DEVELOPMENT OF MAKE-UP REMOVER
FROM PERILLA OIL

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Abstract

The objectives of this study was to develop make-up remover from perilla oil and test its physical-chemistry properties, stability, efficiency and satisfaction in 15 volunteers. Balm formulation 3.3 containing 31.50 % perilla oil provided pH 5.28, melting point at 44ºC and the most satisfied washability properties. This formula was selected to compare its makeup removing property with commercial product. The volunteers satisfied the perilla oil makeup remover balm with higher average overall score of 4.87 ± 0.35 than 4.60 ± 0.51 of the commercial product. The perilla oil balm makeup remover gave the highest washability effectiveness when studied by using weighing and colour difference and absorption scanning methods of eyebrow pencil, lipstick and foundation cleansing. The volunteers satisfied the perilla oil balm with very good scale scoring higher than 4.60 of 5.00. The balm also exhibited comparable satisfaction test with the commercial product in all parameters tested.

Keywords: Make-Up Remover Product/Perilla Oil/Hot Production Methods

Introduction

Nowadays, most of the people around the world use cosmetics in their daily life. (Tejal, et al., 2013) There are many categories in the cosmetic group for instance skin care, perfume, toiletries, and make-up depend on the objective of classified. Make-up is the one category which

is growing rapidly every year because the traditional and behavior of the consumer change. (Meshioye, 2016) The online social medias are influenced people to change their behavior such as dressing up or wearing makeup, especially women or working people because the good personality and appearance lead attention from the other and making more believable. (Perloff, 2014) However, there are many factors can affect to makeup like hot weather which can melt the cosmetic or sweat to make the stain on the face. Therefore, there are many people take the powder all day or using waterproof makeup to maintain cosmetic on their face. It can be seen that if makeup remover product isn't effective, the people will get many skin problems like acne, or irritation because the residue on the face. (Cutis, 2018) Moreover, the cosmetic company develop cosmetic to be more long lasting and well protected from water and sweating. (Kubba, Kumar, Thappa, & Sharma, 2009)

Therefore, the makeup remover will be to more effective to remove all of residue on the face. (Market Research Report, 2019) There are many type of make-up remover such as creams, solution, lotion or oil make-up remover that consist in the market for removing however, almost remover didn't develop the removing from the skin after dispersing the cosmetic and user have to wiped off by tissue paper or cotton patch many time however, this method damages the skin by the rubbing and incomplete remover including wasting the resource like cotton or tissue paper. (Davies & Johnston, 2011)

Under these problems, it is necessary of make-up remover to be rinse with water and dissolve or disperse cosmetics in oil phase so the make-up remover have to be lipophilic, which decrease interfacial of oil's tension. (Watanabe, et al., 2005) However, the make-up remover has to be rinsed by hydrophilic state. Therefore, to have both lipophilic and hydrophilic, the ideal of cleansing have to satisfy these two features at the same time. From this concept, oil is the important ingredient that should concern. (Bialek, Bialek, Jelinska, & Tokarz, 2015) Therefore, from the literature review about interested oil in Thailand, the researcher found the trend of natural product when considering at benefit and possibility the perilla seed oil is interesting substance to be apply as make-up remover. There are perilla trees in Thailand especially north and eastern north area, moreover, there are perilla trees in the other country in Asia as Laos, Myanmar, Vietnam, China, India and Japan. However, the potential of perilla tree in Thailand is increasing but only dietary supplement use. (Miyazawa, 1991)

When considering the opportunity of perilla seed oil in the market and the problems of the skin irritation by wipe off, and wasting resource of the make-up remover like cotton or tissue paper, the researcher interested in developing make-up remover from perilla seed oil to solve these problems and satisfy need and want of the consumer.

