ORIGINAL ARTICLE

Formulation of Sun Screen Gel by Incorporating Titanium Dioxide Nanoparticles and Its Evaluation

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Abstract

Aims: The aim of sunscreen gel by incorporating active ingredient in the range of nano particle size, i.e., titanium dioxide is against sun screen and suntan preparations is to assist the skin painful effects and as a good cosmetic for well-being. **Materials and Methods:** The combination of ingredients were used, such as titanium dioxide, raspberry oil, Jojoba oil, carbopal, polyethylene glycol, methyl paraben, glycerin and rose oil, and distilled water. The titanium dioxide nanoparticles were used as good cosmetic and act against sun screen effect as a sunscreen agent, Jojoba oil and raspberry oil were also used as sunscreen agents, in the present investigation. Carbopal was used as gelling agent and poly ethylene glycol as emollient, glycerin for glossy effect and moisturizing agent, methylparaben as preservative, rose oil as flavoring agent and distilled water used as aqueous phase. The formulations were made by mixing the mentioned ingredients in different proportions by coding F1 to F6 to conclude the best among the prepared formulations for further scope of research. The prepared formulation for evaluated for suitable parameters such as physical appearance/visual inspection, skin irritation, determination of pH, perfume stability, spreadability, viscosity, and sun protection factor (SPF). **Results and Discussion:** All the formulations showed a white color appearance with a pleasant odor and good perfume stability. The pH for F1 to F6 were 6.2, 6.5, 6.4, 6.8, 6.5, and 7.0, and all the formulations do not show any skin irritation and spreadability shows from average to very good. The viscosity values for F1 were 3101, for F2 were 3216, for F3 were 3315, for F4 were 3418, for F5 were 3400, and for F6 were 3627. For the SPF, an *in vitro* method is performed using the Mansur equation, and the obtained grade of SPF is high-to-very high protection. **Conclusion:** After performing all the necessary evaluation tests to decide the best formulation among the prepared, the F6 formulation was selected as best formulation.

Key words: Carbopal, jojoba oil, nanoparticles, polyethylene glycol, raspberry oil, sunscreen agents, titanium dioxide, viscosity and sun protection factor

INTRODUCTION

Among the most significant areas of contemporary research is nanotechnology, which is concerned with matter manipulation at the nanoscale. Nanoparticles are defined as particles with a size between 1 and 100 nm.[1,2] In recent years, titanium dioxide nanoparticles have become one of the most commonly used

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nanomaterials. Titanium dioxide nanoparticles are utilized annually in the cosmetic industry. Every day use involves the functionalization of numerous items with titanium dioxide nanoparticles. Titanium dioxide nanoparticles are found in household items such as toothpaste, shaving creams, sun protection creams, shampoos, and conditioners.[3-5] Titanium dioxide nanoparticles are also frequently employed as food additives to improve the flavor, color, and brightness of a range of food items.[6,7]

A substance that helps protect the skin from the sun's harmful rays. Sunscreen reflects, absorb, and scatter both ultraviolet A and B radiation to provide protection against both types of radiation. Using lotions that contain sunscreens can help protect the skin from premature aging and damage that may lead to skin cancer. Sun-block formulae must be created for repair, reduction of sunburn, sun tanning, skin melanoma, and early lines and wrinkles, as well as increasing the degree of sun protection factor (SPF). Sunscreens are frequently applied to the skin to protect it from the sun's harmful rays and to reduce the risk of skin disorders caused by the sun's rays. Broad spectrum sunscreens are now being researched to reduce the long-term effects of high ultraviolet (UV) radiation.

The use of sunscreens as photo protectants has evolved significantly over the past few decades. With increasing awareness of the protection afforded by sunscreens against sunburns, skin aging, and melanomas, the demand for sunscreen formulations will invariable increase, and there exists a significant opportunity for the pharmaceutical industries to fulfill this demand by manufacturing quality, safe, and esthetically appealing sunscreen formulations. Sunscreen lotion is a sort of product that protects against the sun's harmful rays by containing ultraviolet radiation (UV rays).

The use of sunscreen is a necessity these days to protect our skin from the harsh UV rays. The herbal sunscreen will only protect the skin from the effects of harmful UV rays. Therefore, manufacturers all over the world have begun manufacturing herbal sunscreens to prevent side effects caused by synthetic chemical products.

