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SPF evaluation of sunscreen foundations via UV-Vis spectrophotometry

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ABSTRACT: This study evaluates the accuracy of SPF labelling in seven sunscreen foundations purchased online, consisting of five BPOM-registered and two unregistered samples. Accurate SPF labelling is crucial to ensure effective protection against harmful UV radiation, which can cause skin damage such as photoaging, sunburn, and an increased risk of skin cancer. The research began by optimising analytical methods to determine the appropriate solvent and wavelength for the UV filter octyl methoxycinnamate (OMC). Validation confirmed that a 96% ethanol-chloroform (1:1) solvent with a wavelength of 309.5 nm was effective. The method demonstrated high linearity (r=0.9996), with limits of detection (LOD) and quantitation (LOQ) at 0.190 μg/mL and 0.635 μg/mL, respectively. Precision (CV=0.96%) and accuracy (100.05%-101.37%) met acceptable standards. The SPF results were as follows: A=25.47, B=22.14, C=41.71, D=49.63, E=24.97, F=2.57, G=5.69. The BPOM-registered samples generally met or exceeded their SPF claims, providing reliable protection, while the unregistered samples displayed significant inconsistencies with their labelled SPF values, raising concerns about their effectiveness and safety.

Keywords: sun protection factor (SPF), sunscreen foundation, UV-Vis spectrophotometry

1 INTRODUCTION

As a tropical country, Indonesia receives high exposure to ultraviolet (UV) light throughout the year. Excessive UV exposure can cause skin damage (Sander 2020). Therefore, the use of cosmetics containing sunscreens is necessary (Geoffrey *et al.* 2019).

Along with the increasing awareness of the risks of UV radiation, the cosmetic industry has developed various cosmetic products containing SPF. SPF is a measure of UV light energy required to achieve sunburn on skin using sunscreen (FDA 2017). Based on research conducted by Kim *et al.* (2021), the use of sunscreen and makeup products, especially foundations containing SPF, significantly increases protection against UV rays.

Foundation is one type of makeup that often claims to have SPF. Foundation serves to even out skin tone. The presence of SPF in foundation serves as a second layer of protection from UV rays on the face (Anwar and Rizkamiarty 2020).

Concerns about SPF accuracy in cosmetic products necessitate evaluating sunscreen foundation claims in Indonesia to ensure effective UV protection. This aligns with SDG 3 by promoting informed health decisions and sustainable consumer protection.

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2 METHODS

2.1 *Materials*

OMC standard (MFCI), chloroform pro analysis (Smartlab), ethanol 96% pro analysis (Merck), distilled water, stearic acid, dimethicone, methylparaben, liquid paraffin, brown pigment, propylene glycol, cetyl alcohol, span 80, tween 80, xantan gum, and sunscreen foundation preparations of various brands.

2.2 *Instrumentation*

UV-Vis spectrophotometer (Hitachi U-2910), cuvette, analytical balance (Sartorius), micropipette (IKA), vortex (Heidolph), overhead stirrer (IKA RH digital), hot plate, filter paper, spatula, and set of glassware (Pyrex).

2.3 *Sample collection*

Seven foundation samples from Shopee were purposively sampled, focusing on Indonesian brands with SPF labels, including BPOM and non-BPOM registered products, liquid formulation, and sales exceeding 1000 units/month. The selection emphasised products containing octyl methoxycinnamate (OMC), a widely used sunscreen agent known for its excellent UV absorption curve and good oil solubility. However, the study is limited by purposive sampling, a small sample size, and reliance on Shopee sales data, which may not fully represent the broader market, though it offers insights into a specific foundation market.

2.4 *Simulated foundation formula*

Table 1. Simulated foundation formula.

