Formula preparation of sunscreen (experimental phenomenon and property description) and evaluation of its efficacy

Yizhu Chen

University College London, London, WC1X 0BF, UK

Abstract: Sunscreen is a substance that helps protect the skin from the sun's harmful rays. Sunscreens reflect, absorb, and scatter both ultraviolet A and B radiation to provide protection against both types of radiation. Using lotions, creams, or gels that contain sunscreens can help protect the skin from premature aging and damage that may lead to skin cancer ("NCI Dictionary of Cancer Terms", 2021). This study demonstrates basic knowledge about sunlight and sunscreen including kinds of sunlight, the mechanism and evaluation methods of sunscreen, et al. Experiments were carried out to investigate three variables (solvent, temperature, UV filter) to test the relationship between the changing factors of sunscreen and the relative influences on sunscreen's effectiveness. Key results indicated that temperature has the least effect on the effectiveness of sunscreen, which means the change of temperatures causes the SPF to change insignificantly. Solvents with different polarity can lead to different SPF, with polar and non-polar solvents absorbing more UV, and semi-polar solvents have the weaker ability to absorb UV. Different types of UV filters cause various changes in the effectiveness of sunscreen. The key findings in this study provided insights for the formulation and effect evaluation of the sunscreen for consumers. Also, it could be useful for consumers to choose the sunscreen that is the best fit for them.

Keywords: Sunscreen, SPF, UV Filters

1. Introduction

Nowadays, with the continuous emergence of skin cancer and the rising death data, more and more people begin to pay more and more attention to sunscreens. Data shows from 2016 to 2021, global sunscreen consumption will increase from approximately 44,000 tons to approximately 52,000 tons, with an average annual compound growth rate of approximately 4%. Long term exposure to the sun can lead to skin cancer, the information show that about 90 percent of nonmelanoma skin cancers are associated with exposure to ultraviolet (UV) radiation from the sun (Koh et al., 1996), and one UK study found that about 86 percent of melanomas can be attributed to exposure to ultraviolet (UV) radiation from the sun (Parkin et al., 2011). However, Regular daily use of a sunscreen with SPF 15 or higher can reduce the risk of developing melanoma by 50 percent (Green et al., 2011). There are many reasons for using sunscreen. For example, too much time in the sun will result in sunburn, severe skin reddening and inflammation of the skin. According to Medical News Today: "The ultraviolet rays from the sun damage the skin, which turns red as the body directs more blood to the affected area to repair the damage." When it comes to developing wrinkles, fine lines and crow's feet, one of the most effective preventative formulas is sunscreen. Lengthy exposure to UVA rays prematurely ages your skin and results in the loss of collagen and skin elasticity. In fact, approximately up to 90% of visible signs of aging are caused by sun damage. In addition, blotchy skin or dark spots may spring up on the face, hands and other parts of the body regularly exposed to the sun. ("The Benefits of Sunscreen: Why Do I Need To Wear SPF Every Day?" 2021) Thus, there is a growing trend for demand of sunscreen in the market. A large number of customers lead to a more competitive market.

That causes more and more cosmetics brands launch sunscreen products. To show more comparative advantages and earn more revenue, each sunscreen's brand try to make products more personalized through innovation to improve the positive image of their branding. For example, some brands began to modify the SPF value of each product according to the different needs of customers. SPF is defined as a measure of how much solar energy (UV radiation) is required to produce sunburn on protected skin (i.e., in the presence of sunscreen) relative to the amount of solar energy required to produce sunburn on unprotected skin. As the SPF value increases, sunburn protection increases. ("Sun

Protection Factor (SPF)", 2021). Generally, SPF15~20 is suitable for daily protection. SPF 35-50 is sufficient to protect the skin from UV damage in outdoor leisure activities such as swimming and playing ball games. Hence, people can choose the most suitable products for different activities. As a result, it is very important to understand how these companies change the SPF of same products.

The main topic discusses in this report is "Formula preparation of sunscreen (experimental phenomenon and property description) and evaluation of its efficacy ". Experiments are needed to complete the project. In this investigate, the basic knowledge and research status of sunscreens are reviewed, describing these properties in detail of the product in different steps and evaluate the efficacy of sunscreen.

