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Research

Formulation and Evaluation of Sunscreen Nanoemulsion on Sunflower Oil (*Helianthus annus*)

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Abstract

The study involved the creation of a Nanoemulsion sunscreen using sunflower oil, which contains bioactive compounds with various pharmacological benefits, such as anti-bacterial properties and improved antioxidant effects. The Nanoemulsions were prepared using sunflower oil through a spontaneous emulsification method with three different formulations (F1 (Tween80 8%,sorbitol8.8%), F2 (Tween 80 7.2%, sorbitol8%), F3 (Tween80 6.4%,sorbitol7.2%)).These Nanoemulsions were then evaluated for their physical and chemical properties, solubility, stability, pH, viscosity, percentage drug content, FT-IR and UV analysis, as well as SPF value determination. The results showed that the Nanoemulsion sunscreen remained stable during a 4-week storage period at room temperature, low temperature, and high temperature. Furthermore, the Nanoemulsion preparation exhibited higher SPF values compared to other formulations, indicating its potential effectiveness as a sunscreen for cosmetic use.

Keywords: Sunflower oil, Nanoemulsion, Sunscreen, SPF, Spontaneous emulsification method

INTRODUCTION

Nanoemulsion

Nanoemulsions are also appertained to as miniemulsions, ultrafine mixes, and submicron mixes. Phase geste studies show that the surfactant phase structure (bicontinuous microemulsion or lamellar) at the inversion point caused by either composition or temperature determines the size of the driblets. Exploration on the creation of nanoemulsions using the phase inversion temperature system has shown a relationship between the lowest drop

size and total oil painting solubilization in a microemulsion bicontinuous phase, anyhow of whether the original phase equilibrium is single or multiphase[1]. The oil in- water (O/ W) type of nanoemulsions was first developed, with drop sizes ranging from 50 to 1000 nm on average. [2-3].

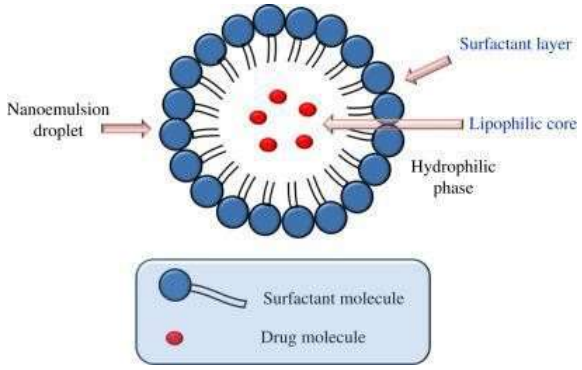


Fig 1: Structure of Nanoemulsion

Sunscreen

Sunscreen is a liquid or cream that is applied to the skin to shield it from the sun's damaging rays and avoid sunburn. But in the United states, the phrase "sunburn lotion" usually refers to lotion that maximizes tanning and UV exposure by moisturizing rather than blocking it[4]. Sunscreen, sometimes referred to as suntan lotion, sun-cream, or sun block, is a type of topical product that helps prevent sunburn by reflecting or absorbing part of the UV radiation from the sun. It can be applied as a lotion, spray, gel, or foam stick. Now a days, a particular type of cosmetic product called sunscreen has become very popular because it offers health benefits in addition to aesthetic ones[5].

Three forms of ultraviolet (UV) radiation make up the harmful solar radiation: UV A, UV B, and UV C.

UV C light is filtered by the ozone layer and does not reach the earth's surface, it is not too much of a skin concern. Burning hot spots on the skin are caused by UVB radiation. The deeper layers of the epidermis and dermis are exposed to UVA radiation, which prematurely ages the skin. Skin cancer has been linked to ultraviolet radiations as a causal factor. UV filter materials (sunscreens) can be further divided into UVA, UVB, and broad-spectrum protection (UVA and UVB) filters based on their ability to absorb shorter or longer wavelengths. The most popular method of preventing UV radiation-induced skin damage is topical sunscreen application[6].

