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Effectiveness of Raspberry Seed Oil in Natural Sunscreen Formulation Using Different Percentage of Zinc Oxide

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Abstract: A natural sunscreen is made from natural ingredients that are used to replace the common sunscreen that uses multiple chemicals that has negative effects such as allergic reaction and free radicals besides can absorb the harmful Ultraviolet (UV) rays before it penetrates to the skin. Then, this project to solve the issues related to skin disease caused of harmful UV rays such as sunburn and skin cancer which is directly influenced by a climate change need to be done. The objectives of this research are to study the sunscreen effect of natural ingredients that contains red raspberry seed oil and other organic oil with aloe vera, to develop a minimum amount of zinc oxide of the sunscreen that has higher than 30 Sun Protection Factor (SPF) and a high UV-A Protection, to characterize the functional group of the sunscreen product by using Fourier Transform Infrared Spectroscopy (FTIR). The natural ingredients and zinc oxide 20%, 10%, 15%, 5%, and 0% are mixed into the sunscreen formulation to absorb UV-B and UV-A. The results from the UV-Vis and FTIR are to identify the absorbance and functional groups that obtained in the product which shows the product having a broad-spectrum UV protection and 44.67 SPF plus having Methyl Mercapto as an active functional group in the product. In conclusion, the best formulation of natural sunscreen consists of 100% natural ingredients like red raspberry seed oil with aloe vera, virgin coconut oil, olive oil, and a 5% amount of zinc oxide to replace the chemical compound.

Keywords: Natural Sunscreen, UV-rays, UV-B, UV-A, FTIR, SPF and UV-A Protection, Spectrometric

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1. Introduction

The natural sunscreen is used to reduce the damaging effect to human skin by blocking or absorbing the UV-Rays from sunlight[1]. The variety of sunscreen produced is a result of the rising of UV-Rays intensity especially the natural sunscreen and non-natural. Natural based sunscreen has a low risk of poisoning and is free of harmful chemical compounds. High energy of UV radiation can cause skin aging, burning and skin cancer [2]. High usage of inorganic zinc oxide can form free radical when exposed with UV-A radiation and it can damage the DNA and skin cells [3]. Conventional Sunscreen already uses a mixture of inorganic compounds such as zinc oxide that can have a long-lasting effect on our skin. Because of that, it causes our skin to be damaged to certain compounds or minerals especially for sensitive skin due to its free radical [3]. Some users have experienced allergic reactions due to the large amount of zinc oxide formulation in the conventional sunscreen, so it is appropriate to reduce the usage of zinc oxide in a sunscreen. Skin disease like sunburn and skin cancer related problems because of UV radiation and temperature rising is the main purpose of this project by finding the best solution and suitable ingredients to solve that problem [4].

Raspberry seed oil extract can reduce the amount of zinc oxide in a sunscreen because it is helpful in blocking both UV-A and UV-B radiation multiplying with coconut oil as anti-aging, olive oil in preventing skin cancer, and aloe vera as moisturizer. So, the objectives of the study are to know how these ingredients affect the performance of the sunscreen using UV-Vis Spectrophotometer. Raspberry offers UV-A and UV-B protection similar to Titanium Dioxide which has higher than 30 Sun Protection Factor (SPF) and offers UV-A Protection so it is suitable to formulate these ingredients with a small amount of zinc oxide while still effectively absorbing the UV radiation [5]. Small amount of zinc oxide formulation is used to cover the UV-A protection of the sunscreen because raspberry seed oil cannot offer higher UV-A protection than UV-B protection. Other objectives are to characterize the functional group in these natural ingredients by using Fourier Transform Infrared Spectroscopy (FTIR).

The product is expected to achieve all the objectives by showing the higher absorbance unit in the testing using UV-Vis Spectroscopy. The higher absorbance unit can produce higher Sun Protection Factor (SPF) and UV-A protection according to Mansur formula [6]. The raspberry seed oil based natural sunscreen can replace and lowering the use of conventional sunscreen which has many negative effects on the human and environment. Moreover, with the low use of zinc oxide which around 5% in the formulation will make the product much cheaper than the conventional sunscreen.

