Application of phytocosmetic formulations based on Coffea arabica leaves extract

Tiffany P. Sandoval1, Sully M. Cruz2, Mónica M. Ramos-Medina1,2, Silvia A. Pinales-Tobar1,2, Lorena Rochac2, Armando Cáceres1,2

1Facultad de Biología, Química y Farmacia, Universidad Galileo, Guatemala
2Facultad de Ciencias Químicas y Farmacia, Universidad de San Carlos de Guatemala, Guatemala

Abstract

Background: Coffee (Coffea arabica L.) leaf is well known for its cosmetic uses and skin benefits, for example its ability to help maintain natural skin’s humidity. The aim of this study was to design and evaluate the cosmetic effectiveness of the formulation of creams containing different concentrations of C. arabica leaves ethanol extract using hydrating, emollient raw materials and other excipients. Extracts from C. arabica seeds and Calahuala (Phlebodium decumanum (Willd.) J. Sm.) rhizome have demonstrated sun protection effect, so they are included in the formulation.

Materials and Methods: Several formulations were made in which the raw materials and the concentration of the extracts varied from 1 to 5%, and thus, achieve a formulation to evaluate its effectiveness in tests such as pH skin compatibility, sun protection factor, among others. For optimal sun protection effects, extracts from C. arabica leaves and P. decumanum rhizome were added.

Results: Results revealed that Formulation 2B, containing C. arabica leaves and C. arabica seeds, obtained a higher value in the UV protection Test, it also presented a better consistency, stability, less physical changes and an optimal pH value when compared to the other formulations.

Conclusions: Based on the test results, herbal formulations using raw materials are suitable for its dermatological use, especially for dry/sensitive skin types based on the pH values, providing solar protection among other benefits which are still under analysis.

Keywords: Cream, Phytocosmetic, Photoaging, Sun protection

Background

Many botanicals incorporated into skin care products are referred to as cosmeceuticals, a term first introduced by Albert Kligman two decades ago. Cosmeceuticals are defined as intermediary substances between drugs and cosmetics, and, as such, do not require the Food and Drug Administration (FDA) approval before being marketed. Before botanicals can be incorporated into a topical product, they must undergo numerous chemical processing steps, which greatly influence the outcome of the botanical extract to be formulated. Ultimately, the efficacy of the plant extract added to a topical preparation depends on its concentration. Sometimes the botanical is added in such small amounts that it provides more marketing than skin benefit.1

Coffee (Coffea arabica L.) leaf is well known for its cosmetic uses and skin benefits, beyond the classical use as a beverage. Coffee is a complex mixture of chemicals and contains substantial amounts of caffeine, chlorogenic acid, and other phenolic compounds. Chlorogenic acid, caffeic acid, and caffeine, which contribute to the potential of C. arabica to effectively prevent photoaging.2

Skin aging is a progressive process that can be divided into intrinsic and extrinsic aging. UV irradiation is the major cause of extrinsic aging; the one caused by UV irradiation is called photoaging. UV irradiation reduces the production of collagen, which is the major component of the dermis. Ultraviolet B (UVB) irradiation induces reactive oxygen species (ROS) production, which promotes downstream signal transduction in the dermis, causing skin damage and photoaging. In addition, bench works, and epidemiological studies have provided evidence supporting the use of certain botanical ingredients, such as coffee berries and leaves, and the findings regarding the proposed biological mechanisms have been applied in the cosmetics industry.3

Coffee is recognized as a symbol of identity for Guatemala. The varied landscapes provide for a wide range of microclimatic variation and altitudes that produce distinct coffees. The large plantations traditionally associated with coffee production produce, for the most part, Prime and Extra Prime coffees. Despite
the names, these relatively low-altitude varieties (grown at 2500–3500 feet above sea level), are at the low end of the coffee value chain. To the singularized high end, this coffee is grown at higher altitudes, the best above 4500 feet. Two main species of coffee are cultivated today, arabica (Coffea arabica L.) and robusta (Coffea canephora L.). Arabicas are marked by much wider range of flavors, grown at higher altitudes and usually under shade, the climatic and growing conditions produce deeper, more concentrated flavor, with notes ranging from dark berries and chocolate to citrus. Arabicas are further graded based on the altitude of production; higher-altitude coffees have more concentrated flavor and are considered of higher quality. All Guatemalan high-rise coffees develop a delicious aroma, pleasant acidity, a lot of body and delicate sweetness, a combination that produces an exquisite and balanced taste. In each of the eight regions these attributes create a special complexity. These are divided in Region 1: South west, Region 2: South-west/Central, Region 3: Central, Region 4: South east, Region 5: North west, Region 6: North, Region 7: North east.

