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EFFECT OF WATER CONTENT ON WATER IN OIL (W/O) EMULSION PROPERTIES BASED ON COCOA BUTTER FOR COSMETIC RAW MATERIAL

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ABSTRACT

Cocoa butter consist of stearic acid, oleic acid, palmitic acid, linoleic acid and vitamin E. It can be used as additional ingredient or raw material for cosmetics formulation in emulsion form. One of the emulsion type that widely used as base for skincare product is water in oil (W/O) emulsion. The purpose of this study was to evaluate the effect of water concentration on the W/O emulsion and its stability based on cocoa butter for cosmetic raw material. W/O emulsion was made by mixing water phase in oil phase at temperature 70°C until homogeneous for 1 hour then allowed to harden at room temperature. In this study the water concentration was varied 12%, 16%, 20%, 24%, 28% and 32%. The characterization carried out were physical appearance, hardness, homogeneity, droplet size, melting point, chemical functional group and sensory test. The study showed that water concentration affected W/O emulsion final product in parameter of texture, melting point and droplets size. The formulation with higher water content produce softer texture, lower melting point, lower hardness and increase droplets size. However, all formulation showed good homogeneity and stability for 5 weeks observation at room temperature. There was no significant change in color, aroma and consistency of physical appearance and there was no phase separation occurred. The absence of evolution in FTIR spectra also indicated that the W/O emulsion also chemically stable during the storage. Finally, the formulation assessment was done based on the sensory test where the formulation with water content 20% was the most preferred formulation with an average score of 6.61.

Keywords: cocoa butter; cosmetics; W/O emulsion; sstability; sensory test.

INTRODUCTION

Cocoa butter is derived from the seeds of Theobroma cacao tree. It is a pale yellow vegetable fat that mainly consists of stearic acid (18:0), oleic acid (18:1), palmitic acid (16:0), and linoleic acid (18: 2). It also contains vitamin E and low amounts of lauric acid and myristic acid [1]. Cocoa butter remains solid at room temperature (below 25ºC) and melts at body temperature (37ºC). Cocoa butter has been widely used as raw material in several applications such as food (as chocolate), pharmaceuticals (as a base for suppositories and topical bases), and cosmetics (as excellent emollient) [2,3].

Cocoa butter is quickly absorbed by the skin because the melting point of cocoa butter is lower than human body temperature, which is 30-35ºC. In addition, the composition of cocoa butter is close to the composition of skin fat thus it is safe to use [4]. Cocoa butter can be applied as additional ingredient or raw material for cosmetics, such as lipstick [5], hand body lotion [6], shampoo [7], and sunscreen [8]. According to Ramlah (2017) the addition of cocoa butter to hand body lotion produced a product that could improve moisture, oil content and skin smoothness and improved skin elasticity [6].

Emulsion is the most common preparation used in cosmetics and skin care because of their varied consistency from liquids, lotions to creams. Emulsion is a complex mixture of two immiscible phases, with one phase dispersed in the other phase. There are two types of emulsion namely "Oil in water" (O/W) emulsion and "water in oil" (W/O) emulsion. A system consist of dispersed oil droplets in the water phase is called oil in water emulsion or O/W emulsions, while a system consist of water droplets dispersed in the oil phase is called water in oil or W/O emulsion [9]. O/W emulsions are commonly used as water-washable drug bases and for general cosmetic purposes and W/O emulsions are widely used as emollients and for dry skin treatment [10].

W/O emulsions can be used to encapsulate compounds in fat-based product, in this case is cacao butter. Emulsion system comprise of several elements such as oil, water and surfactant. Water phase play an important role in W/O emulsions because the high mobility of water droplets can promote W/O emulsion destabilization [11]. Thus, the stability study becomes key factor. W/O emulsion in cosmetic application required to be stable over a long period [12]. This is prominent considering that preparations are usually produced in large quantities and take a long time to reach consumers [13]. Changes in physicochemical properties are observed whether there are changes in the basic formulation through stability test. Cosmetic preparations are said to be stable if they are still have the same properties and conditions within acceptable limits as at the time of manufacture during the storage and use period [14].