Objectives of the Research

1. To develop formulation of make-up remover from Perilla seed oil
2. To evaluate the effectiveness of cleansing the skin
3. To assess the satisfaction of finished product

Literature review

Removing make up is the important step in the skincare process to serve the purpose of impurity skin as ideally removed completely the makeup product which applied afterwards. (Xing, Vaught, Krogmann, & Chambers, 2019) However, there are many makeup product use oil substance and silicone resins which are significantly improved the long lasting property of product with the result that makeup can hardly be removed. (Watanabe, et al., 2005) Therefore, the makeup remover should improve efficacy by surfactant type cleansers like oil based cleansers. Furthermore, oil-based makeup removers do not leave the skin feeling fresh but oily, and often have been used in combination with surfactant-type cleansers. In other words, complete makeup removal and a fresh skin feel are considered to be incompatible in conventional formulation technologies. (Taghipour & Loh, 2019) To obtain compatible systems, the makeup remover can be prepared with a specific composition with a complete hydrophilic-lipophilic balance in an oil/surfactant/water system. The continuous phase has an oil- and water-continuous structure, showing affinity for both aqueous and oily substances. Determination of remover capability and fresh skin feel revealed that the makeup could be completely removed and that the amount of oil remaining on skin surface after wash-off was much less than with previous oil-based removers. (United State Patent No. 5,217,641 , 1991)

The principle of cleansing makeup with a solvent-type remover is dissolving and dispersing oily impurities into the oil base. They are directly applied to the skin, followed by dissolving the impurities by rubbing, and then removed. The cleansing process of two kinds of cleansing creams, water-in-oil (W/O) and oil-in-water (O/W) types, which are both typical solvent-type make-up

removers. (Avi, Robert, & Howard, 2009) Their fundamental cleansing mechanisms are the same in terms of dissolving and dispersing oily impurities within the oily phase of the emulsion, but the consecutive removal process is different. W/O makeup remover can easily dissolve oily impurities due to its oil-based outer continuous phase, though it is incapable of being rinsed off and is usually wiped off. An O/W makeup remover, on the other hand, cannot dissolve oily impurities immediately after the application because of its water-based continuous phase. The rubbing induces evaporation of water and coalescence of oil droplets in the emulsion, which leads to faze inversion from O/W to W/O then the oil-based impurities are dissolved in it. Since a W/O emulsion formed by phase inversion still contains hydrophilic emulsifier that can produce an O/W emulsion, the cleansing cream can disperse in water by rein version during the rinsing process. An O/W cleansing cream, therefore, can be removed by either the wipe-off or rinse-off method, but the efficiency of the latter has not yet been seriously considered

HLB value of perilla seed oil was determined and it was found to have HLB of 6.0 (Saengsorn, 2016). The system of emulsion containing 5% w/w of steareth-2 and steareth-21, 5% w/w of perilla seed oil exhibited good stability after being subject to accelerated stability test for 1 month. Chemical analysis of fatty acids and nutrients in seeds of Nga-mon, *Perilla frutescens* L. Britton, from 14 cultivated areas of northern Thailand found that Nga-mon seed oil prepared by cold compression showed 27.50% w/w yield containing 76.33% omega-3 and 12.90% omega-6 as the main components. By the analysis of nutrients in seeds, there were 38.05 % carbohydrate, 33.99% lipids, 16.63% protein and 17.58% dietary fiber, while the minerals were 108.53% magnesium, 1.72% potassium, 0.15% calcium, 0.14% iron and 0.03% zinc (Maitree, Chakkrit, Payungsak, & Komsak, 2015).

Method
1. Extraction of perilla seed oil

Perilla seed was obtained from Mae Chan district, Chiang Rai province during January 2018. Perilla oil was prepared by using cold-pressing method by screw press extractor. After screw pressing, the obtained mixture was centrifuge at 8000 rpm for 30 min to collect clear supernatant as perilla oil.

2. Formulation of makeup remover balm

Three 3 basic balm base formulas (B1-B3) containing 40 -50% of oil were developed. The concept of formulation was balm base, massage and washable. However, each formula contained different ingredients that would provide the different texture and property. The B1 was balm with water in the formulation comprising thickening agent of stearyl alcohol and cetyl alcohol, emoillent, surfactant and emulsifier. The B2 was balm base containing silicone with emoillent, wax and surfactant. The B3 was balm with wax base, emoillent and surfactant. The best texture and property was selected for further experiment. The final formulation was supposed to be good efficacy, easy to rinse and good texture.

