ORIGINAL PAPER

Effect of Using Ethanol Extract of Artocarpus Heterophyllus Leaves and Olea Europea Fruit Oil Combination on Facial Skin

Tri Widyawati[1,2], Milahayati Daulay[3]

[1]Departement of Pharmacology and Therapeutic, Faculty of Medicine, Medan, Indonesia

[2]Master Program in Tropical Medicine, Faculty of Medicine, Universitas Sumatera Utara, Medan, North Sumatera, 20155, Indonesia

[3]Departement of Physiology, Faculty of Medicine, Medan, Indonesia

Corresponding author: dr. Tri Widyawati, M.Si, Ph.D. Departement of Pharmacology and Therapeutic, Faculty of Medicine, Medan, Indonesia. Jl.dr. Mansur no 5 North Sumatera Medan Indonesia. E-mail: tri.widyawati@usu.ac.id. ORCID ID (https://orcid.org/0000-0001-6230-6943).

doi: 10.5455/aim.2023.31.168-171

ACTA INFORM MED. 2023, 31(3): 168-171

Received: JUL 25, 2023

Accepted: SEP 04, 2023

ABSTRACT

Background: *Artocarpus heterophyllus (A. heterophyllus)* leaves and *Olea europea* (OE) fruit oil are natural sources that have been traditionally used for health and skin care purpose. **Objective:** To assess the potential synergistic effect of combining ethanol extract of *A. heterophyllus* leaves (AHLE) and OE fruit oil in the formulation of clay masks, specifically in terms of their effect on facial skin. **Methods:** AHLE was obtained by the maceration method, while OE was purchased commercially. Total phenol and flavonoid content were calculated and a DPPH assay was conducted to evaluate the antioxidant properties. Furthermore, the four formulas prepared were F1 (AHLE 5%), F2 (OE 10%), F3 (AHLE 5% + OE 10%), and F4 (AHLE 2.5% + OE 5%). Adult women received weekly facial treatments with the formulated mask for one month. The effect of these treatments was evaluated based on several skin parameters, including moisture, oiliness, texture, collagen levels, pigmentation, sensitivity, and the presence of wrinkles. Furthermore, the data obtained were analyzed using the Wilcoxon sign-ranked test. **Results:** AHLE contained total phenolic, flavonoid, and antioxidant activity higher than OE. Clay masks in all formulations showed homogeneity and do not contain coarse grain. After four weeks of treatment, the efficacy of the formulations demonstrated a significant effect. F1 exhibited a reduction in wrinkles by 36.27%, while F3 improved oily skin by 21.39%, enhanced skin texture by 44.32%, reduced pigmentation by 30.30%, and decreased skin sensitivity by 49.18%. Furthermore, F4 demonstrated an increase in skin moisture levels by 27.89% and a boost in collagen production by 32.00%. **Conclusion:** The combination of AHLE and OE at 5% and 10% demonstrated superior effectiveness compared to their individual use.

Keywords: Antiaging, Artocarpus heterophyllus, Clay mask, Facial skin, Olea europea.

1. BACKGROUND

Skincare products are medicinal preparations intended for topical use that are beneficial and provide protection against degenerative skin conditions (1). Currently, herbal skin treatments that used natural ingredients are popular and are claimed to have intrinsic efficacy by their routine daily use (2). *Artocarpus heterophyllus* (*A. heterophyllus*) and *Olea europea* (OE) or olive oil are natural ingredients frequently used for skin care due to their bioactivities benefitting skin health (3).

A. heterophyllus, commonly known as *nangka* in Indonesia, is a plant of the genus *Artocarpus* which contains sterols, prenylflavones, carotenoids, tocopherols, and flavonoids. These chemical constituents are responsible for their pharmacological activities. The previous study demonstrated its leaves effect that benefits facial skincare by improving moisture, smoothness, pore size, blemishes, and wrinkles (4-6)

Olive oil is obtained from the grinding of olives (*Olea europaea*) derived from traditional plants in the Mediterranean region. It resembles the natural oil produced by the skin, hence it is easily absorbed. This oil has been used for various purposes, such as medicine, cooking ingredients, soap, fuel for traditional lamps, as well as cosmetics. It is also useful for softening dry and cracked skin on the elbows and heels

