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Evaluation of carboxymethyl xyloglucan as SPF booster in Oxybenzone cream

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Abstract
The increasing consumer awareness on the risk of sun exposure related diseases like skin cancer, skin aging has lead to development of various sunscreen products. The efficiency of the sunscreen products depends on the sun protection factor (SPF) value. Use of multiple sunscreens in formulation leads to penetration of these in systemic circulation leading to the toxicity. Ingredients called SPF boosters are reported to enhance the efficacy of sunscreen in the formulation. Carboxymethylxyloglucan (CMXG) is a derivative of natural polysaccharide from tamarind seed. Present work describes formulation and SPF determination of benzophenone O/W sunscreen cream containing CMXG in different concentrations (0.5% w/w, 1.75%w/w and 3%w/w). The formulations show increase in viscosity as well as SPF with increase in CMXG concentration. Hence, it can be concluded that the CMXG can act as both viscosity and SPF enhancer in sunscreen cream.

Keywords: Sun protection factor (SPF), Carboxy methyl xyloglucan, Sunscreen

Introduction
Sunlight is composed of various wavelengths ranging from ultraviolet light through infrared to visible light. Exposure to solar radiation is recognized to have harmful effects on the human skin. Amongst all, ultraviolet light is the most harmful to the skin and causes sunburns, skin ageing and over the long term, skin cancer [1] The distinguished major bands of UV spectrum are UVA (400-320 nm) UVB (320-290 nm) and UVC (290-200 nm)[2,3]. Regular application of sunscreen may help to prevent the harmful effects of ultraviolet radiation to some extent.
Oxybenzone is one of the most common chemicals found in commercial chemical sunscreens. It provides broad-spectrum UV coverage. Being effective against UVA and UVB radiation, it works by absorbing UV radiation and dispelling it as heat.
The efficacy of a sunscreen is usually expressed by the sun protection factor (SPF), which is expressed as ratio of UV energy required for producing a minimal erythemal dose (MED) on protected skin, divided by the UV energy required to produce a MED on unprotected skin.
The minimal erythemal dose (MED) is the lowest time interval or dose of UV light irradiation sufficient to produce a minimal, perceptible erythema on unprotected skin [4, 5]. The higher the SPF, the more effective is the product in preventing Sunburn.

$$SPF = \frac{MinimalerytemaldoseMEDofprotectedskin}{MEDofunprotectedskin}$$

A sunscreen with an SPF of 15, blocks about 93% of UVB radiation, while one with an SPF of 30 blocks about 97% of UVB radiation. This difference of 4% may make the difference between an aesthetically pleasing sunscreen and an undesirable one, as products with higher SPF generally tend to be uncomfortable due to the higher concentration of the active ingredient [6, 7]. Apart from the sunscreen active, a class called “SPF Boosters” also significantly affects the SPF of sunscreens. These SPF boosters as the name implies greatly elevate the Sun Protection Factor. Some of the commonly used SPF boosters are surfactants, stabilizers and film formers [8, 9].

Film formersare important SPF boosters & are inevitably used in all the sunscreen formulations [10,11]. Synthetic film formers such as octocrylene are important for imparting body and stability to product. Mostly film formers are cellulose derivatives like hydroxy propyl methyl cellulose, hydroxy propyl cellulose, methyl cellulose, etc. Some natural polysaccharides are also used as film formers viz xanthan gum, guar gum. These polysaccharides swell and thicken in contact with water, due to which product remains in contact with the skin for a longer period.

The present study deals with incorporation of carboxy methyl xyloglucan (CMXG) as SPF booster in which is a carboxy methyl derivative of natural polysaccharide tamarind seed xyloglucan (CMXG) has carbonyl groups which are considered to be an essential molecular requirement for a compound to absorb UV radiation. This derivative has two benefits when incorporated in a sunscreen formulation. Firstly, it has certain absorbance in the UV region by virtue of which it contributes to numerical elevation of the SPF. Secondly, it works on the principle of viscosity enhancement which ensures better product contact & hence greater effectiveness of the product [12,13]

The aim of this work is to evaluate effect of CMXG and its concentration on SPF of oxybenzone creams.

Materials

Carboxy methyl xyloglucan was obtained from Encore Natural Polymers. Pvt. Ltd, Ahmedabad, India, Captex 200 was gifted by Abitech corporation Ltd., all other chemicals were obtained from local sources.

Methods

Preparation of creams: three oxybenzone was formulated in a vanishing cream formula varying the content of CMXG (F1, F2 and F3) given in table 1.

