

STABILITY AND PERFORMANCE OF PALM-BASED TRANSPARENT SOAP WITH OIL PALM LEAVES EXTRACT

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ABSTRACT

Oil palm leaves (OPAL) is one of the oil palm waste components that can be extracted for natural phenolics. The OPAL extracts have been successfully extracted via four different extraction procedures; extraction with ethanol (OPAL M1), deoiled and followed by extraction with ethanol (OPAL M2), deoiled and extraction with ethanolic hydrochloric acid (OPAL M3) and aqueous extraction (OPAL M4). In this study, pH, moisture content, hardness, foaming power and stability, antioxidant activity and colour stability of transparent soaps with OPAL extracts were carried out. The results indicated that all transparent soaps with OPAL extracts had similar pH in the range of 9.88 to 9.98. However, there was a significant reduction of moisture content (14.6%-16.7%) compared to transparent soap control (18.3%) due to the evaporation of water during the melting and mixing. The hardness of transparent soaps with OPAL M3 and OPAL M4 was found to be softer than transparent soaps with OPAL M1 and OPAL M2. By adding OPAL extracts, the foaming ability and stability were not affected. Transparent soap formulated with OPAL M1 extract exhibited the highest percentage of antioxidant activity (3.7%). The use of OPAL extracts is recommended in transparent soap as it provides natural colourant.

Keywords: foaming, OPAL extracts, transparent soap.

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INTRODUCTION

Malaysia and Indonesia contribute 80% of world palm oil production and dominate international trade. Sustainability and environmental issues demand the oil palm industry to look for the latest technologies, beneficial nutritional aspects, and producing value-added products for niche and new markets (Parveez *et al.*, 2020). Thus, oil palm leaves (OPAL) are a by-product of the oil palm industry, which can be exploited for the extraction of phenolics. Several studies reported that OPAL extract is rich in antioxidant activity (Ng and Choo, 2010), shows anti-microbial activities towards

gram-positive bacteria with good UVA and UVB protection for topical applications (Yusof *et al.*, 2016).

Soap is the first skin cleansing agent, discovered by Babylonians as early as 2800 BC. They made soap from boiled animal fats with wood ashes. Then, around 1500 BC, Egyptians used vegetable or animal oil and alkaline salts to produce soap with a high pH value for bathing, washing, and treatment of skin diseases. Generally, soap can be produced from three different routes, *i.e.*, saponification of oils, neutralisation of fatty acids and saponification of fatty methyl ester. Soap is the ultimate environmentally friendly surfactant because of its simple and cheap production, excellent biodegradability, low toxicity and has excellent surfactant properties (Wolfrum *et al.*, 2016). Solid soaps are available in the form of opaque, translucent and transparent soap. However, transparent soap is

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preferred due to luxurious appearance and sold at the upper-middle market segment as beauty soap (Hasibuan *et al.*, 2014). Besides, transparent soap contains a high amount of glycerin which functions as a humectant to counter the drying effects of soap towards the skin.

Soap is commonly used to reduce water surface tension and to lift dirt and oils off from our skin surface so that it can be easily rinsed away. In order to add value to the traditional soap, active ingredients from plant extract were introduced to promote healthy skin. Nowadays, cosmetic products with plant bioactive are receiving higher market demand (Emerald *et al.*, 2016). The inclusion of plant extracts in topical formulations as a source of vitamins, antioxidants and antimicrobials have been shown to improve skin tone, texture and appearance of human skin (Ribeiro *et al.*, 2015). It is well known that plant extracts are a valuable source of active compounds such as phenolics, which function as antioxidants. Many studies have been reported on the effectiveness of plant extracts, especially leaf extract in soap formulation. Pomegranate (*Punica granatum*) leaves extract was formulated in liquid and bar soaps and displayed antibacterial and antioxidant activities, whereas the solid soap only displayed antioxidant activity (Wijetunge and Perera, 2016). A combination of leaf and bark extracts of *Cassia fistula*, *Ficus religiosa* and *Millettia pinnata* in transparent soap were reported to have good antimicrobial effect (Afsar and Khanam, 2016). Anggraini *et al.* (2015) studied the characteristics and antioxidant activity of green tea leaf extract in transparent soap, and they found that 2% of green tea extract has higher antioxidant activity [15.21% 2,2-diphenyl-1-picrylhydrazyl (DPPH) scavenging activity] compared to transparent soap without green tea extract (0.83% DPPH scavenging activity). The antioxidant was used in soap formulation to prevent the oxidation of unsaturated fatty acids and enhance soap shelf life (Adigun *et al.*, 2019). Another common antioxidant used in soap products is butyl hydroxy toluene (BHT). However, this synthetic antioxidant was not popular in topical applications because it may induce allergic reactions to the skin (Yamaki *et al.*, 2007).