Measurement	Parameter				
Moisture	Serious dry	Lack of moisture	Skin dermis dry	Epidermis dry	Enough moisture
	0-11	11-37	37-56	56-75	75-100
Oily	Less oil	Balance oil	Oily skin	Over oil	Oleaginous pustule
	0-13	13-37	37-39	79-91	91-100
Skin texture	Perfect	Slightly irregular	Coarse	Obviously irregular	Abnormal disorder
	0-12	12-24	24-36	36-66	66-100
Collagen	Disjunctive dent	Fiber loosen	Serious lose	Slightly lose	Enough fiber
	0-12	12-26	26-43	43-78	78-100
Wrinkle	Without wrinkle	Wrinkle appear	Fine wrinkle	Irregular wrinkle	Serious wrinkle
	0-12	12-24	24-26	36-66	66-100
Pigment	Normal	Pigmentation	Sight speckle	Speckle on dermis	Serious spots
	0-16	16-37	37-68	68-90	90-100
Sensitivity	Normal skin	Sensitive skin	Slightly redness	Couperose skin	Red spots
	0-16	16-37	37-68	68-90	90-100

Table 1. Distribution and explanation according to skin condition (Skin Observed System)

(7). In addition, olive oil can inhibit oxidative stress, and promote dermal reconstruction, to retain moisture and skin elasticity in conjunction with the process of regeneration (8). It contains fat, vitamin E, and vitamin K, as well as iron, calcium, and potassium in very small amounts. Furthermore, it has oleic acid, with smaller quantities of other fatty acids such as linoleic and palmitic acid. This oil also contains phenolic compounds such as hydroxytyrosol and oleuropein, carotenoids, squalene, phytosterols, chlorophyll, and α-tocopherol. The compounds were known to have antioxidant properties that delay or prevent oxidative stress against reactive oxygen species or free radicals (9). In dermatology, this action benefits the treatment of skin diseases including acne, psoriasis, and seborrhea eczema (10).

Currently, clay mask is one of the preparations that are commonly used. This product is categorized as a wash-off cosmetic, which involves rinsing with water. It is formulated with a clay-based material commonly known in the market as a mud mask preparation (9, 11). Furthermore, it is composed of mixtures of clay minerals containing kaolinite. Clay masks with potential topical applications have a specific dermatological indication in reducing oil secrets, blackheads, spots, and seborrhea (12). Based on the literature, there has been no report on the study of combination of *A. heterorrhophyllus* leaves and Olive oil in the form of clay mask. This study holds significant importance in demonstrating the synergistic efficacy of the ingredients, which could be valuable for both the community and the industry.

2. OBJECTIVE

This study aims to evaluate the antiaging effect of *Artocarpus heterophyllus* leaves extract and Olive oil combination in the form of a clay mask.

3. MATERIALS AND METHODS

3.1. Materials

AHLE leaves were collected from Deli Serdang, Medan, and the plant was identified by Herbarium Medaneses, Universitas Sumatera Utara. Meanwhile, olive oil (RS Extra Virgin Olive Oil Rafael Salgado) was imported by PT. Gautama Indah Perkasa, Indonesia.

3.2. Extract preparation

Leaves were dried at room temperature and smashed manually. The maceration method extracted simplicia powder with ethanol 96% for 5x24 h at room temperature, and the macerate obtained was then evaporated with a rotary evaporator. Subsequently, the concentrated extract was placed in dark bottles, and stored in a refrigerator at 4^0C before being used for experimentation (13).

3.3. Phytochemical screening

Extracts were subjected to phytochemical screening to identify secondary metabolites qualitatively and quantitatively.

3.4. Antioxidant assay

The antioxidant assay was carried out by DPPH radical scavenging activity. The inhibition of DPPH radical by the sample was calculated based on the formula below:

The result was expressed as IC_{50} values, which is the concentration of antioxidant compounds causing a 50% loss of DPPH activity (Molyneux P, 2004). A compound is said to be very strong, strong, moderate, weak, and inactive when IC_{50}<10 μg/mL, 10-50 μg/mL, 50-100 μg/mL, 100-250 μg/mL, and >250 μg/mL (14).