Aim and objective

Aim

- Plant source are rich in Phytochemicals. Active phytochemical such as flavonoids, polyphenols, saponins, tannins and vitamins are known to responsible for the sunscreen activity.
- The preliminary review of literature indicates that ***Helianthus annuus*** were rich in sunscreen phytochemicals.
- Hence the aim of the present study is to the formulation and evaluation of sunscreen nanoemulsion on ***Helianthus annuus*.**

Objective

- Preliminary phytochemical analysis
- Formulation and Evaluation of Nanoemulsion
- Determination of the Sun Protection Factor(SPF)Value

MATERIALS AND METHODS

Materials

Table1: List of Raw Materials and their Suppliers

S.No	Name of the Raw Material	Name of the Supplier
1	Sunfloweroil	Yarrow Chem Products,Mumbai
2	Tween80	Oxford Lab.Science Ltd,Maharashtra.
3	Sorbitol	Molychem Product,Mumbai.
4	MethylParaben	Yarrow Chem Products,Mumbai

5	PropylParaben	Yarrow Chem Products,Mumbai
6	Aqua demineralized	Medilise Chemicals,Kerala.

Phytochemical analyis[7-9]

Carbohydrates

10 ml of distilled water were mixed with one gram of the extract. After boiling this extract in a test tube with Fehling solutions A and B, color changes were noted. The presence of carbohydrates was indicated by the color brick red.

Alkaloids

In a steam bath, 2 ml of extract and 6 ml of 1% HCl were combined, and the mixture was filtered. Wagner's reagent (1 ml) was added, then thoroughly combined. The presence of alkaloids is indicated by the presence of turbidity and a reddish color.

Flavanoids

After mixing 2 ml of extract with 5 ml of diluted ammonia, two to three drops of NaOH were added. The appearance of yellow indicates the presence of flavonoids.

Tannins

To confirm the presence of tannin, 0.5 g of the extract was dissolved in 10 ml of distilled water. A few drops of 1% ferric chloride solution were then added, resulting in a brownish green or blue-black precipitate.

Glycosides

2.5 ml of distilled water and 2.5 g of extract were combined. After adding 0.5 ml of concentrated sulfuric acid, 1 ml of glacial acetic acid containing a few drops of ferric chloride was added. The formation of a reddish brown layer verifies the existence of glycosides.

Saponins

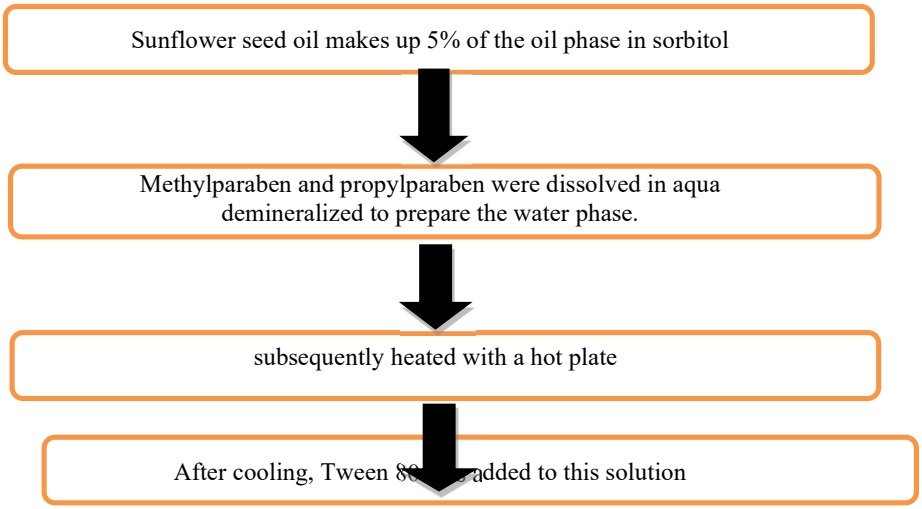
5 ml of distilled water were used to dissolve 0.5 g of the extract. The mixture was given a good shake. Saponins are present when stable, persistent froth forms. Six more drops of olive oil are added and shook to create an emulsion, which verifies the presence of saponins.

