

Türk Doğa ve Fen Dergisi Turkish Journal of Nature and Science

www.dergipark.gov.tr/tdfd

Formulation and Evaluation of a Peel-off Gel Mask Containing Macerate St. John's Wort Oil and Activated Carbon Derived from Pine Cones

Hatice KARAER YAĞMUR^{1*}, Hatice Kübra ÖZER ²

¹Dicle University, Faculty of Science, Chemistry Department, Diyarbakır, Türkiye
²Dicle University, Science and Technology Application and Research Center, Diyarbakır, Türkiye Hatice KARAER YAĞMUR ORCID No: 0000-0002-3728-1825
Hatice Kübra ÖZER ORCID No: 0009-0003-5666-5538

*Corresponding author: profhatice23@hotmail.com

(Received: 21.01.2025, Accepted: 02.09.2025, Online Publication: 26.09.2025)

Keywords Peel-off mask, Active carbon, St. John's wort oil, Hypericum perforatum

Abstract: Skin needs care to protect against environmental pollution. The facial skin can be protected such as cream, peel off mask and lotion facemask. The aim of this study was to obtain an antibacterial peelable gel mask containing macerated St. John's wort oil (Hypericum perforatum oil). This peel-off gel mask consists of ascorbic acid, polyvinyl alcohol (PVA, as preservative), polyethylene glycol (PEG), glycerine (as plasticizer), polysorbate (tween 20, as stabilizer), ethanol, distilled water, macerate St. John's Wort oil and active carbon. The physical properties (homogeneity, spreadability, viscosity, film drying time) and chemical properties (pH value, stability and antibacterial activity properties) of the peelable gel mask were examined. Using of an active ingredient in peel off mask, it strengthens the role of peel off mask by opening the clogged pores. Activated carbon (AC) was added to this formulation as an active ingredient due to its adsorbent activity. In this present study, activated carbon was obtained from pine cone (PC). The specific surface area (SBET) and pore diameter of activated carbon were found to be 536.998 m²/g and 1.8 nm. Microporous activated carbon was obtained. The antimicrobial activity of the St. John's Wort oil was tested against Gram-negative bacteria (Pseudomonas aeruginosa) and Gram-positive bacteria (Staphylococcus aureus) as well as one pathogenic fungus (Candida albicans, ATCC 10231). Disc diffusion method was used to study antimicrobial activity. The resulting peel off mask is a black mask that smells of bergamot essential oil and can be easily spread on the skin. The drying time of the mask was determined as 17 minutes. The prepared peel of mask that can be easily peeled off in one piece is obtained.

Sarı Kantaron Yağı ve Çam Kozalaklarından Elde Edilen Aktif Karbon İçeren Soyulabilir Jel Maskenin Formülasyonu ve Değerlendirilmesi

Anahtar Kelimeler

Soyulabilir maske, Aktif karbon, Sarı kantaron yağı, Sarı kantaron

Öz: Cildin çevre kirliliğinden korunması için bakıma ihtiyacı vardır. Yüz çildi krem, soyulabilir maske ve losyon yüz maskesi gibi ürünlerle korunabilir. Bu çalışmanın amacı, macerat sarı kantaron yağı (Hypericum perforatum yağı) içeren antibakteriyel soyulabilir jel maske elde etmektir. Bu soyulabilir jel maske; askorbik asit, polivinil alkol (PVA, koruyucu olarak), polietilen glikol (PEG), gliserin (plastikleştirici olarak), polisorbat (tween 20, stabilizatör olarak), etanol, damıtılmış su, macerat, sarı kantaron yağı ve aktif karbondan oluşur. Soyulabilir jel fiziksel özellikleri (homojenlik, yayılabilirlik, viskozite, film kuruma süresi) ve kimyasal özellikleri (pH değeri, kararlılık ve antibakteriyel aktivite özellikleri) incelendi. Soyulabilir maskede aktif bir bileşen kullanılması, tıkalı gözenekleri açarak soyulabilir maskenin etkisini güçlendirir. Adsorban aktivitesi nedeniyle bu formülasyona aktif bileşen olarak aktif karbon (AC) eklendi. Bu çalışmada, aktif karbon çam kozalağından (PC) elde edilmiştir. Aktif karbonun özgül yüzey alanı (SBET) ve gözenek çapı 536,998 m²/g ve 1,8 nm olarak bulunmuştur. Mikrogözenekli aktif karbon elde edildi. Sarı kantaron yağının antimikrobiyal aktivitesi Gram negatif bakterilere (Pseudomonas aeruginosa) ve Gram pozitif bakterilere (Staphylococcus aureus) karşı ve ayrıca bir patojen mantara (Candida albicans, ATCC 10231) karşı test edildi. Antimikrobiyal aktiviteyi incelemek için disk difüzyon yöntemi kullanıldı. Elde edilen soyulabilir maske, bergamot esansiyel yağı kokulu ve cilde kolayca uygulanabilen siyah bir maskedir. Maskenin kuruma süresi 17 dakika olarak belirlendi. Tek parça halinde kolayca soyulabilen hazırlanmış bir maske elde edildi.

