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Preparation and Evaluation of Sunflower Oil Nanoemulsion as a Sunscreen

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Abstract

BACKGROUND: There are a lot of different types of sunscreen products (oils, sticks, gels, creams, lotions) which can be found on the world's market. Sunscreen product that contains active chemical ingredients sometimes has harmful effects on the skin. Sunflower oil contains vitamin E and acts as a natural sunscreen which can absorb UVB light. The average droplet size of nanoemulsion is between 100 and 500 nm and do not show the problems of stability (creaming, flocculation, coalescence, and sedimentation), which are commonly associated with macroemulsions.

AIM: The aim of this study was to prepare and evaluate the sunflower oil nanoemulsion as a sunscreen.

METHODS: Sunflower oil nanoemulsions were prepared by spontaneous emulsification method with 3 formulas F1 (Tween 80 38%, sorbitol 22%), F2 (Tween 80 36%, sorbitol 24%), F3 (Tween 80 34%, sorbitol 26%) and 5% sunflower oil as a sunscreen substance. The nanoemulsions were evaluated for particle size, physical stability in room temperature (25 ± 2°C), low temperature (4 ± 2°C) and high temperature (40 ± 2°C) during experiment for 12 weeks of storage, centrifugation at 3750 rpm for 5 hours, viscosity, pH, freeze-thaw test and sun protection value (SPF) value by in vitro.

RESULTS: The results of nanoemulsion evaluation showed that nanoemulsion formula F1 had the smallest average particle size of 124.47 nm with yellowish colour, clear, transparent, pH value (6.5 ± 0.1), viscosity value (225 ± 25 cP), did not show any separation or creaming in the centrifugation, and stable during experiment for 12 weeks of storage at room temperature, low temperature and high temperature. The SPF value of all nanoemulsion preparations was higher than that of the emulsion.

CONCLUSION: The preparation of the sunflower oil nanoemulsion with a ratio of Tween 80 and sorbitol (38: 22) produces a stable nanoemulsion during the experiment for 12 weeks storage at the room, low and high temperature. The nanoemulsion preparation has higher SPF values compared to the emulsion. This nanoemulsion formulation could be considered more effective in sunscreen cosmetic use compare to the emulsion.

Introduction

The harmful solar radiation consists of three types of ultraviolet (UV) radiation: UV A, UV B, and UV C. UV C are not too much of a skin problem because the light is filtered by the ozone layer and does not reach the earth's surface. UVB radiation is responsible for skin damage due to sunburn. UVA radiation reaches the deeper layers of the epidermis and dermis and causes the premature ageing of the skin. Ultraviolet radiations have been implicated as a causative factor for skin cancer. Depending on their capacity to absorb shorter or longer wavelengths, UV filter substances (sunscreens) can be subclassified into UVA filters, UVB filters and filters for broad-spectrum protection (UVA and UVB) [1]. Topical application of sunscreen is most commonly used to avoid skin damage caused by ultraviolet radiation.

Sunflower oil is an oil that is obtained from sunflower seeds by pressing, cold pressing method or extraction with a suitable solvent such as hexane [2]. The composition of sunflower oil is linoleic acid (66%), oleic acid (21.3%), palmitic acid (6.4%), arachidonic acid (4%), stearic acid (1.3%) and behenic acid (0.8%) [3]. The sunflower oil also contains Alpha-tocopherol concentration of 609 mg/kg [4]. Alpha-tocopherol (Vitamin E) in cosmetics acts as an antioxidant and protect against UV radiation. Alpha-tocopherol is very effective against UV-B free radical damage. Vitamin E absorbs strongly in the UV-B region of 280-320 nm [5]. The marketed sunscreens are usually in forms of creams, gels and lotions. Stability of the preparation obtained during the time and shelf life and at extreme climates is contemporarily a matter of efficacy and consumers safety [6].

Nanoemulsion can be defined as oil-in-water (o/w) emulsions with mean droplet diameters ranging from 50 to 1000 nm. Usually, the average droplet size is between 100 and 500 nm. The particles can exist as water-in-oil and oil- in water forms, where the core of the particle is either water or oil, respectively [7]. The nanoemulsion in this study formed by spontaneous emulsification method. The smaller droplet size of emulsions not only suppresses the coalescence or coagulation of emulsion droplets but also suppresses the precipitation of emulsions and also helps to deliver the active agents. Therefore, nanoemulsion is very useful in cosmetics as it has good stability to separation, transparent visual aspect and a small particle size, which makes a high surface area that allows absorption of the active ingredients to the specific area and hence will provide higher sunscreen activity [8], [9], [10]. Sunscreens containing rambutan (Nephelium lappaceum L.) fruit peel extracts with high SPF values in the form of nanoemulsion gel have been studied [11].