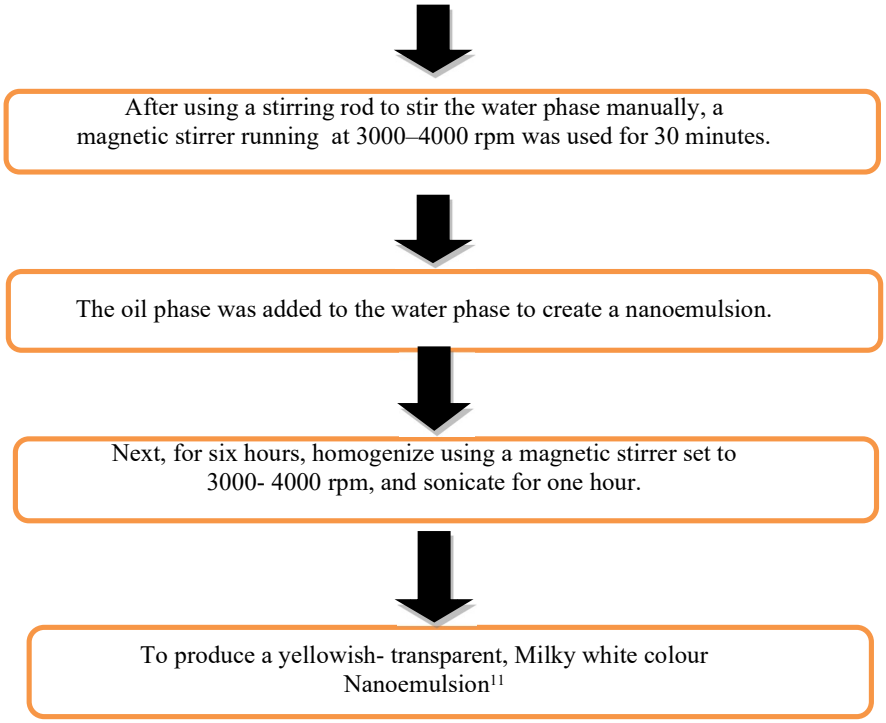
Phenols

4 ml of distilled water were mixed with 2 ml of extract, and a few drops of 10% FeCl3 were added. Green or blue coloration suggests the presence of phenols

Preparation of sunscreen nanoemulsion

Different concentrations of sorbitol and Tween 80 were used to create the nanoemulsion. Three formulas: F1 (tween 80 38%, sorbitol 22%), F2 (tween 80 36%, sorbitol 24%), and F3 (tween 80 34%, sorbitol 26%), each have varying concentrations. The spontaneous emulsification method was used to create the nanoemulsions. A water phase and an oil phase make up an emulsion system. The oil phase was added to the water phase dropwise using the spontaneous emulsification technique.[10]





Formulation of sunscreen nanoemulsion

Table2: Ingredients for Formulation of Sunscreen Nanoemulsion

S.No	Ingredients	Quantity of 30ml(%)		
		F1	F2	F3
1	Sunflower oil	2	2	2
2	Tween80	8	7.2	6.4
3	Sorbitol	8.8	8	7.2
4	Methyl Paraben	0.04	0.04	0.04
5	PropylParaben	0.008	0.008	0.008
6	Aqua demineralized	30ml	30ml	30ml

Table3: Formulation of Sunscreen Nanoemulsion

S.No	Formulation	Surfactants mixture (ratio)	Oil/Surfactants mixture (ratio)	% w/w of components in Nanoemulsion formulation			Drug % w/w
				Oil	S. Mix	Water	
1	F1	1:1	1:5	5.00	30.00	35	1
2	F2	1:1	1:4	5.00	25.00	30	1
3	F3	1:1	1:3	5.00	25.00	30	1

Evaluation of sunscreen nanoemulsion:[12-18]

Physicochemical Parameters

The Sunscreen Nanoemulsion was evaluated various physicochemical parameters such as physical appearance (Colour, Odour and Nature)

Colour Examination

5ml of Nanoemulsion was taken into watch glass and placed against white background in white tube light. It was visually assessed for its colour.

Odour Examination

Two ml of Nanoemulsion was smelled individually. To minimize the impact of the previous smelling, the time gap between two sniffs was kept at 2 minutes.

Spreadability

It can be determined by applying the sunscreen nanoemulsion slowly on the skin. Skin feeling should be noted.

Melting Point Analysis

A Melting Point of the sample is determined by using Capillary Method.