Sunscreen products can be formulated in the form of lotions, creams, sticks, aerosols, gels, powders, and ointments. Sunscreen preparations are designed to be used topically to prevent UV radiation from entering the skin directly by absorbing or reflecting from the skin.

Skin human skin is the largest organ and it covers about 1.7 m^2 of our body. The membrane of the skin is regarded as a physical barrier. The skin is mainly categorized to four layers: subcutaneous fat layer or hypodermis, overlying dermis, viable epidermis, and stratum corneum (non-viable epidermis).

Effects of UV on skin

Sun light is mainly of wavelengths from UV to visible light. UV is the ultraviolet radiation which is of three divisions such as: Ultraviolet A (UVA) is of 320–400 nm, ultraviolet B (UVB) is of 290–320 nm and ultraviolet C (UVC) is of 100–290 nm.

a. UVA has long wavelength that penetrate and reach the bottom layer of the skin, which causes tanning and aging of the skin
b. UVB has a short wavelength and enter to the epidermis and small amount into the dermis.
c. UVC does not reach the surface of the skin. It is blocked completely by the layer of ozone. The effect of sun radiation on human skin is given in Table 1.

Mechanism of photo protection

Sunscreen act by preventing and minimizing the damaging effects of the ultraviolet sunrays following exposure to the sunscreen have been demonstrated to increase the tolerance of the skin to UV exposure. They work on the following two mechanisms:

1. Scattering and reflection of UV energy from the skin surface mineral, based on inorganic sunscreen works on this mechanism; they provide a coating that blocks sun rays from penetrating through the skin
2. Absorption of the UV energy by converting it to heat energy, thus reducing its harmful effects and reducing the depth, which can penetrate the skin organic sunscreen works on this mechanism.

Applications of sunscreens

1. Shields from harmful UV rays
2. Prevent the signs of ageing
3. Reduce inflammation
4. Prevent skin discoloration
5. Lowers skin cancer risk
6. Lowers blotchiness on the face
7. Prevents sunburns

Table 1: Effect of sun radiation on human skin

S. No	Type of radiation	Characteristic wave length	Effects on human skin
1.	UVC	200–290 nm (short wave UV)	DNA damage
2.	UVB	290–320 nm (midrange UV)	Sun burn DNA damage skin cancer
3.	UVA	320–400 nm (long wave UV)	Tanning Skin aging DNA damage, Skin cancer
4.	VIS	400–800 nm	None currently known
5.	IR	800–120,000 nm	Heat sensation

UV: Ultraviolet, UVC: Ultraviolet C, UVB: Ultraviolet B, UVA: Ultraviolet A, IR: Infrared

Figure 1: Preparation titanium dioxide nanoparticles incorporated sunscreen gel

Figure 2: Determination of pH for the prepared formulations

Figure 3: Performing skin irritation test by applying sunscreen on the skin for 10 min.

8. Prevents tanning
9. Enhances health of the skin.[8]

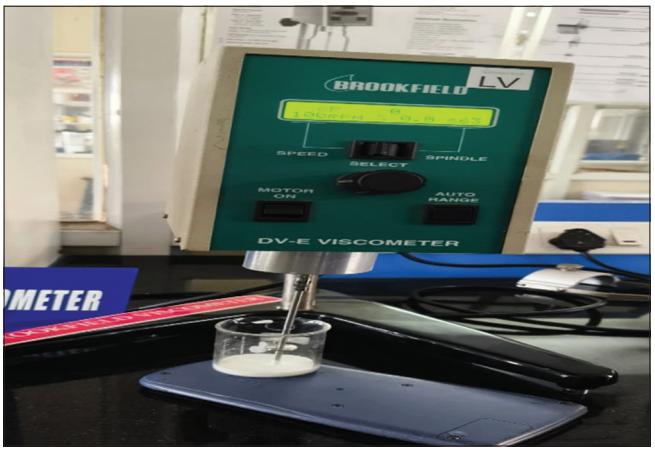

Figure 4: Viscosity test performed for prepared formulations by using Brookfield viscometer

MATERIALS AND METHODS

Materials

Titanium dioxide (Techinstro chemicals), Jojoba oil (Blend it Raw Apotherapy), and Rasp berry oil (RV Essential) were used as sunscreen agents, polyethylene glycol (Reagent Shine Pvt. Ltd) as emollient, glycerin (standard deviation [SD] fine chemicals) as Glossy effect and moisturizing agent, carbopol as thickening agent (SD fine chemicals) methyl paraben as preservative and rose oil (Banjaras touch of nature) as flavoring agent.

