INTERNATIONAL RESEARCH JOURNAL OF PHARMACY
www.irjponline.com
ISSN 2230 – 8407

Research Article

FORMULATION AND EVALUATION OF BLACK SESAME SEED OIL SUNSCREEN EMULGEL USING NATURAL GELLING AGENT

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Article Received on: 16/06/18 Approved for publication: 30/06/18

DOI: 10.7897/2230-8407.096116

ABSTRACT

Herbal molecules are having more potent action and less side effect but because of low bioavailability, high molecular weight, and low lipophilicity they are not considered ideal dosage forms. To overcome this drawback, emulgel formulation has been considered by the formulator. Hence the present investigation focused on formulation of emulgel of sesame oil with fenugreek mucilage as natural gelling agent. These formulation were evaluated for Physical appearance, Measurement of pH, Spreadability, Extrudability study, Rheological study, Determination of In-vitro antioxidant activity, SPF value determination, drug content etc. All the emulgel formulations were white to buff white viscous creamy preparation with a smooth homogeneous consistency and glossy appearance and found to be having good protection against UV-B radiation and SPF value was determined for the formulation F 2 as it has shown good drug content in the range of 95 to 98%.

KEYWORDS: Spreadability, Extrudability, Rheological, Antioxidant.

INTRODUCTION

Plant materials having antioxidant and skin anti-aging activities have been commonly used in cosmetic products for many years. Herbal molecules are known for their potent action and less side effects, but they have demerits like low bioavailability, high molecular weight, and low lipophilicity. To overcome such drawbacks, novel drug delivery system is emerging era of research. One of the such formulation is emulgel.[1] The main aim of utilizing topical delivery system is to overcome first pass metabolism and to avoid risk and inconvenience of I/V therapy and of different conditions of absorption, presence of enzymes, p^H changes, and gastric emptying time.[2] Skin is made up of both hydrophobic intercellular materials within the hydrophobic cornified cells. Each year, more than one million people are diagnosed with skin cancer and near about 10,000 dies from malignant melanoma. Many skin cancers occur on the areas that are most commonly exposed to the sun, such as the face, neck. Ultraviolet exposure is involved in the pathogenesis of skin cancers, causes premature aging of the skin and photo-immunosuppresion. It also plays an important role in the pathogenesis of photosensitive disorders such as polymorphous light chronic actinic dermatitis, eruption, actinic prurigo and phototoxic drug reactions. Both UVB and UVA radiation affect the skin. Therefore, focus has been given to the dangerous effects of the sunrays to avoid harmful skin effects. The use of sunscreen preparations became certainly necessary. Effectiveness of sunscreen is the ability to guard the skin against ultraviolet induced burning which can be determined by the sun protection factor (SPF)[3].

Many commonly used topical agents like ointment, cream, lotion have many demerits. They are very sticky causing discomfort to the patient as applied. Moreover, they also have lesser spreading coefficient and necessary to apply with rubbing which may produce severe dermatitis and also, they exhibit the problem of stability.[4] Due to these reasons within major group of semisolid formulations, use of transparent gels has emerged both in cosmetics and pharmaceutical formulations. Nevertheless, of many benefits of gels a colloidal system shows major shortfalls in delivery of hydrophobic drugs. Therefore, to conquer this limitation, an emulgel formulation is being used so that even a hydrophobic therapeutic moiety can be successfully incorporated through gels. Gels and emulsions in combined form are referred as emulgel.[5] In fact, the incorporation of a gelling agent in the aqueous phase converts a classical emulsion into an emulgel[6]. Emulgel have several properties such as being thixotropic, easily spreadable, easily erasable, emollient, non-greasy, nonstaining and pleasing appearance[7]. Synthetic sunscreen formulation has not been accepted safe for prolonged use[8]. Seeds of sesame have high level of furofuran lignans such as sesamin, sesamolin, phytosterol, Vitamin A, Vitamin E, Vitamin D, Vitamin C, Vitamin E and Vitamin K, triglycerides of single unsaturated oleic acid and double unsaturated linoleic acid. Sesame oil has good shelf life. It is reported to possess antioxidant, antibacterial, anti-inflammatory, hypolipidemic, wound healing and sunscreen properties.[11]. *Sesamum indicum* seed oil used in the present study as it consists of fatty acids like palmitic, oleic, linoleic and stearic acids which are emollients so help to moisturize the skin to keep it smooth and soft. It also contains vitamins A, D, C, E and K which acts as antioxidants. It also composed of minerals like magnesium, zinc, potassium. Black Sesame oil is almost unique in its ability to penetrate all layers of the skin with nutrients. It is reported to have sunscreen activity. The objective of present study is to form emulgel formulation of black sesame oil to overcome limitation of other topical formulations and using natural gelling agent to overcome limitations of synthetic gelling agents like toxicity and irritating properties.