156

1. INTRODUCTION

The skin is a covering layer of the body against the external environment and microorganisms. For women, the face is the most important part of the body in terms of beauty. For this reason, many women try to beautify themselves by using cosmetic products [1]. Cosmetic products are used to improve the appearance of the skin by action of cleansing, beautifying and promoting attractiveness [2]. Facemask is one of the cosmetic products for skin care that are generally often used by women so that the appearance of facial skin becomes healthier and more beautiful [3]. Peel off masks may be used for cleansing and moisturizing the skin [4]. Peel off masks use film-forming polymers so they can be easily peeled off without leaving any residue after complete drying [5]. The peel-off mask is available in gel and dry form. It can be applied onto the face, neck and hands. It is useful in removing blackheads and dead skin [4].

The peel-off mask gel is one of the popular forms of applications used to improve the quality of the face. The face masks in gel form have the advantage of being easily peeled off, is helpful for recovering facial skin, and can be used to minimize pores. In addition, it is also useful for relaxing facial muscles [6]. The gel mask can stay on the face for 15-20 minutes; the drying time may vary depending on its content. Polyvinyl alcohol (PVA) or polyvinyl acetate is usually used as a film former in peeloff facemasks [7]. It can be obtained with synthetic ingredients such as activated charcoal, polyvinyl alcohol, polyethylene glycol, glycerin, tween 20, ascorbic acid and water [4]. Industrial, agricultural and domestic wastes are discharged to the environment due to the rapid development of technology [8]. Agricultural waste materials are economical and environmentally friendly thanks to their chemical composition. Pine seeds are borne in cones. The large quantities of pine cones are produced each year in pine fields cultivated for the paper industry [9]. Activated charcoal is a black powder produced by high temperature pyrolysis of carbonaceous materials [10]. Activated carbon is a material with a high surface area and porous structure. It is widely used in various industrial processes as an adsorbent [8]. The production of commercial activated carbon is quite expensive, and therefore, efforts are being made to develop low-cost and efficient activated carbon by using various alternative sources [11]. In the recent past, researchers have been studying on the preparation of activated carbons from low-cost and ecofriendly biomass/bio-waste. Peanut shells, coffee beans, sunflower seed shells, coconut shells, almond shells, bamboo, waste paper, rice husk, dead leaves, seaweed, etc. resources that can be used for the production of activated carbon [12]. Hypericum perforatum L. (Hypericaceae) (HPL), known as St. John's Wort (SJW), is one of the most studied medicinal plants. H. Perforatum has been used in alternative medicine since ancient times against wounds, depression, burns, internal and external diseases. The last have revealed anticancer. studies antimicrobial. antifungal, anti-inflammatory, antioxidant, healing, antidiabetic and antidepressant activity of H. perforatum extracts. Several methods have been used for

extraction of H. perforatum include; methanol maceration, boiling water extraction, ethanol/water maceration, olive oil maceration, aqueous glycerol extraction, ethanol extraction with soxhlet apparatus and ultrasonic-assisted extraction with methanol/HCl [13]. The traditional way of preparing the St. John's Wort oil is obtained by maceration of fresh flowers of Hypericumn perforatum in sunlight for several weeks. The oil prepared by this method acquires a bright orange-red color [14,15]. According to the old preparation methods, the effect of sunlight can be seen. According to the studies, yellow oil was obtained from dried St. John's Wort flowers, which were kept in hot olive oil for 3 hours owing to the color of the flower pigments. The red color and fluorescence of a St. John's Wort oil is generally ascribed to hypericin. According to literature, St. John's Wort oil contain no hypericin in St. John's Wort oil but only lipophilic compounds with a hypericin-like color and fluorescence. These compounds were considered lipophilic substituted hypericins, which were also present in St. John's Wort flowers [15]. According to studies, with respect to the analysis of traditional Oleum hypericin, the characteristic red color can be attributed to lipophilic components because of the degradation of hypericin by exposure to sunlight [16]. Hypericum perforatum plant has therapeutic effect in wound healing because it contains Hypericin and hyperforin. Hypericins consist of hypericin and pseudohypericin (an oxidized derivative of hypericin) and are found in leaf and petal margins, stamens, dark glands [17]. H. perforatum contains hypericin, pseudohypericin, hyperforin, adhyperforin, flavonoids such as quercetin, quercitrin, biapigenin, rutin, kaempferol, hyperoside, and amento flavone [18, 19]. Among its components, hyperforin has been found to have antidepressant, anti-biotic and antitumor activities. Hyperforin is used in the treatment of skin problems such as atopic dermatitis. Hypericin has antibacterial, antiviral and anti-inflammatory activity, while hyperforin has antidepressant activity [14, 18, 20, 21]. Atopic dermatitis (AD) is a common inflammatory skin disease caused by the overgrowth of S. aureus [22]. This condition was expressed by the term 'eczema' [23]. Researchers have long been aware that bacteria and other microorganisms play a role in atopic dermatitis.

Many authors consider infections important. 'Secondary contribute infection with S. aureus emerges as a cause of atopic dermatitis. Therefore, colonization by S. aureus may both be a cause and a consequence of atopic dermatitis [24, 25]. The skin in patients with atopic dermatitis is constantly colonized by S. aureus, in part because of a deficit in epidermal antimicrobial peptides [26]. Futhermore, an imbalance between S. aureus (S. aureus) and the skin microbiota can create a state of dysbiosis that compromises the skin barrier. The skin dysbiosis that occurs through an increase in the pathogen S. aureus and a variation in the composition and number of skin commensal bacteria also contributes to skin barrier defects and can be a trigger for atopic dermatitis [23]. Hypericum perforatum oil has been determined to have antimicrobial activity on various Gram-positive and Gram-negative bacterial strains [19]. and is widely used

in pharmacy and medicine because of its antibacterial, antifungal and antiviral properties [21].