This study aimed to prepare sunflower oil nanoemulsion by spontaneous emulsification method, and to evaluate their stability and to determine Sun Protecting Factor (SPF) value by *in vitro* using spectrophotometric methods. In vitro testing method have been developed because they are usually used for the rapid control quality test.

Material and Methods

The nanoemulsion was formulated using sunflower oil (PT. Lam Soon, Malaysia), Tween 80, sorbitol; water demineralised were purchased from PT. Bratachem, methylparaben, propylparaben, propylene glycol, sodium CMC, and glycerin were purchased from CV. Rudang Jaya.

Nanoemulsion was formulated by using variations of Tween 80 and sorbitol concentration. The nanoemulsions were formulated by using spontaneous emulsification method [12]. Emulsion system consists of an oil phase and water phase. Spontaneous emulsification technique was done by adding the oil phase to water phase dropwise. Oil phase consists of sunflower seed oil 5% in sorbitol while the water phase was prepared by dissolving methylparaben and propylparaben in aqua demineralised, then heated by using a hot plate. This solution was then cooled down and added with Tween 80. This water phase then stirred manually using stirring rod and continued with a magnetic stirrer at 3000-4000 rpm for 30 minutes. Nanoemulsion was obtained by adding the oil phase into the water phase, then homogenised with a magnetic stirrer at 3000-4000 rpm for 6 hours and sonicated for 1 hour to obtain a transparent, yellowish colour Nanoemulsion.

The oil phase of the emulsion consisted of sunflower oil and span 80. The water phase was prepared by dissolving methylparaben and propylparaben in aqua demineralised, then heated by using a hot plate. This solution was then cooled down and added with Tween 80, propylene glycol and glycerin. This water phase then stirred manually using a stirring rod. The water phase was then added to the sodium CMC and was stirred quickly to avoid the formation of air bubble. The oil phase was then added to the mixture, then stirred until an emulsion was produced.

Evaluation of stability by visual observation, including consistency, odour, colour, and phase separation. All formulas of nanoemulsion and emulsion were stored in a room temperature (25 ± 2°C), low temperature (4 ± 2°C) and high temperature (40 ± 2°C) and evaluated in 0 weeks and 12 weeks.

Determination of nanoemulsions globule size was done by using Vasco$^{\gamma}$ CORDOUAN Technologies Particle Size Analyzer at room temperature for 0, 6, 12 weeks.

Centrifugation test of nanoemulsions and emulsion were done by using a centrifuge (Hitachi CF 16 R X II) at 3750 rpm for 5 hours and observed for phase separation.

Determination of the pH of the nanoemulsion is carried out by using a pH meter (Hanna) and viscosity by using viscometer Brookfield DV-E with specific spindle (spindle 62) after the nanoemulsion was storage for 0, 4, 8, and 12 weeks at room temperature.

The stability of nanoemulsions at low and high temperatures was evaluated using nanoemulsion stored at low temperature (4 ± 2°C) 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 done with 6 cycles of repetition and then observed visually.

The SPF value was determined by weighing one gram of sunscreen preparation, then diluted in ethanol 96% at a final concentration of 200 ppm and analysed by UV Spectrophotometry (Shimadzu) from 290-320 nm with an interval of 5 nm and 10 nm with the interval from 320-400 nm [13]. The SPF determination which is the correlation between the erythemogenic effect (EE) and the radiation intensity at each wavelength (I) and are adjusted according to Eq:

$$SPF = CF \times \sum_{320}^{290} \mathrm{Abs} \times \mathrm{EE} \times \mathrm{I}$$

Where the correction factor (CF) is 10, EE is the erythemogenic effect of radiation on wavelength, I am the intensity of solar light at each wavelength, and Abs is absorption value from the sample.