The mucilages possess various pharmaceutical and cosmetic applications. Mucilage of seeds has been used as binding agent, gelling, emulsifying, granulating, and suspending agent due to its

nontoxicity, low cost, easy availability, emollient and non-irritating properties[10]. Fenugreek (*Trigonella foenum graecum*). seeds have numerous cosmetic and medicinal uses like hypoglycemic, diuretic, gastroprotrective, anti-urolithiatic anti-inflammatory agent as well as antioxidant. It contains 25-30% protein and mucilage mainly composed of soluble galactomannan polysaccharides[11].

Skin constitutes of the major targets for ageing as it is exposed to external oxidant and free radical through UV light, oxygen, chemical pollutants etc. Biological activity study of sunscreen emulgel formulation as antioxidant activity was performed. Sunscreen formula is invented in present study in order to augment sunburn like conditions which is having great potential to quench free radical.

MATERIALS AND METHODS

Sesamum indicum Seeds were collected from local market. Propylene glycol, liquid paraffin, tween 20, span 20 and methyl paraben were purchased from Research Lab Mumbai. All chemicals were used are of analytical grade.

Extraction of *Sesamum indicum* seed oil

Dried powdered material of seed of *Sesamum indicum* (50 g) was extracted with 50 volumes of petroleum ether (40-60°C) using a soxhlet apparatus. This process of extraction was continued for 6 hrs. The petroleum ether was distilled out then concentrated by hot plate drying and air-drying at temperature of 40 ± 2°C.

Extraction of fenugreek mucilage

The mucilage of fenugreek seeds was isolated with following well known methods. 20 gm of fenugreek seeds were placed in 200 ml distilled water and boiled with stirring up to slurry formation and kept it to cool for 3 to 4 hrs to separate supernatant liquid. The clear upper solution was decanted and centrifuged at 500 rpm for 20 minutes. The supernatant was separated and concentrated at 60ºC on water bath. The solution was cooled at room temperature and was poured into thrice the volume of acetone with continuous stirring. The precipitate was washed with distilled water and vacuum dried at 50-6 ºC.

Preparation of Emulgel

Preparation of emulgel was same in all the formulations. The gel bases were prepared by incorporating fenugreek seed mucilage in distilled water separately with constant stirring at a moderate speed using mechanical shaker[13]. Formulations F1, F2, F3 were prepared by fenugreek seed mucilage and the dispersion was cooled and leftover night. The pH of all the formulations was maintained to 6 - 6.5 by using triethanolamine (TEA). The oil phase of the emulsion was formulated by dissolving Span 20 in light liquid paraffin and *Sesamum indicum* seed oil was mixed in oil phase. While the aqueous phase was produced by dissolving Tween 20 in purified water. Methyl paraben was dispersed in propylene glycol and mixed with aqueous phase, being lipophilic it was dissolved in oil phase. Both the oily and aqueous phases were individually heated to 70° to 80°C, then the oily phase was added to the aqueous phase with constant swirling until it was cooled to room temperature. The resultant emulsion was mixed with the gel in 1:1 ratio with subtle stirring to obtain the emulgel[14].

The composition of different formulations has been discussed in Table 1.

Table 1: Composition of different formulations

Ingredient (%w/w)	F1	F2	F3
Sesamum indicum seed oil	5 ml	5 ml	5 ml
Fenugreek mucilage	1.5	2.0	2.5
Liquid paraffin	7.0	7.0	7.0
Tween 20	0.5	0.5	0.5
Span 20	1.0	1.0	1.0
Propylene glycol	5.5	5.5	5.5
Methyl paraben	0.003	0.003	0.003
Distilled water	q.s.	q.s.	q.s.