Preparation of sunscreen gel

The gel was prepared using the dispersion process, dispersing the weighed amount of Carbopol 934 for swelling in distilled water for 2 h. Once the carbopolget swelled, it was held for stirring on magnetic stirrer. The prescribed

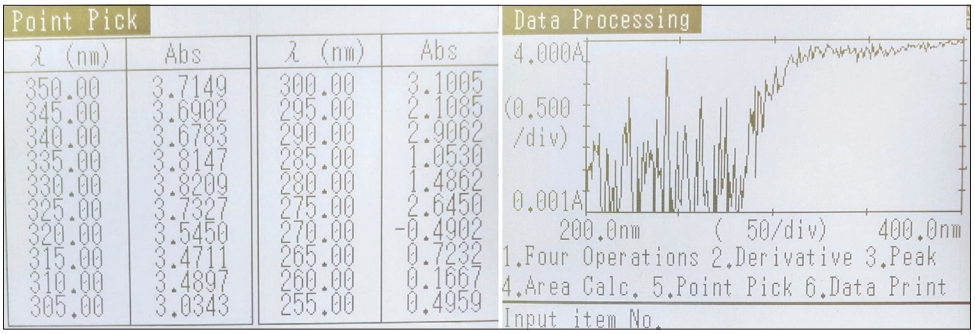

Figure 5: F1 formulation absorbance data obtained by ultraviolet method ranging from 290 to 320 nm

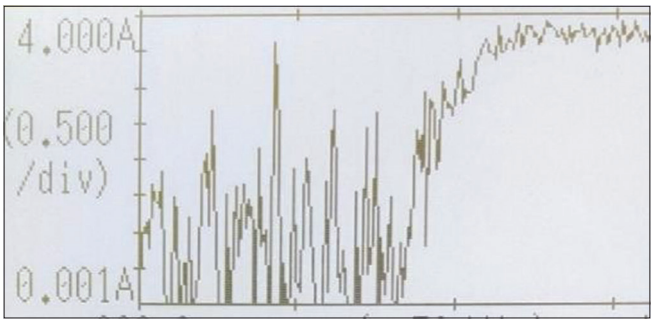

Figure 6: F2 formulation absorbance data obtained by ultraviolet method ranging from 290 to 320 nm

amount of titanium dioxide nanoparticles were incorporated to the dispersion to bring 1% of the substance into the gel. Then add propylene glycol and jojoba oil, raspberry oil were added into the mixture and stirred at 500 rpm using a magnetic stirrer. Add the remaining ingredients and continuous stirring until the formulation was completely dispersed to obtain the homogeneous gel and was shown in figure 1. The composition of sunscreen gel formulations was shown in table 2.[9-12]

Evaluation of sunscreen gel[13-15]

Physical appearance/visual inspection

The formulation was prepared and observed for color, odor, and appearance and stability.

- Color: The prepared sunscreen was evaluated for its color. The color was checked visually
- Odor: Odors was found by smelling the product
- Stability: The product was maintained in different temperature conditions to check its stability.

Determination of pH

Two grams of sunscreen is taken in 50 mL beaker, take the pH paper and put the sample on pH paper to determine the PH.

Skin irritation

It is carried out by applying sunscreen on the skin for 10 min.

Perfume stability

After 30 days, the sunscreen cream underwent testing to record its fragrance.

Spreadability test

Good consistent, if prepared sunscreen cream doesn't leave pieces, flawless application doesn't cause the sunscreen cream to deform.

Intermediate consistent, if few fragments are left behind, proper application, and minimal sunscreen cream deformation.

Unfortunately, if sunscreen is severely deformed application is difficult or improper, and there are numerous fragments left behind.

Viscosity

The formulation is an oil in water emulsion hence the viscosity was low resulting in rapid release of complete drug in 6 h. It is seen that among all the formulations F6 showed higher viscosity compared to other formulations. Those values were ranging between 3100 cp to 3625 cp. The viscosity of the formulation.