Solubility

The solubility of nanoemulsion sunscreen is determined by using various solvents.

Thermodynamic stability

The selected formulation is subjected to different thermodynamic stability tests.

Measurement of pH

pH of various nanoemulsions formulations are determining by using digital pH meter.1 gm of nanoemulsion is dissolved in 100 ml of distilled water and pH was measured. The measurement of formulation is done in triplicate to avoid error.

Viscosity determination

Viscosity of nanoemulsion is determined by using Brookfield viscometer. 20 ml of nanoemulsion is filled in a 25ml beaker and the viscosity is measured using spindle number 6 at10 rpm.

Percentage drug content

1 ml of nanoemulsion is mixed with 10 ml of suitable solvent. Aliquots of different concentration are prepared and by using suitable dilutions after filtering the stock solution; absorbance is measured by UV spectroscopy. Drug content is calculated by using the equation obtains from linear regression analysis of calibration curve.

Invitro Diffusion studies

The diffusion studies of the prepared nanoemulsions are performed by using Franz diffusion cell with the aid of cellophane membrane. Nanoemulsion sample (5ml) is taken in cellophane membrane and the diffusion studies are carried out at 37 ± 1 °C using 250 ml of(25%) methanolic phosphate buffer (pH 7.4) as the dissolution medium. 5 ml of each sample was withdrawn periodically at 1, 2, 3, 4, 5, and 6 hrs and each sample is replaced with equal volume of resh dissolution medium in order to maintain sink condition. Samples are analyzed by UV-spectrophotometer at 271 nm for drug content.

Determination of drug- excipients compatibility study

Fourier Transform infrared Spectroscopy is the best method to evaluate the drug excipient in compatibility test. The FT-IR spectrum of Sunflower oil Nanoemulsion sunscreen was compared with FT-IR spectrum of physical mixture of Tween 80, Sorbitol, Methyl Paraben, Propyl Paraben and Water demineralized. Spectra were recorded within the rangefrom4000 to 500 cm^{-1}.

Stability Studies of Nanoemulsion

stability of nanoemulsions at low and high temperatures was evaluated using nanoemulsion stored at low temperature (4 ± 2°) in the refrigerator for 24 hours, then directly stored in high temperature (40 ± 2°C) in the climatic chamber for another 24 hours (1 cycle).This test was donewith6 cycles of repetition and then observed visually.

Determination of spf value[19]

The effectiveness of a sunscreen is usually expressed by Sun Protection Factor (SPF) which is the ratio of UV energy required to produce a Minimal Erythemal Dose (MED) in protected skin to unprotected skin. A simple, rapid and reliable in vitro method of calculating the SPF is to screen the absorbance of the product between 290-320 nm at every 5 nm intervals. SPF can be calculated by applying the following formula

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

Where,

CF = Correction factor(10).

EE (λ = Erythmogenic effect of radiation with wavelength λ.

Abs (λ) = Spectrophotometric absorbance values at wavelength λ.

The values of EE xλ are constants.

RESULTS AND DISCUSSION

Phytochemical analysis

Table 4: Phytochemical Analysis of Helianthus annuus

S.No	Chemical Constituents	Results
1	Carbohydrates	+
2	Alkaloids	+
3	Flavanoids	+
4	Tannins	+
5	Glycosides	-
6	Saponins	+
7	Phenols	+
8	Proteins	-

Note: (+)Present (-)Absent

Evaluation parameters on nanoemulsion

Organoleptic Properties

Table 5: Organoleptic Characteristics of Sunscreen Nanoemulsion

S.No	Test	Observation	Inference
1	Colour	Milky White	Passes
2	Odour	Odorless	Passes
3	Nature	Amorphous	Passes

Spreadability

The Spreadability is good and sticks well on skin.

Melting Point Analysis

The melting point of Nanoemulsion Sunscreen was observed to be 171.5ºC which complies with melting range of standard 170 -175ºC.

