MICROENCAPSULATION TECHNIQUE FOR SUNSCREEN GEL DEVELOPMENT: ENHANCING UV PROTECTION

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ABSTRACT

The current research looked at the sunscreen gel formulation using microbead technology via the ionotropic gelation method. Herbal oils such as jojoba oil and raspberry seed oil were used in our study due to their sun-protective qualities, which provide protection against both UV-A and UV-B rays. The pH, viscosity, stability, homogeneity, spreadability, extrudability, scanning electron microscopy and SPF determination of the prepared herbal sunscreen gel were also evaluated. Sunscreen gel has a pH range of 6.20-6.23, a viscosity range of 3060-3684 cps, and an extrudability study of 12-16. The F5 formulation demonstrated good spreadability, consistency, homogeneity, appearance, and pH, according to the evaluation, with no evidence of phase separation. During the study, the formulation F5 provided the best UV protection with SPF 11.

Keywords: Sunscreen gel, microencapsulation, SEM analysis, Sun Protection Factor

INTRODUCTION

Sunscreen is a popular skincare product that shields the skin from the damaging effects of ultraviolet (UV) radiation. Traditional sunscreen formulations, on the other hand, may not provide optimal UV protection due to issues such as poor stability, insufficient coverage and potential skin irritation. While handling these issues, researchers have developed novel approaches, such as microencapsulation technology, to improve sunscreen performance[1].

Microencapsulation involves encapsulating active ingredients, such as UV filters, within small particles to improve their stability and efficacy. This technique has been widely used in various industries, including cosmetics, pharmaceuticals and food. In the case of sunscreen, microencapsulation can help to protect UV filters from degradation due to exposure to heat, light and other environmental factors[2].

The design and development of a sunscreen gel using microencapsulation technology involves several steps, including the selection of appropriate UV filters, encapsulation materials and techniques. The formulation must also be tested for stability efficacy and safety[3,4].

Once the formulation is developed, it can be evaluated through various *in vitro* and *in vivo* tests to determine its ability to provide enhanced UV protection. These tests include sun protection factor (SPF), water resistance, and photostability measurements[5-7].

In general, microencapsulation technology holds great promise for enhancing the efficiency and safety of sunscreens in the design and development of sunscreen gels. This strategy may aid in lowering the risk of skin cancer and other negative effects of UV radiation[8-10].

MATERIALS AND METHODS

Carbopol® 934 was procured from Yarrow Chem Products, India. Glycerin, triethanolamine, sodium benzoate, zinc oxide and calcium chloride were procured from Spectrum Reagents & Chemicals Pvt. Ltd., India, Tween®80 and titanium dioxide were procured from Sigma-Aldrich Chemicals Pvt. Ltd., India. Red raspberry seed oil was procured from Greenwood Essential Pvt. Ltd., India and banjaras rose water (Vishal Personal Care Pvt. Ltd., Hyderabad) was used. Sodium alginate was procured from Nice Chemicals Pvt. Ltd., Kochi, India and Jojoba oil was procured from Sista Organic, Faridabad, India.

Preparation of sodium alginate drug solution

Sodium alginate (3 g) was dissolved in 30 mL distilled water in a beaker by stirring continuously for 30 minutes.

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The ingredients, such as zinc oxide, titanium dioxide, jojoba oil, and Tween® 80, were added in appropriate quantities. A constant stirring was maintained until the entire amount was dissolved[11].

Preparation of jojoba oil enclosed sodium alginate beads

The prepared homogenous mixture was extruded to the ion solution counter 5 % calcium chloride solution through hypodermic syringe (No. 20). The formed microbeads were kept for 30 mins. in counter ion solution to complete gelation reaction. The prepared microbeads were filtered and dried in hot air oven[11].

Preparation of sunscreen gel

To prepare the gel and get uniform mass, 1.1 g of Carbopol® 934 was soaked in water for 5 to 6 h, and incorporated into glycerin, then triethanolamine were added for neutralization and cucumber extract, fragrance and preservatives were added with constant stirring[11].

Incorporation of microbeads in sunscreen gel formulation

The prepared microbeads were added to the sunscreen gel base with continuous stirring until a homogenous transparent jelly was formed as shown in Table I.