3. Physical chemical properties

Properties of formulations including melting point, color, texture, odor by sensory observe and pH were evaluated at room temperature ($25 \pm 2°C$) and climate chamber (at $40 \pm 2°C$ and 75% relative humidity) under the determined conditions and time intervals. (n:3). The formulations that get high score at least 1 formulation to compare with commercial was chosen to test the stability.

Heating-Cooling Cycle Test: The 6 cycles test was performed by alternating conditions between $4°C$ (±2 $°C$) for 24 hours and 45 $°C$ (±2 $°C$) for 24 hours of each cycle. The viscosity of formulation were a and phase separation, color, and odor were visually observed at cycle 0 and cycle 6.

4. Irritation test

Patch test was performed on 15 volunteers both male and female aged between 18–50 years old. Enrolled volunteers were done closed patch test by using Finn chamber with 0.2 g per each of emulsion base, perilla oil, perilla formulation sodium lauryl sulfate as positive control and deionized water as negative control. M.I.I (Mean Irritation Index) was measured.

5. The cleansing ability

Cleansing ability assessment was done by 15 volunteers (same group with safety test) including control rubbing press by washing forearm with soap, then dry it with paper napkin. The makeup was applied into the forearm, then apply remover 10 mg., gently rub and rinse with water 50 ml. then, compare with commercial product and pure perilla seed oil by the same method.

Subjective sensory and satisfaction assessment was evaluated by comparing chosen formulation by perilla seed oil with commercial makeup remover product and pure perilla seed oil. Evaluate satisfaction by questionnaire that was done by 15 volunteers (same group with safety test and ability test).

Results and discussion

1. Development of make up remover containing perilla seed oil

To develop the makeup remover with easy washable. Therefore, the design of formulation was a balm. Balm makeup remover formulation is appropriate more than liquid formulation because less oily feeling and texture of formulation good for massage that help to absorb the cosmetic and residue on the skin. The B1 formulation was represent the balm with water in formulation. The B2 formulation was represent the balm with silicone base in formulation. The B3 formulation was represent the balm with wax base. Oil in the formulation B3 was 42% and the ratio between mineral oil and perilla oil was altered by increasing perilla oil 10 to 40 in each formulation. From the result of chemical and physical property test and the application properties of the developed cleanser as shown in Table 1 , it could be concluded that the B3.3 cleanser formulation was the most proper physical and chemical properties.

Table 1 Chemical and physical properties of developed perilla oil make up remover.

Formulation	pH	Melting Point (°C)	Colour	Spread	Spread score
B1	5.88	55	Opaque white	Clumpy, unscattered texture	0
B2	5.48	46	Opaque white	Clumpy texture, begin to scatter.	3
B3	5.57	45	Opaque white-yellow	Clumpy, poorly scattered texture.	6
B3.1	5.49	43	Opaque white-yellow	Clumpy, poorly scattered texture.	6
B3.2	5.35	43	Opaque white-yellow	Unclumpy, scattered texture.	8

B3.3	5.28	44	Opaque yellow	Fairly scattered texture, easy to spread	9
B3.4	5.22	41	Opaque yellow	liquid texture, easy to spread	10

Note: Spread Score is measured by the ability to spread at the starting point. Score is ranged from 1-10 with 10 is the most scattering recipe.

2. Stability Test

Because of the B3.3 formula provided the most paper properties to be makeup remover balm, this formula was then tested for its stability and efficiency. Physical stability after being stored in the refrigerator at 4 ˚C for 24 hours and 45 ˚C for 24 hours, tested for 6 cycles. The result was shown in Table 2 that the viscosity was not changed and phase separation was not occurred.

Table 2 Stability test of make-up remover containing perilla seed oil

Formula	Result				
	Physical properties	Color	Odor	pH	Viscosity
B3.3	Easy to spread, No phase separate	Opaque yellow	Perilla oil smell	5.3	Not change

Note: The rancidity level

0 = No rancidity   +1 = Mild Rancidity

+2 = Moderate Rancidity   +3 = Heavy Rancidity

3. Irritation test

From the irritation test with 15 volunteers by patch test, the mean irritation index: M.I.I of negative control (DI water), perilla oil, balm base and B3.3 were not irritation compared with positive control (SLS 1%) that was irritation with M.I.I 1.5. all the result shown as Table 3.

