RESEARCH ARTICLE

SYNTHESIS OF SUNSCREEN CONTAINING OCTYL SALICYLATE AND ZINC OXIDE – COMPARISON OF SUN PROTECTION FACTOR

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Abstract
With global warming on the rise and depletion of ozone layer, the wrath of the only star in our solar system is on its epitome. The main objective of this project was to develop a non-toxic and nourishing UV sunscreen with the incorporation of organic and inorganic filters and natural ingredients which protects the skin from carcinogenic UVA and UVB radiation and at the same time exfoliating and nourishing the skin. The formulation that was investigated: Octyl Salicylate with Zinc Oxide. The active ingredients were dispersed, that is Octyl Salicylate and Zinc Oxide, into a medium. That medium was a water-oil cream, on which the active ingredients were dispersed. The cream was formulated using hydrosol(80%) - rose water, humectant(4%) - honey, liquid oil(10%) - virgin coconut oil, emulsifier(4%) - Glyceral Stearate, thickener (2%) - Proplyene Glycol. The UV absorption of the cream was measured using UV-Visible Spectrophotometer, while the SPF of the formulated sunscreen was measured using a linear regression model obtained between maximum absorption in the UVB region and the SPF value of the commercial sunscreen available in the market. The cream showed broad spectrum absorption especially in the UVB region. Comparison of UV spectra indicates that sunscreen synthesized in this study has superior UV protection than those of commercial samples.

Introduction:
Lifestyle changes during the past five decades, with an increase in exposure to sunlight because of outdoor activities and worsening sunbathing habits often result in skin cancers. As a result of chronic UV exposure, skin aging, “wrinkles, uneven skin pigmentation, loss of skin elasticity and a disturbance of skin barrier functions” result. These “changes in the skin that superimpose the alterations of chronological aging” refer to photo aging. [1-5]

A variety of environmental stresses, particularly UV light, can damage sun-exposed areas of the skin, such as the face and neck, and accelerate premature aging. The skin Immunosuppression and skin cancer genesis are widely studied as the main toxic effects of ultraviolet radiation (UVR) on adult skin.[3] UV radiations comprise of 100-400nm wavelength, and can be further divided into UVC (100-290nm), UVB (290-320nm) and UVA (320-400nm). The shorter the wavelength, the more harmful the UV radiation. However, shorter wavelength UV radiation is less able to penetrate the skin. Short-wavelength UVC is the most damaging type of UV radiation. However, it is completely filtered by the atmosphere and does not reach the earth's surface. [6-12]
UVA wavelength is the longest of the three at 320–400 nm. UVA rays account for up to 95% of the UV radiation reaching the Earth’s surface. They pass through the ozone layer and reach the earth. They are present with relatively equal intensity during all daylight hours throughout the year. UVB wavelength is 290-320 nm. UVB rays are intensity varies by season, location, and time of day. UVB rays, the chief cause of skin reddening and sunburn, tends to damage the skin’s more superficial epidermal layers. They can also lead to the development of skin cancers. It plays a contributory role in tanning and photoaging. UVB rays are responsible for causing most skin cancers. UVC wavelength is 200-290 nm that have the shortest wavelength.\[16-20\] They are the most dangerous among all the rays. However, these rays do not reach the earth’s surface as they are completely absorbed by the ozone layer. It is therefore, not considered to be a factor in solar exposure to human beings and thus is not of any relevance genetically and does not much attention as far as sunscreen are concerned. Octyl Salicylate is most effective as a UV-B (290–320nm) filter. Ana Flo Sierra [21] et. al. showed that formulations containing octyl salicylate for topical administration would be a promising formulation with good in vitro, ex vivo and in vivo pharmaceutical results for prevent the undesirable adverse effects of UV skin irradiation because melatonin not only acts as a potent antioxidant itself, but also is capable of activating an endogenous enzymatic protective system against oxidative stress. Sunscreen, a part of photoprotection strategy, also known as sunblock is a lotion, spray, gel, foam, stick or other topical product that absorbs or reflects some of the sun's ultraviolet radiation and thus helps protect against sunburn. The use of sunscreen products has been advocated by many health care practitioners as a means to reduce skin damage produced by ultraviolet radiation (UVR) from sunlight.[22-26] It has become an indispensable part of our everyday lives as diligent use of sunscreen can also slow or temporarily prevent the development of wrinkles, dark spots and sagging skin. It is a chemical defence, penetrating the skin and absorbing the UV rays before they reach and damage the dermal layers.[10-12] Since the skin is the largest organ of the human body, the importance of maintaining homeostasis and protecting the skin from ultraviolet radiation is important. Imbalances can result in wrinkles, hair loss, blisters, rashes, life-threatening cancers, and disorders in immune regulation.