2. What is sunscreen?

Sunscreen, also known as sun cream, sunblock or suntan lotion. ("Sunscreen - Wikipedia", 2021) Sunscreen is a chemical defense, including avobenzone, oxybenzone, and para-aminobenzoic acid (PABA), which are ingredients used to absorb the sun's rays, so it can penetrate the skin, absorbing or reflecting the UV rays before they reach and damage the dermal layers ("Sunscreen Versus Sunblock: What's the Difference?", 2021). Thus, it helps to prevent sunburn and premature aging (such as wrinkles, leathery skin), most importantly skin cancer. ("Tips to Stay Safe in the Sun: From Sunscreen to Sunglasses", 2021).

3. Basic knowledge about UV and the importance of sunscreen

The sun emits non-ionizing electromagnetic radiation (EMR) composed of UV (100-400 nm), visible (400-780 nm) and infrared (780-5000 nm) radiation. With regard to human health, the most relevant and concerning form of EMR is UVR (4-6). Ultraviolet radiation is composed of wave-lengths between 100 and 400 nm that are further divided into UVC (100-290 nm), UVB (290-320 nm) and UVA (320400 nm). Because wavelengths below 290 nm are absorbed by atmospheric ozone and do not reach the earth's surface, UVC from sunlight is of little practical concern. (Jackson, 1995). As stated, the primary source of UVB and UVA radiation is the sun, to which exposure is considered largely unavoidable. The amount of UVR reaching a given location on earth varies seasonally, geographically and diurnally. For example, UVR intensity is highest at the equator and high altitudes and decreases with increasing latitudes. The intensity of UVB is considered highest during the summer months and on a daily basis between 1100 and 1500 h. Importantly, however, UVA intensity is more consistent throughout the day and from season to season compared to UVB. Meteorological and atmospheric conditions including cloud cover, pollution, humidity and temperature modify the spectrum and intensity of terrestrial sunlight, particularly the UV component.(Gibson et al., 1984). This incidental exposure can account for as much as 80-90% of an estimated yearly exposure to UVR (LEACH et al., 1978);(Diffey, 1987) and, not coincidentally, over 60% of non melanoma skin cancers (NMSC) appear at these sites (3,12,13). (Dayan, 1993); (Czarnecki et al., 1991);(Jackson, 1995)

Skin is the organ most affected by environmental sunlight. Interaction between UVR and skin involves mutagenic lesions as well as indirect genotoxic effects mediated by oxidative stress (Cadet et al., 2005). It is well known that UVR can damage many skin molecules and structures, including DNA (Cadet et al., 2005). UVR can modify purines or pyrimidines, can disrupt the link between genes, or can even delete parts of the genome (Marrot & Meunier, 2008);(Mouret et al., 2006). All these damages are usually reversible, thanks to the DNA-repair mechanisms. Unfortunately, sometimes the repair mechanism fails and inability to further read and transcribe can occur, leading to cell death or abnormal behavior like hyper proliferation or malignant transformation. Exposure to UVR has pronounced acute, chronic or delayed effects on the skin. The UVR-induced skin effects manifest as acute responses such as inflammation, i.e. sunburn (Hruza & Pentland, 1993), pigmentation (Gilchrest et al., 1996), hyperplasia (Adhoute et al., 1992) immunosuppression (Cooper et al., 1992); (Whitmore & Morison, 1995) and vitamin D synthesis and chronic effects, primarily photocarcinogenesis and photoaging. These acute and chronic effects are dependent on the spectrum and cumulative dose of UVR. Like skin cancer, chronic exposure to solar UVR is thought to accelerate aging of human skin. This skin photoaging is characterized by dryness, roughness, irregular pigmentation such as freckling/lentigenes, actinic keratoses, wrinkling, elastosis, inelasticity and sebaceous hyperplasia (GILCHREST, 1996).

4. Protection mechanism

According to the protection mechanism of sunscreen, there are two categories of sunscreen agents: organic and inorganic. The organic sunscreens are referred to as soluble or chemical sunscreens. The inorganic sunscreens are commonly known as physical, mineral, insoluble, natural or non chemical.Zinc oxide and TiO2, exist as odorless white powders comprised of a Gausian or normal distribution of particle sizes.

4.1 Inorganic sunscreen

Firstly, the inorganic sunscreens are generally viewed as harmless pigments that cannot enter the skin and are largely unaffected by light energy like organic sunscreens may be. The two most commonly used inorganic sunscreens are titanium dioxide (TiO2) and zinc oxide (ZnO). Although these two metal oxides differ substantially in their appearance and attenuation spectra (Grady, 1947). It protects the skin by reflecting and scattering ultraviolet light. The UV shielding mechanism of titanium dioxide and zinc oxide can be explained by the solid energy band theory. Because they are all wide band gap semiconductors, the band gap of rutile TiO2 and ZnO are 3.0ev and 3.2eV, respectively, corresponding to the absorption of 413nm and 388nm UV. At the same time, they also have a strong ability to scatter ultraviolet rays. When ultraviolet rays irradiate nano-sized titanium oxide and zinc oxide particles, because their particle size is smaller than the wavelength of ultraviolet rays, the electrons in zinc oxide and titanium dioxide particles are forced to vibrate and become a secondary wave source, emitting electromagnetic waves in all directions, so as to achieve the effect of scattering ultraviolet rays.