2. Materials and Methods

The materials and methods, otherwise known as methodology, in this section will describe all the necessary information that is required to obtain the results of the study.

2.1 Materials

The equipment and materials used in this project are listed as follows. The tools needed are five beakers of 50 mL, spatula, five glass rods, stand, analytical balance, pH meter, pipette, water bath, vortex, UV-Vis spectrophotometry and FTIR. While the materials used are 40 mL raspberry seed oil, 50 mL olive oil, 250 mL virgin coconut oil, 30mL aloe vera, beeswax, distilled water and 3.94 g zinc oxide.

2.2 Methods of the study

Flowcharts in **Figure 1** shows the procedure of the experiment to produce natural sunscreen. First, sample preparation was required by preparing five 50 mL beakers and labelled with samples A, B, C, D, and E. All materials need to be weighed and measured and mixed in a beaker according to the formulation for each sample. Then, in this experiment, it is necessary to re-examine the formulation and re-formulate the sunscreen into 100% natural ingredients with the addition of UV-filter zinc oxide and stability. Then, the formulation for each ingredient becomes 0% zinc oxide for Sample A, 5% for Sample B, 10 % for Sample C, 15% for Sample D and 20% zinc oxide for Sample E. Next, heated the sample in a beaker used a water bath at 70ºC for 15 minutes, and stirred used a glass rod. After that, mixed using a vortex for 10 minutes. Let it cool, then test the pH of the sample using a pH meter and store it in a bottle for 24 hours at room temperature. The next day, determined SPF with used UV-vis 3900H Spectrophotometer by putting the sample in the cuvette and distilled water as a blank. Absorption data were obtained at range 290 to 320 for UVB range to determine SPF and 320 to 400 for UVA range for UVA protection in every 5 nm. Lastly, characterized the functional group in the sample product by used FTIR spectra that is a Cary 630 FTIR.

