Original Article

# To develop and evaluate ultraviolet protective skincare topical formulation

**Shivam Singla, Kirandeep Kaur, Veerpal Kaur***

Department of Pharmaceutics, ISF College of Pharmacy, Moga, Punjab, India

**Correspondence:**
Veerpal Kaur, Department of Pharmaceutics, ISF College of Pharmacy, Moga - 142 001, Punjab, India.
Phone: +91-9888236802.
E-mail: vkaur013@gmail.com
**How to cite this article:** Singla S, Kaur K, Kaur V. To develop and evaluate ultraviolet protective skin care topical formulation. Pharmaspire 2020;12(1):32-36.

**Source of Support:** Nil,
**Conflicts of Interest:** None declared

## ABSTRACT

**Objectives:** The main objective of this study was to develop a safe UV protective product with maximum free radical scavenging property. To generate a safe and long acting product against UV radiations. **Methods:** Selenium Nanoparticles were synthesized by using method chemical reduction. Se Nanoparticles are tested for its particle size, PDI and antioxidant activity. Then these Nanoparticles are incorporated into w/o based cream along with titanium oxide and other excipients. The final formulation was evaluated for its physical appearance, viscosity, texture analysis, globule size and distribution, pH, spreadability and SPF determination. **Results:** Results of evaluation of elemental Se showed nanoparticle size in nanoscale. The final product that is UV protective topical cream showed required physicochemical properties. **Conclusion:** The outcomes of this work conclude that the Se nanoparticles and titanium oxide combination was very good as the results indicated.

**Keywords:** Sunscreen, ultraviolet absorbent, topical formulation, skincare

## INTRODUCTION

Ultraviolet (UV) radiations are defined as the part of the electromagnetic spectrum between X-ray and visible light (40–400nm).[1] There has been an increasing trend of sunburn due to escalated heat wave's risk across the globe. Further, sunburn, if not treated, leads to skin cancer. UV radiations can induce reactive oxygen species which are the key mediators of oxidative damage to the skin. Repeated exposure to UVA and UVB increase the risk of sunburn as shown in Figure 1.[2] Topical protectants are most commonly used to protect the skin from harmful UV radiation. A physical sunscreen composition reflects and absorbs UV radiation. The most commonly used are sunscreens, often in the form of ointments, lotions, creams, or spray.[3]

Titanium oxide is the most popular UV adsorbent material that can be used in the formulation of sunscreen cream.[4] It is a semiconductor photocatalyst due to its long-term stability, non-toxicity, and good photocatalytic activity.[1] These properties make $Tio_2$ a very excellent absorber for UV radiation.

| Access this article online | |
|---|---|
| **Website:** www.isfcppharmaspire.com | **P-ISSN:** 2321-4732<br>**E-ISSN:** XXXX-XXXX |

The efficacy of a sunscreen agent is usually expressed in the term of sun protection factor (SPF). As higher is the SPF value it is as much as the sun protective.

Moreover, the topical application of the proposed study was introduced to produce a long-term localized application. The final formulation was designed with selenium nanoparticles and suitable UV adsorbent material. The protective efficacy of the formulation was adjusted by measuring SPF.

## MATERIALS AND METHODS

### Materials

The following materials are used to prepare sunscreen formulation: selenius acid and titanium (IV) oxide (Sigma Aldrich Pvt. Ltd.), sodium borohydride, sodium citrate, sodium lauryl sulfate, and wheat germ oil (HiMedia Laboratories, Mumbai).