Table 1: Formula for oxybenzone creams

Sr. No.	Ingredient	F1	F2	F3
1	Oxybenzone	5	5	5
2	Captex 200	25	25	25
3	Stearic acid	15	15	15
4	CMXG	0.5	1.75	3
5	Methyl paraben	0.025	0.025	0.025
6	Propyl paraben	0.015	0.015	0.015
7	Glycerine	12.5	12.5	12.5
8	Perfume	Qs	Qs	Qs
9	Water	q.s. 100	q.s. 100	q.s. 100

Oxybenzone, captex and stearic acid were dissolved together and heated to 70 °C, the parabens, Span: Tween blend and glycerine was dissolved separately and heated to 70° C. The mixtures were mixed under mechanical stirring and perfume was added during cooling.

Evaluation of prepared creams

pH determination

pH of 1% dispersion of the cream was determined by using pH meter (Deluxe pH meter). The pH meter was calibrated before each use with standard pH 4,7 and 10 buffer solutions. 1% dispersion of the formulation was made in distilled water and pH was measured.

Determination of spreadability [14]

The spreadability of the formulation was determined using a fabricated apparatus as described in the literature. The apparatus (fig 1) consisted of two glass slides (7.5 × 2.5 cm), one of which was fixed onto the wooden board and the other was movable, tied to a thread which passed over a pulley, carrying a weight. One g of formulation was placed between the two glass slides. 100 g weight was allowed to rest on the upper slide for 1 to 2 minutes to expel the entrapped air between the slides and to provide a uniform film of the formulation. The weight was removed and the top slide was subjected to a pull obtained by attaching 30 g weight over the pulley. The time required for moving slide to travel premarked 6.5 cm distance was noted. The readings obtained were indications of relative spreadability of different formulations.

$$S = \frac{M * L}{T}$$

Where, M = wt. tied to upper slide L = length of glass slides T = time taken to separate the slides.

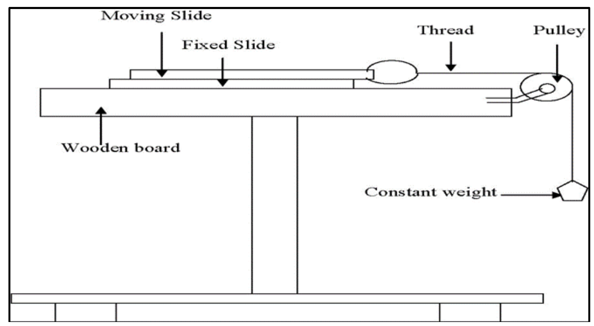

Fig 1: Spreadability apparatus

Determination of viscosity [15]

Brookfield digital viscometer (RVDV Pro plus), equipped with a T-Bar spindle was used to determine viscosity (cp) of the formulations. The viscosity was measured at 10 rpm after 30 seconds. Measurements were performed at ambient temperature and in triplicate.

Determination of globule size

Estimation of globule size was performed using a trinocular microscope. Initial calibration using occulometer & stage micrometer. Formulation was mixed with water to produce 1% dispersion, smeared on the slide & observed under the trinocular microscope to estimate globule size.

In vitro testing of SPF [16, 17, 18, 19]

SPF of the formulation was evaluated spectrophotometrically. The method estimated the ultraviolet absorption by a uniform thin film (about 8.8 mg) applied on one side of quartz cuvette, a clean cuvette served as a control [20, 21] Absorbance was recorded between 290-320 nm & SPF was estimated using the Mansur equation:

Sun protecting factor (SPF) = $CF * \sum^{320} EE\ (\lambda)\ x\ I(\lambda)\ x\ Abs(\lambda)$

Where,

EE (I)- erythemal effect spectrum;

$SPF = CF \sum 320\ EE\ (\lambda) * I(\lambda) * Abs(\lambda)$ I

Where (I) -solar intensity spectrum,
Abs-Absorbance of sunscreen product;
CF-correction factor.
The value of EE x I are constant.