4.2 Organic sunscreen

Chemical UV absorbent is divided into ultraviolet A (UVA) absorbent and ultraviolet B (UVB) absorbent. Organic sunscreens are often classified as derivatives of anthranilates, cinnamates, dibenzoylmethanes, p-amino-benzoates or salicylates .These materials absorb, reflect, or scatter UV radiation that is incident on the skin.

People will add more plant ingredients and antioxidants into the suncreams, because plant ingredients can increase the sun protection factor, while avoiding the use of chemical ingredients. A variety of antioxidants are added to some newly launched facial skincare products on the market. These antioxidants increase the SPF value of products.

5. Evaluation method of sunscreen

The protective efficacy of a sunscreen product is determined by the absorption properties of the UV filter substances, by the homogeneity of the distribution of the sunscreen on the skin and by the annihilation of the absorbed and/or scattered high-energy photons in the skin. The SPF for a sunscreen is defined as the ratio of sun exposure that skin can tolerate before burning or minimal erythema is apparent with and without sunscreen protection.

6. Types of sun protection evaluation

6.1 Vivo tests

The definition of vivo Evaluation of Sunscreens is defined as a series of increasing latencies by irradiating with ultraviolet light. The test site was limited between the back, waist and shoulder line. The experimental site will be divided into three areas: the first area is directly exposed to ultraviolet light, the second area is irradiated after smearing the test sample and the third area was irradiated with SPF standard reference substance. The dose of ultraviolet radiation increased in turn, and the irradiated skin developed delayed erythema reaction due to superficial vascular dilation. The distribution of the sunscreen on the skin was analyzed on skin area 1 (15 and 30 min after topical application) with a multi-photon tomograph (Dermalnspect, JenLab GmbH, Jena, Germany) appropriate for the noninvasive in vivo measurement of human skin.

6.2 Vitro tests

One of these, the critical wavelength (CW), has been proposed to evaluate sunscreens for their UVA absorption, based on the methods outlined by Diffey. In this method, the transmission through a substrate, both with and without the sunscreen, is measured on a wavelength-by-wavelength basis using a light source with a continuous output over the terrestrial UVR spectrum. The attenuation spectrum of the sunscreen is then determined.

6.3 Tape Stripping and Spectroscopic Measurement

The distribution of the sunscreen on the skin was analyzed by tape stripping on skin area. The tape-stripping procedure was performed 30 min after sunscreen application, as described previously. Adhesive films (Tesafilm No. 5529; Beiersdorf, Hamburg, Germany) were pressed onto the skin of the forearm with a roller and were subsequently removed. The removed tape strips contained one layer of corneocytes and the corresponding amount of sunscreen penetrated into this cell layer. The tape stripping was repeated up to 8 times on exactly the same skin area.

7. Factors that can affect the sunscreen effectiveness

7.1 Type of solvents

For sunscreens, O/W and W/O emulsions are the most popular vehicles and will be formulations of choice for some time to come (LOWE, 1990). Polar solvents will blue-shift the maximum absorption peak λ max of some polar sunscreens (in the short-wavelength direction), and red shift the low-polar sunscreens (in the long-wavelength direction). So most sunscreens increase UV absorption in polar and non-polar solvents, and semi-polar solvents (hexylene glycol and C12-C15 alkyl benzoate) will reduce UV absorption. For most skin care formulations, emulsions are based on a mixture of different oils. In our case, the oil content contains paraffin liquid for its stability, low cost, and function as a moisture barrier. Coconut oil is employed for its high compatibility with the skin. Isopropyl myristate leaves no sticky or greasy feeling after application. In water phase, propyleneglycol is used for some combined properties such as its ability to attract moisture, retard evaporation, and its moderately adhesive effect.

7.2 Type of UV filter

7.2.1 TiO2

In 1978, an OTC Panel (US Federal Register, 1978) reported that "...as a physical sunscreen agent, titanium dioxide (TiO2) is a safe,opaque and effective product that provides a barrier to sun-sensitive individuals against sunburns, because it reflects and scatters UVA and UVB radiation 290–400 nm) rather than absorbing the rays...". However, TiO2 is a much stronger absorber of UV light than is a scatterer of UVB and UVA radiation. Particle sizes of TiO2 that are tenfold greater (range 200–500 nm) are best at reflecting visible light.

