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Calculation of SPF Value and Root Analysis on Determining Vitamin C Levels in Sunscreens Using the Fishbone Diagram

***Hanin Shohwati Muthi'ah*[1], *Ghaida Azzahra*[2], *Hana Rahmawati*[3], *Nailah Husnayain*[4]**

[1,2,3,4]Faculty of Pharmacy, Universitas Muhammadiyah Surakarta, Indonesia

[1]k100220196@student.ums.ac.id, [2]k100220261@student.ums.ac.id,
[3]k100229259@student.ums.ac.id, [4]k100229260@student.ums.ac.id

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Abstract

Objective: This research was conducted to determine the SPF value in 2 different samples of sunscreen and identify factors that affected the failure to determine vitamin C contents in each sample. **Theoretical framework:** This research is grounded in the principles of cosmetic chemistry and analytical instrumentation. Sunscreen efficacy is evaluated through SPF measurement using UV-Vis spectrophotometry. Vitamin C, a common antioxidant in sunscreens, requires precise analytical conditions for detection. Root cause analysis using the Fishbone Diagram identifies systematic errors in lab procedures, guiding improvements in cosmetic product testing and quality assurance. **Literature review:** Sunscreens are cosmetic preparations used to protect the skin from ultraviolet exposure to sunlight. The protective ability of sunscreen products is measured in terms of SPF (Sun Protection Factor). One of the many ingredients found in sunscreen is Vitamin C. Vitamin C is widely used in cosmetic formulations, due to its natural antioxidant content and its ability to protect the skin from damage caused by free radicals, as well as to enhance the skin regeneration process. **Methods:** The methodology used to determine the SPF value is UV Spectroscopy, and to analyze the problem, we conducted a root analysis using Kaoru Ishikawa's fishbone diagram. **Results:** We found that the two samples contain low SPF values (MC: 2.514, DN: 1.913) which are contrary to compendia recommendations. Then for the analysis, six main root causes of vitamin C determination are identified. **Implications:** The findings emphasize the need for consistent analytical methods and improved quality control in cosmetic testing. Identifying the root causes of failure provides a basis for refining laboratory practices. The low SPF values also suggest a need for further evaluation of product formulation and labelling accuracy. **Novelty:** Our novelty in this research is the analysis of causes of failure within an experiment conducted in the lab.

Keywords: sunscreen, spf (sun protection factor), vitamin c, root analysis, uv-vis spectrophotometer.

INTRODUCTION

Countries with tropical climates, such as Indonesia, are exposed to high levels of sunlight intensity throughout the year [1]. This results in significant exposure to ultraviolet (UV) radiation, which can have detrimental effects on skin health [2]. Prolonged exposure to UV

radiation can lead to various skin issues, including premature aging (photoaging), skin darkening, and photocarcinogenesis [3]. Photoaging occurs when UV radiation accelerates the aging process of the skin, causing it to lose its elasticity, become saggy, and form wrinkles [4]. In addition to this, UV radiation can lead to photocarcinogenesis, which refers to cellular and DNA damage that may result in the development of skin cancers, such as melanoma [5].

Aside from its effects on the skin, UV exposure also poses other health risks, such as the development of cataracts, which can impair vision [6]. This makes it essential for people living in tropical regions to be aware of the potential harm caused by unprotected sun exposure. The risk is even greater because tropical climates experience high levels of sunlight consistently throughout the year, increasing the duration and intensity of UV exposure [7].

To mitigate these harmful effects, one of the most effective solutions developed by the pharmaceutical industry is the use of sunscreen. Sunscreen acts as a protective barrier for the skin by either reflecting or absorbing UV radiation [8]. When applied properly, sunscreen prevents harmful rays from penetrating the skin, thus reducing the risk of photoaging, skin darkening, and skin cancer [9][10]. Sunscreens are formulated with ingredients that either reflect UV rays, such as zinc oxide or titanium dioxide or absorb them, such as avobenzone or oxybenzone [11][12]. These active ingredients help prevent harmful UV radiation from reaching the deeper layers of the skin [13].