OPAL extract is also a potential new source of antioxidants for food and cosmetic applications. In 2011, Jaffri *et al.* analysed the composition of OPAL extract using high-performance liquid chromatography and found that the main phenolic compounds were epigallocatechin (0.08%), catechin (0.30%), epicatechin (0.01%), epigallocatechin gallate (0.28%) and epicatechin gallate (0.05%). In 2018, Ahmad *et al.* studied the effect of OPAL extract on the colour of transparent soap. The results showed that 0.1% OPAL extracts gave a better and acceptable colour compared to 0.5% OPAL extracts. However, the researchers did not investigate the stability and

performance of the transparent soap with OPAL extracts. It is important to ensure that the formulated soap with OPAL extracts has good stability and functionality. In this study, 0.1% of OPAL extracts prepared from four different extraction methods were used in transparent soap formulation. The purpose of this study was to investigate the effects of OPAL extracts on the quality of transparent soap such as pH, hardness, moisture content, foaming ability, anti-oxidant and colour stability compared to control and soap with synthetic antioxidant.

MATERIALS AND METHODS

Chemical and Apparatus

OPAL powder was obtained from Fyllo (M) Sdn. Bhd. Hexane was purchased from Fisher Scientific, USA. Ethanol with 99.9% purity, was obtained from ACI Labscan, Thailand. Sodium hydroxide (NaOH) with 99% purity was obtained from Merck, Germany. BHT with 99% purity was obtained from Sigma-Aldrich, Germany. Commercial green tea extract with known composition (water, propylene glycol, 25% of green tea extract, phenonip and EDTA) was purchased from Active Concepts, USA. DPPH radical was supplied from Sigma-Aldrich, USA. Fatty acids were purchased from Emery Oleochemicals (M) Sdn. Bhd. Glycerin was purchased from Croda, Singapore. Ethylenediaminetetraacetic acid disodium salt (EDTA) was purchased from Ajax Finechem Pty Ltd, Australia. Sodium laureth sulfate (SLES) was purchased from BASF, Germany and lactic acid was obtained from Purac Biochem, United Kingdom. Deionised water was used in this study. The spectrophotometric determination was performed on a UV-Visible Spectrophotometer, model UV-1800 from Shimadzu Corporation, Japan. Chroma Meter CR300, Konica Minolta, Inc. Japan was used to measure the colour luminosity of transparent soap. Texture analyser TA.XT plus from Micro Stable Systems, United Kingdom was used to measure the hardness of soap. While the pH of the soap solution was measured using pH meter by Mettler Toledo AG, Switzerland. Moisture analyser (XM50 from Precisa Gravimetrics AG, Switzerland) was used to measure the moisture content of transparent soap.

Preparation of Transparent Soap Base

The transparent soap base was prepared according to the method described by Ahmad *et al.* (2018). Briefly, 29% (w/v) NaOH solution was added into the melted fatty acids to form soap. Then, a premixed solution containing glycerin, EDTA, water, and SLES was added into the vessel. The mixture was heated and stirred to obtain a homogeneous

solution. Lactic acid was used to adjust the pH of the mixture to pH 9-10. Lastly, the mixture was poured into a mould and cooled down to room temperature (RT). The solid transparent soap base was wrapped with plastic to avoid sweating.

OPAL Extracts

In this study, four extraction methods of OPAL, as described by Ahmad *et al.* (2018), were adopted without any modification. OPAL M1 was prepared by extracting 20 g OPAL powder in 200 ml ethanol at 78°C for 2 hr. OPAL M2 was prepared by soaking 20 g OPAL in 200 ml hexane at RT overnight to remove fatty materials. Then, the phenolics were extracted in 200 ml ethanol at 78°C for 2 hr. The phenolic extraction method for OPAL M3 was similar to the OPAL M2, but 5 ml of 6 M HCl was introduced during the extraction of phenolic. Lastly, OPAL M4 was prepared by mixing 20 g OPAL powder with 200 ml deionised water at 100°C for 2 hr. All the crude OPAL extracts obtained were stored in the dark at RT.