7.2.2 Chemical filter

Chemicalorganic filters are classified into either UVA(benzophenones, anthranilates and dibenzoylmethanes) or UVB filters (PABA derivatives, salicylates, cinnamatesand camphor derivatives). Among the sunscreen products tested, ethylhexyl methoxycinnamate and butylmethoxydibenzoylmethane are used most frequently, at 80.3% and 24.0% respectively. This is because these two ingredients are safe, stable and have high UV absorption efficiency has become the most commonly used sunscreen agent in sunscreen cosmetics. Chemical UV filters are almost always used in combination because no single active agent, used at levels currently allowed by the FDA, provides high enough SPF (sun protection factor) protection or broad-spectrum absorption.(US Food and Drug Administration,2000)

7.2.3 P-amino benzoic acid (PABA)

One of the first UV filters widely used was p-amino benzoic acid, better known as PABA. It is no longer used to any great extent globally, as it is clearly known that irradiation of PABA solutions results in photo-degradation.; The most widely used organic UV filter in the analysed samples was BMDM owing to the fact that this compound has a broad absorption spectrum (maximum absorption in

359 nm) and is one of the few organic filters allowed that protects against UVA.

7.2.4 Avobenzone

It has been known for several years that exposure to UV radiation will cause avobenzone to breakdown. However, when tested on a human skin model, the 'positive' results were no longer observed. Thus, it is possible that photostability of sunscreen products containing avobenzone is acceptable or perhaps constantly improving or the risk of photo allergy is low or a combination of these. And avobenzone in repeat exposure animal studies has been shown to be protective when tested in phototoxicological

7.2.5 Octyl methoxycinnamate

Octyl methoxycinnamate (0.01–9.0%) also was used UV filters in the sunscreen formulations investigated, found in 44% of all products investigated.

7.2.6 Temperature

Experiment: samples were exposed in vitro to the defined temperatures, in their proper package, using an incubator (P-Selecta®, Barcelona, Spain). Groups of maximum average and extreme temperatures (29 and 40°C, respectively) were exposed to these conditions for a period of 5 h for predetermined days (1, 2, 5, 6, 7, 8, 9, 12, 13, 14 and 15), staying at 25°C in the remaining time. As can be seen, generally, SPF values remained stable throughout the whole period of study. However, when the sunscreen was exposed to the temperature of 25°C, upon 24 h, a slight decrease of approximately 5% in SPF was identified (P< 0.05), as compared to the initial SPF value. A similar SPF reduction (4.2%) was perceived in the group of 29°C when comparing initial SPF with that measured on day 15 (P< 0.05). Nevertheless, in spite of the statistical significance of the values, these determinations do not compromise the general trend of results, which indicate the maintenance of SPF. An exception occurs for the maximum average temperature as compared to the initial SPF value, as previously referred, which is significant but not compromising.

8. Method

- (1)Prepare the water phase mixture by adding samples of different mass fractions according to the formula. First try to stir with a stir bar. If the substance cannot be completely dissolved, you need to use a heater. Manually stir under heating conditions.
- (2)Prepare the oil phase mixture according to the ratio in the formula and heat it with a heater to dissolve the sunscreen in the mixture and use a blender to stir the mixture into a delicate paste.
- (3)Mix the oil and water phases and make sure that the mixture is a fine paste without any precipitation. If there is solid precipitation, it is necessary to continue heating and stirring until the target product is formed
- (4) When the delicate paste is formed, take the part out and place it in another beaker for stability testing. The time cycle is one week, every day you need to observe whether there is any change in its properties and sun protection effect
- (5) The other part of the sunscreen is used to evaluate the effect of sun protection. By comparing the intensity difference received by the UV test paper with or without sunscreen, the sunscreen effect of the prepared sunscreen can be demonstrated by receiving the same intensity and the same time of UV light irradiation

9. Results

In this experiment, I made a sunscreen based on the formula in the literature and evaluated the efficacy of this sunscreen. The standard mass of the sample is 100g. This is a sunscreen with an oil-in-water phase, so the oil phase needs to be blended into the water phase. I will show the properties of oil phase, water phase and combined below. I will compare the results of the test paper with and without sunscreen under the same intensity of ultraviolet radiation to show the effectiveness of sunscreen at final.