For the present study, the bio-waste pinecone was used as the raw precursor for the preparation of activated carbon. The active carbon was characterized with FT-IR, DLS, TGA and BET measurements. The objective of this study was to develop and formulate St. John's Wort oil peel-off mask by using activated carbon obtained from pine cones and evaluate by test methods as an alternative of facial skin care product. The antimicrobial test was performed by disc diffusion method and the growth inhibitory effects of *Hypericum perforatum* oil on *P. aeruginosa* and *S. aureus* and *Candida albicans* were investigated.

2. MATERIAL AND METHOD

2.1. Material and Method

The pine cone (PC) shells used in the preparation of activated carbon were collected from pine trees in the Dicle University campus (Diyarbakır, Turkey). Pine tree cones were collected July 2021. Ascorbic acid, polyvinyl alcohol (PVA), polyethylene glycol (peg), glycine, polysorbate (tween 20), ethanol, distilled water.

2.2. Preparation of the activated carbon (AC)

The pine cones (PC) were washed thoroughly with distilled water to remove impurity; the cone biomass was then dried at 100°C for 24 h in an oven. The dried cones ground in a crusher. The pine cone shells were placed in a tube oven (which was set in a horizontal stainless-steel tubular reactor (7 cm x100 cm) and heated from room temperature to 500 °C at a heating rate of 5 oC min-1 in an N₂ atmosphere and carbonized at 500 °C for 1h [11, 27]. The charred sample was mixed with HCl (0.1 M) solution to remove impurities for 5 hours. Then the activated carbon was washed with distilled water and dried at 120 °C for 48 hours. The dried activated carbon was powdered and stored in a desiccator (Fig. 1).

2.3. Preparation methods for St. John's Wort oil by traditional maceration

St. John's Wort upper flowered parts (with flowers and leaves) were collected in the region of Turkey, central İstanbul in July 2022. The plant materials were dried at room temperature for 3 days. The natural olive oil obtained from olives of Manisa region in Turkey was used

in the macerate preparation. About 400 grams of the dried plant materials (*Hypericum perforatum*), were mixed with 1.5 L of olive oil in a transparent glass bottle and kept under sunshine for 40 days from the mid- July to beginning September. In this process, the mixture was stirred in the glass every other day. At the end of 40 days the macerate oil, which turned to red color, was filtered and then stored in an amber glass bottle in a dark place at room temperature until use (Fig.2) [13, 28, 29]. Fig. 3 shows biologically active compounds of Hypericum perforatum.



Figure 1. Preparation of activated carbon (AC).



Figure 2. Preparation of St. John's Wort oil.

Figure 3. Biologically Active Compounds of *Hypericum perforatum*.



Figure 4. Preparation and application of the gel mask.

2.4. Formulation of Peel off Gel Mask

- I. This phase involves the addition of (14%) of polyvinyl alcohol (film former) to distilled water (70%, as base) in the beaker at 80°C temperature with a constant vigorous stirring. Further, this mixture is allowed to cool down at 40°C.
- II. In this phase a mixture of St. John's Wort oil (or glycerine, smoothing agent or a humectant) and PEG (as a surfactant) in the ratio of 3:1 is added to phase I at 40°C temperature and mix well.
- III. Add (0.5%) polysobate (tween 20) (Polymer and emulsifier)
- IV. Add ethanol (solvent) 1ml and add (1%) distilled water with (0.1%) ascorbic acid into the phase III mixture and mixed well. Moreover, distilled water was added until 100% w/w total volume.
- V. Add activated charcoal (5%) and stirred well and cooled for few minutes [4, 6, 30]. Bergamot (essential oil) was added to mixture to be 1%. Fig. 4 shows preparation and application of the mask on face and hand.

2.5. Characterization

The active carbon was characterized by Fourier transform infrared spectroscopy (FT-IR Perkin Elmer, ATR sampling in the range of 4000 to 400 cm-1), Brunauer-Emmett-Teller (BET), dynamic light scattering (DLS), particle size of the active carbon was determined Malvern mastersizer 3000 hydro. Thermal analysis of the active carbon was determined by PerkinElmer Diamond Thermal Analysis (between 20-1000 °C (in N₂, 10 oC min-1). The surface area and pore volume of the active carbon were obtained from Brunauere EmmetteTeller (BET) (NOVA model). Hanna HI 2002-02 Desktop pH meter was used for pH study. Koehler Instrument KV1000 Digital Constant Temperature Kinematic Viscosity Bath was used for viscosity measurements.