Given the potential long-term consequences of excessive sun exposure, the consistent use of sunscreen is highly recommended as a preventive measure. Sunscreen should be applied generously and reapplied every two hours, especially when spending time outdoors or engaging in activities that may cause sweating or water exposure [14][15]. Moreover, it is important to choose a sunscreen that offers broad-spectrum protection, covering both UVA and UVB rays, which are responsible for skin damage and skin cancer [16].

Therefore, individuals in tropical countries need to understand the importance of protecting their skin from the harmful effects of UV radiation. By using sunscreen consistently and taking other precautions, such as wearing protective clothing and avoiding peak sunlight hours, individuals can significantly reduce their risk of developing skin-related health issues and promote overall skin health.

Significance and Novelty. This research is significant in the field of cosmetic product analysis as it highlights the importance of accurate SPF evaluation and the challenges in determining vitamin C content in sunscreen formulations. The low SPF values obtained from both samples (MC: 2.514, DN: 1.913) indicate potential discrepancies between claimed and actual protective capabilities, raising concerns about product reliability and consumer safety. Additionally, the inability to detect vitamin C content effectively calls attention to the need for standardized and validated laboratory methods [16].

The novelty of this study lies in the integration of a root cause analysis using Kaoru Ishikawa's Fishbone Diagram to systematically identify and address factors contributing to experimental failure. This approach is rarely applied in cosmetic testing and provides a structured methodology for continuous improvement in laboratory practices. By combining technical analysis with problem-solving tools, this research not only evaluates product quality but also contributes to the advancement of reliable analytical techniques in the cosmetic industry [14][15].

LITERATURE REVIEW

Ultraviolet (UV) radiation consists of UVA with a wavelength of 320-400 nm, UVB with a wavelength of 280-320 nm, and UVC with a wavelength of 100-280 nm [17]. UVB is more cytotoxic than UVA, and UVB can cause sunburn on the skin, leading to DNA damage [18].

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The protective ability of sunscreen products is measured by the Sun Protection Factor (SPF), where the higher the SPF value, the greater the protective effect of the sunscreen against UV radiation exposure [19]. The recommended SPF value depends on skin colour type; for dark skin, an SPF30+ sunscreen is recommended, while for light skin, an SPF50+ sunscreen is suggested [20]. Additionally, the SPF value should be adjusted to the surrounding climate conditions, and in tropical climates like Indonesia, an SPF30+ is recommended. However, it is often found that the SPF value stated on the packaging label does not correspond to the actual value [21].

The content of sunscreen products includes vitamin C, which functions as a natural antioxidant to protect the skin from free radicals that cause skin damage and promote skin regeneration. Moreover, vitamin C can enhance the action of sun protection agents by preventing photo-oxidation reactions through its photoprotective effects [22]. The higher the concentration of vitamin C, the better its skin-brightening effect [23]. Vitamin C derivatives are considered more stable than the original compound, as they can be hydrolyzed by skin enzymes, releasing natural vitamin C onto the skin. One of its derivatives, Ascorbyl glucoside, is more suitable for water-based formulations, has lower penetration, and can release vitamin C onto the skin after 24 hours [24].

Various challenges in the laboratory can significantly affect the results of research, such as sample variations, errors in analysis methods, lack of method validation, and external factors like environmental instability or disturbances during the research process. Identifying the root cause is crucial to understanding the factors that lead to discrepancies in research results. One method that can be used is root cause analysis. Several tools commonly employed include the fishbone diagram or cause-and-effect diagram (Ishikawa), the 5 Why's analysis, Failure Mode Effect Analysis (FMEA), and Pareto charts [25].

The fishbone diagram is a diagram shaped like a fishbone with various parts mapped out by the factors of incidents in the research. The fishbone diagram is used as an analytical technique in research processes and it is effective by utilizing identified problems, helping to find the root cause of an issue. This diagram is also commonly known as the "Ishikawa Diagram." The factors used in the fishbone diagram include man, material, method, machine, and environment [26].