The purpose of this study was to evaluate the effect of water ratio on the properties and stability of the W/O emulsion based on cocoa butter for cosmetic raw material. Characterization carried out included physical appearance, hardness, homogeneity, melting point, droplets size, chemical functional group and sensory test.

METHODOLOGY

W/O emulsions were processed by mixing oil phase and water phase. The W/O emulsion from this process was observed for its properties and stability for five weeks with certain parameters. The detailed material, composition, methodology and characterization are described below.

Material

The materials used in this study namely, cocoa butter from Biotechnology Research Center LIPI (Indonesia), beeswax technical grade (Brataco, Indonesia), avocado oil (Brataco, Indonesia), Euxyl PE 9010® (Schűlke, USA), Soluvit richter NP® (CLR, Germany), caprylic acid (Brataco, Indonesia), Span 80® (Merck, Germany), propylene glycol (Brataco, Indonesia) and water.

The Making of Emulsion

The variation of the formulation is shown in Table-1. Oil phase consisted of cocoa butter, beeswax, Euxyl PE 9010®, Soluvit Richter NP®, avocado oil, caprylic acid, and Span 80®. Water phase consisted of propylene glycol and water. Oil phase was mixed and stirred at temperature of 70-80°C until homogeneous. While water phase was stirred separately at temperature 50ºC. The water phase was added gradually into the oil phase while maintaining the temperature at 70°C. The mixture was stirred until W/O emulsion formed for 1 hours then cooled and allowed to harden at room temperature.

Table-1. Variations in w/o emulsion composition (% weight).

Materials	F1	F2	F3	F4	F5	F6
Cocoa Butter	24	24	24	24	24	24
Beeswax	24	24	24	24	24	24
Avocado Oil	22.8	18.8	14.8	10.8	6.8	2.8
Euxyl PE 9010®	0.4	0.4	0.4	0.4	0.4	0.4
Soluvit Richter NP®	4	4	4	4	4	4
Caprilic Acid	4.8	4.8	4.8	4.8	4.8	4.8
Span 80®	4	4	4	4	4	4
Water	12	16	20	24	28	30
Propylene Glycol	4	4	4	4	4	4

Characterization

W/O emulsion were tested for their physical properties by observing changes in color, texture and comfort on the skin during room temperature storage in the first week until the fifth week. Homogeneity test was done by smearing the sample on a transparent flat glass surface and the presence of coarse particles was observed, hardness test using Food Technology Corporation Texture Analyzer based on ASTM D1321-10, melting point test using Melting Point Analyzer Fisher Scientific, droplets size analysis using Horiba LA-960 and analysis of chemical functional groups using Fourier Transform Infra-Red (FTIR) Shimadzu IR Prestige-21.

The sensory test using the hedonic test method based on SNI 01-2346-2006 about organoleptic and/or sensory testing. This test for determining the user's response to the sample after application to the skin. This test was performed on women and men with age ranged from 21-40 years. The test was done by filling the assessment form with score 1-9 where each number represent the level of preference of the tested sample.

RESULTS AND DISCUSSIONS

Physical Stability Test of W/O Emulsion

The stability test aimed to evaluate W/O emulsion characteristics under by observing the physical endurance of the product under certain condition and duration. Test was carried out at room temperature by observing changes in color, texture, aroma and comfort on skin for 5 weeks. The results of W/O emulsion stability test are shown in Table-2.

The result of color observation on the formulation showed a white to yellowish color which was the natural color of cocoa butter and beeswax. The results of the aroma observation gave distinctive cacao aroma which was derived from the basic ingredients of cocoa butter. Meanwhile, texture observation was related to skin comfort parameter. The texture of this formulation depends on the water content. Formulation with low water content produced a soft texture but very oily corresponded to higher oil composition. Meanwhile formulation with high water content gave mushy texture caused sticky and uncomfortable impression when applied on the skin. Water has the role as plasticizer in emulsion system. The addition of plasticizer into a rigid material could modify a hard material to a highly viscous liquid which had softer texture. The plasticization occurred because of two factors.