A recent evaluation of antioxidant activity and chemical composition of ethanol extracts from the seven coffee regions of Guatemala, demonstrated diverse results which might depend on environmental conditions as well as coffee characteristics. The extract yields from leaves were above 28%; beans extract yields were lower (26.25%-29.83%) than leaves; fixed oil yields varied from 1.30%-4.18% and oleoresin from 1.31%-7.01% Extraction yields are quite promising in all leaf’s samples, particularly from regions VI (48.32%) and leaves from the same region demonstrated the highest amount of total phenolic compounds (TPC) (81.05 μg gallic acid/mg of extract), which correlated with antioxidant activity. The highest antioxidant activity [TPC, 1,1-diphenyl-2-picrylhydrazyl (DPPH) and 2,2’-azino-bis(3-ethylbenzothiazoline-6-sulphonic acid (ABTS)] values of leaves and beans were also from region VI. The chemical characterization (caffeine, by lead acetate method according to Yao et al; chlorogenic acid by standard method according to Núñez et al and total sugars by phenol-sulfuric method according to Núñez et al), showed the highest amount of caffeine in the leaves were from region VI (0.72%). For this reason, the region VI (Baja Verapaz) was chosen for this experimental research.

For optimal sun protection effect, extracts from C. arabica seeds and Plebodium decumanum (Willd.) J. Sm. Rhizome are considered suitable natural options to add to the formulation. The aim of this study is to design and evaluate the cosmetic effectiveness of the formulation of creams containing different quantities of C. arabica leaves ethanol extract using hydrating, emollient raw materials and other excipients. Extracts from C. arabica seeds and calahuala (P. decumanum) rhizome have demonstrated sun protection effect.

Materials and Methods

Vegetal Material

For the plant material and extraction, the organic farm Rincón Grande from Salama, Baja Verapaz (region VI), growing Caturra variety was selected; 1 kg of leaves and beans were collected at fruiting time, and dried in a forced-air convection oven at 40°C and ground. According to the highest total solid extraction, ethanol 50% was chosen for the preparation of extract. A tincture obtained by percolation was concentrated on a BÜCHI R-215 rota-evaporator and placed on a drier until humidity reached less than 5%.

Formulation

The different plant materials used were three; Baja Verapaz region C. arabica leaves extract – ethanol 50%, Baja Verapaz region C. arabica seeds extract – ethanol 50%, and P. decumanum rhizome fluid extract (1:1) (Figure 1).

Three material combinations search for the most stable and aesthetically pleasing appearance-formulation. Raw materials such as shea butter and beeswax can be used on the oily phase to give body to the final product. Both are helpful in keeping the skin hydrated. Beeswax is also a humectant, meaning it attracts water from the atmosphere to itself. Shea butter, on the other hand, is rich with skin cell building essential fatty acids, which in turn prevents

Figure 1. Formulation material options.
cellular water loss. Emulgade® is a non-ionic cream base for traditional O/W emulsions with high emollient flexibility and good electrolyte stability, which proved to be optimal for this research. Also, isopropyl myristate is a synthetic oil used as an emollient, thickening agent, or lubricant in beauty products. For the aqueous phase, demineralized water is added to all three options to give a proper O/W balance in the emulsion. Glycerin works as a humectant agent and, for Option 3, Carbopol is considered to create a gel-cream formulation. This to compare if the functional results are similar with Options 1 and 2.

In the thermolabile phase, C. arabica and P. decumanum extracts were positioned, so their chemical composition wouldn't be altered due to high temperature instability if it is added above 40°C. Tocopheryl acetate achieves two different functions, although technically not a preservative it is a powerful antioxidant that helps slow down oxidation in oils and therefore help prolong the life of the products; and it is also beneficial at reducing UV damage to skin. Collagen, fragrance, Kathon CG and triethanolamine were among the list of materials used for said phase.