METHODOLOGY

Instruments and Materials

The instruments used in this experiment include an analytical balance (Ohauss), a UV Spectrophotometer (Shimadzu), and a High-Performance Liquid Chromatography (HPLC) system Shimadzu SPD-20A (Shimadzu, Europe) equipped with a mobile phase of methanol: water (1:9 ratio) and a stationary phase of Purospher® RP-18. The HPLC specifications are: flow rate of 1 mL/min; pump pressure of 108 Kgf/cm²; detection wavelength at 265 nm. Additional equipment includes a syringe (Hamilton), drip pipette, spatula, filter paper, filter membrane, and HPLC vials. Laboratory glassware such as 25 mL, 10 mL, and 5 mL volumetric flasks were also used.

Materials used include cosmetic product samples from different brands of sunscreen cream, namely "Devnen" and "My Choice," 2N HCl, distilled water (aquadest), 96% ethanol, and a standard solution of vitamin C.

SPF Determination

1. Sample Preparation

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An amount of 100 mg of the sample was transferred into a 10 mL volumetric flask and dissolved in 96% ethanol up to the mark. The solution was sonicated and then analyzed using UV spectrophotometry [27][28].

2. SPF Value Measurement

Absorbance was measured using a UV Spectrophotometer at 5 nm intervals within the wavelength range of 290–320 nm, with each point measured in triplicate. The data obtained were then processed using the Mansur equation [29]. Results were further analyzed using the Mansur equation as follows [30].

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

Where:

CF = Correction Factor (10)

EE = Erythmogenic effect of radiation with wavelength λ

I = UV radiation intensity on λ nm

Abs = Absorbance

The value of EE × I is a constant and has been predetermined, as shown in Table 1.

Table 1. EE x I value in the wavelength range of 290 – 320 nm

Wavelength (nm)	EE × I
290	0,0150
295	0,0817
300	0,2874
305	0,3278
310	0,1864
315	0,0837
320	0,0180
Total	**1**

Root Cause Analysis in the Determination of Vitamin C Content

To identify the causes of errors in determining the Vitamin C content in this study, a Fishbone Diagram was used. The Fishbone Diagram, also known as the Ishikawa Diagram, serves to analyze the cause-and-effect relationships behind the errors in determining Vitamin C levels using HPLC [31]. The analysis begins by identifying the problem as the main effect. Then, the potential causes of the problem are categorized into six areas: man, material, method, environment, machine, and measurement. These potential causes are identified through brainstorming [32]. The analysis is visually represented in the shape of a fishbone, which helps facilitate further investigation of the issue. The problem is shown as the head of the fish, while the causes are placed along the bones [33].

RESULTS AND DISCUSSION

Determination of SPF Value

Sun Protection Factor (SPF) is a widely recognized and standardized indicator used to assess and quantify the level of protection a product or substance provides against the harmful effects of ultraviolet (UV) radiation, particularly UVB rays, which are primarily responsible for causing sunburn and contributing to skin damage and the development of skin

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cancer [34]. SPF values serve as a critical measure in evaluating the effectiveness of sunscreens and other UV-protective formulations. A higher SPF value indicates a greater ability of the product to absorb or reflect UV radiation, thereby offering more effective protection by delaying the onset of sunburn and minimizing long-term skin damage caused by prolonged exposure to the sun [35].

This study was conducted to measure the SPF values of sunscreen products under the brands “My Choice” and “Devnen” using a UV-VIS spectrophotometer. The absorbance of the samples was measured under UV light within the wavelength range of 290–320 nm, covering both the UVA and UVB regions [36]. The absorbance data in Table 1 were used to determine the SPF values, which were calculated using the Mansur equation [37] as presented in Table 2.

Table 2. SPF value on both samples (Mychoice and Devnen)

Sample	SPF value
My Choice	2,514
Devnen	1,913

The results indicate that the sunscreen sample "My Choice" has a higher SPF value compared to the "Devnen" sample. However, both samples fail to meet the required protection standards based on SPF values, falling short of both low and high protection categories as defined by the SPF classification under the European Commission Recommendation [38].