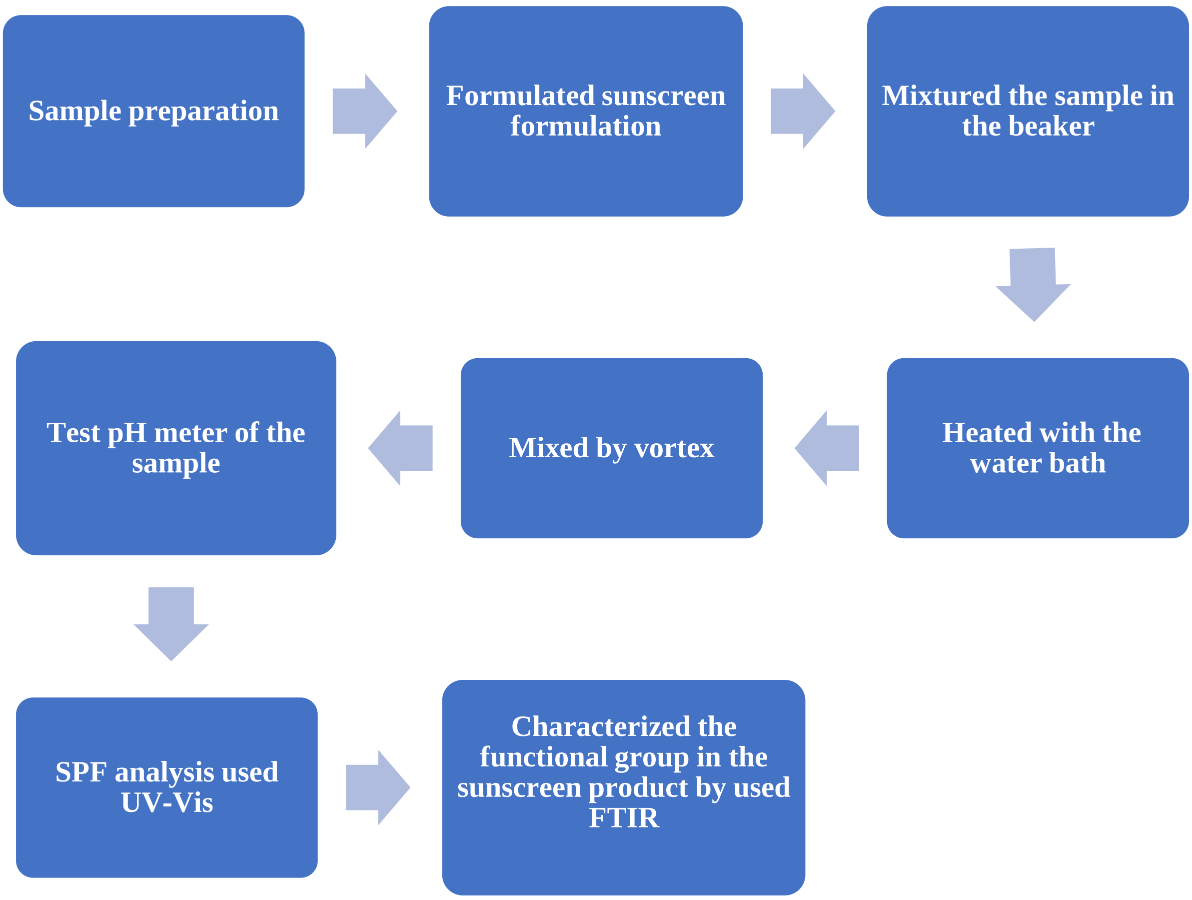


Figure 1: Flowcharts of the experiment

2.2.1 Production of Natural Sunscreen

In this experiment, the production of natural sunscreen was used the different amount of zinc oxide based on the formulation in the **Table 1** below:

Table 1: Formulation each sample with different percentage of zinc oxide

Sample	Percentage of zinc oxide
A	0%
B	5%
C	10%
D	15%
E	20%

The formulation is divided into 4 phases that is water phase, oil phase, UV filter and stability. For the water phase, the ingredients used were distilled water and aloe vera. Then, for the oil phase ingredients are red raspberry seed oil, olive oil and coconut oil. The ratio of oil phase and water phase is 3:1 that is the oil phase is 100% natural ingredients. Next is the material used for stability is 3% beeswax [7]. Based on **Table 1**, the UV filter used is zinc oxide. This material is used with different percentages for each sample to obtain the SPF value that achieves the objective.

2.3 Determination of SPF Value

This will be determined from the absorbance graph from UV Spectrophotometer analysis. The data were obtained from the result graph at UV-B wavelengths range, 290 nm to 320 nm, this can determine the SPF value results for each sample. The SPF value can be calculated through formula below:

$$\mathrm{SPF} = \frac{\sum_{290}^{320} E(\lambda)\ X\ I(\lambda)\ X\ d\lambda}{\sum_{290}^{320} E(\lambda)\ X\ I(\lambda)\ X\ 10^{-A(\lambda)}\ X\ d\lambda} \qquad \textbf{Eq. 1}[8]$$

Where: "*E* (λ)" is the erythema action spectrum at wavelength 320-400 nm ; *P*(λ) is the persistent pigment darkening action spectrum at wavelength 320-400 nm; "*I* (λ)" is the spectral irradiance of UV source at wavelength 320-400 nm; "*dλ*" is the wavelength step (1 nm) and "*A* (λ)" is the absorbance of the test samples at wavelength 320-400 nm [8]. The value of *EE* (λ) X I (λ) are constants like the **Table 2** below:

Table 2: Value of *EE* (*λ*) *X I* (*λ*) at (290 nm – 320 nm) [9]

Wavelength (nm)	*EE X I* (normalized)
290	0.0150
295	0.0817
300	0.2874
305	0.3278
310	0.1864
315	0.0837
320	0.0180
Total	1.0000

Table 2 shows the constant value of *EE X I* (*EE X I* = $E(\lambda)\ X\ I(\lambda)$) in each wavelength from 290 nm to 320 nm (UV-B region) with 5 nm interval which helps in calculation for SPF using SPF formula.

2.4 Determine UVA Protection

UVA protection is determined from the absorption graph from UV Spectrophotometer analysis. Based on the observation of results from the UVA wavelength range of 320 nm to 400 nm, this can determine the UVA protection results for each sample.