Option 1 was made using beeswax and shea butter as the oily phase, glycerin and demineralized water as the aqueous phase and collagen, tocopheryl acetate, Fragrance and the extracts as the Thermolabile elements added at the end.

Option 2 was elaborated using beeswax, Emulgade®, isopropyl myristate and shea butter as the oily phase, glycerin and demineralized water as the aqueous phase and collagen, tocopheryl acetate, Kathon CG and the extracts were added at the end because of its thermolabile characteristics.

Option 3 was prepared with isopropylmystrate and shea butter as the oily phase, carbopol and demineralized water as the aqueous phase and collagen, tocopheryl acetate, Kathon CG, triethanolamine and the extracts we added at the end.

All three preparations followed the same procedure, were in a water bath, the inputs of the oil phase were placed inside a Beaker and heated until it reached a temperature of 70°C. Then, in another Beaker, the elements of the aqueous phase were heated in a water bath to reach 70°C. Both phases being at the same temperature, the Beaker was slowly added with the oily materials in the Beaker with the aqueous phase, stirring constantly. Stirring was continued until the mixture reached 40°C, where the thermolabile elements were added little by little.

Extract Combinations

Four combinations were added to each formulation, varying their total concentration from 1-5% of extract (Figure 2). Combination A included C. arabica leaves extract alone in concentrations 1%, 2.5% and 5%. Combination B had C. arabica leaves extract and C. arabica seed extract with a total concentration of 5%.

Combination C included C. arabica seeds extract and P. decumanum rhizome extract with 5% total concentration. Combination D mixed all three extracts (C. arabica leaves, C. arabica seed and P. decumanum rhizome) in a 5% total concentration.

pH Test

pH test was performed using a direct method, analyzing each formulation with a desk pH Meter HACH Sens ion-3.

UV Protection Test

The photoprotection afforded by topical sunscreens against solar UV radiation exposure can be determined in vivo or in vitro. In vitro methods involve the measurement of absorption or the transmission of UV radiation through sunscreen product films in quartz plates or biomembranes, and methods in which the absorption characteristics of the sunscreen agents are determined based on spectrophotometric analysis of dilute solutions. Mansur et al (2020) developed a very simple mathematical equation which substitutes the in vitro method proposed by Sayre, utilizing UV spectrophotometry and the following equation:

\[
SPF_{spectrophotometric} = \frac{\sum EE(\lambda) \times I(\lambda) \times Abs(\lambda)}{CF} = \frac{\sum EE(\lambda) \times I(\lambda)}{CF}
\]

where: EE (l) - erythemal effect spectrum; I (l) - solar intensity spectrum; Abs (l) - absorbance of sunscreen product; CF - correction factor (= 10). It was determined so that a standard sunscreen formulation containing 8% homovalvate presented a sun protection factor (SPF) value of 4, determined by UV spectrophotometry.

The values of EE x I are constants. They were determined by Sayre, and are showed in Table 1.

For sample preparation, 1.0 g of all samples was weighed, transferred to a 100 mL volumetric flask, diluted to volume with ethanol 50% for the first analysis and then with demineralized water for the second analysis, followed by ultrasonication for 5 minutes and then filtered through filter paper, rejecting the first 10 mL. A 2.0 mL aliquot was transferred to 50 mL volumetric flask and diluted to volume with ethanol on the first analysis and with demineralized water on the second analysis.

The UV Thermo Scientific GENESYS 10S
Formulation based on Coffea arabica leaves

spectrophotometer was programmed to read between 290-320 nm, with 5 nm intervals, using 1 cm quartz cell, and ethanol 50%/demineralized water as a blank. A reading was made at each point, followed by the application of Mansur equation. This test was done in triplicate.

Results and Discussion

Formulation

Option 1 did not maintain a homogeneous consistency when it reached room temperature, resulting in a separation of the oily and aqueous phase. The formulation was discarded.

Option 3 was made with the idea to create a cream-gel. Since it contained Shea butter, they were not compatible, resulting in a gel containing particles of grease. The formulation was discarded.