2.6. Preparation of Inoculum

The antimicrobial properties of the St John's Wort oil were tested against gram-positive bacteria (*S. aureus* (ATCC 25923)), gram-negative bacteria (*P. aeruginosa* (ATCC 27853)) as well as one pathogenic fungus (Candida albicans (ATCC 10231)) and they are purchased from Kwik-Stick. Bacteria and fungus were pre-cultured in Tryticase Soy Broth (TSB) (Merck) overnight in a rotary shaker at 37°C. Afterward, each bacteria strain was adjusted at a concentration of 108 cells mL⁻¹ using 0.5 McFarland standard [31]. The fungal inoculum was prepared from the 48 h culture at a final concentration of 106 cells mL⁻¹.

2.7. Antimicrobial Screening

The antibacterial and antifungal activities of the St. John's Wort oil were determined by using Agar well diffusion method [32]. The suspension was prepared from an overnight culture for each microorganism. Mueller Hinton Agar (MHA) (Biolife) and Sabouraud Dextrose Agar (SDA) (Biokar) were swabbed with the bacterial and fungal suspension and wells were made using a sterile pipet tip (6 mm in diameter) into agar plates containing inoculums. Then, 100 mL of St John's Wort oil in tween 20 extracts were added to respective wells. The plates were placed in the refrigerator for 30 min to let the extracts diffusion well into the agar. Then, the plates were incubated at 37°C for 18-24 h. Antimicrobial activity was detected by measuring the zone of inhibition (including the wells diameter) appeared after the incubation period. Tween 20 was employed as a negative control.

3. RESULTS

3.1. The structure and characterization

PVA, which acts as a gelling agent, is used to form an elastic film layer so that the formed film can be easily removed without cracking. In the prepared mask, Glycerin / Centaury oil acts as a moisturizer to preserve skin moisture. The preservative used in the peel-off gel mask formula is Ascorbic acid. Ascorbic acid (vitamin C) is an ingredient of antiaging cosmetic products used for its collagen synthesis, depigmentation, and antioxidant

properties. Because of these properties, it has been used as a stabilizer in various pharmaceutical dosage forms [33, 34]. A combination of preservatives is used to enhance the effect against bacteria and fungi [1, 5, 30]. The preservatives are used in gel masks to increase the effect against bacteria and fungi.

The FT-IR spectrum of active carbon (AC) was given Fig. 5. The peaks at 3283 cm⁻¹ and are related to the OH stretching vibration in alcoholic, phenolic and carboxylic functional groups and the CH stretching of aldehyde. The band at ~1700 cm⁻¹ is attributed to the C=O stretching vibration. The peaks at 1622, 1517, 1443 and 1372 cm⁻¹ are attributed to the C=C stretching vibration, the C-H asymmetrical and symmetrical bending vibrations, respectively. The peaks at 1242, 1027 and 812 cm⁻¹ are attributed to stretching vibrations of C-O in volatile species, carboxyl acids and bending vibration of CH aromatic ring, respectively [35]. The band 2681 cm⁻¹ is related to C-H vibrational stretching for alkanes and alkyl respectively. The bands at 1572 cm⁻¹ account for C=C and C-O stretching vibrations of carboxyl group. The bands around 1159 cm⁻¹ are attributed to C-O stretching in alcohol and asymmetric stretching of ester and ether functional group. The bands between (400 and 800) cm⁻¹ are due to bending vibration of -OH groups, stretching vibration of C-O and C-H bonds [11].

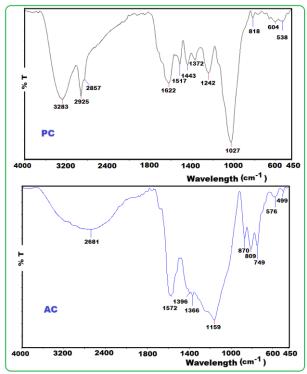


Figure 5. FT-IR spectra of pine cone (PC) and active carbon (AC).

The thermal behaviour of active carbon was determined using thermogravimetric analysis. The TGA-DTGA curves were given in Fig. 6. Thermal decomposition of pine cones has a weight loss % at 120–150 °C due to physically adsorbed water and structured water [35, 36]. The initial degradation temperature (Tonset) of the composite was found 540 °C. Tmax value is determined from the DTGA curve, was determined 599 °C. Furthermore, temperature values corresponding to weight

losses of 5% (T5) and 10 % (T10) were determined 489 and 661°C. The event in the range of 160–286 °C, 507–757 °C and of 817–901 °C were owing to the decomposition of hemicellulose and cellulose, decomposition of lignin and the carbon constituent [35].

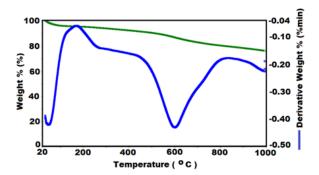


Figure 6. TGA-DTGA curves of active carbon.

The specific surface area (SBET), total pore volume and average pore diameter of AC were found to be 536.998 m²/g, 4.9x10 cm3g⁻¹ and 1.8 nm, respectively. According to literature, the pore size distributions were determined in nitrogen adsorption/desorption isotherms, as micropore (0-2 nm), mesopore (2-50 nm) and macropore (50-300 nm) [37]. According to BET results, diameter pores of activated carbon show micro. These pores on the activated carbon surface absorb chemicals, which enables it to draw bacteria and impurities from the skin, improve acne, minimize pores, treat skin conditions [38]. Activated carbon peel off mask show a very efficient effect on to the skin. The most important characteristic of an activated carbon is its adsorption capacity. Using it as an active ingredient in peel off mask, it adds to its value by enhancing the role of peel off mask by absorbing dust particles and opening the clogged pores [4]. Activated carbon has micro pores, which increase its surface area and therefore its adsorptive properties. Activated carbon is added to cosmetic preparations because of its ability to adsorb oils and clogged pores [38].