### Preparation of selenium nanoparticles

Selenium nanoparticles were prepared using sodium borohydride reduction method. 0.1 M selenius acid solution was prepared in

ethanol and water (1:2). Solution containing 0.6% w/v sodium citrate and 0.4% w/v sodium lauryl sulfate was preheated to 55°C. Then, this preheated solution was added very slowly to the solution of selenius acid in a mixture of ethanol and water to form required molar concentration, that is, 0.05M selenius acid. Sodium borohydride solution was prepared by dissolving 0.4 g sodium borohydride in 100 ml distilled water in a separate beaker. Sodium borohydride (freshly prepared) is then added drop-wise into the above mixture of selenius acid solution and was mixed continuously. Finally, bright red color nanoparticles were prepared. The whole procedure was done in the dark. The mixture was then centrifuged for 30 min at 12000 rpm, and the pellet was washed with absolute alcohol to remove water. The prepared Selenium nanoparticles were used immediately for the preparation of sunscreen formulation.[5]

## Characterization of nanoparticles

### *Particle size and polydispersity index (PDI)*

Particle size and size distribution of prepared selenium nanoparticles were determined at 25°C in the quartz cuvette using Delsa-Nano Zetasizer. The procedure was done in a triplicate manner and the mean value was reported.

### *Nitric oxide assay*

To determine the antioxidant activity of selenium nanoparticles, nitric oxide assay was performed. Two milliliters of 10 mM sodium nitroprusside in 0.5 ml phosphate buffer saline (pH-7.4) were mixed with 0.5 ml of the sample at three different concentrations and then incubated at 25°C for 150 min. 0.5 ml was taken out from the incubated mixture and added into 1 ml sulfanilic acid reagent (33% in 20% glacial acetic acid) and again incubated at 25°C for 5 min. At last 1 ml of naphthyl ethylenediamine dihydrochloride (0.1% w/v) was mixed and incubated at 25°C for 30min. Then, absorbance at 540 nm was measured with a UV spectrophotometer. The nitric oxide radicals scavenging activity was calculated by the following formula.[6]

$$Nitric\ Oxide\ Scavenged\ (\%) = \frac{A\ control - A\ test}{A\ control} \times 100$$

Where $A_{control}$ = Absorbance of control reaction and
$A_{test}$ = Absorbance of test sample reaction.

## Preparation of sunscreen cream

The topical formulation composition consists of ingredients, as listed in Table 1. Water in oil emulsion-based topical cream was formulated. A measured amount of titanium oxide, Stearic acid, cetosteary alcohol, lecithin, Isopropyl myristate, PEG 1500, and wheat germ oil were dissolved in the oil phase and heated up to 70°C. PEG 400, triethanolamine, and glycerin were added in a separate beaker and heating up to 70°C. After the melting oil phase and maintaining temperature for both the phases, the aqueous phase was added slowly to the oil phase with continuous stirring until a uniform, the smooth emulsion was formed. The mixture was kept aside to cool at 45°C. Then, the measured amount of selenium nanoparticles was added and mixed well. At last, propylparaben and methylparaben were added and the formulation was then transferred in the final container.

**Table 1: Composition of sunscreen cream[7]**

| S. No. | Ingredients | Quantity used (%W/W) |
|---|---|---|
| 1. | Selenium nanoparticles | 4 |
| 2. | Titanium oxide | 4 |
| 3. | Stearic acid | 3 |
| 4. | Isopropyl myristate | 4 |
| 5. | Cetosteary alcohol | 2 |
| 6. | Lecithin | 0.02 |
| 7. | PEG 1500 | 3 |
| 8. | Wheat germ oil | 9 |
| 9. | Propylparaben | 0.05 |
| 10 | PEG 400 | 4 |
| 11. | Triethanolamine | 0.42 |
| 12. | Glycerin | 4 |
| 13. | Methylparaben | 0.02 |
| 14. | Freshly boiled and cooled water | q.s. |

## Characterization of sunscreen cream

### *Physical appearance*

The prepared formulation was visually inspected for its physical appearance and the result was recorded.

### *Viscosity*

The Brookfield Rheometer (R/S Plus) was used to determine the viscosity of the prepared formulation. The determination was done in the triplicate manner, and mean value was reported.

### *Texture Analysis*

The Brookfield Texture Analyzer (Texture Pro CTV1.3 build 15) was used to determine the texture of prepared formulation. A compression type of apparatus was used in the texture analyzer. The test speed and recovery speed were set to be 0.5 mm/s.