3.0 Results and Discussions

The parameters determined the best formulation of natural ingredients to produces higher than 30 SPF and protect skin from UVA radiation. The ingredient's benefit consumers by following the standard of successful natural sunscreen.

3.1 SPF Value and UVA Protection

From the **Figure 2**, Sample A has the lower absorbance in both UV-A and UV-B regions. Sample B has the higher absorbance in UV-A region (320 nm – 400 nm) and high absorbance in UV-B region (290nm – 320nm) thus Sample B is chosen as perfectly suitable for the sunscreen which has the 5% formulation of zinc oxide.

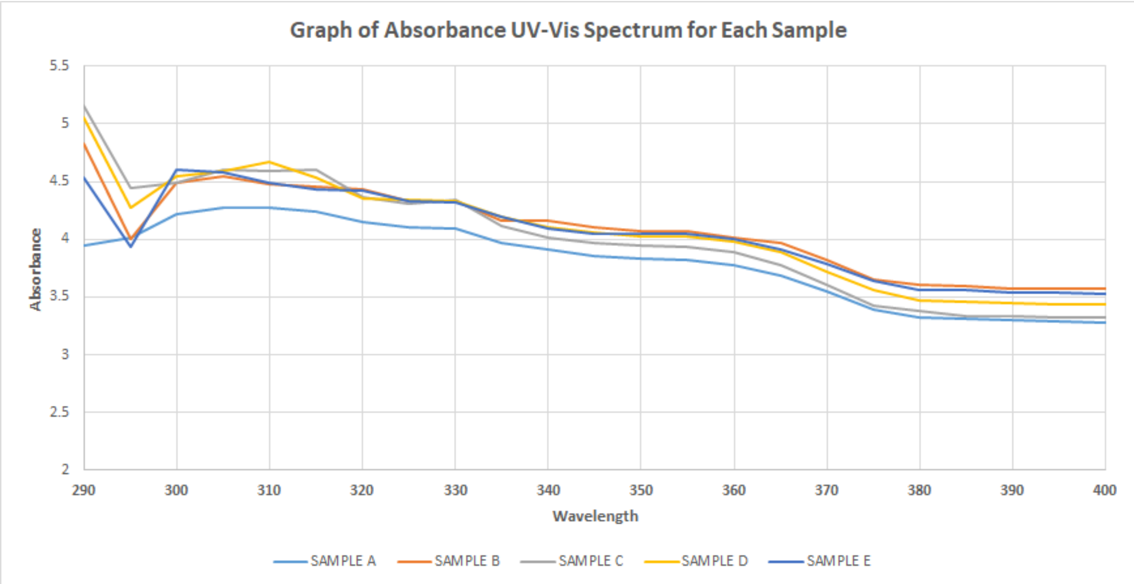


Figure 2: Absorbance Spectroscopy of Each Sample

3.1.1 Sun Protection Factor Determination (290nm – 320nm)

The samples absorbance at 290 nm – 320 nm wavelength which is UV-B region is used to calculate the SPF value of the samples because SPF value only can be determined at UV-B region. The absorbance of each samples at UV-B region (290nm – 320nm) and Sun Protection Factor were tabulated in a **Table 3** and **Table 4**.

Table 3: Absorbance unit of the samples

Wavelength (λ, nm)	***EE X I***	**Absorbance**				
		Sample A	Sample B	Sample C	Sample D	Sample E
290	0.015	3.944	4.833	5.162	5.055	4.538
295	0.0817	4.017	4.003	4.449	4.272	3.936
300	0.2874	4.223	4.487	4.491	4.544	4.606
305	0.3278	4.275	4.542	4.608	4.587	4.578
310	0.1864	4.239	4.483	4.587	4.670	4.492
315	0.0837	4.153	4.461	4.608	4.533	4.428
320	0.0180	4.101	4.434	4.368	4.354	4.420

Table 3 above shows the absorbability of each sample for each wavelength around 290 nm - 320 nm with 5 nm interval accompanied by the constant EE x I normalized.

