical evaluation.

Parameters	0.2% w/w	0.4% w/w	0.8% w/w	Control*
Color	Light green	Medium green	Dark green	White
Odor	Mild leafy smell	Leafy smell	Strong leafy smell	No smell
Texture	Rough	Rough	Rough	Smooth
Phase separation	None	None	None	None
Homogeneity	Uniform	Uniform	Uniform	Uniform
Stiffness	Firm	Firm	Firm	Firm
Grittiness	Significant grittiness	Significant grittiness	Significant grittiness	No grittiness
Greasiness	Non-greasy	Non-greasy	Non-greasy	Non-greasy
pH	9.09	9.02	9.01	9.12
Foaming index	18 cm	14 cm	14 cm	20 cm
Total fatty matter	38.8%	36.8%	35.02%	40.02%
Moisture content	22.42%	26.67%	28.03%	20.30%
Total alkali content	0.28%	0.26%	0.20%	0.30%

**no extract added/ SF1*

The foaming index is a measure of how soap creates foam. Although the amount of foam may not directly indicate how effective the soap is at cleansing, it is a factor for consumers to consider. In the case of KaPaMa soaps, their foaming index ranges from 14 to 18 cm, which is lower compared to the control soap at 20 cm. This difference could be due to the concentration of extracts used in the KaPaMa soap formulation. As the concentration of extracts increases, it can impact the surfactant properties of the soap, resulting in reduced foaming ability. This observation is supported by the finding that as extract concentration rises, the foaming index of KaPaMa soap decreases. While consumers often associate foaming soaps with cleaning abilities, it is crucial to note that a soap's effectiveness in removing dirt and oils is not solely determined by its ability to produce foam.

Fig. 1. Six different soap base formulations.

Total Fatty Matter (TFM) is how much fat substance the soap has, which is an indication of soap quality. The amount of fatty acids in the soap contributes to its cleansing and moisturizing properties. The TFM of the KaPaMa soaps ranges from 35.02%-38.8%, which is closer to 40.02% of the control. The difference in TFM is responsible for high moisture content. The presence of unreacted NaOH in the mixture results in low TFM values. Higher concentration of fatty acids has good effects on the skin in the sense of rehydration and overall enhanced cleansing properties. According to ISO standards, good quality soaps must have a TFM above 76% (Kundu *et al.*, 1977; Arasaretnam and Venujah, 2019). It shows that KaPaMa soaps can be harmful, particularly to dry skin. According to studies, reduced TFM in soaps is owing to the inclusion of several additives in soaps, such as color, preservatives, and fillers (Idoko *et al.*, 2018), as well as unreacted sodium hydroxide residue remaining in the soap (Vivian *et al.*, 2014).

Moisture content is a parameter for evaluating a product's shelf life (Oyedele *et al.*, 2021). High moisture content in the soap could lead to a reaction of excess water with unsaponified fat to give free fatty acid and glycerol in a process called hydrolysis of soap on storage. The soap's high moisture content causes the formation of free fatty acids and glycerol when it reacts with unsaponified fat, lowering its quality and shortening its shelf life (Vivian *et al.*, 2014). As a result, the moisture content of a soap must be kept to a minimum (Dema *et al.*, 2022).

The soap should have a moisture level of 10.5%-12.5%, according to ISO requirements (Firempong and Mak-Mensah, 2011). However, the moisture content results of the soaps range from 22.42%-28.03%, which increases as the concentration of the extract added increases. This range is also high compared to the control (20.30%). This could be due to the hygroscopic nature of the herbal extracts used in the formulation. The higher the concentration of the extract, the more hygroscopic the soap becomes, leading to an increase in moisture content. This could also be attributed to the method of preparation, where the soap was not dried properly after saponification. This means, longer curing of the produced soaps before determination of moisture content is recommended.

Fig. 2. Herbal soaps infused with different KaPaMa extract concentrations.

The cleansing properties of soap are influenced by its total alkaline content, which reveals the presence of alkaline compounds, like sodium or potassium hydroxide oxides, carbonates, or bicarbonates (Mwanza and Zombe, 2020). This content signifies the completion of saponification, the chemical reaction that takes place during soap-making when alkali reacts with fatty acids (Oyekunle *et al.*, 2021).

If the overall alkali level is higher, it may suggest saponification, resulting in a harsh and unpleasant soap. The KaPaMa soaps have a content ranging from 0.20% to 0.28%, which is lower compared to the control at 0.30%. Interestingly, as the concentration of extracts increases, the total alkali content decreases. This could be attributed to compounds in the extracts reacting with the soap component and leading to a lower overall alkali content.

According to the Bureau of Indian Standards (BIS), quality soaps should have more than 5% alkali content. Similarly, the International Organization for Standardization (ISO) specifies that soap should contain 2% alkali content (Mwanza and Zombe, 2020). Lower values indicate soap quality, which suggests that it will not cause skin corrosion.