Solubility

Solubility of Nanoemulsion sunscreen was found to be in different solvents are given below-

Table 6: Solubility analysis of Sunscreen Nanoemulsion

S.No	Solvent System	Specification as per USP	Result
1	Sunflower Oil	Insoluble	Insoluble
2	Tween80	Soluble	Soluble
3	Sorbitol	Very soluble	Very soluble
4	MethylParaben	Soluble	Soluble
5	PropylParaben	Soluble	Soluble
6	Water Demineralised	Slightly soluble	Slightly soluble

Thermodynamic stability

Table 7: Thermodynamic stabilities Sunscreen Nanoemulsion

Formulations	Heating Cooling Cycle	Centrifugation
F1	Stable	Phase separation formed
F2	Stable	No phase separation
F3	Stable	No phase separation

pH, Viscosity and % drug content

Table 8: pH, Viscosity and %drug content of Sunscreen Nanoemulsion

Formulations	pH	Viscosity(cp)	Drug content%
F1	8.5	497.87	95.99%
F2	7.40	985.02	96.71%
F3	7.85	626.35	96.37%

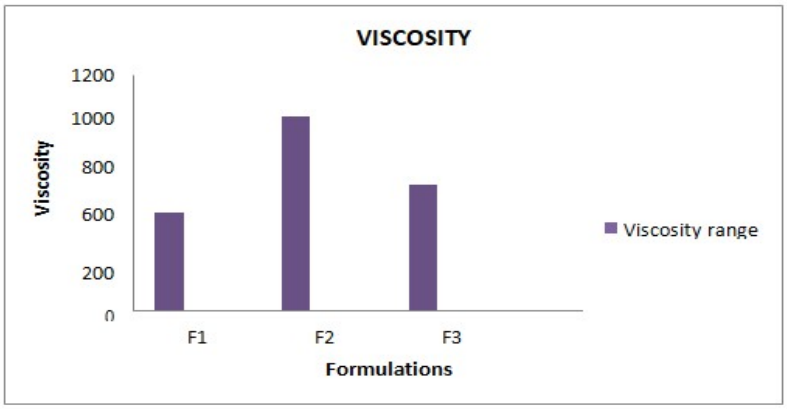


Fig 2: Viscosity of nanoemulsion formulation

- pH of prepared nanoemulsion formulations was found in the range of 7.40–8.53
- The percentage drug content of prepared nanoemulsion formulation was found to be 95 to 97%.
- The mean average viscosity was found to be 700 to 750cp, F2 batch shows highest viscosity.

In-vitro drug release

Table 9: In-vitro drug release (in %)

Time in hrs	F1	F2	F3
0.5	3.62	8.20	5.72
1	9.54	14.11	7.94
1.5	20.42	25.49	16.04
2	28.56	38.25	27.15
2.5	40.02	52.72	40.33
3	51.53	59.54	50.02
3.5	67.18	69.08	64.21
4	71.19	75.22	71.12
4.5	76.35	85.12	76.41
5	82.08	90.21	81.63
5.5	90.75	94.65	90.04
6	96.10	98.08	95.20

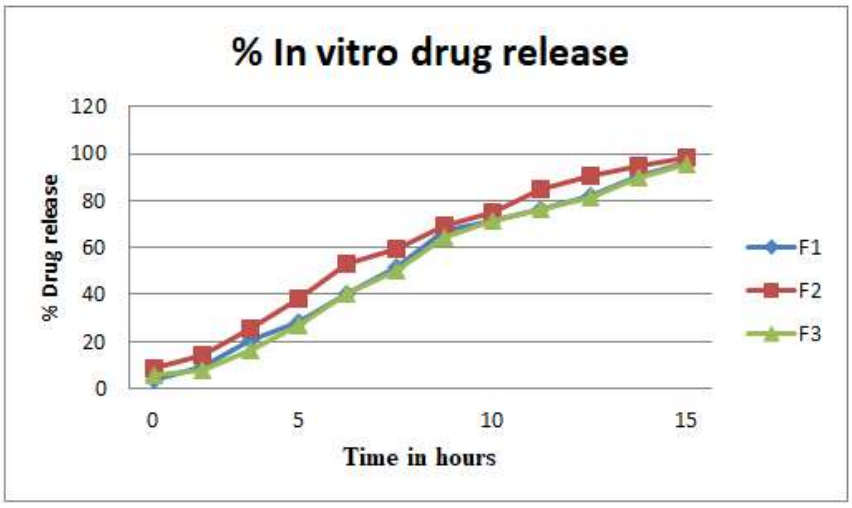


Fig 3: *In-vitro* Drug Release

Result of *in-vitro* drug release from different formulations are tabulated in table 12and graphically shown in fig.3.The prepared formulation batch F2 shows the better release profile as compared to other preparation F1 and F3.