Preparation of Transparent Soaps with OPAL Extract, BHT and Commercial Green Tea Extracts

Six transparent soaps were prepared namely base soap without active (control), blend of transparent soap base with 0.1 g of OPAL extracts (OPAL M1, OPAL M2, OPAL M3, and OPAL M4), soap base with 0.1 g of BHT and soap base with commercial green tea extract. About 100 g of transparent soap base was melted at temperature 60°C-70°C. Then, the active was added into the melted soap. The homogeneous mixture was poured into a mould and cooled to RT. The soap was wrapped with plastic after the soap had hardened and stored in a box at RT for analysis. Table 1 shows the soap samples prepared for this study.

TABLE 1. TRANSPARENT SOAP SAMPLES

Transparent soap	Description
Control	Soap base (without active)
S1	Soap base with 0.1 g of OPAL M1
S2	Soap base with 0.1 g of OPAL M2
S3	Soap base with 0.1 g of OPAL M3
S4	Soap base with 0.1 g of OPAL M4
S5	Soap base with 0.1 g of BHT
S6	Soap base with 0.1 g of commercial green tea extract

pH

The pH of soap solution was determined using a pH meter (Mettler Toledo AG, Switzerland). Buffer solutions pH 4 and 7 were used to calibrate

the pH meter. Electrode InLab® Routine Pro was washed with distilled water and dried. The soap solution was prepared by diluting 1.0 g of soap sample in 10 ml of distilled water. The pH meter electrode was dipped into the soap solution, and the pH was recorded after constant reading was achieved.

Hardness

The hardness of transparent soap without OPAL extracts, with OPAL extracts, BHT and commercial green tea extract were evaluated by penetration method using a texture analyser TA.XT plus (Micro Stable Systems, UK). A stainless-steel needle (P/2N) with a diameter of 2 mm was used for the measurement. The needle was dipped 7 mm into a soap sample with a moving rate of 2 mm s⁻¹. The test was carried out at RT. This method was adapted from ASTM standard method D1321-95. The maximum force was defined as hardness. The data were expressed in Newton (N).

Moisture Content

The moisture content of transparent soap, with and without OPAL extracts and soap with BHT and commercial green tea extract was determined by using a moisture analyser (XM50 from Precisa Gravimetrics AG, Switzerland) with a halogen heater. Two g of soap sample was accurately weighed with a precision of 1.0 mg and placed in aluminium dishes in the moisture analyser. The temperature of heating was 105°C. The weight of the soap sample was measured once it has reached constant weight (g). The result is represented in % of moisture content and reported in average value.

Foaming Power and Stability

The foaming power and stability of soap solutions were assessed using a method developed by Benn *et al.* (2017) with slight modification. Foaming power is a measure of foam height taken immediately after 200 ml of the test solution was stroked for 30 times at a constant rate using a perforated base rod. The test solution was prepared by dissolving 0.5 g of product with 200 ml of water in a 500 ml measuring cylinder. The foam was let to rest for 5 min before the foam height was measured, which indicated foam stability. The analysis was performed in triplicate and data reported the average value. The foaming power and foam stability of the transparent soaps with OPAL extracts were tested in deionised water and 50 ppm water hardness (water with calcium carbonate, CaCO₃).

Antioxidant Activity

The method for antioxidant activity, as described by Enujughha *et al.* (2012) was adopted with few modifications. An ethanolic solution of DPPH (18.18% v/v) was prepared and stored at 10°C in the dark. Soap samples were prepared in ethanol solution with a concentration of 2 mg ml⁻¹. About 0.15 ml of the sample solution was then added to a 2.85 ml ethanolic DPPH solution. The mixture was shaken and left to stand at RT for 1 hr in the dark. The colour changes from deep violet to light yellow were measured spectrophotometrically at 515 nm. The absorbance of DPPH solution without soap sample (control), soap without OPAL extracts, soap with BHT and soap with commercial green tea extract were also measured. All measurements were performed in three replicates and the average was counted. The percentage of inhibition of the DPPH was calculated according to the formula as followed:

$$\text{DPPH inhibition, \%} = \left[\frac{A_{(\text{control})} - A_{(\text{sample})}}{A_{(\text{control})}} \right] \times 100$$

where $A_{(\text{control})}$ is the absorbance of the control, and $A_{(\text{sample})}$ is the absorbance of the sample.