Table 3 Irritation test of make-up remover base and the base containing perilla seed oil.

Sample	M.I.I	Result

DI water (negative control)	0	Non Irritation
SLS 1% (positive control)	1.50 ± 0.37	Irritation
Perilla Oil	0.15 ± 0.64	Non Irritation
Balm Base	0.10 ± 0.42	Non Irritation
B3.3	0.15 ± 0.23	Non Irritation

4. Cleansing ability

The assessment of cleansing ability by observe and scoring the result after use in volunteers. The cleansing ability was shown in Table 4 that the highest score was 5.

Table 4. Cleansing ability of make-up remover containing perilla seed oil compared to commercial make-up remover.

Remover	B3.3	Commercial
Eyebrow	4.20 ± 0.24	3.20 ± 0.62
Lipstick	4.50 ± 0.25	3.90 ± 0.15
Foundation	5.00 ± 0.04	4.90 ± 0.20
Average	4.60 ± 0.12	4.00 ± 0.26

5. Preference test

In this study, the B3.3 was selected from all 7 formulation to be tested and the best results compared to commercial cosmetics and other. The satisfaction evaluation of cleanser users are shown in Table 5.

Table 5. Score of 15 volunteers in satisfaction evaluation

Part 1 Basic Information		
	No	Yes
Have you ever used cleanser product?	0	15
Have you ever had irritation from wiping cosmetics with cotton?	6	9
	12	3

Have you ever used a cleanser with ingredient from perilla oil?		
Have you ever had skin problems such as acne from using cleanser products that contain oil?	5	10
Require cleanser products which can wipe out	11	4

Part 2 Satisfaction assessment	B3.3	Commercial	P-value
Satisfaction of product's texture	4.67 ± 0.62	4.53 ± 0.52	0.500
Satisfaction of product's color	4.60 ± 0.51	4.80 ± 0.41	0.165
Satisfaction of product's smell	4.47 ± 0.64	4.73 ± 0.46	0.189
Satisfaction of the cleanliness of the skin after cleansing with the product	4.87 ± 0.35	4.53 ± 0.74	0.263
Eyebrow pencil cleansing effectiveness	4.80 ± 0.41	4.60 ± 0.51	0.082
Lipstick cleansing effectiveness	4.67 ± 0.49	4.47 ± 0.64	0.500
Foundation cleansing effectiveness	4.87 ± 0.35	4.67 ± 0.49	0.082
non-greasy feeling of the skin after using the product	4.73 ± 0.46	4.33 ± 0.72	0.055
Skin moisturizing after cleansing with the product	4.60 ± 0.51	4.80 ± 0.41	0.165
Overall product satisfaction	4.87 ± 0.35	4.60 ± 0.51	0.189

Remark: Part 2 is a satisfaction rating. Each person will give 1-5 score. 1 means the least satisfaction and 5 means the most satisfaction.

Conclusion

Perilla oil prepared from perilla seed was light yellow and medium viscous clear liquid. The makeup remover balm containing perilla seed oil 31.50% (formulation 3.3) provided the most satisfaction properties including pH of 5.28, melting point at 44°C, speadibility, cleansing effectiveness texture and colour stability.

The perilla oil balm makeup remover showed higher cleansing ability to wash eyebrow pencil, lipstick and foundation than that of commercial product, perilla oil and water, respectively, when using weighing and colour difference and absorption scanning methods.

The results from the visual assessment after washing eyebrow, lipstick and foundation by the perilla oil balm remover and the commercial product in 15 volunteers showed higher score in cleansing ability of the perilla oil balm. For satisfaction test by 15 volunteers, the perilla oil blam were satisfied by all volunteers in all parameters including texture of the product, the efficiency of cleansing and overall satisfaction of the product. This were comparable to those of the commercial product.

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