The determination of ph, skin irritation, viscosity, was shown in figures 2-4.

Determination of SPF

The efficacy of a sunscreen is expressed by the SPF. An *in vitro* method of determining SPF of the sunscreens is by using Mansur equation.

Procedure

Weigh about 2.0 g of the sample in a 100 mL volumetric flask and add methanol to about 3/4th volume of the flask. Sonicate the contents for about 15–20 min and make up to the mark using methanol. Filter the solution through Whatman No1 filter paper and collect the filtrate by rejecting the first few mL of the filtrate. Take 5 mL of the aliquot in a 50 mL volumetric flask and make up to the mark using methanol. Then take 5 mL of the diluted solution in to

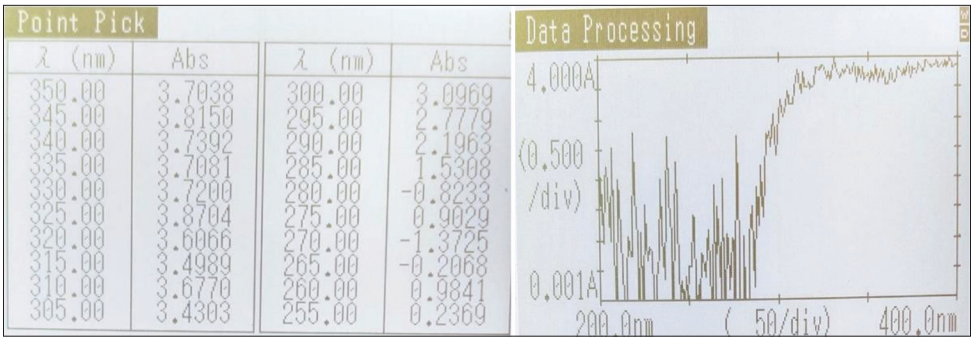

Figure 7: F3 formulation absorbance data obtained by ultraviolet method ranging from 290 to 320 nm

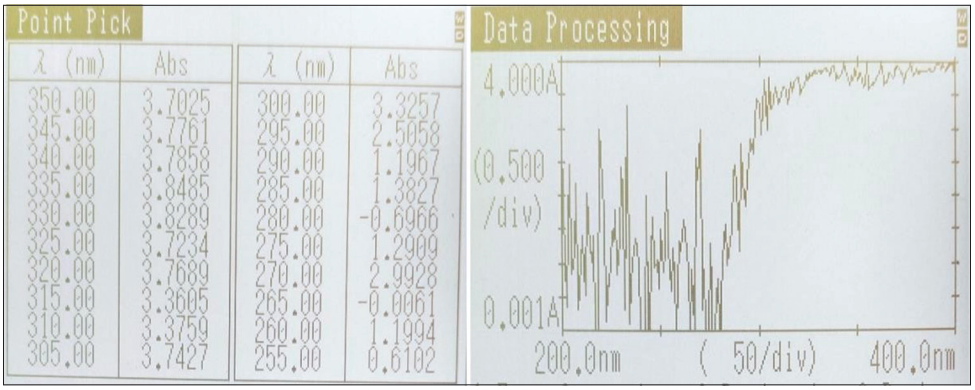

Figure 8: F4 formulation absorbance data obtained by ultraviolet method ranging from 290 to 320 nm

Table 2: Formulation and composition of titanium dioxide nanoparticles incorporated sunscreen gel

S. No.	Name of the ingredients	F1	F2	F3	F4	F5	F6
1	Titanium dioxide	5 g	5.25 g	5.5 g	5.75 g	6 g	6.25 g
2	Raspberry oil	1.5 mL	1.75 mL	2 mL	2.25 mL	2.5 mL	2.75 mL
3	Jojoba oil	4 mL	3.5 mL	3 mL	2.5 mL	2 mL	1.5 mL
4	Carbopal	3 g	3 g	3 g	3 g	3 g	3 g
5	Poly ethylene glycol	0.5 mL	0.5 mL	0.5 mL	0.5 mL	0.5 mL	0.5 mL
6	Methyl paraben	0.8 mg	0.8 mg	0.8 mg	0.8 mg	0.8 mg	0.8 mg
7	Glycerin	3 mL	3 mL	3 mL	3 mL	3 mL	3 mL
8	Rose oil	QS	QS	QS	QS	QS	QS
9	Water	Upto 100 mL	Upto 100 mL	Upto 100 mL	Upto 100 mL	Upto 100 mL	Upto 100 mL

the 25 mL volumetric flask and made up to the mark using methanol. The absorption spectra of sample solution were obtained in the range of 250–400 nm using 1 cm quartz cell, and methanol as blank. The absorption data were obtained in the range of 290–320, every 5 nm, and 3 determinations were made for each sample. The SPF of the samples were calculated using the below equation (a mathematical expression derived by Mansur).