Table 4: Sun protection factor value of the samples

Sample	**Sun Protection Factor**
A	42.2 SPF
B	44.67 SPF
C	45.61 SPF
D	45.63 SPF
E	45.02 SPF

Table 4 above shows the Sun Protection Factor Value of each sample calculated using the Mansur formula. Sample A has the lowest SPF value and Sample D has the highest SPF value compared to others. However, Sample B is chosen to be the sunscreen product because it has a higher UV-A protection among others and has high Sun Protection Factor (SPF) which is around 44.67 SPF.

3.1.2 UV-A Protection Determination (320 nm – 400 nm)

The parameter to determine how much the sunscreen offers UV-A Protection is considered as Protection Grade for UV-A (PA). Usually, it is determined using the absorbance at UV-A region around 320 nm – 400 nm wavelength with the specific formula and constant value. Due to the limitation of the research, it is hard to determine it without a proper equipment such as solar irradiance stimulators so it is no use to show the absorbance in each UV-A region wavelength because it cannot be calculated, and the data is too long. There is another method to determine it by only claiming that the sunscreen has the broad-spectrum protection due to highest absorbability of the sample chosen. Based on the graph from **Figure 2** claimed that Sample B protects the skin from UV-A radiation more effectively because at the wavelength 320 nm to 400 nm proved that it has UV-A protection due to the highest peak of the absorbance among other samples.

To be conclude, after determining the SPF value and UV-A protection, the right formulation of natural sunscreen is chosen from Sample B. Sample B is chosen to be the sunscreen product because it has a high Sun Protection Factor (SPF) which is around 44.67 SPF even the Sample D is the highest which is 45.63 SPF. The reason is Sample B has a highest UV-A protection among other samples, so it is sufficiently meeting the objectives of the study, which is achieved higher than 30 SPF, having highest UV-A Protection and using less amount of zinc oxide that only has 5%. To be precise, Sample B is greener than Sample D that is using 15% zinc oxide where it is the major reason of the sample selection.

3.2 Physicochemical of the Sunscreen Formulations

All the test formulations were having the same procedure and the same amount of natural ingredients. The difference is just the amount of zinc oxide. The combination of zinc oxide with red raspberry seed oil as a major active ingredient and other minor ingredients like aloe vera extract, virgin coconut oil, and olive oil will give an excellent result of being a broad-spectrum sunscreen. Each of the ingredients in the product has its specific function to improvise the product quality among the other conventional sunscreen that already sells in the market. The figure below shows the picture of the sample after the experiment. **Figure 3** shows from the right side is the clear one was distilled water (blank) then the next beside is sample A until sample E.

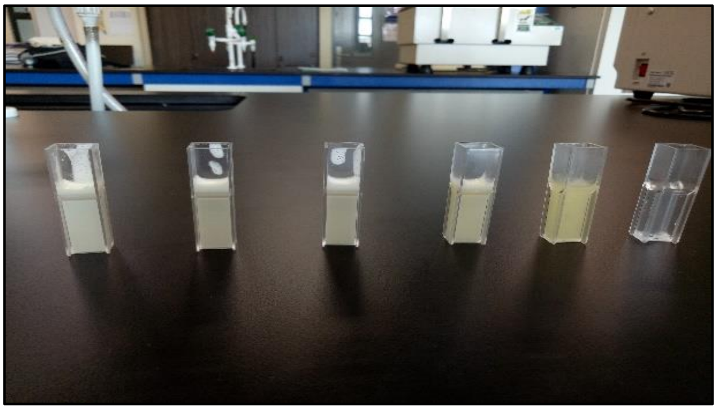


Figure 3: The results of the samples

In addition, the table below shows the result of physicochemical (pH value, colour, texture, and SPF value) after experimenting.