EVALUATION OF EMULGEL

i)Physical appearance

All the prepared emulgel preparations were inspected visually for their homogeneity, consistency, color and phase separation[15].

ii) Measurement of pH

The pH of all emulgel formulations was determined by digital pH meter. 1 gram of emulgel was dissolved in 100 ml of distilled water and it was placed aside for two hours. The pH measurement was done in triplicate and average values were reported.[16]

iii) Spreadability

Spreadability is the term expressed to signalize the degree of area to which gel readily spread on applied skin or affected body part. The therapeutic effectiveness of a formulation majorly depends upon its Spreadability. Spreadability is exhibited in terms of time in seconds taken by two slides to slip off from emulgel formulation and placed in between the slides under the orientation of certain load. Lesser the time taken for separation of two slides, better the spreadability. It is calculated by using the formula. S = M. L / T

Where M = wt. tied to upper slide

L = length of glass slides

T = time taken to separate the slides[1].

iv) Extrudability study

All the formulations were filled in the collapsible tubes. The extrudability of the formulation was calculated in terms of weight in grams required to expel a 0.5cm ribbon of gel within 10 seconds[1].

v) Rheological study

The viscosity of different emulgel formulations were determined using a brookfield viscometer at 37 °C [6].

vi)Drug content determination

The drug content was evaluated in each of the formulations. About 1 gm of emulgel was weighed and transferred to 100 ml volumetric flask to which about 70 ml of phosphate buffer pH 7.4 was added, after continuous shaking the volume made up to 100 ml with pH 7.4 phosphate buffer. The content was filtered. An aliquot 1 ml was pipetted out from the filtrate and suitably diluted in pH 7.4 phosphate buffer. The content of *Sesamum indicum* oil was determined using spectrophotometry at 227 nm against blank. The blank solution was prepared same as above, using emulgel without the drug[17].

vii)Evaluation of sun protection factor

The absorption spectra of samples in solution were obtained in the wavelength of 290 to 320 nm, after every 5 nm. 3 determinations were made at each point using ethanol as a blank. SPF values were calculated using Mansur equation.

$$SPF_{in\ vitro} = CF \times \sum_{290}^{320} EE(\lambda) \times I(\lambda) \times abs(\lambda)$$

Where EE: erythemal effect spectrum; I: solar intensity spectrum; Abs: absorbance of sunscreen product; CF: correction factor (= 10)[18].

The values of EE x I are constants and are showed in Table 2.

Table 2: Values of EE x I

Wavelength(nm)	EExI(normalized)
290	0.0150
295	0.0817
300	0.2874
305	0.3278
310	0.1864
315	0.0839
320	0.0180

viii) Determination of In-vitro antioxidant activity

a) DPPH radical scavenging assay

The antioxidant properties were measured using the free stable radical 1, 1-diphenyl-2- picrylhydrazyl (DPPH) of 5 equally diluted samples of O/W sunscreen emulgel formulations. One ml of 100 µM DPPH in methanol was disperesed with equal volume of the diluted sample solution in pH 7.4 phosphate buffer, mixed well and the test tubes were kept for 30 min in a dark room. After incubation at 37° C for 30 minutes the absorbance of each solution was recorded using double beam UV spectrophotometer at 517 nm. The blank reading were also taken and the remaining free radical of DPPH was determined by using the following formula,

DPPH radical scavenging activity (%) = [Abs (control) – Abs (test)/ Abs (control)] × 100.

Where Abs is the absorbance and IC50 value is the concentration of the sample required to quench 50% DPPH free radical. The assays were done in triplicate. An IC50 values was calculated by plotting means of % inhibitions of each assay versus concentrations which is prepared by the dilution of each of the formulation[19].

b) Determination of reducing power

Reducing power of the sample was determined as per the method as one ml of different concentrations of sample (to produce final concentration 5-25mg/ml) was mixed with 2.5ml of potassium ferricyanide (1%) and 2.5 ml of phosphate buffer (pH 6.6). The mixture was incubated for about 20 minutes at 500 ° C and 2.5ml of TCA (10%) was added to it and centrifuged at 800 rpm for 10 minutes. 2.5 ml of supernatant was added to 2.5 ml of water and 0.5 ml of ferric chloride solution (0.1%). Absorbance was recorded at 700nm. [20].

RESULT AND DISCUSSION

All the emulgel formulations were white to buff white viscous creamy preparation with a smooth homogeneous consistency and glossy appearance. Results have been discussed in Table 3.

Table 3: Physical characteristics of emulgels

Formulation	Colour	Homogeneity	Consistency	Phase Separation
F1	White	Good	Good	No
F2	White	Excellent	Good	No
F3	Buff White	Excellent	Good	No

The pH of all emulgel formulations was in the range of 6.55 to 6.70 which lies in the normal pH range of the skin and would not produce any skin irritation. There was no significant change in pH values. All the formulations showed good extrudability. Formulation F2 showed good spreadability, viscosity and drug content. The drug content of the formulated Emulgel was estimated spectrophotometrically at 227 nm. It is higher in F2 formulation. The results are discussed in table 4.