$$SPF\ (spectrophotometric) = CF \times \sum (\lambda)\ 320\ 290 \times (\lambda) \times Ab\ (\lambda)$$

Where, CF is correction factor (=10);
EE (λ) - erythemal effect of radiation with wavelength λ

Scanning electron microscopy test (SEM)

The titanium dioxide nanoparticles are incorporated in sunscreen gel which are detected using SEM analysis in YV University, and the given pictures are Figures 11 and 12.

RESULTS AND DISCUSSION

The innovative research work is made to prepare the sunscreen gel by using ingredients such as Titanium dioxide, raspberry oil, jojoba oil, glycerin, rose oil, polyethylene glycol, carbopol,

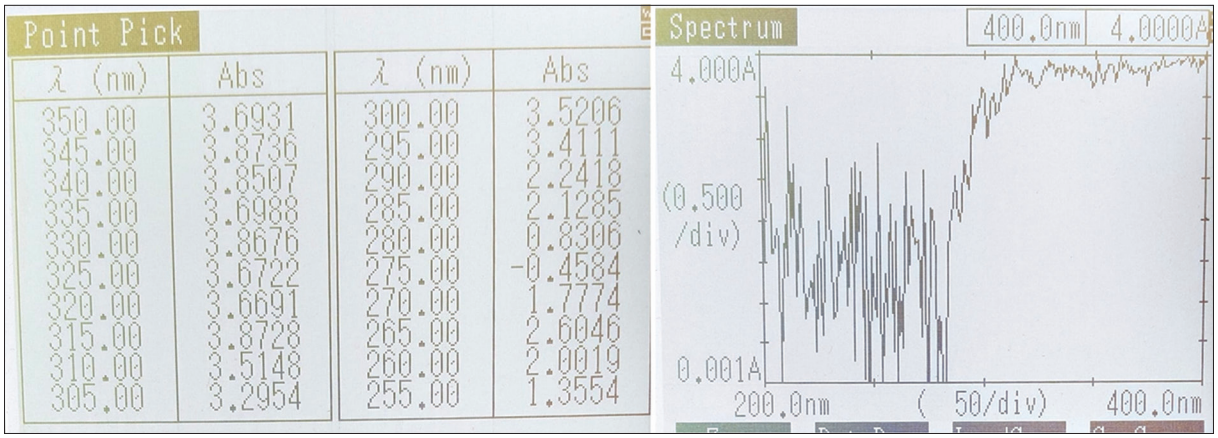

Figure 9: F5 formulation absorbance data obtained by ultraviolet method ranging from 290 to 320 nm