Result and discussion

Preparation of creams

Oxybenzone was found to have highest solubility in Captex 200 (182 mg/ml) and least solubility in liquid paraffin (43 mg/ml). Captex 200 has high solubilization capacity owing to its low molecular volume (417.62 w/v) & natural surfactant enhancer activity. The presence of hydroxyl groups in captex 200 is another reason for good solubility of oxybenzone. In surfactants oxybenzone had higher solubility in span 80 (45 mg/ml) in comparison to span 20 (23 mg/ml) that can be attributed to the lower HLB of former (4.3) indicating the hydrophobic character. Similarly tween 80 could dissolve more oxybenzone (15 mg/ml) than in tween 20 (3 mg/ml) hence pair of span 80 and tween 80 was decided to form the cream [22, 23]. The span and tween 80 was used in a ratio of 1:3 as it could provide the required HLB (12.5) of captex 200 [24, 25]. CMX was included as an SPF booster in creams in different concentrations viz 0.5, 1.15, and 3.0%

Table 2: Evaluation of prepared sunscreen creams

Formulation/Parameter	F1	F2	F3
pH	6.8	6.5	6.37
Spreadability (unit)	43.85	15.75	8.25
Viscosity (cps)	46,310	54,936	1,67,857
Globule size (mic)	73.78	57.93	53.03
SPF	20.92	33.36	40.91

Evaluation of prepared creams: The evaluation parameters for the prepared sunscreen creams is summarized in table 2

Determination of pH

The pH of the emulsion was in the range of 6.0-7.0 which may be attributed to the acidic nature of CMXG. pH of the skin 5.5-6.5. Hence, this pH was found to be acceptable.

Spreadability

Spreadability ranged between 6.31 to 45.34. CMXG had much higher retarding effect than the surfactant bled proportion. The surfactant due to decrease in globule size may have increased viscosity and thereby reduction in spredability [26]

Mean globular diameter

The volume and size of internal phase has lot of impact on stability and viscosity of cream. The software use for globule size determination, Analyzer "ipv PSA" from image Provision Technology. Globule size was recorded over 25 fields which increase precision and minimized incident of error. For Oxybenzone cream, the effect of surfactant blend concentration on decrease in globule size was higher compare to CMXG [27] it form film around globules.

Viscosity

Viscosity is resistance to flow. As concentration of CMXG increases viscosity also increases and lower the thixotrophy higher the spf value.

SPF

Spectrophotometric method was used for estimation of SPF which involved cream application on glass side of cuvette. SPF increased chiefly as a function of CMXG. For any material to function as a sunscreen, certain molecular attributes are a pre-requisite. Common functional group being carbonyl group, aromatic group and conjugation [28]. Carboxy methyl xyloglucan a semisynthetic derivative of xyloglucan, is bio-compatible and possesses carbonyl groups which are an essential requirement for radiation absorption (Fig.2) [29]. Hence, it was incorporated as an SPF booster and increase in CMXG concentration led to substitutional elevation in *in vitro* SPF.

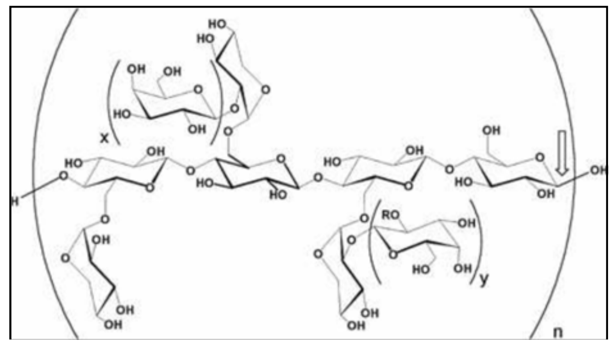

Fig 2: structure of carboxy methyl xyloglucan

Statistical Analysis of data

Statistical analysis of the evaluation test data was done by using one way anova followed by Dunnett test. The results were found to be significant.

Conclusion

Most of the sunscreens comprise of multiple sunscreen actives to claim a numerically superior SPF. It not only virtue of the active alone, but also due to its excipients. A class of excipients known as 'SPF booster' substantially elevates the SPF in addition to the actives. In addition to these, modification of inherent formulation parameters may also elevate the SPF.

The aim of this dissection was to enhance the SPF of mono-active sunscreen formulations by incorporating a semi-synthetic SPF booster. Base on evaluation parameters, it was concluded that the SPF of mono- active sunscreen formulation could be enhanced by simple incorporation of an SPF booster which not only provided a numerically superior SPF but also had lesser side effect due to its semi-synthetic nature and biocompatibility. Similarly, modifying surfactant blend proportion in creams helped to decrease globule diameter which in turn, increased surface area and this led to better absorption of UV radiation with the same concentration of the active. Incorporating CMXG in the gel formulation not only contributed to the sunscreen activity but also decreased the concentration of carbopol required for gelation without compromising its spredability or aesthetic appeal.

Thus, SPF of a sunscreen formulation need not be elevated only by increasing only by increasing the number and/or concentration of sunscreen actives, but can also be enhanced by modifying formulation aspects of dosage form.

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