Ingredients	Concentration (%)
OMC	5
Stearic acid	2
Cetyl alcohol	4
Liquid paraffin	20
Span 80	1,35
Dimetichone	5
Brow pigment	0,05
Tween 80	3,65
Methylparaben	0,18
Propylene glycol	5
Xantan gum	0,3
Aquades	Ad 100%

2.5 *Preparation of simulated foundation*

The oil phase and brown pigment were melted until they reached a temperature of 70°C. The water phase except propylene glycol was dissolved with distilled water and heated to a temperature of 70°C, then propylene glycol was added while maintaining the temperature. When both phases reached a temperature of 70°C, the oil phase was added little by little into the water phase while stirring with an overhead stirrer at a constant speed of 1500 rpm for 30 minutes. Then OMC was added while stirring until a homogeneous mixture was obtained until it reached room temperature (25-30°C) (Anwar and Rizkamiarty 2020).

2.6 *Solution preparation*

Extraction solvents were made consisting of 3 variations :96% ethanol p.a; 96% ethanol-chloroform (1:1); and 96% ethanol-chloroform (1:3) and a standard solution of OMC with a concentration of 100 μg/mL was made.

2.7 *Determination of maximum wavelength*

OMC standard solution with a concentration of 6 μg/mL was measured with a UV-Vis spectrophotometer in the wavelength range of 200 - 400 nm.

2.8 *Extraction solvent optimisation*

The simulated foundation was weighed as much as 0.5 g. Then vortexed for 5 minutes and filtered with filter paper. The solution was pipetted as much as 0.5 mL and put into a 50 mL volumetric flask, then the extracting solvent was added until the limit mark. the solution was measured at the maximum wavelength of octyl methoxycinate using a UV-Vis spectrophotometer. %recovery of the three extracting solvents was calculated, one extracting solvent was chosen that had the best %recovery for the validation process and SPF analysis.

2.9 *Method validation (linearity, LOD, LOQ, accuracy, precision)*

The linearity was determined from the calibration curve of OMC standard solution made in concentration series of 3, 4, 5, 6, 7, 8, and 9 μg/mL. The calibration curve resulted in a linear equation $y = a \pm bx$. The correlation coefficient (r) was calculated from the linear regression equation to measure the linearity of the calibration curve (Raposo 2016).

The LOD and LOQ were calculated using the linear regression equation of the OMC calibration curve and calculated using the formula: LOD = 3 x SD/b and LOQ = 10 x SD/b.

The precision test was carried out by measuring the concentration of OMC in the foundation with a simulated variation rate of 100% for 6 repetitions.

The accuracy test was conducted with the spike method at three sample levels, namely 80%, 100%, and 120% and each was repeated three times. Sample preparation was performed as in the extraction solvent optimisation procedure.

2.10 *SPF analysis on sunscreen foundations*

Sample preparation was carried out as in the optimisation procedure. Then, the absorption of the sample was measured using a UV-Vis spectrophotometer every 5 nm in the wavelength range 290-320 nm and each test was repeated three times. Then, the absorption value obtained is recorded and the SPF calculation is carried out based on the Mansur Equation 1 below (Cefali *et al.* 2019).

$$\mathrm{SPF} = \mathrm{CF} \sum EE(\lambda) \mathrm{x}\, I\, (\lambda) \mathrm{x}\, Abs\, (\lambda) \tag{1}$$

Description:
EE: Erythema effect spectrum
I: Light intensity spectrum
Abs: sample absorbance
CF: correction factor (=10)

3 RESULT AND DISCUSSION

3.1 *Determination of maximum wavelength of OMC*

Figure 1 and Table 2 showed the maximum wavelength of OMC in the mixture is not much different from the maximum wavelength of OMC according to USP 41 which is

310 nm (USP 41). The difference in wavelength is due to a hypsochromic shift (Akash *et al.* 2020).

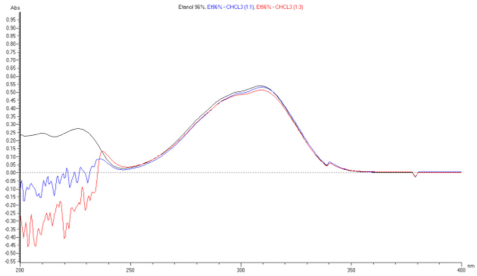

Figure 1. Wavelength spectrum of OMC.

Table 2. Maximum wavelength optimization of OMC 6 µg/ml.