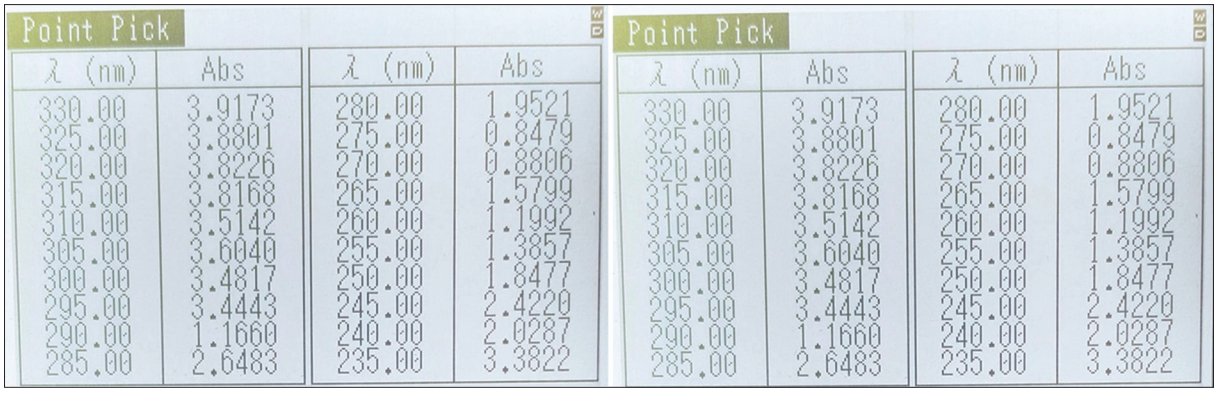

Figure 10: F6 formulation absorbance data obtained by ultraviolet method ranging from 290 to 320 nm

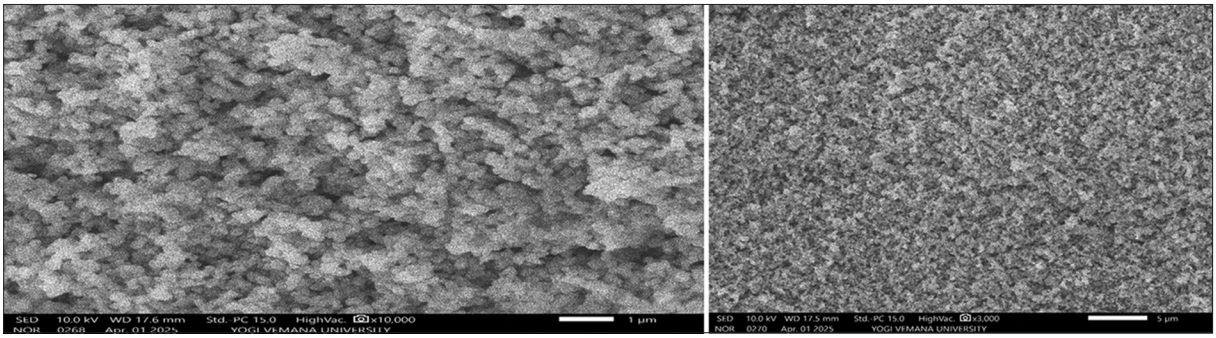

Figure 11: The SEM analysis of titanium dioxide nanoparticles

Table 3: Evaluation of sunscreen gel formulations for physical appearance, odor, pH. viscosity (cps), and spreadability

S. No.	Formulation	Physical appearance	Odor	PH	Viscocity (cps)	Spreadability
1.	F1	White color	Pleasant	6.2	3101	Average
2.	F2	White color	Pleasant	6.5	3216	Good
3.	F3	White color	Pleasant	6.4	3315	Good
4.	F4	White color	Pleasant	6.8	3418	Average
5.	F5	White color	Pleasant	6.5	3400	Average
6.	F6	White color	Pleasant	7.0	3627	Very good

Table 4: Absorbance data from 290 to 320 obtained by the ultraviolet method, and calculation of SPF for all the formulations ranging from F1 to F6 and also represented the grades of all the formulations

S. No.	Wavelength	EE×I	Formulation F1		Formulation F2	
			Absorbance	EE×I × A	Absorbance	EE×I × A
1.	290	0.015	2.9062	0.043593	1.8575	0.043593
2.	295	0.0817	2.1085	0.1722644	2.5235	0.1722644
3.	300	0.2874	3.1005	0.8910837	2.9811	0.8910837
4.	305	0.3278	3.0343	0.9946435	3.5020	0.9946435
5.	310	0.1864	3.4897	0.6504800	3.0852	0.6504800
6.	315	0.0839	3.4711	0.2912252	3.5260	0.2912252
7.	320	0.018	3.5450	0.06381	3.5767	0.06381
			Total	3.04322900	Total	3.174049
S. No.	**Wavelength**	**EE×I**	**Formulation F3**		**Formulation F4**	
			Absorbance	**EE×I × A**	**Absorbance**	**EE×I × A**
1.	290	0.015	2.1963	0.0329445	1.1697	0.0179505
2.	295	0.0817	2.7779	0.22704443	2.5058	0.2047238
3.	300	0.2874	3.0969	0.890046906	3.3257	0.95580618
4.	305	0.3278	3.4303	1.12445234	3.7427	1.22685010
5.	310	0.1864	3.6770	0.6853928	3.3759	0.629267
6.	315	0.0839	3.4989	0.293557	3.7605	0.281945
7.	320	0.018	3.6066	0.0649188	3.7689	0.0678402
			Total	3.3233789	Total	3.478105
S. No.	**Wavelength**	**EE×I**	**Formulation F5**		**Formulation F6**	
			Absorbance	**EE×I × A**	**Absorbance**	**EE×I × A**
1.	290	0.015	2.2418	0.033627	1.1660	0.001749
2.	295	0.0817	3.4111	0.2786787	3.4443	0.28139
3.	300	0.2874	3.5206	1.01182044	3.4817	1.0006
4.	305	0.3278	3.2954	1.08023212	3.6040	1.181391
5.	310	0.1864	3.5148	0.65515872	3.5142	0.65504
6.	315	0.0839	3.8728	0.32492792	3.8168	0.32022
7.	320	0.018	3.6691	0.0660438	3.8226	0.68806
			Total	3.7531317	Total	4.144191