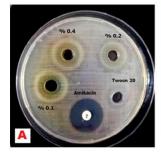
The particle size distribution of the sample was evaluated using a light scattering particle size analyzer equipped with an aqueous sample dispersion unit. Results were expressed as the median (d50%) and as the span, which is an index indicative of the particle size dispersity. Span is calculated with equation 1 follow:

$$Span = (d90\% - d10\%)/d50\%$$
 (1)

where dx% (x%=10, 50, or 90%) means that the volume percentage of particles with diameters up to dx % is equal to x%, and the smaller the span, the narrower the particle size distribution [5]. The active carbon was diluted using deionized water. Size distribution of the active carbon was found as < 17 < 68 and < 204 mm for 10, 50 and 90% cumulative mass, respectively. Light scattering particle size analysis was carried out, and it was verified a particle size disparity (span=2.75).

The antimicrobial activities of the St. John wort oil was investigated by using disc diffusion method on Gram-

positive bacteria (S. aureus), Gram-negative bacteria (P. aeruginosa) as well as C.albicans. The antimicrobial activities of St John's Wort oil was studied by the agar well diffusion method. St. John's Wort oil diluted with tween 20 as 0.1 %, 0.2% and 0.4%. Three standard reference antibiotic, amikacin was used as reference controls for the tested bacteria. The antibacterial activity was evaluated by measuring the diameter of inhibitory zones in millimeters using digital calliper. Inhibition zone diameters (mm) were measured and summarized on Table 1. Atopic dermatitis is caused by abnormal overgrowth of S. aureus, a common cause of skin infections [22]. The results showed that St. John's Wort oil sample has antimicrobial activity on both test microorganisms (Fig.7). In the case of *C. albicans*, the inhibition of the fungus's development was no observed. The healing rate of chronic wounds is affected by bacterial infections (such as S. aureus, E. coli, and P. aeruginosa), pain, inflammation, and blood flow, and thus infection and inflammation control can assist in accelerating healing [39]. The effect of antibacterial activity of essential oils depends on the growth of bacteria (bacteriostatic) or destroying bacterial cells (bactericidal). In addition, essential oils extracted from oregano, basil, and coriander plants possess an inhibitory effect against P. aeruginosa and S. aureus [40]. Moreover, it was reported that essential oils were more effective against Gram-positive bacteria than Gram-negative bacteria, which also supports our findings [41]. These results prove that St John's Wort oil could be a good candidate to inhibit target microorganisms. Experiments were repeated 3 times.



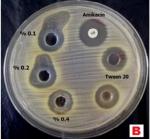


Figure 7. Antibacterial effects of membranes on *P. aeruginosa* (A) & *S. aureus* (B).

Table 1. Inhibition zone diameters of membranes on *S. aureus* and *P. aeruginosa.*

	Inhibition zones (mm)				
	Tween 20 (0)	Amikacin (antibiotic)	St.John's wort oil concentration (%)		
			0.1	0.2	0.4
S. aureus	17	20	21	20	15
P. aeruginosa	14	23	20	20	18

3.2. Evaluation parameters

The mask formula was placed in a plastic container, stored in the refrigerator and measured stability parameters such as odor, color, pH, time of preparation dried, and viscosity evaluated during 4 weeks storage with observations every 2 weeks. Since the prepared peel-off mask was stored in the refrigerator for 4 weeks, the evaluation parameters were repeated at least 3 times during this period.

- i. Color and odour: The Mask is black in color because of activated carbon. It is bergamot oil smells because of bergamot essential oil.
- ii. **The consistency of the gel mask:** Smooth and lightly applied.
- Peel Test: Peel-off facemask gel is one of the iii. popular forms of topical formulation, which is applied to hand is peeled off after few minutes of its application. According to literature, these masks have many advantages like giving moisturizing effects, cleanness, removing dead cells. They are also used for the skin problems such as acne, wrinkles, atopic dermatitis [42]. Peeling gel was applied evenly to the back of the hand. The peel was allowed to dry. According to the studies, the ideal drying time of the peel able mask gel is 15-30 minutes [43]. The drying time of the gel mask was then determined using a stopwatch. The drying time of the peel-off gel mask is 17 minutes. At the end of the period, the dried mask was removed from the skin surface. It was observed that the mask was easily removed without breaking as shown in Fig. 4.

3.3. Determination of viscosity

The viscosity of the peel-off gel mask was measured using a Koehler viscometer. As the amount of gelling agent increases, the viscosity of the gel mask increases. PVA is a film-forming or gelling agent and has considerable flexibility. This will cause a greater binding and retention of the fluid by the gelling agent, so the viscosity of the mask increases [43]. The viscosity value of the peel off gel mask was determined 111.57 cSt. Temperaturedependent viscosity measurement was not made. Measurements were taken at room conditions.It was observed that the viscosity increased during the 16-week storage period. The pH value should be (4.0 to 7.0) to avoid any skin irritation [44]. In addition, should be lower than 5.0 for better skin condition. The pH of the gel mask was determined using a pH meter. pH measurements were taken at room conditions. pH measurement was made at 1% concentration using distilled water [1]. The pH test results on all peel off gel mask showed a pH of 6.74. According to Hariyad and et.al., pH 4.5-6.5 was safe for use on the skin [45].