10. Property

10.1 The properties of the water phase

The standard unit is grams. Kasone, glycerin, NaCl, Fragrance and deionized water are all liquid. Therefore, the mixture of them is also a liquid first, and there is no need to stir and heat to form a liquid without precipitation. Xanthan gum is a white powder. After adding it to the mixed liquid, you only need to manually stir it with a stirring rod for several times, and no solid particles will appear. 1% of Carbo is a solid gel, which is very difficult to melt. After adding it to the liquid, I tried to stir it manually with a stir bar, but after five minutes there was still a gel solid. So I decided to put it in a water heater, heat it at a high temperature of 75 degrees for 5 minutes, and then use a 1500r/min blender for about 7-8 minutes to stir, and finally get a fragrant white and delicate mixture.

In this step, it is very important to obtain a product without any solid precipitation. Because before getting a successful sunscreen product, I have had several failed experiences. If there are still insoluble substances in the product of this step, then in the final step of combining the water phase and the oil phase, those insoluble substances are more difficult to dissolve in the mixture, which will cause the final product to fail to form delicate White paste, the sun protection effect is also close to zero.

10.2 The properties of the oil phase

Compared with the water phase, the oil phase is more difficult to dissolve. Basically all the raw materials are viscous, and they tend to stick to the wall of the beaker when they are poured into the beaker, which causes a relatively large error. So choose a small beaker. Except for paraffin, UV filter (Escalol 567), and emulsifier, the other raw materials are all fluid, and it is easier to form a liquid without precipitation, so add them together first. Paraffin wax is solid, sunscreen is white powder, emulsifier is dark yellow paste. I added the three substances according to the amount, and heated the mixture at 90 degrees for 15 minutes using a water bath heater. In the heating process, it is also necessary to use a stirring rod to stir the mixture as much as possible. After the heating is complete, add the mixture to the blender as well, but this time it takes about 20 minutes to stir. Because sunscreens are hardly soluble solids, paraffin wax solids also require high temperature and stirring to dissolve in the liquid. Finally got a pale yellow viscous paste.

In this step, it is still necessary to stir the product until there is no solid precipitation, and the reason is the same as that of the water phase.

10.3 The properties of the combination of water phase and oil phase

In this step, just need to add the oil phase to the water phase. But in the process of adding, it is still necessary to heat and stir at the same time, so as to prevent the sudden drop of temperature and the precipitation of solids during the process of adding, which will weaken the sun protection effect. At a temperature of 60 degrees, a magnetic stirrer was used to stir at a speed of 1500r/min for about 10 minutes, and finally a light yellow delicate paste with fragrance was obtained.

The product is a dark-yellow delicate paste, and has a fragrance because of the addition of fragrance. The skin feel is very delicate, can push it off when apply it on the skin and spread it evenly.

11. Efficacy evaluation of sunscreen

Use UV paper to compare the UV intensity measured with and without sunscreen under the same intensity of UV radiation. In the test paper, the stronger the UV intensity, the darker the purple color block will be.In the absence of any shielding and protection, the test paper shows a dark purple color, which is the highest intensity that can be measured in the test paper. Another test paper coated with sunscreen on the test area showed a light purple color block after being irradiated with ultraviolet light of the same intensity and time, which means that the intensity of the irradiation in this area is much weaker than that. This color speaks volumes about the success of the sunscreen and its effectiveness.

12. Stability of sunscreen

Put the final sunscreen product into a beaker, check its properties every day, whether it is still a

delicate paste, whether there is any solid precipitation, etc. In general, sunscreen is relatively stable. After a week of daily observation, no solid particles were precipitated, and the overall volume did not decrease or increase. However, after the fourth day of preparation, the sunscreen was not as delicate as before, but rather solidified and felt a bit like a jelly to the touch. But the sunscreen effect does not make a big difference. A week later, it still has no major changes, and the signs of sunscreen weakening are not obvious.

13. Conclusion

In the experiment, sunscreen was prepared according to the formula, and the characteristics at different stages were recorded, and the sunscreen efficacy was tested. Experiments have proved that temperature has a relatively large influence on sun protection effect and stability, because when the temperature is not high enough, the particles cannot be dissolved. But when the temperature is sufficient and all reactants can be reacted completely, the influence of temperature will decrease instead.

The results of the experiment have certain practical significance. It can provide suggestions for cosmetic companies to help them develop products that are more in line with customer needs, especially those start-ups without the branding effect, to attract their most reliable consumers and build up the consumers' loyalty. And it also gives customers an opportunity to learn more about daily necessities.

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