Colour Stability

Colour stability of the soap was evaluated at day 7 and monthly for 3 months. The measurement was carried out using a Chroma Meter CR300 (Minolta, Japan). The Chroma Meter measures the sample surface of 8 mm in diameter at wavelengths (400-700 nm) of transmitted light, standard observer (0°), under illuminant D65 and white plate calibration. The soap was placed on a white tile and measurement was taken from the top of the soap. The readings of the soap samples were recorded in the CIELAB system in the form of L* and b* values. The system measures L* which refers to luminosity black (0) to white (100) and b* positive which refers to yellow colour. A total of ten readings were taken and a mean value was calculated. Yellowness Index (YI) which indicates the degree of yellowness was determined using Equation 1 (Rhim *et al.*, 1999).

$$\text{YI} = 142.86 \text{ b}^*/\text{L}^* \quad (1)$$

where b* is the yellow colour scale, and L* is the luminosity scale.

Statistical Analysis

The data obtained from the above studies were analysed statistically using Microsoft Excel version 2013 for analysis of variance (ANOVA) single factor

and Student t-Test. The differences were considered significant if the probability, $p < 0.05$.

RESULTS AND DISCUSSION

pH and Moisture Content of Transparent Soap

The pH and moisture content of transparent soap samples were determined after one week of storage at RT. The results obtained from analyses of pH and moisture content of transparent soaps are presented in Table 2. The pH of all transparent soaps analysed in this study fall within the range between 9.88 and 10.02. It was observed that the addition of 0.1% OPAL extracts, BHT and commercial green tea extract in the transparent soap have significantly reduced the pH of soap samples compared to control. In the preliminary screening of five different commercial transparent soaps which were sourced locally, the pH of the soaps was determined in the range of 9.19-9.60. Kulthanan *et al.* (2014) reported that the pH for bar soaps marketed in Thailand was in the range of 9.8 to 11.3. In addition, Dlova *et al.* (2017) reported that commercial bar soaps obtained in South Africa had pH values ranging from 9.36 to 10.75. Thus, the pH of the formulated transparent soaps in this study is in agreement with the commercial bar soaps. Several studies have shown that the use of alkaline soaps increases skin pH. Korting *et al.* in 1990 and 1996 reported the impact of long-term and short-term effects of alkaline (pH 8.5), acidic (pH 5.5), and neutral (pH 7.0) cleansers on skin pH. It was reported that skin pH increased significantly after washing with alkaline cleanser. However, a slight increase was also found after the usage of an acidic product as well as after washing the skin with a neutral cleanser. Another study carried out by Takagi *et al.* (2015) found that continuous application of soap at pH 10.3 for 6 hr did not adversely affect the skin pH. After 6 hr, the skin pH returns to normal acidic conditions. Thus, the alkaline pH of soap does not significantly affect healthy skin.

TABLE 2. MEAN pH AND MOISTURE CONTENT OF TRANSPARENT SOAPS

Transparent soap	Mean pH ± SD	Mean moisture content (%) ± SD
Control	10.02 ± 0.01 ^a	18.36 ± 0.27 ^a
S1	9.88 ± 0.03 ^b	14.64 ± 0.09 ^b
S2	9.90 ± 0.02 ^b	15.70 ± 0.10 ^c
S3	9.93 ± 0.03 ^{b,c,d,e}	15.72 ± 0.23 ^c
S4	9.96 ± 0.01 ^c	16.73 ± 0.12 ^d
S5	9.98 ± 0.01 ^d	16.68 ± 0.10 ^d
S6	9.92 ± 0.01 ^c	18.31 ± 0.35 ^a

Note: Values with the same superscript letter are not statistically significant at the 5% level; SD – standard deviation.