Swelling-index

Swelling-index of microbeads indicates the ability of the microbeads to absorb distilled water and expand. For the purpose of determining a swelling index, microbeads were weighed prior to being suspended at 25 mL in an acid buffer pH 7.4. With a stainless-steel grid, the beads were carefully dabbed so that excess surface liquid is eliminated at different periods without pressing hard. A swollen bead was weighed with an electronic microbalance. The investigational study was carried out in triplicate, and the average values were used in data analysis[12,13].

$$\text{Swelling index} = \frac{\text{Weight of wet microbeads}}{\text{Weight of dry microbeads}} \times 100$$

Scanning electron microscopy[12,13]

Particle size, surface morphology and texture were determined using scanning electron microscopy with a scanning microscope Model: JSM series (JSM-IT300LV); manufacturer: JEOL (Japan Electron Optics Laboratory).

Determination of pH

0.5 g of herbal sunscreen gel was weighed and mixed with 50 mL of distilled water. pH of the sunscreen gel was then checked at room temperature using a pH meter[14-17].

Determination of viscosity

The viscosity determinations were carried out for the sunscreen gel with Brookfield viscometer using spindle no. S64 at 50 rpm. The readings were observed and recorded[14-17].

Table I: Formulations

Ingredient	F_1	F_2	F_3	F_4	F_5
Sodium alginate (g)	3	3	3	3	3
Jojoba oil (mL)	0.5 mL	0.5 mL	0.5 mL	0.5 mL	0.5 mL
Tween® 80 (mL)	0.1 mL	0.1 mL	0.1 mL	0.1 mL	0.1 mL
Zinc oxide (g)	1.5	-	1.5	1.5	1.5
Titanium dioxide (g)	-	3	3	3	3
Calcium chloride (g)	5	5	5	5	5
Red raspberry seed oil (mL)	1.5 mL	1.5 mL	1.5 mL	2.0 mL	2.5 mL
Carbopol® 934 (g)	1.1	1.1	1.1	1.1	1.1
Glycerin (mL)	5 mL	5 mL	5 mL	5 mL	5 mL
Triethanolamine (mL)	1 mL	1 mL	1 mL	1 mL	1 mL
Sodium benzoate (g)	0.5	0.5	0.5	0.5	0.5
Rose water (mL)	Q. S	Q. S	Q. S	Q. S	Q. S
Distilled water	100	100	100	100	100

Determination of spreadability

Two sets of glass slides were taken. On one of the slides, 0.5 g of the sunscreen gel formulation was applied. Sunscreen gel was positioned between the two slides, forming a sandwich with gel. For 5 minutes, on the upper slide, a weight of 125 g was inserted, pressing the gel evenly between the two slides to create a thin coating. The circle's diameter was measured as an indication of gel spreadability[14].

Determination of extrudability

The sunscreen gel was placed in a neat and clean, lacquered aluminum collapsible tube. The quantity of cream extruded was measured via the tip for the identification of extrudability.

$$\text{Extrudability} = \frac{\text{Quantity of gel that emerged from the tube}}{\text{Total amount of gel to be filled in the tube}} \times 100$$

Determination of stability studies

Stability evaluation for gel was carried out by placing the sunscreen gel sample at the room temperature and at 40 °C ± 2 °C, 75 ± 5 % RH for 30 days.

Determination of SPF by UV spectroscopic method

Sunscreen gel of 1 g accurately weighed was transferred to a 100 mL volumetric flask and dissolved with water, then subjected to 5 min. of ultrasonication and filtered. 5 mL solution was transferred to a 50 mL volumetric flask and volume was made up with water. Using a 1cm quartz cell and water as a blank, the UV -absorption was measured in the range of 290 nm to 450 nm[18,19].

RESULTS AND DISCUSSION

Swelling index

The swelling index for the sunscreen gel was performed, and percentage of swelling index was found to range from 540 to 720 %, as shown in Table II.

Scanning electron microscopy

The prepared microbeads were subjected to SEM analysis for identification of their particle size and shape. The particle size ranges from 800-1000μm, as shown in Fig. 1.