Results

The nanoemulsions were prepared in 3 formulas, F1 (Tween 80 38%, sorbitol 22%), F2 (Tween 80 36%, sorbitol 24%), F3 (Tween 80 34%, sorbitol 26%). The composition of nanoemulsions containing sunflower oil was shown in Table 1. All the nanoemulsions were yellowish, clear dan transparent.

Table 1: Composition of nanoemulsions containing sunflower oil

Material	Quantity of 100 mL (%)		
	F1	F2	F3
Sunflower oil	5	5	5
Tween 80	38	36	34
Sorbitol	22	24	26
Methylparaben	0.1	0.1	0.1
Propylparaben	0.02	0.02	0.02
Aqua demineralized	Up to 100 mL	Up to 100 mL	Up to 100 mL

The formula for emulsion preparation was shown in Table 2. The emulsion formed was milky-white in colour and was not transparent.

Table 2: Formula of emulsion preparation

Material	Quantity of 100 mL (%)
Sunflower oil	5
Tween 80	1.25
Span 80	3.73
Methylparaben	0.1
Propylparaben	0.02
Propylene glycol	10
Glycerin	13
Sodium CMC	1
Aqua demineralised	Up to 100 mL

All the nanoemulsion and emulsion were evaluated physical stability by visual observation, including consistency, colour and phase separation. Nanoemulsions were stored in room temperature (25 ± 2°C), low temperature (4 ± 2°C) and high temperature (40 ± 2°C) while the emulsion was stored in room temperature for 12 weeks. The results were shown in Figure 1A, 1B, 1C, and 1D. The results of physical stability evaluation of emulsion were shown in Figure 2A, 2B, and 2C.

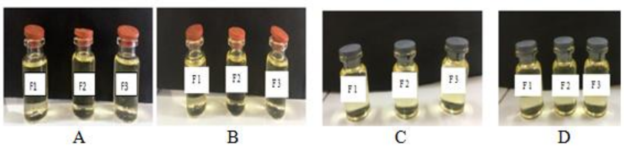

Figure 1: Appearance of the prepared sunscreen nanoemulsions (F1, F2, F3) containing sunflower oil; A) Before storage; B) After storage for 12 weeks at room temperature; C) After storage for 12 weeks at high temperature; and D) After storage for 12 weeks at low temperature)

Figure 2: Appearance of the prepared sunscreen emulsion containing sunflower oil; A) Before storage; B) After storage for 8 weeks; and C) After storage for 12 weeks) at room temperature

The results of the centrifugation test showed that all the nanoemulsion were stable with no phase separation or creaming after centrifugation, but the emulsion was not stable with the formation of phase separation (Figure 3).

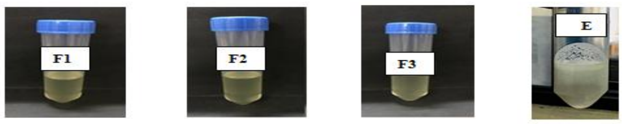

Figure 3: Appearance of the prepared sunscreen nanoemulsion (F1, F2, F3) and emulsion (E) containing sunflower oil after centrifugation

Nanoemulsion (F2) has the smallest average globule size. Globule size of all nanoemulsion was an increase after storage at room temperature (Table 3).

Table 3: Globule size distribution and average globule size of sunflower oil nanoemulsion F1, F2, and F3 during storage for 12 weeks at room temperature

Formula	The ratio of Tween 80 and Sorbitol	Time (week	Globule size distribution (nm)	Average globule size (nm)
F1	38/22	0	42.67 - 281.91	124.47
		6	93.35 - 741.51	307.17
		12	123.06 - 933.50	393.78
F2	36/24	0	38.91 - 323.68	128.88
		6	74.15 - 489.91	216.64
		12	107.18 - 1122.32	429.35
F3	34/26	0	67.58 - 467.86	200.05
		6	112.23 - 576.26	325.07
		12	117.52 - 1560.21	571.95

There was an increase in pH and a decrease in viscosity from nanoemulsion after storage at room temperature (Table 4).