The first was an affinity of water as plasticizer to the macromolecular compound and its interaction with charged and polar groups. The second is an increase of the gap between macromolecules and a decrease in inter-molecular interaction because of water dilution [15]. The observation for five weeks showed that there was no significant change in color, aroma and texture and there was no phase separation occurred, yet the addition of water significantly affect the hardness of W/O emulsion.

Table-2. W/O emulsion physical stability test.

Sample	Weeks	Observation			
		Color	Texture	Aroma	Comfort
F1	1-5	Yellow, stable until the fifth week	Soft, oily	Typical cacao, stable until the fifth week	Too oily, uncomfortable to apply
F2	1-5	Yellowish white, stable until the fifth week	Soft, slight oily	Typical cacao, stable until the fifth week	Easy to seep-oily, uncomfortable to apply
F3	1-5	Yellowish white, stable until the fifth week	Soft, slight oily	Typical cacao, stable until the fifth week	Easy to seep, comfortable to apply
F4	1-5	Yellowish white, stable until the fifth week	Soft, slight oily	Typical cacao, stable until the fifth week	Easy to seep, comfortable to apply
F5	1-5	Yellowish white, stable until the fifth week	Too soft	Typical cacao, stable until the fifth week	a little sticky, uncomfortable to apply
F6	1-5	Yellowish white, stable until the fifth week	Too soft	Typical cacao, stable until the fifth week	a little sticky, uncomfortable to apply

Hardness of W/O Emulsion

The physical appearance observation showed that the water concentration on W/O emulsion affected the hardness of the final product. It was quantified using texture analyzer based on ASTM D1321-10 with modification as shown in Table-3.

Table-3. Hardness of W/O Emulsion.

Formula	Hardness (N)
F1	4.763 ± 0.204
F2	4.586 ± 0.008
F3	3.265 ± 0.204
F4	3.640 ± 0.035
F5	3.402 ± 0.074
F6	1.843 ± 0.015

Formula F1 and F2 hardness was in the range 4.5-5.7 N, Formula F2, F3, F4 hardness was in the range 3.2-3.6 N, while formula F6 had the lowest hardness value. The ratio between water and oil strongly affect hardness and spreadability of the emulsion product. As the water content increased, the product consistency shifted to liquid state. Therefore, less fat content supported fat crystal network which directly affected the final product firmness. It was proved that increasing water content from 12% to 32% decrease the product hardness [16].

Droplet Size of W/O Emulsion

The addition of water as plasticizer also affects the droplet size of the emulsion. The result of droplet size analysis is shown in Table-4.

Table-4. Droplet Size of W/O Emulsion.

Formula	Mean Diameter (μm)
F1	2.46896 ± 0.00148
F2	3.54008 ± 0.02764
F3	3.80650 ± 0.00322
F4	7.15205 ± 0.00385
F5	8.14223 ± 0.13529
F6	11.17629 ± 0.03534

The formula with the lowest water content had the smallest droplet size that was 2.46896 ± 0.00148 μm and the formula with the highest water content had the biggest droplet size that was 11.17629 ± 0.03534 μm. Similar result also reported by Silva *et al*., (2019), where larger droplets size in irregular shape were found in emulsion sample with water concentration of 25-30% (w/w) [17]. The presence of large droplet size occurred caused by coalescene in emulsion system due to high water concentration thus it changed the uniformity of particle size distribution. It was proven by the droplet size distribution analysis shown in Figure-1 where formulation with water content 12-20% showed unimodal distribution

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while the formulation with water content above 20% showed multimodal distribution. This phenomenon was related to a reduction of emulsifier concentration in the formulation in consequent of the increase of water concentration [18].