### *Globule size and globule distribution*

Delsa-Nano ZetaSizer was used to determine the globule size and distribution of prepared formulation. The low volume quartz cuvette was used for determination. The test was done at room temperature in triplicate manner.

### *pH determination*

Digital pH meter was used to determine the pH of the prepared formulation. The determination of pH was done in the triplicate manner and repeated 4 times after every 3 days.

### *Spreadability*

A special apparatus used by Kaur and Rath.[5] was used to determine the spreadability of the prepared formulations. For this, the measured amount of formulation was placed on the ground glass plate, and thus, the formulation was sandwiched between the glass plates having the same dimensions of ground glass plate attached with a hook.[8] A weight of 300 g was placed on the top of two plates for around 5 min so that the cream spread evenly throughout the plate. The top plate was subjected to separate through pulling action by 20 g with the help of a string attached to the hook,

and the time (in seconds) required by the top plate to cover a distance of 10 cm was noted. Spreadability was calculated using the equation.

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

Where, $S$ = Spreadability, $M$ = Weight tied to upper slide, $L$ = Length of glass slides and $T$ = Time taken to separate the slides from each other.

### *SPF determination*

SPF was determined using the UV spectrophotometric method.[9] For SPF determination, we use diluted solutions of the prepared formulation. Weigh 1 g of cream and makeup volume up to 100 ml with ethanol in a volumetric flask, after mixing filter this solution through Whatman filter paper and then take a 5 ml sample from the above solution and again makeup volume up to 25 ml in a volumetric flask. Then, this solution was subjected to determine SPF. The test was done in the triplicate manner at 290–320 nm range using UV-spectrophotometer. Then, the Mansur equation was used to determine the SPF value of the formulation:

$$\text{SPF} = \text{CF} \times \sum_{320}^{290} \text{EE}(\lambda) \times \text{abs}(\lambda)$$

Where, CF = 10 (correction factor),
EE ($\lambda$) = Erythemogenic effect of radiation at wavelength ,
I ($\lambda$) = Intensity of solar light at wavelength $\lambda$ and
Abs ($\lambda$) = Absorbance of the sample at wavelength $\lambda$.

The values for the term "EE×I" are constants, which were determined by Sayre RM *et al*.[10]

## RESULTS AND DISCUSSIONS

### Preparation of selenium nanoparticles

Selenium nanoparticles were prepared using a chemical reduction method. In this method, the presence of reducing agent ion form of selenium reduced to the elemental form of selenium. The results of this experiment suggest that the reduction of Se (IV) pH plays an important role and sodium citrate played a vital role in the preparation of the optimized size of the selenium nanoparticles. In this process, nucleation is prevented due to opposite charges of citrate ions. This leads to inhibition of the growth of nuclei. Kai Kai *et al*. used sodium citrate as a reducing agent producing mono-disperse elemental selenium particles with a narrow size distribution of 50 nm. This is further interesting to observe that the change in pH[5-7] significantly alters the size resulting in larger nanoparticles. Results of particle size and size distribution indicated that reducing agent alone is not sufficient to control particle growth and homogeneity. Surfactant helps to improve the size distribution of selenium nanoparticles by decreasing the mobility of the nanoparticles. Saeedeh used sodium lauryl sulfate as a stabilizing agent, producing selenium nanoparticles with a size range of 170 nm.[11]

### Characterization of selenium nanoparticles

#### *Particle size and PDI*

Zeta sizer was used to characterize the particle size and PDI. Beyond critical limits, particle size was found to be present in the range of 100–150 nm with 0.1–0.2 PDI, as shown in Figure 2. There was no aggregation seen in the formulation.

#### *Nitric oxide assay*

To determine the antioxidant activity of selenium nanoparticles, nitric oxide assay was performed. The result shows that selenium nanoparticles exhibit excellent nitric oxide scavenging activity which is satisfactory for the formulation. Xiaona *et al*. used chitosan to synthesize selenium nanoparticles of stabilized morphology. Free radical scavenging activity shows 0.2 m nanoparticle dispersion capable to reduce 2,2-diphenyl-1-picryl-hydrazyl-hydrate up to 87%.