Identification of drug through UV spectroscopy

Standard calibration curve of Nanoemulsion Sunscreen using Sunflower Oil as pure drug in Methanol:

Table 10: Standard calibration curve of Sunflower oil

Concentration (µg/ml)	Absorbance (nm)
2	0.206
4	0.442
6	0.673
8	0.896
10	1.108
12	1.312

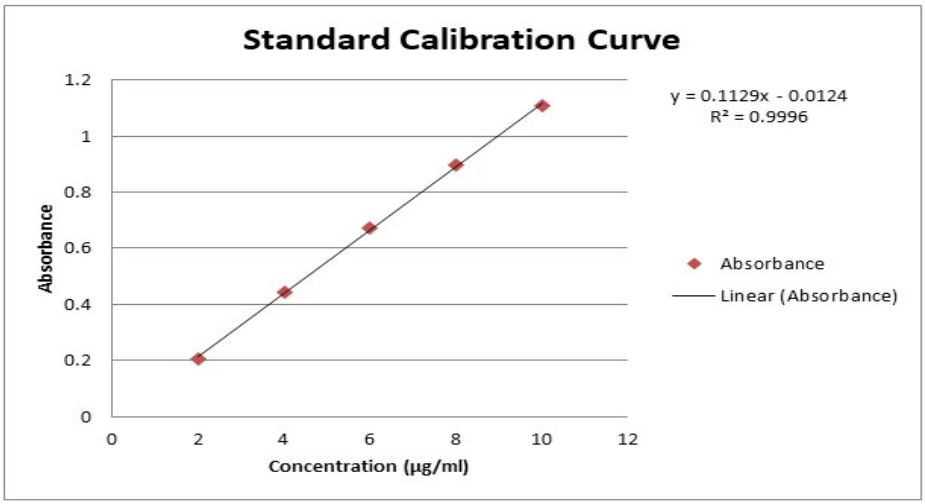
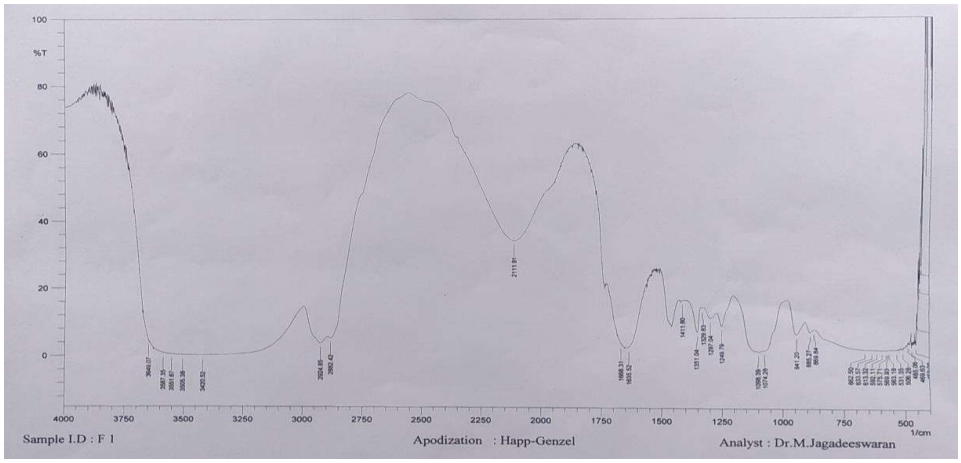