Table 3. SPF classification based on European Commission Recommendation 2006/647/EC

Protection Level	SPF Value
Low Protection	6,10
Medium Protection	15, 20, 25
High Protection	30, 40
Very High Protection	50+

According to Standard Nasional Indonesia, SNI 16-4399-1996 [39][40], a good SPF value should be at least 4. However, the SPF values obtained from the sunscreen samples, both of the brands "My Choice" and "Devnen," did not meet this requirement. These two brands exhibited SPF values that failed to comply with the standards set by both the European Commission and the Indonesian National Standard (SNI).

Root Cause Analysis of Vitamin C Content Determination

1. Content Determination

Vitamin C content was determined on two sunscreen creams branded "Devnen" and "My Choice" using the HPLC (High-Pressure Liquid Chromatography), employing a mobile phase of methanol: water in a 1:9 ratio and a stationary phase of Purospher® RP-18. The specifications included a flow rate of 1 mL/min, a pump pressure of 108 Kgf/cm², and a UV-Vis detector at a wavelength of 265 nm [41][42].

The calibration curve was prepared using a series of standard concentration solutions. The ascorbic acid standard solution was obtained from UMS (Universitas Muhammadiyah

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Surakarta) Pharmaceutical Analysis Laboratory with a concentration of 0.1% and was diluted to 0.001% before HPLC analysis. A series of ascorbic acid concentrations ranging from 10–300 µg/mL was attained, consisting of seven concentrations: 0.02%, 0.004%, 0.001%, 0.0008%, 0.00064%, 0.000512%, and 0.000409%. This curve was constructed based on the linear regression equation $y = bx + a$, where y is the chromatogram peak area and x is the concentration [43].

Before analysis with the HPLC, sample preparation was carried out for orientation purposes. One gram of sample was taken and diluted in a 25 mL volumetric flask by adding 10 mL of distilled water and shaking the mixture. The solution was stirred until homogeneous, then 10 mL of HCl was added, followed by additional distilled water up to the 25 mL mark. Filtration was performed using filter paper and a filter membrane of 0.45µm.

The determination procedure failed to produce an interpretable calibration curve and concentration results. The chromatograms of both the standard solution and the sample injection did not yield peak areas with good resolution, and the retention times were inconsistent. Consistent retention time is essential as a qualitative indicator of the presence of Vitamin C in the sample. The retention time is considered acceptable when the deviation is within ±0.1 minutes [44].

The attempt to generate a calibration curve was repeated on two separate days. The resulting regression equation was $y = 596812.56x - 290951.16$ with $r = 0.903$. This result did not meet the requirement for a good calibration curve, which is $r \geq 0.98$ [45]. A poor calibration curve leads to inaccurate determination of Vitamin C content. Therefore, the attempt to quantify Vitamin C in the sunscreen creams "Devnen" and "My Choice" in this experiment was unsuccessful. The root causes of the failure were analyzed using an Ishikawa (Fishbone) Diagram.

2. Fishbone Analysis

The Fishbone Diagram or Ishikawa Analysis method was developed by Japanese professor Kaoru Ishikawa around the 1960s during his tenure at the University of Tokyo. This method is a graphical representation that illustrates the cause-and-effect relationship or factors contributing to errors in research [46]. Based on our study, several issues were identified as contributing to the failed Vitamin C analysis using HPLC:

a) Man
Human error by personnel conducting the HPLC sample injection may have led to inaccuracies in the invalidated Vitamin C analysis method. This was caused by inadequate understanding, improper sample preparation, and inconsistency among personnel involved in the sample preparation process.

b) Material
Materials used in the Vitamin C content analysis potentially influenced the errors. Ascorbic acid is prone to oxidation, and solvents may have been impure or contaminated with other compounds, which could be detected during HPLC analysis.

c) Method
The methodology used in this study may have contributed to the inaccurate determination of Vitamin C content. The use of HCl as a solvent might have been excessively acidic, potentially leaving residues in the column that interfered with the results. Furthermore, efforts to achieve high yields by using various injection volumes may have also influenced the outcome.

d) Measurement
Detection of the sample using the HPLC system was carried out at a wavelength of 265 nm with a measurement time of 6 minutes at a flow rate of 1 mL/min. Time constraints in laboratory usage and the relatively long injection time (6 minutes) contributed to the inability to obtain the desired concentration data.