3.4. Physicochemical stability

The gel mask stored at low temperature $(4\pm 1^{\circ}C)$ did not show signs of instability. When exposed to sunlight, the drying time of the gel mask increases as the ethanol will evaporate in the gel [5]. It was observed that there was no change in the color and odor of the peel-off gel mask during 16 weeks of storage in the refrigerator. The results showed that the longer the storage time, the longer the drying time of peel-off gel masks [1].

4. DISCUSSION AND CONCLUSION

The peel off mask was successfully obtained containing activated carbon added St. John's Wort oil and showed a good spreadability. St. John's Wort oil has been found to be effective on *S. aureus* and *P. aeruginosa*. We found that peel-off mask gel containing St. John's Wort oil had antibacterial activity with an inhibition zone of 20 mm against *S. aureus* and 21mm against *P. aeruginosa*. Due to activated carbon adsorbing properties, it is being used in all sorts of beauty products from face masks to cleansers and even soaps. It is suggested that the prepared peel-off mask gel formulation was physico-chemically stable. Based on the physical evaluation and antibacterial activity of the peel-off mask gel preparations, a formula which has good characteristics was selected.

Research involving human participation and/or animals

None

Ethical approval

None of the studies conducted in this article include any studies conducted on animals or humans. Since the product obtained in this study is not considered a commercial product, no ethics committee certificate has been obtained.

Conflict of interests

The authors have no conflict of interest to declare.

Authors contribution statement

Hatice Karaer Yağmur: Conceptualization, Data curation, Investigation, Methodology, Resources, Software, Visualization, Validation, Writing-original draft, Writing-review & editing. Hatice Kübra Özer: Data curation, Methodology, Validation, Writing-review & editing.

REFERENCES

- [1] Ridwanto R, Lubis M, Syahputra R, Inriyani H. Utilisation of biopolymer combination as a material for making gel peel off mask. Proceedings of The 5th Annual International Seminar on Trends in Science and Science Education. AISTSSE 18-19 October 2018, Medan, Indonesia.
- [2] Somwanshi S, Kudale K, Dolas R, Kotade K. Formulation and evaluation of cosmetic herbal face pack for glowing skin. International Journal of Pharmacy and Biological Sciences. 2018:8 (4):49-52. https://doi.org/10.7897/2277-4343.083199.
- [3] Rum I, Suherman H. Formulation and evaluation of peel-off gel mask from whole milk yogurt and seaweed (Eucheuma cottonii) as antioxidants sources. Pharmacy & Pharmacology International Journal. 2021;9(4):132–135. https://doi.org/10.15406/ppij.2021.09.00338.
- [4] Kulkarni S, Gupta D, Bhawsar S. Formulation and evaluation of activated charcoal peel off mask. International Journal of Pharmacy Research & Technology. 2019; 9 (2): 44-48. https://doi.org/10.31838/ijprt/09.02.06.

- [5] Beringhs A, Rosa J, Stulzer H, Budal R. Sonaglio D. Green clay and aloe vera peel-off facial masks: response surface methodology applied to the formulation design. AAPS Pharm Sci Tech. 2013;14(1):444-455. https://doi.org/10.1208/s12249-013-9930-8.
- [6] Budiman A, Aulifa D, Kusuma A, Kurniawan I, Sulastri A. Peel-off gel formulation from black mulberries (Morus nigra) extract as anti-acne mask. January National Journal of Physiology Pharmacy and Pharmacology. 2017; 7(10):987-994. https://doi.org/10.5455/njppp.2017.7.04131230520 17.
- [7] Vieira R, Fernandes A, Kaneko T, Consiglieri V, Pinto C, Pereira C, et al. Physical and physicochemical stability evaluation of cosmetic formulations containing soybean extract fermented by Bifidobacterium animalis. Brazilian Journal of Pharmaceutical Science. 2009; 45(3):515-525.https://doi.org/10.1590/S1984-82502009000300018.
- [8] Iqbal M, Ashiq M. Adsorption of dyes from aqueous solutions on activated charcoal. Journal of Hazardous Materials B. 2007; 139: 57–66. https://doi.org/10.1016/j.jhazmat.2006.06.007.
- [9] Demirak A, Keskin F, Şahin Y, Kalemci V. Removal of ammonium from water by pine cone powder as biosorbent. Mugla Journal of Science and Technology. 2015; 1(1): 5-12. https://doi.org/10.22531/muglajsci.209992.
- [10] Gaudreault P. Activated Charcoal Revisited. Clinical Pediatric Emergency Medicine. 2005; 6(2):76-80. https://doi.org/10.1016/j.cpem.2005.04.005.
- [11] Bhomick P, Supong A, Baruah M, Pongener C, Sinha D. Pine Cone biomass as an efficient precursor for the synthesis of activated biocarbon for adsorption of anionic dye from aqueous solution: Isotherm, kinetic, thermodynamic and regeneration studies. Sustainable Chemistry and Pharmacy. 2018; 10:41–49.
 - https://doi.org/10.1016/j.scp.2018.09.001.
- [12] Bhat M, Yadav N, Hashmi S. Pinecone-derived porous activated carbon for high performance all-solid-state electrical double layer capacitors fabricated with flexible gel polymer electrolytes. Electrochimica Acta. 2019; 304:94-108. https://doi.org/10.1016/j.electacta.2019.02.092.
- [13] Eroglu E. Girgin S. A unique phenolic extraction method from olive oil macerate of *Hypericum* perforatum using DMSO: Assessment of in vitro anticancer activity, LC-MS/MS profile, total phenolic content and antioxidant capacity. South African Journal of Botany. 2021; 139: 6-11. https://doi.org/10.1016/j.sajb.2021.01.015.
- [14] Orhan I, Kartal M, Gülpinar A, Cos P, Matheeussen A, Maes L. et al. Assessment of antimicrobial and antiprotozoal activity of the olive oil macerate samples of *Hypericum perforatum* and their LC-DAD-MS analyses. Food Chemistry. 2013;138 (2–3):870-875.
 - https://doi.org/10.1016/j.foodchem.2012.11.053.