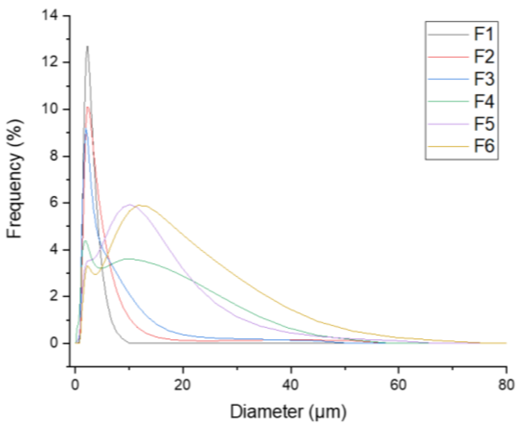

Figure-1. W/O emulsion droplet size distribution.

Homogeneity of W/O Emulsion

The homogeneity test aimed to observe and determine whether the ingredients were mixed properly. The test was carried out by smearing W/O emulsion on a flat glass object, then the glass object was pressed with another glass object. According to Nurany *et al*., (2018) homogeneous product had no coarse grain on the glass surface that had been smeared by the product [19]. The homogeneity test result is shown in Figure-2.

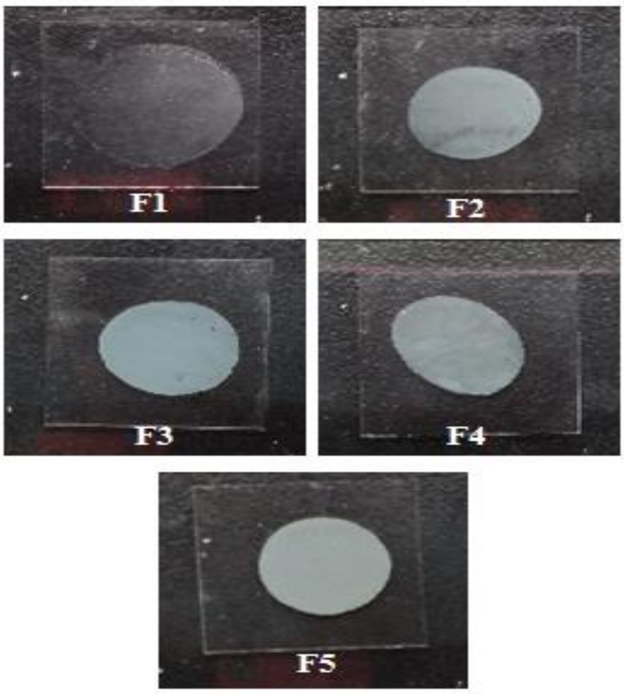

Figure-2. Homogeneity test of w/o emulsion.

Based on the results of the observations all formulations had good homogeneity which was characterized by a smooth, even surface and the absence of coarse grains. This indicated that all ingredients mixed well. Homogenous emulsion caused by good dispersion between oil droplets and water by reducing the surface tension with the addition of sufficient amount of emulsifier. The well-homogenized emulsion consisted of molecules with appropriate small particle and had no tendency to increase its size over time which leads to separation [20]. According to the previous discussion, the droplets size of W/O emulsion in this study ranged between 2-11 µm.

Melting Point of W/O Emulsion

The melting point is one of the test for cosmetic quality which represents the stability of product during the manufacturing, storage, usage period and transportation. The composition of the material is an important factors which could yield different melting point value [21]. The melting point results obtained from W/O emulsion formulations are presented in Table-5.

Table-5. Melting point for w/o emulsion.

Sample	Melting Point (ºC)	
	Week 1	Week 5
F1	55	53
F2	58	52
F3	54	52
F4	51	50
F5	49	49
F6	45	45

The melting point of raw material in the formulation affected the melting point value of the final product. The melting point of beeswax is 62-64ºC, while the melting point of cocoa butter is 31-35ºC. The test result showed the W/O emulsion had melting point in the range of 45-55ºC. Similar result was reported by Kamairudin et al., (2014) where the cosmetic preparation with raw material beeswax and other wax had melting point ranged from 41-51ºC [22].

Formulation with low water concentration had higher melting point compared with the formulation with high water concentration. This caused by the formulation with low water concentration corresponded with higher fat and emulsifier content which lead to formation of more stable polymorphs. While, the formulation with higher water content caused more emulsifier adsorption at the interface thus reduced the amount emulsifier available to form crystallization in bulk fat, hence decrease the melting point [18].