The active ingredients of sunscreen are organic and inorganic UV filters. Organic filters absorb whereas inorganic filters reflect and scatter the UVB radiations.[27] Sunscreen formulations with broad protection spectrum unite both organic and inorganic UV filters. In this study, sunscreen containing octyl salicylate as organic UV filter and zinc oxide as inorganic filter is synthesized and compared with three commercial sunscreen of known SPF values (SPF=20, 30 and 40). Octyl Salicylate is an ester formed by the condensation of a salicylic acid with 2-ethylhexanol. Zinc oxide is non-toxic, chemically inert and biocompatible.

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**Fig. I:** UV rays wavelength spectrum.
Materials and Methods:

Materials:
The chemicals used in this study are: Octyl Salicylate, Zinc Oxide, Coconut oil, Glycerol stearate, Propylene glycol, Isopropyl alcohol, Honey and Double distilled water. Three samples of commercial sunscreen having SPF-20, SPF-30 and SPF-40 are procured from the market for comparison. All the components used in the preparation of sunscreen are recognized as safe as per FDA regulations.

Octyl Salicylate is procured from Sisco Research Laboratory having more than 99% purity. It is a colourless oily liquid with a slight floral odour. Extra pure zinc oxide (purity 99.9%) with particle size ranging 10-60nm was used. 100% pure organic virgin coconut oil, cold pressed, centrifuge extracted, unrefined and chemical free having 82% of saturated fatty acid with smoke point(175°C) is used. Laboratory grade (minimum purity 99%) propylene glycol and glyceryl stearate are used. Honey (100% pure, Dabur India make) do not include high fructose corn syrup, sugar, jaggery or added preservatives. Double distilled water (using glass apparatus) of resistivity 18MΩ-cm is used in this study.

Treatment of zinc oxide powder:
To maintain uniformity, increase surface area and reduce the size of the particles, sample of zinc oxide is subjected to high energy ball milling using a planetary ball mill. 5g powder is placed in a teflon container along with 25mL isopropanol. Hard 10 mm dia zirconia balls were used. The milling is performed for 24h. Powder is recovered by evaporating solvent at ~110°C.

Preparation of Octyl Salicylate solution:
A 50 mg ± 1 mg sample of Octyl salicylate was weighed accurately into a 100-ml volumetric flask and then diluted to the mark with solvent. The resulting stock solution was then diluted 1:100 to yield a final sunscreen concentration of 5 mg/L.

Preparation of sunscreen:
The sunscreen is prepared by the emulsification of an oil phase and an aqueous phase. The oil phase consists of coconut oil (10%), glyceryl stearate (4%) and propylene glycol(2%) while water phase contains double distilled water(80%) and honey(4%). Both oil and aqueous phases are heated separately in a water bath at 75°C for 15 minutes. The aqueous phase is gradually added to the oil phase with constant stirring and the temperature is maintained at 75°C for the effective homogenization. The mixture is continuously stirred until it was congealed at the room temperature. The octyl salicylate (0.5%) and zinc oxide (0.5%) powders were then added immediately to the emulsion, before it became cold; the emulsion was stirred during the whole process to ensure a uniform mixing of all components. The emulsions were then cooled down at room temperature. Sunscreen with octyl salicylate and zinc oxide powder and a cream with no powders are also prepared. The commercial sunscreens are designated as SPF-20, SPF-30 and SPF-40 having SPF values 20, 30, and 40 respectively. The sunscreen prepared in this study is designated as Sun Block.