3.5. Preparation of clay mask

Clay mask preparation was prepared in four formulas of *F1:* AHLE 5%, F2: Olive oil 10%, F3: AHLE 5% and Olive oil 10%, and F4: AHLE 2.5% and Olive oil 5%. The ingredients for the preparation are bentonite, xanthan gum, kaolin, glycerine, sodium lauryl sulfate, titanium dioxide, green tea oil, and aquadest (9). Furthermore, the parameters observed to ensure that the clay mask was safe to use were physical appearance, homogeneity, pH, and irritation test.

3.6. Experimental procedure

The inclusion criteria are healthy women, age over 20 years, with no history of allergies, and not under external treatment. Meanwhile, the exclusion criteria are irritated and allergic skin reactions during treatment. Clay mask was applied once a week and then observed before and after application using a skin analyzer, as shown in Table 1. The study was approved by the Health Research Ethical Committee, Faculty of Medicine, Universitas Sumatera Utara, Medan, Indonesia: No. 1051/KEP/USU/2021.

3.7. Statistical analysis

Data were analyzed using IBM SPSS Statistics 26 software, and the results were presented as mean standard deviation. Statistical analysis by Wilcoxon Signed Ranks Test to was then con-

ducted to investigate the comparison before and after treatment.

4. RESULTS

4.1. Phytochemical screening

AHLE traces the presence of flavonoids, tannins, glycosides, saponins, and steroids/triterpenoids, while olive oil contains flavonoids, tannins, and steroids/triterpenoids. AHLE contains 3.27 mg GAE/g and 24.29 mg QE/g extract of total phenolic and flavonoid, while olive oil contains 0.32 mg GAE/g and 31.09 mg QE/g.

4.2. Antioxidant Assay

DPPH AHLE at 48.09 mg/mL was lower than olive oil at 163.37 mg/mL.

4.3. Clay Mask Formulation Evaluation

The results of the preparation test can be seen in Table 2. Skincare effectiveness of ethanol extract of *A.heterophyllus*, olive oil, and a combination of both based on moisture, oil, skin texture, collagen, wrinkle, pigment, and sensitivity showed an increase or improvement of facial skin within 4 weeks of using clay mask formulation. Statistically, before and after 4 weeks of using clay mask showed a significant difference with $p<0.05$, as shown in Table 3. The percent improvement from F1 (36.27%) showed the highest result in the wrinkle parameter, while the highest from F3 is in oil (21.39%), skin texture (44.32%), pigment (30.30%), and sensitivity (49.18%). The percent improvement from F4 showed the highest improvement in moisture (27.89%) and collagen (32.00%) parameters, while F2 showed improvement lower than other formulations.

Parameters	Time	F1	F2	F3	F4	p
Moisture	Before	53.0±9.6	57.0±7.1	51.3±1.9	49.0±3.7	0.03
	After	61.7±10.0	66.7±10.9	63.0±3.7	62.7±2.5	
Oily	Before	59.7±8.0	63.3±6.1	57.7±4.2	59.7±4.1	0.02
	After	52.0±5.0	50.3±2.1	45.3±4.8	51.3±1.2	
Skin Texture	Before	37.3±1.7	33.3±7.3	29.3±0.5	30.3±1.2	0.02
	After	23.0±2.9	20.7±4.5	16.3±2.5	22.7±3.7	
Collagen	Before	34.7±4.2	32.7±6.6	37.3±2.9	33.3±4.2	0.03
	After	40.0±0.8	42.3±2.5	41.0±1.4	44.0±3.6	
Wrinkle	Before	34.0±2.4	33.3±4.5	34.7±2.4	31.0±3.6	0.03
	After	21.7±1.2	27.0±1.6	27.0±2.9	24.0±0.8	
Pigment	Before	21.7±3.4	21.7±1.2	22.0±3.6	19.3±1.7	0.02
	After	16.7±5.4	18.3±0.5	15.3±2.9	15.0±1.6	
Sensitivity	Before	19.3±2.1	15.0±1.4	20.3±5.6	15.3±1.9	0.02
	After	13.3±3.9	12.7±1.9	10.3±0.3	9.3±1.7	

Table 2. Skincare effectiveness of each clay mask formula p: Wilcoxon signed the test

Group	Percentage (%)						
	Moisture	Oily	Skin Texture	Collagen	Wrinkle	Pigment	Sensitivity
F1	16.35	12.85	38.39	15.38	36.27	23.08	31.03
F2	16.96	20.53	38.00	27.00	19.00	15.38	15.56
F3	22.73	21.39	44.32	28.13	22.12	30.30	49.18
F4	27.89	13.97	25.27	32.00	22.58	22.41	39.13