Option 2 resulted in a homogeneous soft, fluid cream. This formulation was used with the different combination of extracts, which results are shown below.

Physical appearance and pH results shown in Table 2, demonstrate that all formulations have a varying pH value from 5.20 in C. arabica leaves 1% as the lowest, to pH 7.06 in C. arabica leaves 5% as the highest value.

Table 2. Formulation of extracts combination physical results

<table>
<thead>
<tr>
<th>Combination</th>
<th>Formulation</th>
<th>Appearance</th>
<th>Smell</th>
<th>Color</th>
<th>pH</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>Leaves 1%</td>
<td>Semi-liquid, lotion-like consistency</td>
<td>Wax-like</td>
<td>Pine 203</td>
<td>5.20</td>
</tr>
<tr>
<td>A2</td>
<td>Leaves 2.5%</td>
<td>Thick, cream-like consistency</td>
<td>Wax-like</td>
<td>Birch 201</td>
<td>5.84</td>
</tr>
<tr>
<td>A3</td>
<td>Leaves 5%</td>
<td>Thick, bumpy, unstable</td>
<td>Wax-like</td>
<td>Cherrywood 103</td>
<td>7.06</td>
</tr>
<tr>
<td>B</td>
<td>Leaves + seeds (5%)</td>
<td>Soft, fluid, cream-like consistency</td>
<td>Wax-like</td>
<td>Dark Beach 158</td>
<td>5.92</td>
</tr>
<tr>
<td>C</td>
<td>Leaves + rhizome (5%)</td>
<td>Soft, fluid, cream-like consistency</td>
<td>Wax-like</td>
<td>Larch 159</td>
<td>6.11</td>
</tr>
<tr>
<td>D</td>
<td>Leaves + seeds + rhizome (5%)</td>
<td>Thick, cream-like</td>
<td>Wax-like</td>
<td>Dark Beach 158</td>
<td>6.03</td>
</tr>
</tbody>
</table>

formulation appearance seems to be soft and esthetically pleasing to the eye, very important in the cosmetic market.

Formulations with an extract concentration higher than 5% were unstable, since the aqueous phase separates from the oil phase after a couple of days.

The skin has a thin protective layer which is called the acid mantle. It is made up of sebum (free fatty acids) excreted from the skin sebaceous glands, which mixes with lactic and amino acids from sweat to create the skin's pH, which ideally should be slightly acidic – at about 5.5.\(^1\)

If the topical product's pH is more acidic than the skin, it is prone to redness and irritation. In the same way, if the pH of the product is more basic than the average skin pH value, it will produce dryness and even wrinkles.

The balanced pH level of the facial skin and most parts of the body is considered to be 5.5. That may vary depending on the skin type. In oily skin, the pH is between 4.0 and 5.2; normal skin - from 5.2 to 5.7; and dry skin - from 5.7 to 7.0.

All formulations pH level goes between 5.20–7.06, so they may be suitable for normal/dry skin types, since oily skin has a lower pH level.

UV Protection Test

The SPF is a quantitative measurement of the effectiveness of a sunscreen formulation. To be effective in preventing sunburn and other skin damage, a sunscreen product should have a wide range of absorbance between 290 and 320 nm. The \textit{in vitro} SPF is useful for screening test during product development, as a supplement of the \textit{in vivo} SPS measure.\(^18\)

As seen on Figure 3 there is a notable difference on the test results when using Ethanol 50% versus demineralized water. Demineralized water seems to give higher values on the extracts ethanol is a very polar molecule due to its hydroxyl (OH) group, with the high electronegativity of oxygen allowing hydrogen bonding to take place with other molecules. Attracting both polar and non-polar substances. And water is a universal solvent, more polar than Ethanol.

The FDA states that the SPF value is the minimal erythema dose for protected skin after application of 2 milligrams per square centimeter of the final formulation of the sunscreen product. Also, a moderate
sun protection product, should provide an SPF value of 12 to be acceptable.\textsuperscript{19} Since demineralized water gave the best results for the UV Protection Test, the formulation products were analyzed using this solvent, giving the results shown on Figure 4, where \textit{C. arabica} leaves + seeds (5%) and \textit{C. arabica} leaves + seeds + \textit{P. decumanum} rhizome (5%) formulations needed a minimal amount for every mg/cm\textsuperscript{3} to achieve this required SPF value (0.08 mg/cm\textsuperscript{3} and 0.14 mg/cm\textsuperscript{3} respectively), while the other formulations needed a greater amount to meet this standard.