Table 2 shows that the mean moisture content of transparent soap S1 – S5 reduced significantly compared to control and transparent soap S6. These variations may be due to the evaporation of water during melting of the soap base and mixing of active. It was observed that the transparent soap S1 had the lowest moisture content. According to Ahmad *et al.* (2018), the OPAL M1 was waxy, and thus incorporation of this active into transparent soap needs prolonged heating, which resulted in low moisture content. However, commercial green tea extract was easy to mix with the soap base. Thus, less evaporation of water was observed for transparent soap S6. The transparent soaps moisture content falls within the range reported by Kuntom *et al.* (1996), which was 8%-14%. It was reported that the appearance of active, either waxy or not, also affected the moisture content of transparent soap. The moisture content values of all transparent soap samples in this study are within the ranges obtained by Vivian *et al.* (2014) *i.e.* 10.91%-22.69% moisture for the commercial soap sold in Kenya, Osuji *et al.* (2013) *i.e.* 18.3%-22.5% moisture for soap with palm oil sludge, tallow and palm kernel oil as well as Kuntom and Kifli (1998) *i.e.* 10%-18% moisture for the soap blends with palm stearin and palm kernel fatty acids.

Physical Properties of Soap

The results from the analyses of hardness for palm-based transparent soap with 0.1% of OPAL extracts, 0.1% commercial green tea, BHT and control are depicted in Figure 1. A reduction in penetration force indicates that the soap is softer, as less force is used to penetrate a fixed distance. It was observed that the addition of OPAL extracts, BHT and commercial green tea extract into the transparent soap base significantly reduced the hardness of bar

soap compared to control at seven days of storage. In accordance with the present results, a previous study by Anggraini *et al.* (2015) has demonstrated that the addition of green tea extract in transparent soap reduced the soap hardness. A possible explanation for this result is that the addition of an active ingredient in the form of liquid may cause a reduction in soap hardness. In addition, the results of this study showed that the hardness of palm-based transparent soap remained stable during the six months storage and within the average hardness for commercial transparent soaps in Malaysia which is 3.80-6.96 ± 0.01 N.

Besides hardness, palm-based transparent soaps with 0.1% of OPAL extracts, 0.1% commercial green tea extract, BHT and control were also assessed for their foaming ability and foam stability in deionised water *vs.* 50 ppm water hardness (Figure 2). The water hardness will prevent the lathering of soap (Srinivasan *et al.*, 2013). Thus, the study was carried out to determine whether hard water would adversely affect the lathering of palm-based transparent soap. The water hardness concentration of 50 ppm of CaCO₃ was chosen in this study based on a survey carried out by Ong *et al.* (2007), which reported that the hardness of tap water in Klang Valley, Malaysia was in the range of 48-92 ppm of CaCO₃. Based on this study, there was a statistically significant difference in foam performance of palm-based transparent soap in deionised and hard water, where the presence of 50 ppm CaCO₃ decreased the foaming power and foam stability of palm-based transparent soap samples. Based on analysis of variant, there is no significant difference in foaming ability and foam stability for the palm-based transparent soap with OPAL extracts (S1 - S4) and control. Thus, indicating that the addition of OPAL extracts did not affect foaming power and foam stability.