### Characterization of sunscreen formulation

#### *Physical appearance*

The formulation without selenium nanoparticles was found to be white. When nanoparticles are added, the color of formulation turned to light yellow.

#### *Viscosity*

The viscosity of the formulation was found to be 40 pa.s, as shown in Figure 3. The results show that the formulation had good rheology.

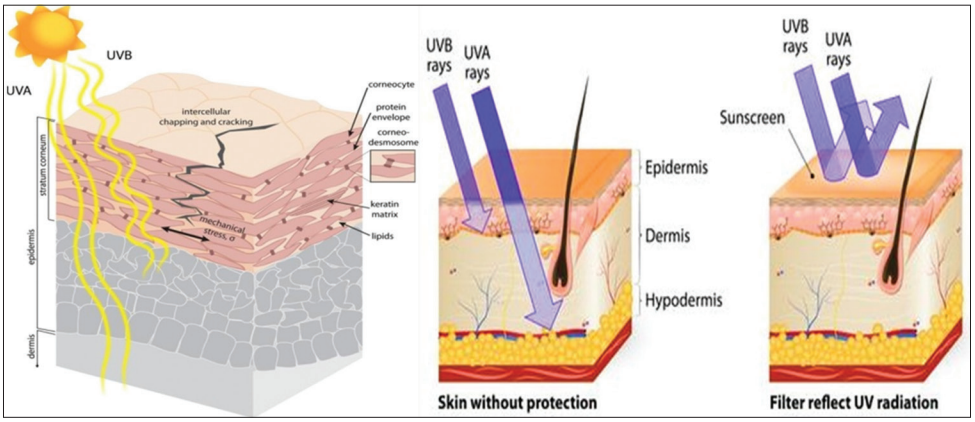


**Figure 1:** Indicating the harmful effects of ultraviolet (UV) radiation on skin and UV penetration into the skin

Cumulants Results

| | | | |
|---|---|---|---|
| Diameter | (d) | : 143.7 | (nm) |
| Polydispersity Index | (P.I.) | : 0.269 | |
| Diffusion Const. | (D) | : 3.442e-008 | (cm²/sec) |
| Residual | | : 7.130e-003 | (O.K) |

Measurement Condition

| | | |
|---|---|---|
| Temperature | : 25.2 | (°C) |
| Diluent Name | : WATER | |
| Refractive Index | : 1.3328 | |
| Viscosity | : 0.8838 | (cP) |
| Scattering Intensity | : 11229 | (cps) |

Distribution Results (Contin)

| Intensity Distribution | | | Volume Distribution | | | Number Distribution | | |
|---|---|---|---|---|---|---|---|---|
| Peak | Diameter (nm) | Std. Dev. | Peak | Diameter (nm) | Std. Dev. | Peak | Diameter (nm) | Std. Dev. |
| 1 | 222.3 | 215.9 | 1 | 80.7 | 37.4 | 1 | 60.4 | 15.9 |
| 2 | 0.0 | 0.0 | 2 | 0.0 | 0.0 | 2 | 0.0 | 0.0 |
| 3 | 0.0 | 0.0 | 3 | 0.0 | 0.0 | 3 | 0.0 | 0.0 |
| 4 | 0.0 | 0.0 | 4 | 0.0 | 0.0 | 4 | 0.0 | 0.0 |
| 5 | 0.0 | 0.0 | 5 | 0.0 | 0.0 | 5 | 0.0 | 0.0 |