Table 4: Emulgel Parameters

Formulation	pH	Extrudability	Spreadability (gm.cm/sec)	Viscosity (Pa.s)	Drug content (%)
F1	6.55±o.52	Excellent	42.11	1124	95±o.28
F2	6.69±o.32	Excellent	48.93	1149	98±o.61
F3	6.70±o.73	Excellent	45.29	1160	96±o.59

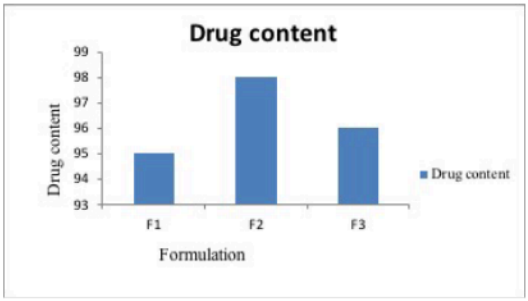

Fig. 1: Drug content of formulations

SPF value determination

The SPF value was determined for the formulation F 2 as it has shown good drug content. The absorbance of the solutions were then measured at 290 – 320 nm with the range of measurement 5 nm, that appropriate with the wavelength of UVB radiation. SPF value is described in table 5, while The CF value used in this measurement was 10 as recommended.

Table 5: SPF value calculation of formulation F2

Wavelength (λ nm)	EE x I	Abs (Average)	EE x I x Abs x CF
290	0.0150	0.618	0.0927
295	0.0817	0.529	0.4321
300	0.2874	0.479	1.3766
305	0.3278	0.380	1.2456
310	0.1864	0.315	0.5871
315	0.0839	0.277	0.2324
320	0.0180	0.243	0.0437
SPF Value			**4.0103**

SPF –sun protection factor, EE – erythemal effect spectrum, I – solar intensity spectrum, CF- correction factor.

In-vitro antioxidant activity

The ability of phenolic compounds to quench reactive species by hydrogen (H^+ions) donation was measured through DPPH radical scavenging activity assay. Antioxidant activity was evaluated with % inhibition. In DPPH method, the absorbance was measured at 517 nm. Lower absorbance of the reaction mixture indicates higher free radical scavenging activity. The obtained results of absorbance and % inhibition showed decrease in concentration of DPPH radical due to scavenging ability of formulation and standard ascorbic acid, as a reference compound. IC_{50} (µg/mL) for ascorbic acid was found to be 8.87± 0.21 and for formulation it was 4.58±1.23 as described in fig 2. For the measurement of the reducing ability, the reducing capability of a compound may serve as a significant indicator of its potential antioxidant. The reducing power of extracts were increased with increase in concentration. It is described in fig 3.

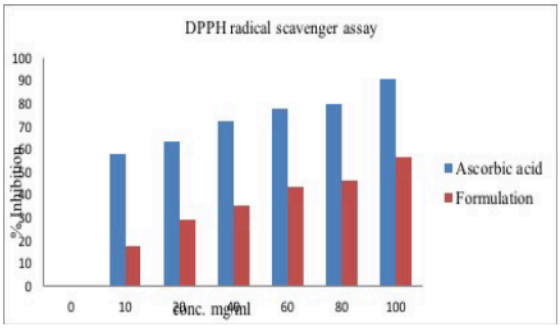

Fig. 2: DPPH Radical Scavenger assay

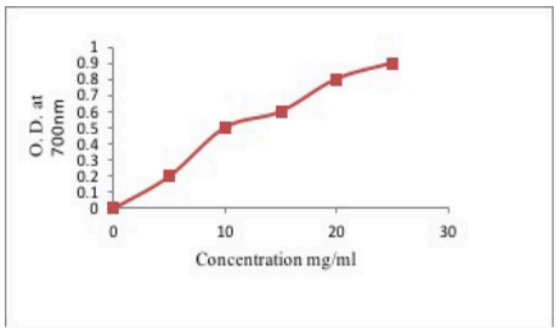

Fig. 3: Reducing power Assay

CONCLUSION

The formulated emulgel have potency to protect against UV-B radiation. The emulgel was physically stable with sun protection factor is 4.01033.

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Cite this article as:

Reshma Jadhav *et al*. Formulation and evaluation of black sesame seed oil sunscreen emulgel using natural gelling agent. Int. Res. J. Pharm. 2018;9(6):197-201 http://dx.doi.org/10.7897/2230-8407.096116

Source of support: Nil, Conflict of interest: None Declared