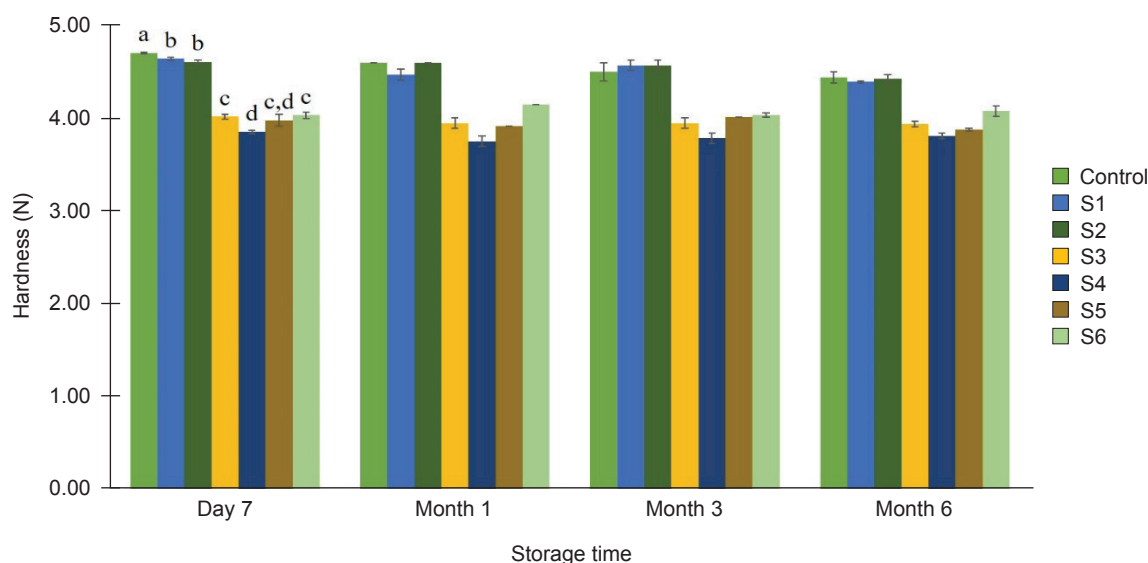


Figure 1. The hardness of palm-based transparent soap with 0.1% of OPAL extracts, 0.1% commercial green tea, BHT and control. Lower hardness value indicates the softness of soap. Values with the same superscript letter are not statistically significant at the 5% confidence level.

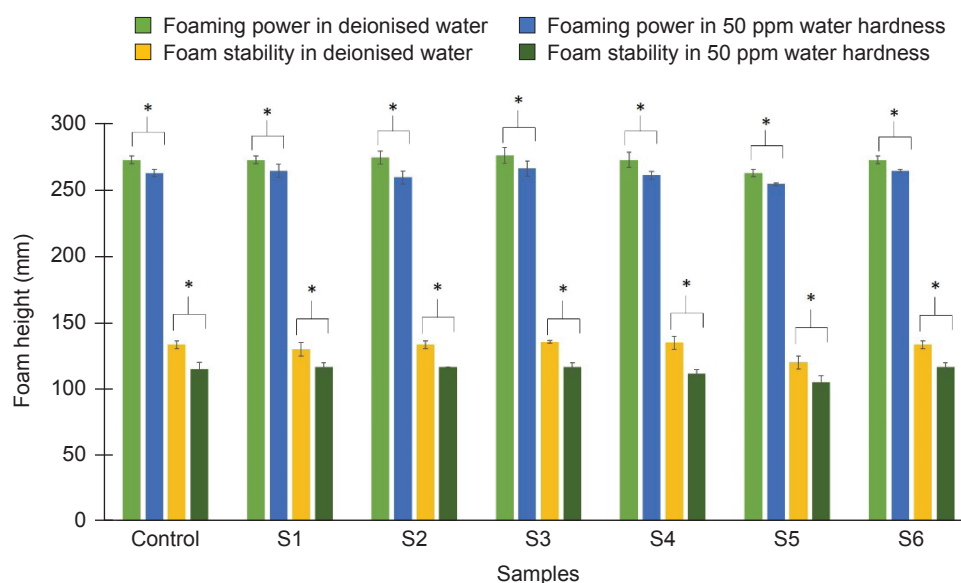


Figure 2. Foaming power and stability of palm-based transparent soap with 0.1% of OPAL extracts, 0.1% commercial green tea extract, BHT and control in deionised water vs. 50 ppm water hardness. * statistically significant at the 5% confidence level.

Antioxidant Activity

The addition of OPAL extracts in transparent soap was determined for their capacity to inhibit DPPH radicals and transparent soaps with BHT and green tea extract were selected as the benchmarks. BHT is a synthetic antioxidant, extensively used in many cosmetic and food products due to its chemical stability and inexpensive (Ghosh *et al.*, 2020; Yamaki *et al.*, 2007). While commercial green tea extract was chosen due to the known composition of the ingredient and commercially available extract. Based on these results, it was found that the addition of OPAL extracts into transparent soap significantly helped to increase the antioxidant activity (Table 3). In addition, S1 soap formulated with OPAL M1 extract exhibited the highest DPPH inhibition compared to other soaps with OPAL extracts and transparent soap with green tea extract. Irine *et al.* (2003) ascribed that OPAL extract has 8% higher content of phenolic compounds than green tea. Fadda *et al.* (2014) studied the reaction time and kinetic behaviour of plant extracts (green tea, pomegranate and lemon) with DPPH radical. They reported that the antioxidant activity was strongly influenced by DPPH concentration and reaction time. They also presented plant extracts that will perform either fast or slow kinetic behaviour. Thus, the antioxidant activity of transparent soap with green tea probably has slow kinetic behaviour, which may need more than 1 hr to react with DPPH radical. In addition, the commercial green tea extract contains 25% active material. Due to the low content of the active ingredient, it has caused low antioxidant activity. Since the purpose of this work is to investigate the antioxidant activity of transparent soap with OPAL