Solvent	λ (nm)	Abs
Ethanol 96% p.a	309	0,541
Ethanol 96% - Chloroform (1:1)	309,5	0,532
Ethanol 96% - Chloroform (1:3)	309,5	0,513

3.2 *OMC solvent optimisation results on simulated foundation*

Optimisation results using 96% ethanol-chloroform solvent mixture (1:1) showed a % recovery of 100.68% (Table 3) which met the recovery requirements because it was in the range of 97%-103% (Riyanto 2014). This shows that the 96%-chloroform ethanol solvent mixture with a ratio of 1:1 has a solubility that matches the foundation matrix and is able to dissolve the active substance and extract it from the cosmetic matrix (Pratiwi *et al.* 2016).

Table 3. Optimisation of OMC solvent in simulated foundation.

Solvent	Mean %recovery
Ethanol 96% p.a	95,39 ± 0,119
Ethanol 96% - Chloroform (1:1)	100,68 ± 0,072
Ethanol 96% - Chloroform (1:3)	93,13 ± 0,078

3.3 *Validation of analysis method*

The method validation test results (Table 4) showed a linearity correlation coefficient of 0.9996, meeting the AOAC standard ($\geq$ 0.998). The limit of detection (LOD) of OMC is 0.190 µg/mL and the limit of quantification (LOQ) is 0.635 µg/mL, which means that concentrations below the LOD cannot be detected, while above the LOQ provide accurate data (Forootan *et al.* 2017). The precision test resulted in a coefficient of variation (KV) value of 0.96%, meeting the criteria of a good analytical method (KV $<$ 2%) and indicating that the method has good precision and accuracy. The accuracy test showed OMC recovery % of 101.37 ± 0.032% for 80% simulation, 100.05 ± 0.021 for 100% simulation, and 100.81 ± 0.016% for 120% simulation, all within the range of 97-103%, indicating the method is accurate and reliable (Riyanto 2014).

Table 4. Validation test results.

Parameter	Result	Requirement
Linearity	r = 0,9996	r $\geq$ 0.998
LOD	0,190 μg/mL	-
LOQ	0,635 μg/mL	-
Precision	KV = 0,96%	KV < 2%
Accuracy	100,05% -101,37%	97% - 103%

3.4 *Determination of SPF on samples*

According to Nur'azizah (2023), SPF 15 means 1 in 15 UV rays can penetrate the skin, with a protection effectiveness of 93.3%. BPOM-registered samples (A-E) showed SPF values close to or exceeding label claims, providing 95.4%-97.9% UV protection (Amique and Khurshid 2022). Samples D and E had SPF values close to the label, with 97.9% and 95.9% protection (Figure 2). Non-registered samples (F, G) had significant discrepancies, offering only minimal (61%) to moderate (82.4%) protection. According to the FDA (2024), SPF 15 or above is recommended to protect skin from sunburn and skin cancer. Figure 3 shows samples A-E are classified in the ultra protection category (SPF > 15), while samples F and G only protect the skin from sunburn with minimal and moderate protection, and cannot prevent skin cancer or photoaging.

Figure 2. Testing samples.

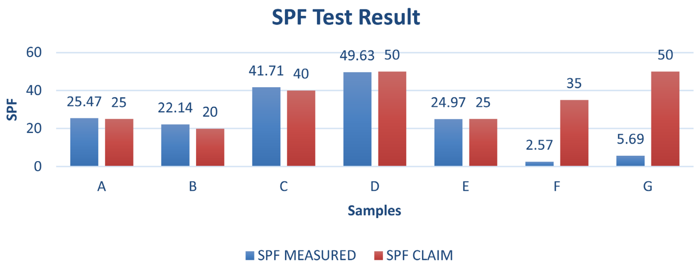

Figure 3. SPF test result.

4 CONCLUSION

BPOM-registered foundations (A-E) provide ultra-protection (SPF > 15), effectively preventing sunburn, skin cancer, and photoaging. Non-registered samples (F, G) show significant SPF discrepancies, offering only minimal to moderate protection, highlighting the need for stricter regulatory oversight.

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