- [15] Maisenbacher P, Kovar K. Analysis and stability of Hyperici oleum, Planta Med. 1992; 58(4): 351-354. https://doi.org/10.1055/s-2006-961483.
- [16] Isacchi B, Bergonzi M, Carnevali F, Esch S, Vincieri F, Bilia A. Analysis and stability of the constituents of St. John's wort oils prepared with different methods. Journal of Pharmaceutical and Biomedical Analysis. 2007; 45: 756–761. https://doi.org/10.1016/j.jpba.2007.08.025.
- [17] Gîtea D, Şipoş M, Mircea T, Paşca B. The analysis of alcoholic extracts of hypericum species by: UVv/Vis spectrophotometry. Analele Universității din Oradea-Fascicula Biologie Tom. XVII / 1, 2010: 111-115.
- [18] Milosevic T, Solujic S, Sukdolak S. In vitro study of ethanolic extract of *Hypericum perforatum* L. on growth and sporulation of some bacteria and fungi. Turkish Journal of Biology. 2007: 31: 237-241.
- [19] Eğri Ö, Erdemir N. Production of *Hypericum perforatum* oil-loaded membranes for wound dressing material and in vitro tests. Artıfıcıal Cells, Nanomedicine, And Biotechnology. 2019; 47(1):1404–1415. https://doi.org/10.1080/21691401.2019.1596933.
- [20] Zhang J, Feng C, Ge P, Wang Q, Liu Y, Xu H, Zhou X. High purity separation of hypericin from *Hypericum perforatum* L. Extract with macroporous resin column coupling preparative liquid chromatography. Process Biochemistry. 2021; 103: 107–113.
 - https://doi.org/10.1016/j.procbio.2021.02.012.
- [21] Jarzębski M, Smułek W, Baranowska H, Masewicz L, Kobus-Cisowska J, Ligaj M, et al. Characterization of St. John's wort (*Hypericum perforatum L.*) and the impact of filtration process on bioactive extracts incorporated into carbohydrate-based hydrogels. Food Hydrocolloids. 2020; 104: 105748. https://doi.org/10.1016/j.foodhyd.2020.105748.
- [22] Shimamori Y. Mitsunaka S, Yamashita H, Suzuki T, Kitao T, Kubori T, et al. Staphylococcal Phage in Combination with Staphylococcus epidermidis as a Potential Treatment for *Staphylococcus aureus*-Associated Atopic Dermatitis and Suppressor of Phage-Resistant Mutants. Viruses. 2021; 13 (7). https://dx.doi.org/10.3390/v13010007.
- [23] Magnifico I, Petronio G, Venditti N, Cutuli M, Pietrangelo L, Vergalito F, et al. Atopic dermatitis as a multifactorial skin disorder. Can the analysis of pathophysiological targets represent the winning therapeutic strategy. Pharmaceuticals. 2020; 13(411). https://doi.org/10.3390/ph13110411.
- [24] Lubbe J. Secondary infections in patients with atopic dermatitis. American Journal of Clinical Dermatology. 2003; 4 (9):641-654. https://doi.org/10.2165/00128071-200304090-00006.
- [25] Nowicka D, Chilicka K, Dziendziora-Urbinska I. Host-microbe interaction on the skin and its role in the pathogenesis and treatment of atopic dermatitis. Pathogens. 2022; 11 (71). https://doi.org/10.3390/pathogens11010071.