**Figure 2:** The particle size and polydispersity index of selenium nanoparticles

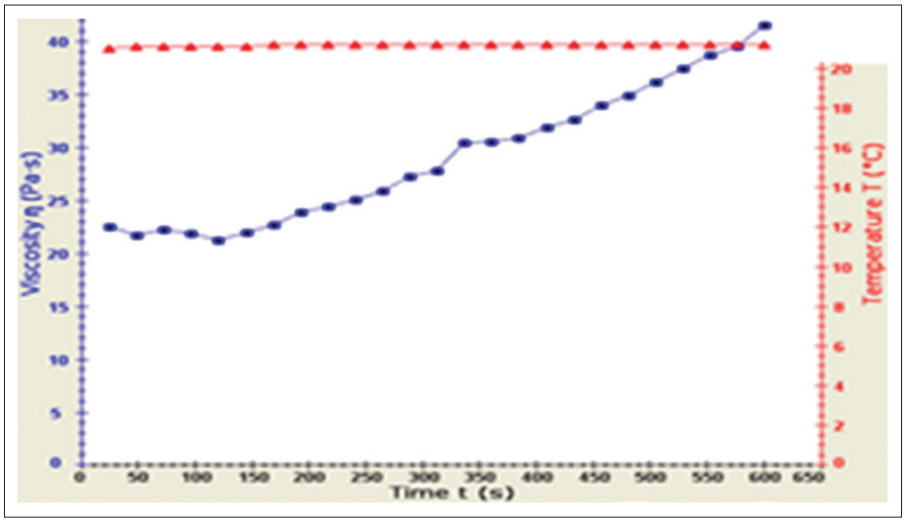


**Figure 3:** The viscosity of cream having a good flow of formulation

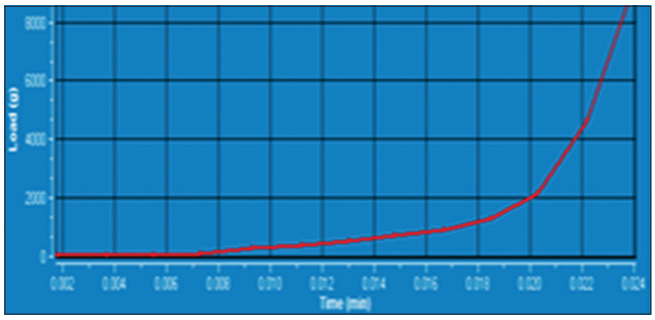

**Figure 4:** The dilatant behavior of the prepared formulation

Good rheology indicates better emulsification of the oil phase and aqueous phase with the help of emulsifier.

### *Texture analysis*

The prepared formulation was tested for analysis of its texture using the Brookfield texture analyzer. The result shows in Figure 4 that indicates the dilatant behavior of the cream. The dilatant nature of the cream ensures better spreadability and required retention time at the site of application which is very important for sunscreen formulation.

### *Globule size analysis*

Delsa-Nano Zetasizer was used to determine the globule size of the prepared formulation. The result shows that the globule size of the cream was found to be 207 nm at 0.1 PDI, as indicated by Figure 5. The PDI indicated the good stability of the formulation.

### *pH*

The result of pH analysis shows that there is an increase in pH as the amount of triethanolamine increases. pH plays an important role in the stability of the topical formulation. The pH was found to be 6.7 ± 0.003.

### *Spreadability*

The spreadability of the prepared formulation was calculated using the equation written in section 2.5.6. Spread ability is required