Chemical Functional Group of W/O Emulsion

The chemical functional group of W/O emulsion were analyzed using FTIR and the results of the analysis

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are shown in Figure-3 and Table-6. The infrared band interpreted based on Lambert *et al.*, (1998) [23]. Based on the analysis results, all formulations had similar results with several dominant functional groups such as OH in alcohol and phenol, $-CH_3$ and $-CH_2$ in aliphatic compounds, $-CH_3$ attached to O or N, and C=O. Based on the results of FTIR analysis, W/O emulsion was produced through the physical mixing process of various raw materials, of which there were no new functional groups that indicated the formation of new compound. The FTIR analysis was conducted on the week 1 and week 5 to observe any chemical change during the storage. It was seen that the functional group before and after the storage for five week exhibited similar functional group with no significant shifting. It could be said that the W/O emulsion was chemically stable during five weeks storage at room temperature. Similar behavior also reported by Masmoudi *et al.*, (2005) in stability test on O/W emulsion sample at 0-25ºC where there was no evolution in FTIR spectra. This was caused by emulsion oxidation didn’t occurred at room temperature, however emulsion might started to oxidize at temperature above 40ºC [24].

Table-6. Wavelength functional groups of w/o emulsion.

OH in alcohols or phenols						
	F1	**F2**	**F3**	**F4**	**F5**	**F6**
Week 1	3377.50	3378.47	3377.50	3377.50	3283.95	3381.36
Week 5	3388.93	3398.57	3383.29	3389.08	3375.57	3388.93
$-CH_3$ and $-CH_2$ in aliphatic compound						
Week 1	2919.39	2919.39	2919.39	2919.39	2918.42	2919.39
Week 5	2918.30	2918.30	2917.46	2919.39	2920.35	2920.23
$-CH_3$ attached to O or N						
Week 1	2851.88	2852.84	2850.91	2851.88	2850.91	2850.91
Week 5	2854.65	2856.58	2852.84	2853.81	2854.77	2856.58
C=O						
Week 1	1743.72	1743.73	1743.74	1743.75	1743.76	1743.77
Week 5	1741.72	1734.01	1741.80	1741.80	1741.80	1737.86