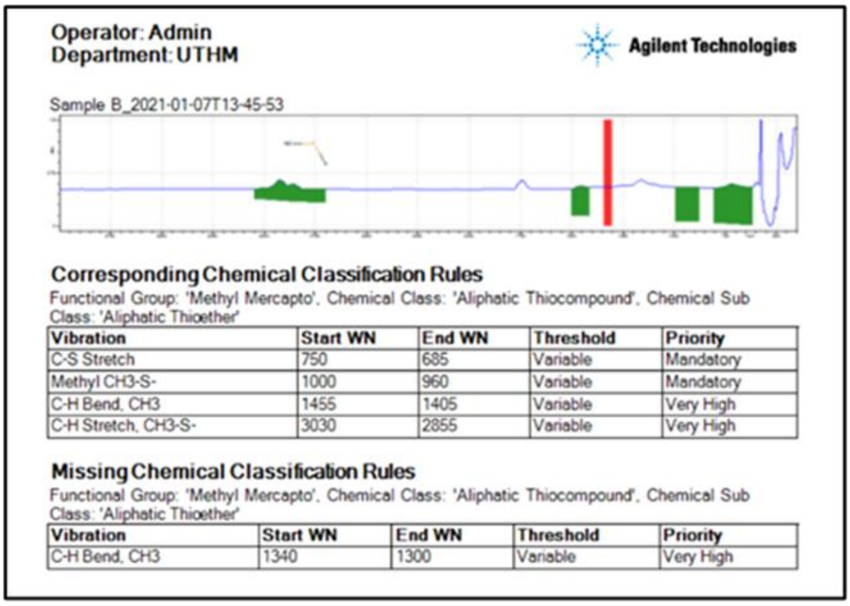
Table 5: Physicochemical of sunscreen formulation

Sample	pH value	Colour	Texture	SPF value
A	4.50	Light yellow	Oily	42.2
B	6.72	Light white	Oily and creamy	44.67
C	7.12	White	Oily and creamy	45.61
D	7.10	White	Oily and creamy	45.63
E	7.55	White	Oily and creamy	45.02

Besides, the physicochemical of this sunscreen is also the average. **Table 5** shows the pH value from Sample B until Sample D is in a suitable pH level for adult skin was the pH requirement should reach a pH of 5 to 7.5 [3]. The sunscreen's colour had the same as another commercial sunscreen that suitable for every skin type which is white and a bit yellowish. While the texture was suitable for standard sunscreen was creamy but oily (not suitable for oily skin) because of the ingredient based on the essential oil.

3.3 FTIR Analysis

From the FTIR analysis, the sunscreen is proven that contains a functional group of Methyl Mercapto that have anti-cancer properties and antimetabolite as medical treatment. Based on the research according to the guidelines for efficacy claims of a sunscreen product is recommended that should be contented UV protection, anti-aging, prevention of skin cancer, and part of medical treatment [10]. A mercapto group is a functional group containing a sulfur atom bonded to a hydrogen atom. General formula: -SH and also known as thiol group, sulfanyl group [11]. The function is an anti-cancer and has medicinal properties is also classified as an "antimetabolite" [12]. The FTIR analysis graph is shown in the **Figure 4** below:


Operator: Admin
Department: UTHM

Agilent Technologies

Sample B_2021-01-07T13-45-53

Corresponding Chemical Classification Rules

Functional Group: 'Methyl Mercapto', Chemical Class: 'Aliphatic Thiocompound', Chemical Sub Class: 'Aliphatic Thioether'

Vibration	Start WN	End WN	Threshold	Priority
C-S Stretch	750	685	Variable	Mandatory
Methyl CH3-S-	1000	960	Variable	Mandatory
C-H Bend, CH3	1455	1405	Variable	Very High
C-H Stretch, CH3-S-	3030	2855	Variable	Very High

Missing Chemical Classification Rules

Functional Group: 'Methyl Mercapto', Chemical Class: 'Aliphatic Thiocompound', Chemical Sub Class: 'Aliphatic Thioether'

Vibration	Start WN	End WN	Threshold	Priority
C-H Bend, CH3	1340	1300	Variable	Very High

Figure 4: FTIR analysis

3. Conclusion

In conclusion, raspberry seed oil based natural sunscreen provided enough UV protection by absorbing the harmful UV-rays and ensure the safety for the consumer without any harmful chemical. With lowering the use of zinc oxide, greener sunscreen will be formed, and offered broad-spectrum UV protection like the conventional sunscreen with the help of organic oil. By formulating guidelines and procedures in this project by conducting research and finding accurate information, the objectives of the study are achieved. Raspberry seed oil is giving a higher protection from the UVB rays and can protect skin from harmful UV-A rays in the sunlight. From the research, Sample B is the best combination of natural sunscreen.

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