Table 4: Viscosity and pH of sunflower oil nanoemulsion F1, F2 and F3 during storage for 12 weeks at room temperature

Formula	Time (week)	pH ± SD	Viscosity (± SD) (cP)
F1	0	6.50 ± 0.10	225.0 ± 25.00
	4	6.53 ± 0.06	437.5 ± 21.65
	8	6.40 ± 0.00	562.5 ± 0.00
	12	6.23 ± 0.06	750.0 ± 50.00
F2	0	6.80 ± 0.00	100.0 ± 0.00
	4	6.80 ± 0.06	250.0 ± 0.00
	8	6.70 ± 0.00	487.5 ± 10.82
	12	6.63 ± 0.06	612.5 ± 0.00
F3	0	7.00 ± 0.00	125.0 ± 0.00
	4	7.00 ± 0.00	225.0 ± 0.00
	8	6.83 ± 0.06	375.0 ± 25.00
	12	6.80 ± 0.00	500.0 ± 0.00

N = 3.

The results of physical stability test of nanoemulsions at low and high temperature showed that nanoemulsions were clear and transparent (stable) during the test, there were no change colour or phase separation. The result of this test was shown in Figure 4.

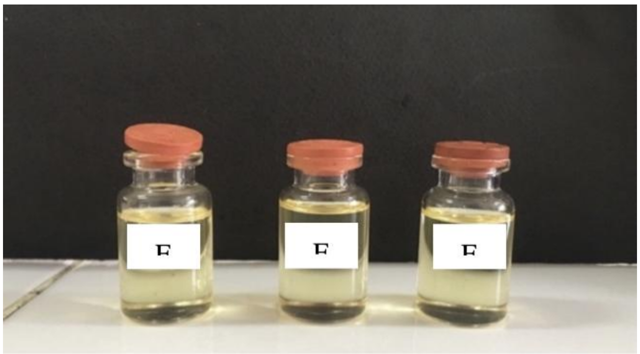

Fig. 4: The results of the freeze-thaw test of nanoemulsions F1, F2, F3 containing sunflower oil

The results of determination of the SPF value are shown in Table 5. The results showed that the SPF value of nanoemulsion is higher than the emulsion.

Table 5: SPF value of nanoemulsions and emulsion containing sunflower oil 5 %

No.	Formula	SPF value			
		I	II	III	Average SPF value
1.	Nanoemulsion F1	5.28	5.30	5.39	5.43 ± 0.03
2.	Nanoemulsion F2	5.19	5.15	4.90	5.08 ± 0.16
3.	Nanoemulsion F3	4.92	4.85	5.50	5.09 ± 0.36
4.	Emulsion	0.06	0.06	0.06	0.06 ± 0.00

Discussion

The formulation of nanoemulsion was prepared based on the spontaneous emulsification method. Spontaneous emulsification can produce nanoemulsion without any external forces. They are formed spontaneously, and they are characterised by ultra-low interfacial tension. The ultralow interfacial tension leads to spontaneous emulsification when the phases (oil and water) are brought in contact.

All nanoemulsions (F1, F2 and F3) were stable during the experiment for 12 weeks storage at 3 different temperatures, which are room temperature (25 ± 2°C), low temperature (4 ± 2°C) and high temperature (40 ± 2°C) during the experiment for 12 weeks. There was no discolouration, changes in consistency, odour and phase separation during the experiment for 12 weeks storage at a variation temperature in the nanoemulsion, but the emulsion showed discolouration for 8 weeks storage and phase separation (not stable) for 12 weeks at room temperature.

Centrifugation test was performed to determine the stability of nanoemulsion. The centrifugation test describes the stability of one year of storage. All of the nanoemulsions were stable with no phase separation or creaming after centrifugation. However, the emulsion was not stable with the formation of phase separation.

The results of the globule size determination show that nanoemulsion (F1) that used the highest surfactant concentration with the ratio of Tween 80 and co-surfactant sorbitol 38: 22 showed the smallest droplet size. The droplet size increased but not significant during 12 weeks of storage at room temperature. High surfactant concentration decreases surface tension and the production of smaller particles.

The results of viscosity and pH evaluation of sunflower oil nanoemulsions show that there was a decrease in pH value and the pH value of formula F1 is close to the neutral pH of human skin normally ranges from 4.5 to 6.0 [14]. The viscosity of all nanoemulsion was decreased during storage for 12 weeks at room temperature

The SPF value of all nanoemulsion preparations was higher than that of the emulsion. This is because nanoemulsion preparation has smaller globule than emulsion, so they absorb more ultraviolet light which results in higher SPF values. This nanoemulsion formulation could be considered more effective in sunscreen cosmetic use compare to the emulsion.

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