Fig 4: Standard calibration curve of Sunflower oil in methanol

Determination of drug-excipients compatibility study



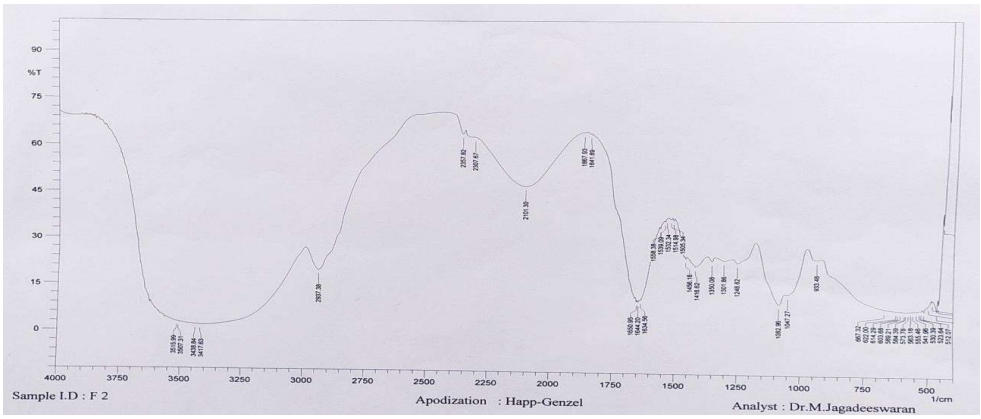


Fig 5: FTIR Analysis of Nanoemulsion formulation (F1)

Fig 6: FTIR Analysis of Nanoemulsion formulation (F2)

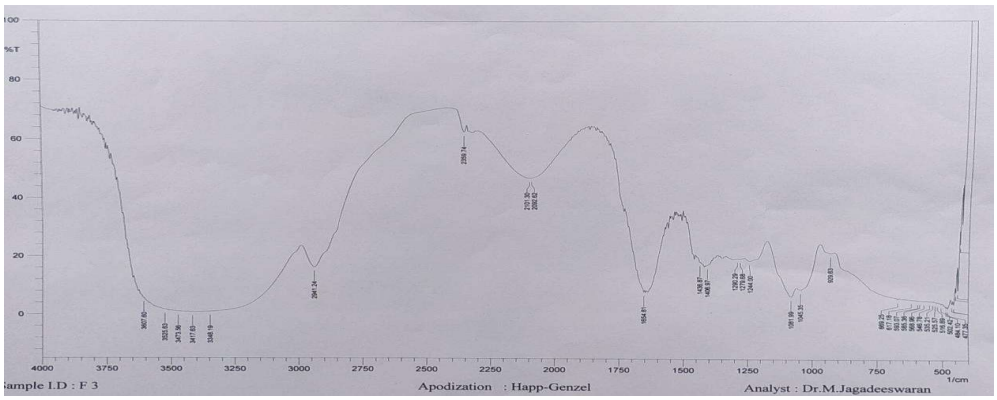
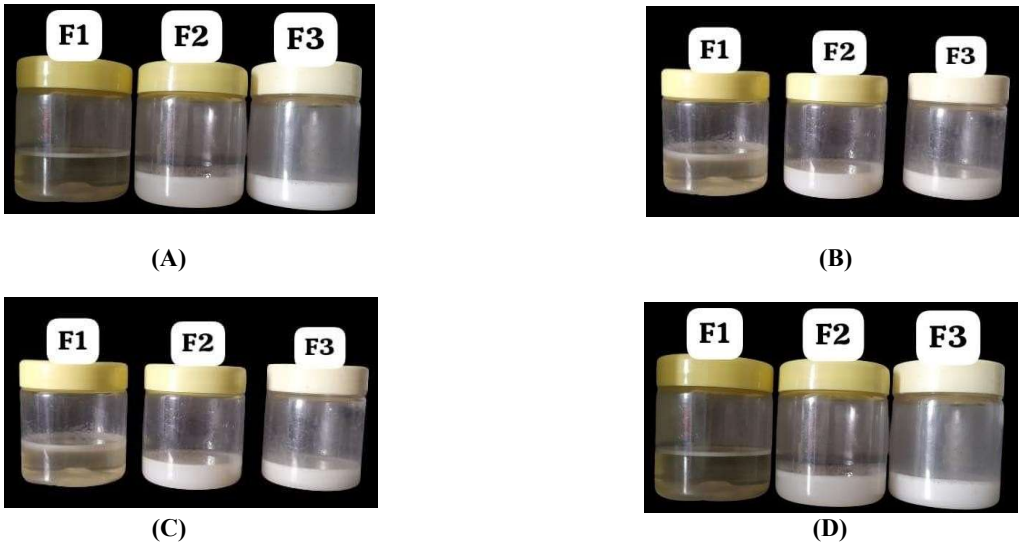


Fig 7: FTIR Analysis of Nanoemulsion formulation (F3)

The FT-IR spectrum of Sunflower oil Nanoemulsion sunscreen was compared with FT-IR spectrum of physical mixture of Tween 80, Sorbitol, MethylParaben, PropylParaben, water demineralized. There was no apperance or disappearance of any characteristics peaks. This shows that there is no chemical interaction between the drug and the surfactant used in the nanoemulsion .The presents of peaks at the excepted range confirms that the materials taken for the study are genuine.