Table II: Swelling behaviour study

Formulation	Percentage of swelling index
F_1	540
F_2	620
F_3	680
F_4	710
F_5	720

Determination of pH

The pH for the sunscreen gel was determined, and the results ranged from 6.18 to 6.23, as shown in Table III.

Table III: Determination of pH

Formulation	pH
F_1	6.23
F_2	6.20
F_3	6.18
F_4	6.21
F_5	6.22

Determination of viscosity

The viscosity of the sunscreen gel was determined for the formulations and results are shown in Table IV, and the results ranged from 3060 to 3684 cps.

Table IV: Determination of viscosity

Formulation	Viscosity (CPS)
F_1	3288
F_2	3296
F_3	3068
F_4	3060
F_5	3684

Determination of spreadability

The spreadability of the sunscreen gel was performed for the formulations and the results are shown in Table V. The spreadability ranges from 6.7 to 7.2 cm.

Table V: Determination of spreadability

Formulation	Spreadability diameter (cm)
F_1	6.7
F_2	6.9
F_3	7.0
F_4	7.1
F_5	7.2

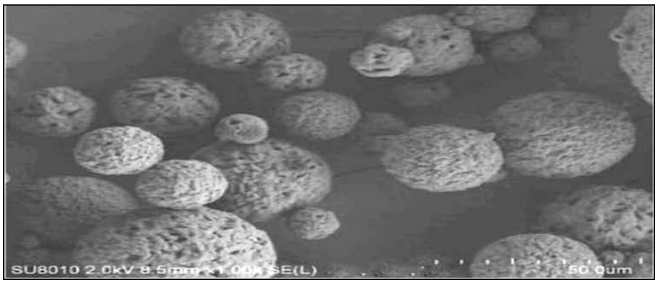

Fig. 1: SEM analysis

Determination of extrudability

The extrudability of the sunscreen gel was performed for the formulations. The results range from 12 to 16 g cm^{-2} as shown in Table VI.

Table VI: Determinations of extrudability

Formulation	Extrudability
F_1	12
F_2	15
F_3	13
F_4	14
F_5	16

Determination of stability studies

Stability studies were performed for the prepared sunscreen gel formulations. The results show no phase separation, and it is stable, as mentioned in Table VII.

Table VII: Determination of stability studies

Formulation	Stability (Room temperature, 40± 2 ˚C, 75 ± 5 % RH)
F_1	Stable and absence of phase separation
F_2	Stable and absence of phase separation
F_3	Stable and absence of phase separation
F_4	Stable and absence of phase separation
F_5	Stable and absence of phase separation

Homogeneity test

Homogeneity test for the sunscreen gel were performed. The results show that they are smooth and homogenous. The results are mentioned in Table VIII.

Table VIII: Tests for homogeneity

Formulation	Homogeneity
F_1	Smooth and homogenous
F_2	Smooth and homogenous
F_3	Smooth and homogenous
F_4	Smooth and homogenous
F_5	Smooth and homogenous

Table IX: Calculation of SPF value for different formulations

Formulation	SPF Value
F_1	1.24
F_2	4.24
F_3	6.75
F_4	9.06
F_5	11.08
F_6 (Marketed formulation)	27.36

Determination of SPF by UV spectroscopic method

The SPF of the sunscreen gel was performed by UV spectroscopic method. The results range from 1.24 to 27.36, as shown in Table IX, since F_5 formulation exhibits higher SPF than F_1, F_2, F_3, and F_4, it is regarded as being superior, whereas F_6 is the marketed product (batch number: MPA167, Manufactured by: Micro Labs Limited, Brand Name: Photoban®-30), on comparison with marketed formulation(F_6), the use of micro-encapsulated method sunscreen gel containing natural ingredients (F_5) provides SPF 11.08.

However, the SPF value is significantly different F_5 vs F_6, indicating that the F_5 formulation prepared by micro-encapsulated sunscreen gel containing herbal components provides 11.08 SPF, which gives maximum 96 % protection from the UV rays, while F_6 marketed product which gives less protection from UV rays even though, it has SPF value greater than F_5. So this result directly shows that the F5 formulation which is made by herbal and micro-encapsulated technique, gives more protection from UV rays.