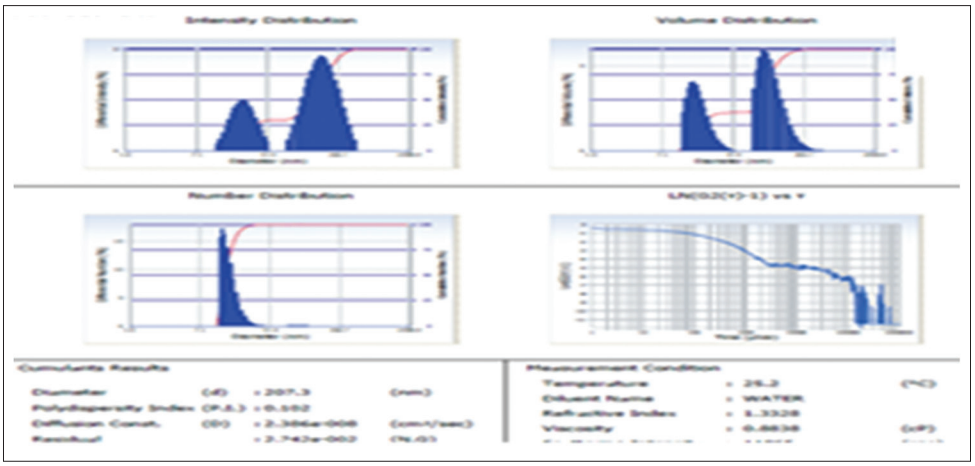

**Figure 5:** The globule size and polydispersity index of the prepared formulation

for a smooth film at the site of application. The spreadability was found to be 9.2 g.cm/s that ensures easy application of the formulation.

### *SPF determination*

SPF is determined by the UV spectrophotometric method. The SPF was calculated using the equation as described in section 2.5.7. The result of SPF determination was found to be 28.8 ± 0.91.

## CONCLUSION

The outcomes of this work conclude that the selenium nanoparticles and titanium oxide combination were very good as the results indicated. This prepared cream formulation has antioxidant properties, UV A adsorbent, and UV B adsorbent properties. Water in oil type emulsion-based formulation is good for the stability of selenium nanoparticles. The composition of the formulation shows required viscosity, spreadability, pH, and texture.

## REFERENCES


1. Wirunchit S, Apivitcholchat C, Chodjarusawad T, Koetniyom W. The study of UV protection materials. InAIP Conference Proceedings. United States: AIP Publishing LLC; 2018. p. 020024.
2. Ribeiro FM, Volpato H, Lazarin-Bidóia D, Desoti VC, de Souza RO, Fonseca MJ, *et al*. The extended production of UV-induced reactive oxygen species in L929 fibroblasts is attenuated by posttreatment with *Arrabidaea chica* through scavenging mechanisms. J Photochem Photobiol B Biol 2018;178:175-81.
3. Serpone N, Dondi D, Albini A. Inorganic and organic UV filters: Their role and efficacy in sunscreens and suncare products. Inorg Chim Acta 2007;360:794-802.
4. Landa B, Abramovich S, Snir DO. UV-Protective Compositions and Their Use. United States Patent US No. 10,675,229. Inventors LANDA LABS 2012 Ltd., Assignee; 2020.
5. Kaur K, Rath G. Formulation and evaluation of UV protective synbiotic skin care topical formulation. J Cosmet Laser Ther 2019;21:332-42.
6. Balakrishnan N, Panda AB, Raj NR, Shrivastava A, Prathani R. The evaluation of nitric oxide scavenging activity of *Acalypha indica* Linn root. Asian J Res Chem 2009;2:148-50.
7. Dike M, Thergoankar R, Deodhar M. Screening of Various Extracts of *Garcinia indica* viz., Leaf, Seed, Stem, Root and Fruit for UV Protective Activity and Incorporation of Extracts in Sun Protective Formulations. In: 3rd International Symposium on Underutilized Plant Species, No. 1241 2015. p. 639-46.
8. Sabale V, Kunjwani H, Sabale P. Formulation and *in vitro* evaluation of the topical antiageing preparation of the fruit of *Benincasa hispida*. J Ayurveda Integr Med 2011;2:124.
9. Ntohogian S, Gavriliadou V, Christodoulou E, Nanaki S, Lykidou S, Naidis P, *et al*. Chitosan nanoparticles with encapsulated natural and uf-purified annatto and saffron for the preparation of uv protective cosmetic emulsions. Molecules 2018;23:2107.
10. Sayre RM, Agin PP, LeVee GJ, Marlowe E. A comparison of *in vivo* and *in vitro* testing of sunscreening formulas. Photochem Photobiol 1979;29:559-66.
11. Pouri S, Motamedi H, Honary S, Kazeminezhad I. Biological synthesis of selenium nanoparticles and evaluation of their bioavailability. Braz Arch Biol Technol 2017;60:1-11.