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e) Machine
The HPLC instrument was a critical factor in the success of the analysis. However, the study revealed system issues that affected performance, such as the instrument not being cleaned regularly. This was identified as a contributing factor to the poor calibration curve and unstable HPLC system.

f) Environment
Environmental conditions during analysis also contributed to the misleading results. The laboratory's exhaust system was not functioning, leading to high room temperatures and poor air circulation during sample preparation, ultimately affecting sample stability.

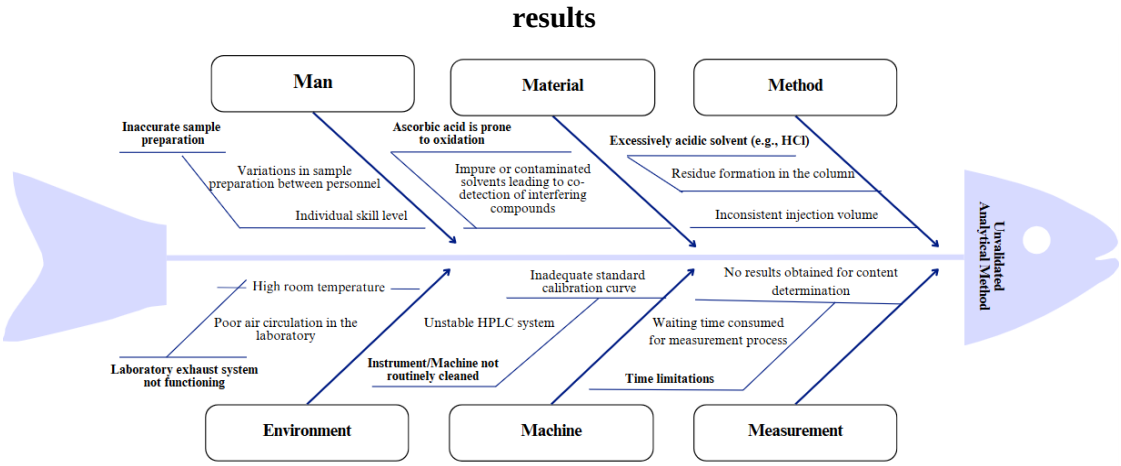

Figure 1. Fishbone Diagram on the root cause analysis of invalid Vitamin C content

CONCLUSION

This study examined the Vitamin C content and Sun Protection Factor (SPF) values of two commercially available sunscreen samples. The findings revealed that the sample labelled "My Choice" demonstrated a slightly higher SPF value (2.514) compared to "Devnen" (1.913). However, both values fall below the minimum SPF threshold as defined by the European Commission Recommendation 2006/647/EC, indicating that neither product meets the standard for classification within the low or high protection categories. Additionally, significant discrepancies were observed in the determination of Vitamin C content, prompting a root cause analysis using the fishbone (Ishikawa) diagram. The analysis identified six primary contributing factors: inadequate sample preparation (Man), the oxidative instability of ascorbic acid (Materials), residue accumulation in the HPLC column (Method), time limitations during measurement (Measurement), insufficient equipment maintenance (Machine), and suboptimal laboratory conditions due to a malfunctioning exhaust system (Environment). These findings underscore the importance of proper analytical practices, equipment upkeep, and environmental control in obtaining accurate and reliable results in cosmetic product testing.

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Author Contribution

All authors contribute equally to the publication of this paper, all authors read and agree to this paper, and all authors declare no conflict of interest.

Conflicts of Interest

All authors declare no conflict of interest.

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