As a result, sunscreens made from natural compounds using the micro-encapsulated technique can offer the maximum level of protection from broad spectrum UV radiation even in SPF range of 11.08, so natural products have less side effects than chemicals used in prepared sunscreen gels.

DISCUSSION

When compared with chemicals, natural components are generally less hazardous and less harmful to use. The possible natural ingredients such like jojoba oil and red raspberry seed oil are used as sunscreen agents, as they contain the active ingredients which have a preventive effect towards the hazards caused by UV radiation. Swelling index of prepared microbeads F_1, F_2, F_3, F_4 and F_5 shows results in the range from 540 to 740 %. SEM examination was performed to evaluate batch F_5. According to the research, the microbead's nearly globular structure and rougher exterior strengthen it's capacity to release medications through the formation of channels. pH in the formulation of all five batches ranged from 6.20 to 6.23, respectively. In this research investigational examination, F_1 to F_5 the viscosity was ranging from 3060-3684 cps, meeting the requirements of sunscreen preparations. Spreadability is the way in which a gel is distributed when applied to skin, and the therapeutic efficacy of a gel is related on this spreadability, F_1 to F_5 were able to spread in the range of 6.7-7.2. Extrudability study gave good results for all five batches and ranged to 12- 16. Homogeneity test results for all five formulations indicated smooth and homogenous nature of the formation SPF for each batch formulation F_1, F_2, F_3 showed 1.24, 4.24 and 6.75, whereas F_4, and F_5 showed 9.08 and 11.08 respectively, indicating that F4 & F5 formulations have more sunscreen activity.

The usage of natural ingredients with the micro-encapsulated technique (F_5) provides SPF 11.08, which is approximately up to 96 % protection, and the formulation F_5 demonstrates greater SPF than F_1, F_2, F_3, and F_4. Even sunscreens made from natural substances with a micro-encapsulated technique can provide 96 % protection from broad spectrum UV radiation even in small SPF ranges.

CONCLUSION

Sunscreen gel formulations containing batch F_5 with a combination of titanium dioxide and zinc oxide are potentially and effectively active as sunscreens with SPF values of 11.08. All five sunscreen gel formulas meet the physio - chemical parameters and are stable at 40° C for 30 days. The most preferred formula is a sunscreen gel containing jojoba oil, red raspberry oil, titanium dioxide, and zinc oxide.

Herbal sunscreen gel for sun protection that met the relevant pharmaceutical characteristics was successfully developed. During the study period, the constructed formulations showed significant consistency, excellent spreadability and absence of evidence of phase separation. Throughout the course of the investigation, there was no discernible change in the formulations' visual characteristics, nature, pH and viscosity. pH value of the prepared formulations was approximately pH 6; this confirms compatibility of the formulations with the skin's secretions.

During a stability study at 30 days, sunscreen gels were found stable. Based on the findings of this study, it is possible to create a micro-encapsulated sunscreen gel containing herbal oil of red raspberry seed oil and jojoba oil that can be used to provide a barrier to protect skin from sunlight.

The sunscreen that was prepared with the micro-encapsulated technique was compared with the normal sunscreen that was available on the market.

However, the SPF value is notably different between F5 and F6, showing that the F5 formulation, made from micro-encapsulated sunscreen gel containing herbal ingredients, provides 11.08 SPF, giving the maximum 96% protection from UV rays, while the F6 marketed product, even though it has an SPF value greater than F5, provides less protection from UV rays. Thus, the F5 formulation, which is manufactured using a herbal blend and a micro-encapsulation technology, provides greater UV protection than commercial formulations with SPF values around 30.

The microencapsulated sunscreen gel, which also incorporated herbal components, was found to be more effective than the marketed formulation. Moreover, the herbal products are known to have fewer skin adverse effects and thus help to improve patient compliance.