\textit{Coffea arabica} leaves formulation proved to be concentration-related since the higher the amount of extract added to the product, the higher the SPF value, starting from 0.2, 0.6, and 0.7 at a 5% concentration.

Based on Table 3, \textit{C. arabica} leaves + seeds (5%) shows a better result when it comes to photo blocking UVB rays compared to other formulations, resulting in SPF 3.9 for every 0.02 mg/mL of product. While the lowest value was shown by \textit{C. arabica} Leaves (1%) formulation with a SPF 0.3 for every 0.004 mg/mL of product.

The chosen formulation was 2B since it obtained an ideal consistency and, as the weeks passed, its components did not separate. pH levels are suitable for dry skin types, since it has a pH 5.92. Also, the combination of \textit{C. arabica} leaves ethanol extract + \textit{C. arabica} seed ethanol extract provides a higher SPF than the other combinations, with a result of 3.9/0.02 mg/mL, followed by \textit{C. arabica} leaves ethanol extract + \textit{C. arabica} seed ethanol extract + \textit{P. decumanum} rhizome extract combination with a result of 2.1/0.02 mg/mL.

Among the rest of the materials used, vitamin E has been demonstrated to be correlated with increased oxidative stress and cell injury. The photoprotective effects of topically applied a-tocopherol have been shown to be a reduction in UV-B induced damage and an inhibition of photo carcinogenesis.\textsuperscript{20,21} Furthermore, oral and topical vitamin E supplementation have been shown to diminish the effects of photoaging, inhibit the development of skin cancer, and counteract immunosuppression induced by UV radiation in animal models.\textsuperscript{11}

These natural bioactive components have the ability to absorb the UV radiations and protect the skin from adverse effects. They also neutralize the free radicals produced in the skin due to UV radiation exposure. The mechanism of action of the natural bioactive components is either through scavenging or chelating the harmful free radicals generated during radiation exposure. DNA damage due
to UV radiation in plants induces the accumulation of UV ray absorbing secondary metabolic products such as flavonoids and other phenolic compounds in plants. These flavonoids and phenolic compounds show outstanding antioxidant and photoprotective properties which can be used as a safe, cost-effective and biologically effective ingredient in sunscreen preparations. Prevention of absorption of UV radiation is a more effective way of controlling skin cancer where naturally occurring agents are considered as less toxic for this purpose.

**Conclusions**

When formulating, it is important to choose an emulsifier suitable for the type of mixture to be made, in this case for an O/W cream. The emulsifier used for this formulation is a non-ionic emulsifier for traditional O/W emulsions with high emollient flexibility and good electrolyte stability. It is important to balance the skin’s pH in order to reduce irritation and not strip the skin of its natural oils.

The wax aroma can be masked using fragrances, without altering the test results. To match the earth pallet colors obtained on the products due to the extract color, it would be advisable to use coffee-like aroma for consumers to associate the color, ingredients and scent with the popular beverage and its skin benefits. Based on the materials used for the formulations and their pH levels, these products can be used on dry/normal/sensible types of skin.

The *C. arabica* leaves ethanol extract formulation alone has proven to be effective against UV radiation, but combined with *C. arabica* seeds ethanol extract boosts this effect, needing less amount of product to achieve SPF 15, which is a higher value than FDA minimum standard.

In order to formulate products with a high UV protection, it is important to evaluate the physicochemical interactions between the extracts and the vehicles, like emollients, emulsifiers, anti-oxidant and oily components; since they can interact and it may affect the product in a negative or positive way.

More tests are being done to see if the formulations could have other benefits, such as antioxidant power and hydration.

**Acknowledgments**

The authors thank the Natural Products Research Laboratory (Lipronat) for letting them use the installations to do the correspondent laboratory analysis throughout the investigation. We also thank the General Directorate of Research (DIGI) of USAC for financial support (grant No. 4.8.63.4.13 of 2018).

**Conflict of Interests**

None.

**References**