Stability Studies of Nanoemulsion



(A) **(B)**

(C) **(D)**

Fig 8: The results of the stability of nanoemulsions F1, F2, F3 containing sunflower oil

The stability of prepared sunscreen nanoemulsions (F1,F2,F3)containing Sunflower oil

A. Before storage
B. After storage for4 weeks at room temperature
C. After storage for4 weeks at high temperature
D. After storage for4 weeks at low temperature

The **results** of physical stability test of nanoemulsions were clear and transparent (stable) during the test, there were slightly change colour or phase separation.

Determination of spf value

Table 11: SPF Value of Sunscreen Nanoemulsion

S.No	Formulations	SPF Value			
		I	II	III	Average SPF value
1	F1	3.72	4.14	3.88	4.44 ± 0.03
2	F2	4.18	4.29	4.72	4.08 ± 0.16
3	F3	3.82	3.84	4.49	4.09 ± 0.36

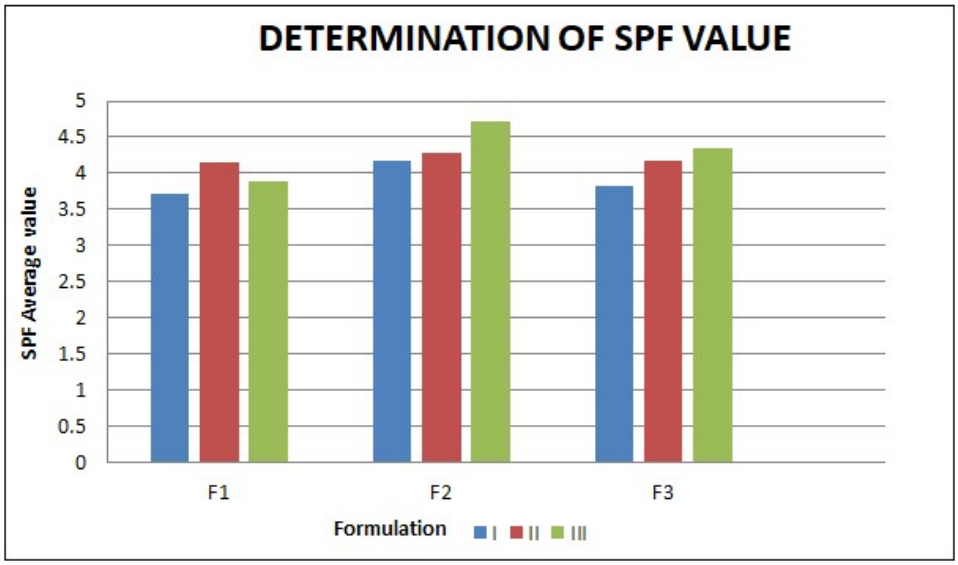


Fig 9: Determination of the SPF Value

SUMMARY AND CONCULSION

The present work concluded that sunflower oil containing nanoemulsion formulation for solubility enhancement was successfully prepared by the spontaneous emulsification method. Nanoemulsion is more acceptable for the topical drug administration. Sunflower oil, Tween 80 (Surfactant), Sorbitol (Co-surfactant) was successfully used as a suitable carrier system for incorporating sunflower oil for topical drug delivery.

Various formulation (F1, F2, F3) were prepared as per the composition and drug loaded in Nanoemulsion. The prepared formulation of F2 shows the better release profile than others. From above result concluded that nanoemulsion drug delivery system can be effective for topical application of Sunscreen is commonly used for avoid skin damage caused by ultraviolet radiation. Further study on an animal being need to perform before its commercial use.

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