extracts, so we have not investigated a proper reaction time for transparent soap with green tea. However, the antioxidant activity of soap with OPAL extracts was still lower than soap formulated using synthetic antioxidant, BHT. Fatiha and Abdelkader (2019) reported that BHT would react rapidly with DPPH and gave the highest DPPH inhibition compared to other compounds, which may require longer reaction times and higher concentrations.

TABLE 3. ANTIOXIDANT ACTIVITY OF TRANSPARENT SOAPS

Transparent soap	DPPH inhibition (%) \pm SD
Control	0.182 \pm 0.000 ^a
S1	3.703 \pm 0.105 ^b
S2	2.004 \pm 0.105 ^c
S3	1.639 \pm 0.000 ^d
S4	1.822 \pm 0.000 ^e
S5	34.244 \pm 0.182 ^f
S6	0.182 \pm 0.000 ^a

Note: Values with the same superscript letter are not statistically significant at the 5% level; SD - standard deviation.

Colour Stability

The addition of OPAL extracts resulted in an increase in YI of palm-based transparent soap with 0.1% of OPAL extracts compared to transparent soaps with BHT, green tea extract and control (Figure 3). Besides OPAL extracts, green tea extract was also reported to enhance the colour of transparent soap at 0.5% concentration (Anggraini *et al.*, 2015). Palm-based transparent soap S2 (soap with OPAL M2) gave the highest YI compared to

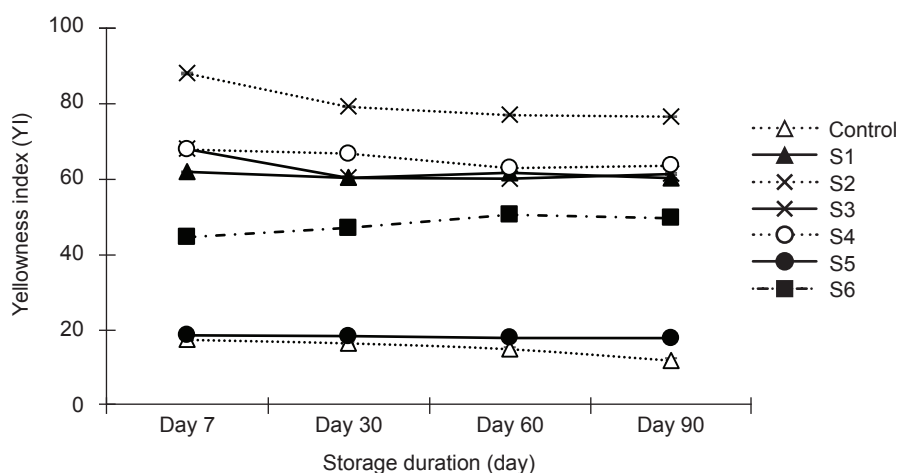


Figure 3. Yellowness index of palm-based transparent soap with 0.1% of OPAL extracts, BHT and control.

other transparent soaps. Furthermore, the yellow colour of palm-based transparent soap, S1 – S5 remains stable during storage from day 30 to day 90. Therefore, OPAL extract can be used as natural yellow colour and the presence of antioxidants in OPAL extract may help in terms of colour stability.

CONCLUSION

The addition of OPAL extract in transparent soap formulations did not affect foaming power and foam stability. However, transparent soap with OPAL M1 extract showed the highest antioxidant activity than transparent soap with other OPAL extracts and commercial green tea extract. In addition, all OPAL extracts showed stable hardness throughout the study and provided natural yellow colour to the transparent soaps within 90 days of storage.

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