REFERENCES

1. Dupont E., Gomez J. and Bilodeau D.: Beyond UV radiation - A skin under challenge, **Int. J. Cosmet. Sci.**, 2013, 1-9.
2. Freitas S., Merkle H. P. and Gander B.: Microencapsulation by solvent extraction/evaporation: reviewing the state of the art of microsphere preparation process technology, **JCR**, 2005, 102(2), 313-332.
3. Raghavamma S. T. V., Anjali M. V., Amrutha N., Hemalatha T. and Nadendla R. R.: Assessment of efficacy and safety of newly formulated natural sun protective cream, **IOSR-JPBS**, 2016, 11(5), 119-124.
4. Smaoui S., Hlima H. B., Chobba I. B. and Kadri A.: Development and stability studies of sunscreen cream formulations containing

three photo-protective filters, **Arab. J. Chem**, 2017, 10(1), 1216-1222.

5. Handali S., Hosseini H., Ameri A. and Moghimipour E.: Formulation and evaluation of an antibacterial cream from *Oxalis corniculata* aqueous extract, Jundishapur, **J. Microbiol**, 2011, 4(4), 255-260.
6. Das M. K., Ahmed A. B. and Saha D.: Microsphere a drug delivery system–a review, **Int. J. Curr. Pharm**, 2019, 11(4), 34-41.
7. Ngoc L. T. N., Tran. V. V., Moon Ju-Y., Chae M., Park D. and Lee Y-C.: Recent trends of sunscreen cosmetic: An update review, **MDPI**, 2019, 6 (64), 1-14.
8. Dutra E. A., Oliveira D., Kedor-Hackmann E. R. M. and Santoro M. I. R. M.: Determination of sun protection factor (SPF) of sunscreens by ultraviolet spectrophotometry, **BJPS**, 2004, 40(3), 381-385.
9. Singh M., Dua J. S., Menra M., Soni M. and Prasad D. N.: Microencapsulation and its various aspects: a review, **IJAR**, 2016, 4(6), 2094-2108.
10. Bhattacharjee D., Preethi S., Patil A. B. and Jain V.: A comparison of natural and synthetic sunscreen agents: A review, **Int. J. Pharm. Res.,** 2021, 13(1), 3494-3505.
11. Marodkar R. and Pardeshi M.: Herbal sunscreen gel with microbeads technology, **IJASRW**, 2020, 3(3), 31-38.
12. Shivhare U. D., Mathur V. B., Shrivastava C. G. and Ramteke V. I.: Preparation of microbeads by different techniques and study of their influence on evaluation parameters, **J. Adv. Pharm. Res.**, 2013, 3(3), 278-288.
13. Shetty P. K., Venuvanka V., Jagani H. V., Chethan G. H., Ligade V. L., Musmade P. B., Nayak U. Y., Reddy M. S., Kalthur G., Udupa N., Rao C. M. and Mutalik S.: Development and evaluation of sunscreen creams containing morin-encapsulated nanoparticles for enhanced UV radiation protection and antioxidant activity, **Int. J. Nanomedicine**, 2015, 1(1), 6477–6491.
14. Yadhav H. K. S., Usman S., Alabdin R. H. E. Z., Ahmed H. S. and Usman H. A.: Formulation and evaluation of topical gel using avocado extract, **AJPHR**, 2020, 8(8), 2321-3647.
15. Aprilita R. Y. E., Rahayu S. T., Saraswati H. And Munim A.: Formulation and evaluation of sunscreen gels containing mangiferin isolated from *Phaleria macrocarta* fruits, **Int. J. Pharm. Investig.**, 2019, 9(3), 141-145.
16. Rasheed A., Shama N. S., Mohanalakshmi S. And Ravichandran V.: Formulation, characterization and *in vitro* evaluation of herbal sunscreen lotion, **Orient. Pharm. Exp. Med.,** 2012, 12, 241–246.
17. Pachpawar N. G., Mahajan U. N. and Kharwade R. S.: Formulation and evaluation of sun protective topical preparation, **Int. Res. J. Pharm.**, 2018, 9(2), 2230 – 8407.
18. Amnuaikit T. and Boonme P.: Formulation and characterization of sunscreen creams with synergistic efficacy on SPF by combination of UV filters, **J. Appl. Pharm. Sci.**, 2013, 3(8), 1-5.
19. Kaur C. D. and Saraf S.: *In vitro* sun protection factor determination of herbal oils used in cosmetics, **Pharmacogn. Res.**, 2010, 2(1), 22-25.