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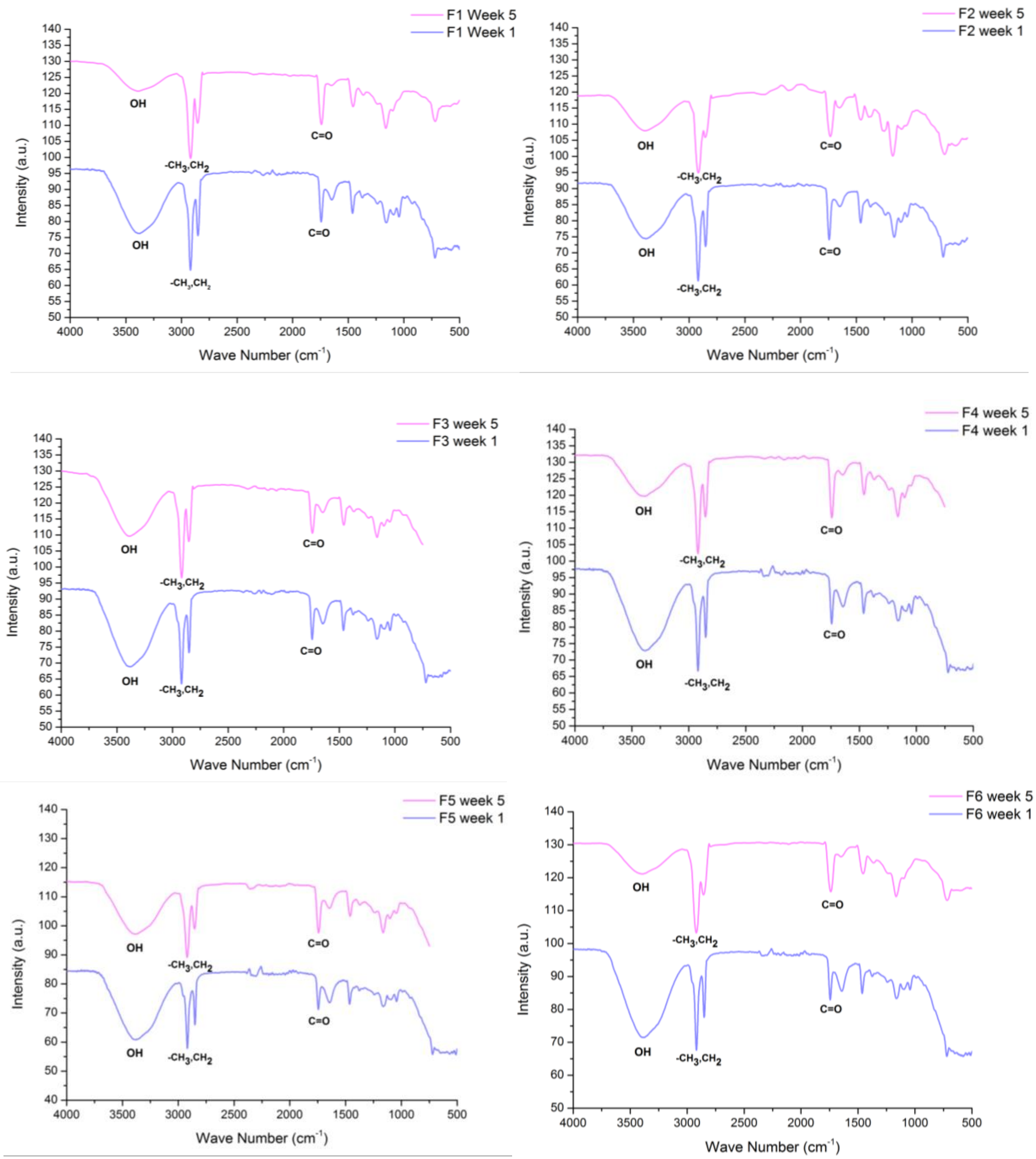

Figure-3. FTIR spectra functional groups of w/o emulsion formulation.

Sensory Test of W/O Emulsion with Hedonic Test Method

The sensory test was carried out on all formulations using hedonic test method. The test refered to SNI 01-2346-2006 about Organoleptic and/or Sensory Testing. The sensory test is a method of testing using the five human senses. The test was done by selecting the assessment score ranged from 1 (very dislike) until 9 (very like), where each number indicated the level of preference for the tested samples [25]. The average product preference for test parameters such as color, texture and comfort on the skin are shown in Table-7.

Table-7. Hedonic test preference score of w/o emulsion.

Formula	Color	Texture	Comfort	Average
F1	5.70	6	6.48	6.06
F2	6.43	6	6.39	6.27
F3	6.91	6.48	6.43	6.61
F4	6.83	6.35	6.30	6.49
F5	6.57	6	5.91	6.16
F6	6.91	6	5.78	6.23

The formulation that the panelists preferred based on hedonic test was the formula F3 with water concentration 20% by score of parameter color, texture and comfort 6.91, 6.48 and 6.43 respectively, while average score for the three parameters was 6.61. This sensory analysis could identify the level of acceptance of a product which was the basis for product development and a reference to the concentration limits of the used ingredients [26].

CONCLUSIONS

Different water content affected W/O emulsion final product in parameter of texture, melting point and droplets size. The formulation with higher water content produce softer texture thus made uncomfortable sensation on skin. The water content in W/O emulsion also corresponded to melting point wherein higher water content decrease melting point and increase droplets size. However, all formulation showed good homogeneity and stability for 5 weeks of observation at room temperature. There was no significant change in color, aroma and consistency of physical appearance and there was no phase separation occurred. The absence of evolution in FTIR spectra also indicated that the W/O emulsion also chemically stable during 5 weeks storage. Finally, the formulation assessment was done based on the hedonic test where the formulation with water content 20% was the most preferred formulation with an average score of 6.61.

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