- [26] Lacour J. Skin microbiota and atopic dermatitis: toward new therapeutic options?. Annales de Dermatologie et de Vénéréologie. 2015;142(1):18-22. https://doi.org/10.1016/S0151-9638(15)30003-X.
- [27] Li K, Tian S, Jiang J, Wang J, Chen X, Yan F. Pine cone shells-based activated carbon used for CO2 adsorption. Journal of Materials Chemistry A. 2016;4(14):5223–5234. https://doi.org/10.1039/C5TA09908K.
- [28] Arsić I. Preparation and characterization of St. John's Wort herb extracts using olive, sunflower and palm oils. Acta Facultatis Medicae Naissensis. 2016;33(2):119-126. https://doi.org/10.1515/afmnai-2016-0013.
- [29] Aydın G, Şirin N, Kekeçoğlu M, Türken T, Sipahi N., Göksu H. The use of natural preservative propolis and *hypericum perforatum* oil in herbal cream production. International Journal of Traditional and Complementary Medicine Research. 2021; 2 (1): 27 35. https://dergipark.org.tr/tr/pub/ijtcmr/issue/61352/80 7509.
- [30] Tanjung Y, Julianti A, Isnayanti I, Ricsa A. Formulation and evaluation of peel off gel facial mask from arabica coffee fruit peel extract (coffea arabica L.). International Journal of Applied Pharmaceutics. 2021;13(4):148-151. https://doi.org/10.22159/ijap.2021.v13s4.43845
- [31] Bhalodia R, Shukla J. Antibacterial and antifungal activities from leaf extracts of cassia fistula l.: An ethnomedicinal plant. J. Adv. Pharm. Technol. Res. 2011; 2:104-109. https://doi.org/ 10.4103/2231-4040.82956.
- [32] Daoud A, Malika D, Bakari S, Hfaiedh N, Mnafgui K, Kadri A, et al. Assessment of polyphenol composition, antioxidant and antimicrobial properties of various extracts of date palm pollen (DPP) from two Tunisian cultivars. Arab. J. Chem. 2015;12(8) 3075-3086. https://doi.org/10.1016/j.arabjc.2015.07.014.
- [33] Ahmad I, Sheraz M, Ahmed S, Shaikh R, Vaid F, Khattak S, et al. Photostability and interaction of ascorbic acid in cream formulations. AAPS PharmSciTech. 2011;12(3):917. https://doi.org/10.1208/s12249-011-9659-1.
- [34] Sheraz M, Khan M, Ahmed S, Kazi S, Khattak S, Ahmad, I. Factors affecting formulation characteristics and stability of ascorbic acid in water-in-oil creams. International Journal of Cosmetic Science. 2014;36: 494–504. https://doi.org/10.1111/ics.12152.
- [35] Sarabandi D, Roudini G, Barahuie F. Activated carbon derived from pine cone as a framework for the preparation of n-heptadecane nanocomposite for thermal energy storage. Journal of Energy Storage. 2019; 24100795. https://doi.org/10.1016/j.est.2019.100795.
- [36] Assefi M, Davar F, Hadadzadeh H. Green synthesis of nanosilica by thermal decomposition of pine cones and pine needles. Advanced Powder Technology. 2015; 26: 1583–1589. https://doi.org/10.1016/j.apt.2015.09.004.

- [37] Zsirka B, Horvath E, Jarvas Z, Dallos A, Mako E, Kristof J. Structural and energetical characterization of exfoliated kaolinite surfaces. Appl. Clay Sci. 2016;124-125: 54-61. https://doi.org/10.1016/j.clay.2016.01.035.
- [38] Sajjad M, Sarwar R, Ali T, Khan L, Mahmood S. Cosmetic uses of activated charcoal. International Journal of Community Medicine and Public Health. 2021; 8: 4572-4574. https://dx.doi.org/10.18203/2394-6040.ijcmph20213569.
- [39] Orchard A, Vuuren S. Commercial essential oils as potential antimicrobials to treat skin diseases. Evidence-Based Complementary and Alternative Medicine. 2017;4517971: 92. https://doi.org/10.1155/2017/4517971.
- [40] Elgayyar M, Draughon F. A, Golden D. A, Mount J. R. Antimicrobial activity of essential oils from plants against selected pathogenic and saprophytic microorganisms. Journal of Food Protection. 2001; 64:1019–1024. https://doi: 10.4315/0362-028x-64.7.1019.
- [41] Burt S. Essential oils: their antibacterial properties and potential applications in foods-a review. Int. J. Food Microbiol. 2004;94: 223–253. https://doi:10.1016/j.ijfoodmicro.2004.03.022.
- [42] Badnore A, Sorde K, Datir K, Ananthanarayan L, Pratap A, Pandit A. Preparation of antibacterial peeloff facial mask formulation incorporating biosynthesized silver nanoparticles. Applied Nanoscience. 2019;9: 279–287. https://doi.org/10.1007/s13204-018-0934-2.
- [43] Rahmasari D, Ermawati D, Weka Nugraheni R, Ramadhaningtyas Putri D, Nur Pratiwi I. Design and development of peel-off mask gel formulation of citronella oil for acne vulgaris. In Proceedings of the 2nd Health Science International Conference (HSIC 2019), 157-163. ISBN: 978-989-758-462-6. https://doi.org/10.5220/0009126101570163.
- [44] Ali S, Yosipovitch G. Skin pH: From Basic Science to Basic Skin Care. Acta Derm Venereol. 2013; 93: 261–267. https://doi.org/10.2340/00015555-1531.
- [45] Hariyadi D, Isnaeni I, Sudarma S, Suciati S, Rosita N. Peel-off emulgel mask of *Cocos nucifera* L. extract using gelling agent carbomer 940 as antiacne against Propionibacterium acnes ATCC 11827. Journal of Advanced Pharmaceutical Technology & Research, 2022;11 (4): 220-225. https://doi.org/10.4103/japtr.JAPTR_51_20from: https://www.fhi.no/nettpub/utbruddsveilederen/