UV Spectra:
The properties of sunscreen are assessed using sunscreen Protection Factor. 0.05g of the synthesized sunscreen (Sun Block) and 3 commercial samples are weighed and transferred to 50ml stoppered flask and dissolved in 20mL isopropyl alcohol. UV absorption spectra of Sun Block, SPF-20, SPF-30 and SPF-40 sunscreens are recorded using Perkin Elmer Lambda 365 UV-Visible spectrophotometer. Four trials were carried out for each sunscreen, in which absorbance of the solution was scanned from 190-400nm. SPF values are calculated using absorbance of the sunscreen at λ ranging between 290-320nm at an interval of 5nm. The absorption by sunscreen is directly related to the concentration of the solution, the maximum absorbance between 290-320nm was noted and it was corrected to the same mass of 0.05g. The corrected absorbance was calculated by the following formula: Ac= measured A x (0.05g/ mass of sample). Uniformity of concentration is maintained in the solutions.

Results and Discussions:
UV- absorption spectra of different sunscreens are presented Figure II. From the spectra, it is evident that absorbance characteristics of the sunscreen based on octyl salicylate and zinc oxide, synthesized in this study (designated as-Sun Block) is superior to the commercial sunscreens SPF-20, SPF-30 and SPF-40 procured from the open market.
Ana Flo Sierra et. al. had recommended the use of octyl salicylate to protect the skin from sun damage. SPF stands for Sun Protection Factor, is a scientific measure and gives an idea of how much lower the risk of skin damage is due to the use of sunscreen. Sunscreens are commonly rated and labelled with a sun protection factor (SPF) that measures the fraction of sunburn producing UV rays that reach the skin. The SPF values are calculated using Mansur mathematical equation\[28\]

$$\text{SPF} = \text{CF} \times \sum \text{EE}(\lambda) \cdot I(\lambda) \cdot \text{Abs}(\lambda)$$

Where, \(\text{EE}(\lambda)\): Erythemal effect spectrum; \(I(\lambda)\): Solar intensity spectrum; \(\text{Abs}(\lambda)\): Absorbance of sunscreen; \(\text{CF}\): Correction factor (=10). The normalized value of \(\text{EE}(\lambda) \cdot I(\lambda) \cdot \text{Abs}(\lambda)\) used \[29-30\] in the calculation of SPF are: \(\lambda_{290}=(0.0150); \lambda_{295}=(0.0817); \lambda_{300}=(0.2874); \lambda_{305}=(0.3278); \lambda_{310}=(0.1864); \lambda_{315}=(0.0839); \lambda_{320}=(0.0180).\) The calculated SPF values are shown in Table 1.

### Table 1: Sun protection factor (SPF) of sunscreens

<table>
<thead>
<tr>
<th>Sample</th>
<th>SPF Calculated</th>
<th>SPF</th>
</tr>
</thead>
<tbody>
<tr>
<td>SPF-20</td>
<td>19.7</td>
<td>20</td>
</tr>
<tr>
<td>SPF-30</td>
<td>30.7</td>
<td>30</td>
</tr>
<tr>
<td>SPF-40</td>
<td>40.7</td>
<td>40</td>
</tr>
<tr>
<td>Sun Block</td>
<td>45.9</td>
<td>--</td>
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</tbody>
</table>

Chou et al\[31\] plotted corrected absorbance at \(\lambda_{\text{max}}\) of several sunscreens from the same brand vs. the SPF values and established a linear relationship of the type,

$$A = 0.0272 S + 0.1146$$

Where \(A\) is corrected absorbance at \(\lambda_{\text{max}}\) and \(S\) is SPF value of sunscreens. The \(A\) for Sun Block is found 1.375. Substituting the value of \(A\) in the equation, the SPF of Sun Block is 46.33. Since there is a direct relation between the corrected absorbance of sunscreen in the UVB region and the SPF of the sunscreen, the corrected absorbance of SPF-20, SPF-30 and SPF-40 is plotted against their SPF values and the same plot of Chou et al.[31] are shown in Figure III.
We also obtained linear relation between absorbance and SPF values,

\[ A = 0.025 S + 0.2228 \]

Substituting the value of \( A \) for Sun Block, its SPF value is 46.088 which is almost similar as obtained from the curve of Chou et al[31].

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