Conclusion

In conclusion, the ethanolic extracts from KaPaMa exhibit potential in soap formulation. The evaluation of characteristics revealed that as the concentration of KaPaMa extract increased, the texture of the soap became rougher, and grittiness became more prominent. However, there were no changes observed in terms of phase separation, uniformity, stiffness, or greasiness. It was determined that the KaPaMa herbal soaps have a pH level and total alkali content, indicating they are not corrosive to the skin. The foaming index of the soap was lower compared to the control group, potentially due to the concentration of extracts used in its formulation. While the total fatty matter of the soap closely resembled that of the control group, its moisture content was higher, likely because of the nature of extracts used in production. Based on these findings, it is recommended to optimize KaPaMa soap formulations to enhance their foaming ability and reduce moisture content.

References

Arasaretnam S, Venujah K. 2019. Preparation of soaps by using different oil and analyze their properties. Natural Products Chemistry & Research **7,** 1.

Carvalho IT, Estevinho BN, Santos L. 2016. Application of microencapsulated essential oils in cosmetic and personal healthcare products – a review. International Journal of Cosmetic Science, **38(2),** 109-119. http://dx.doi.org/10.1111/ics.12232

Chirani MR, Kowsari E, Teymourian T, Ramakrishna S. 2021. Environmental impact of increased soap consumption during COVID-19 pandemic: Biodegradable soap production and sustainable packaging. The Science of the Total Environment **796,** 149013. http://dx.doi.org/10.1016/j.scitotenv.2021.149013

Dema N, Subba JR. 2022. Physiochemical Properties of the Commercial Herbal Soaps of Bhutan. The Research Journal of Sherubtse College **15,** 18-23.

Devipriya Nisha P, Nivetha L, Deepak K. 2021. Formulation, Development and Characterization of Herbal Soap Using *Borassus flabellifer and Curcuma zedoaria*. International Journal of Pharmaceutical Sciences Review and Research **69(2),** 134-139.

Eastin I. 2001. Some Considerations for the More Effective Production and Marketing of Non-Timber Forest Products in the Philippines. College, Los Baños, Laguna Philippines.

Firempong CK, Mak-Mensah EE. 2011. Chemical characteristics of toilet soap prepared from neem (*Azadirachtaindica A. Juss*) seed oil. Asian Journal of Plant Science and Research **1(4),** 1–7.

Guevarra B. 2005. A guidebook to phytochemical screening: phytochemical and biological. Manila: UST Publishing House.

Idoko O, Emmanuel SA, Salau AA, Obigwa PA. 2018. Quality assessment of some soaps sold in Nigeria. Nigerian Journal of Technology **37(4),** 1137-1140.

Kundu MK, Deb AT, Gupta SP. 1977. A simple rapid method for direct determination of total fatty matter in soaps **79,** 285-288.

Mendes BR, Shimabukuro DM, Uber M, Abagge KT. 2016. Critical assessment of the pH of children's soap. Journal of Pediatria. http://dx.doi.org/10.1016/j. jped.2015.08.009.

Muñoz MNM, Alvarado UG, Reyes JI, Watanabe K. 2021. Acute oral toxicity assessment of ethanolic extracts of *Antidesma bunius (L.)* Spreng fruits in mice. Toxicology Reports **8**, 1289-1299. http://dx.doi.org/10.1016/j.toxrep.2021.06.010

Mwanza C, Zombe K. 2020. Comparative Evaluation of Some Physicochemical Properties on Selected Commercially Available Soaps on the Zambian Market **7**, 1-13. http://dx.doi.org/10.4236/oalib.1106147.

Nadeeshani Dilhara Gamage DG, Dharmadasa RM, Abeysinghe DC, Saman Wijesekara RG, Prathapasinghe GA, Someya T. 2022. Global Perspective of Plant-Based Cosmetic Industry and Possible Contribution of Sri Lanka to the Development of Herbal Cosmetics. Evidence-Based Complementary and Alternative Medicine: ECAM, 2022. https://doi.org/10.1155/2022/9940548

Oyedele A, Akinkunmi EO, Doyinsola D, Orafidiya L. 2017. Physicochemical properties and antimicrobial activities of soap formulations containing *Senna alata* and *Eugenia uniflora* leaf preparations. Journal of Medicinal Plants Research **11,** 778-787. http://dx.doi.org/10.5897/JMPR2017.6515.

Oyekunle AO, Ore OT, Ogunjumelo OH, Akanni MS. 2021. Comparative chemical analysis of Indigenous Nigerian soaps with conventional ones. Heliyon **7(4).** https://doi.org/10.1016/j.heliyon.2021.e06689

Razal RA, Palijon AM. 2009. Non-Wood Forest Products of the Philippines. UPLB College of Forestry and Natural Resources, College, Laguna 4031. ISBN No. 978-971-579-058-1

Rajčević N, Bukvički D, Dodoš T, Marin PD. 2022. Interactions between Natural Products—A Review. Metabolites **12(12).** http://dx.doi.org/10.3390/metabo12121256

Roila A, Salmiah A, Razmah. 2001. Properties of sodium soap derived from palm based hydroxystearic acid. Journal of Oil Palm Research **13(2),** 33-38.

Vivian OP, Nathan O, Osano A, Mesopirr L, Omwoyo WN. 2014. Assessment of the physicochemical properties of selected commercial soaps manufactured and sold in Kenya. Open Journal of Applied Sciences **4,** 433-440.