Table 3. The percentage of improvement before and after treatment

5. DISCUSSION

Natural skincare can increase the supply of nutrients required for healthy skin, and improve skin tone, texture, and light radiation (15, 16). The use of natural ingredients requires special attention regarding the extract method, and plant-solvents ratio to ensure the safety, quality, and content of active ingredients (17). Physicochemical analysis can guarantee product safety and market acceptance. This is because the test ensures the safety and quality of the product to screen for the potential for irritation from skin care products. Bioactive extracts and phytochemicals affect the biological function of the skin, providing nutrients for healthy skin (18).

Antioxidants have an important role in protecting against oxidative damage caused by pollutants and UV radiation. It is assumed that exposure can affect the sebum composition and quality of the corneum layer thereby intensifying the signs of skin aging with the formation of pigment spots, wrinkles, enlarged pores, and skin becoming dry and rough (19-21). Polyphenols are an important group of natural compounds commonly found in plants classified as flavonoids, phenolic acids, and tannins. Furthermore, ethanol extract from *A. heterophyllus* leaves and olive oil contains flavonoids, tannins, and phenolic acid as shown in Table 4,5. Flavonoids may function as cofactors for enzymes, exert an impact on angiogenic and inflammatory processes, offer protection against radiation, and provide moisturizing and softening benefits to the skin. These activities are useful for antiaging, anticellulite, anticouperose, and skin-lightening products. Phenolic acid can exhibit depigmenting properties by controlling tyrosinase activity, moisturizing skin, and stimulating the synthesis of collagen and elastin fibers. This compound also showed antiallergic, antimicrobial, anti-inflammatory, and anti-aging. It acts as a photo protector by preventing the formation of UV-induced erythema in the skin. In this study, AHLE has strong antioxidant activity in contrast to olive oil with weak antioxidant activity (14).

Tannins promote tropoelastin synthesis and reduce elastase activity, as well as protect the skin from inflammation caused by external irritants (22). The previous study showed that AHLE improved facial skin smoothness and moisture in clay mask formulation (7,8). Olive oil has good lipid penetration in the epidermis. It contains tocopherols that may act as moisturizers and remove dead skin cells to promote the shrinking of the pores. As an emulsion preparation, olive oil can increase skin moisture, shrink pores, and reduce blemishes, and the number of wrinkles (23). Skin exposed to the sun for a long time causes damage to the collagen and elastin layers. This condition stimulates the accumulation of dead cells in the stratum corneum. Therefore, the skin surface becomes rougher, and drier with larger pores. Patches of uneven pigmentation on the skin occur due to changes in melanin pigment distribution and decreased melanocyte function. The tyrosinase enzyme plays important role in converting tyrosine into 3,4-dihydroxyphenylalanine (DOPA) followed by dopaquinone which then goes through several stages of conversion transformation into melanin. Meanwhile, decreased skin texture and collagen can cause wrinkles. Collagen and elastin fibrils in the skin can be degraded in the presence of

collagenic matrix metalloproteinase (MMP-1). Flavonoids and phenolic acids directly inhibit tyrosinase activity in the melanogenesis process and increase MMP-1 levels to maintain collagen in the skin and inhibit the appearance of signs of aging (24, 25) A combination of *A. heterophyllus* leaves extract and olive oil increases the effect on facial skin compared to each preparation. Therefore, this combination produces synergistic pharmacological activity as a skincare product.

6. CONCLUSION

In conclusion, the combination of *A. heterophyllus* leaves ethanol extract and olive oil in clay mask preparation produces a synergistic effect on facial skin. The best combination is 5% AHLE and 10% Olive oil.

- **Acknowledgment:** The authors gratefully acknowledge to the Universitas Sumatera Utara for supporting this study by USU Grant (Kontrak Penelitian Talenta USU Tahun Anggaran 2021 No.332/UN5.2.3.1/PPM/SPP-TALENTA USU/2021).
- **Author's contribution** T.W contributed to conception, design of the study and manuscript preparation. M.D performed data acquisition, experimental laboratory works, data analysis. All authors have approved the final version of the manuscript.
- **Declaration of interest: The authors report no conflicts of interest.**
- **Financial support and sponsorship:** TALENTA USU Grant.

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