S. No.	Formulations	SPF=CF×Σ^{320}290×EE×I × absorbance	SPF values	Grade
1.	F1	3.04322900×10	30.432	High protection
2.	F2	3.174049×10	31.740	High protection
3.	F3	3.3233789×10	33.23	High protection
4.	F4	3.478105×10	34.78	High protection
5.	F5	3.7531317×10	37.53	High protection
6.	F6	4.144191×10	41.44	Very high protection

SPF: Sun protection factor

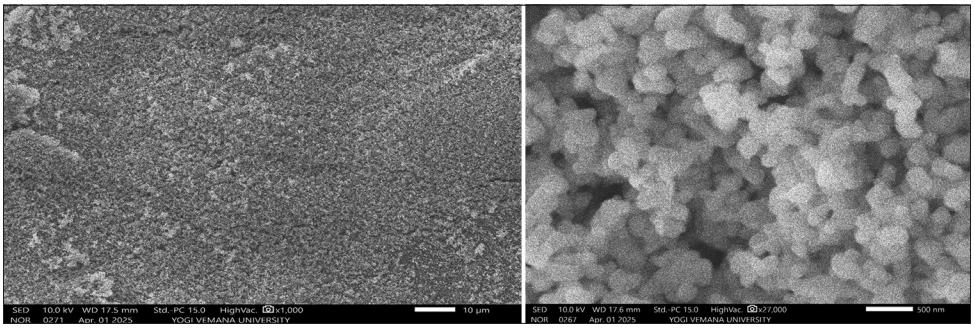

Figure 12: The images of SEM analysis of titanium dioxide nanoparticles

and methylparaben by various combinations. The prepared sunscreen gel was evaluated for physical appearance, pH, skin irritation, perfume stability, spreadability and viscosity, and SPF.

Physical appearance

The formulations of sunscreen cream prepared were appeared in white color [Table 3].

Determination of pH

The pH of the formulated sunscreen cream was determined and found to be in the range of cream 6.2–7.0 [Table 3].

Skin irritation

All the prepared formulations of sunscreen cream and gel does not show any skin irritation when applied on skin and observed for more than 10 min [Table 3].

Spreadability

Formulations of sunscreen cream show from average to very good spreadability. The formulation (F6) shows very good spreadability nature compared to other formulations [Table 3].

Viscosity

The viscosity of sunscreen cream was determined for the formulations and the results are shown in table (6.2) and the results ranged from 3101 to 3627 cps [Table 3].

SPF

The SPF of the sunscreen cream was determined for the formulations and results are shown in [Table 4] and the SPF ranges from 30.432 to 41.44 [Figures 7-12].

CONCLUSION

In this novel work development the formulation was made with the use of ingredients such as titanium dioxide, raspberry oil, jojoba oil, carbopal, polyethylene glycol, methyl paraben, glycerin and rose oil and distilled water in various proportions from formulations F1 to F6. All the developed formulations of sunscreen gel were evaluated for various tests and the results obtained for all were in acceptable range.

For the F6 Formulation, the spreadability obtained is very good and the values of pH are similar to skin compatibility with very high protection result in SPF evaluation. Based on the obtained data on all the evaluation tests, the F6 formulation was selected as best formulation.

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