Research Article

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Formulation and Evaluation of Alcohol-Free Peel-Off Facial Mask Gel Containing Red Torch Ginger Flower Extract

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Abstract: The extract of torch ginger flower (*Etlingera elatior* (Jack) R.M. Smith) showed excellent antioxidant activity. This study utilized freeze-dried powder derived from red torch ginger flower (TFE) as the active component in the formulation of an alcohol-free peel-off facial mask gel. The formulation employed PVA (12% w/w) as a film-forming agent, HPMC (1.5% w/w) as a gelling agent, and polyethylene glycol 400 (20% w/w) as a plasticizer. This combination resulted in an alcohol-free peel-off facial mask gel base with desirable characteristics, including high viscosity, short drying time, and easy removal of the film without breakage. Consequently, this formulation was employed as a gel base for loading TFE. The TFE was loaded at varying concentrations of 0.05%, 0.1%, and 0.2% w/w. This produced a decrease in the pH of the formulation, which can be attributed to the acidic composition present in TFE. After storing the formulation containing TFE at room temperature and 40°C for 28 days, it was observed that the formulation's color became darker while its physical characteristics remained largely constant. Nevertheless, the antioxidant activity of all formulations shown a tendency to decrease upon storage. This suggests that TFE can be incorporated in an alcohol-free peel-off facial mask formulation as an anti-aging skincare product, although further research is required to improve chemical stability of TFE.

Keywords: Torch ginger, peel-off mask, antioxidant activity, anti-aging cosmetic

INTRODUCTION

The aging of the skin is influenced by both external and internal causes, which lead to the production of reactive oxygen species (ROS) free radicals on the skin and a decrease in collagen and elastin creation (1, 2). Consequently, aging skin appears as the development of wrinkles, sagging skin, age spots, and dryness, along with the loss of fat that results in the skin's diminished natural smoothness. Substances that possess antioxidant properties and have the ability to stimulate collagen synthesis or inhibit the natural degradation of collagen could potentially be beneficial as anti-aging products (2). Therefore, a cosmetic product that includes anti-aging ingredients has the ability to inhibit the occurrence aging caused by free radicals.

Face masks offer rapid and extensive moisturization, oil regulation, and skin renewal. Nevertheless, various types of facial masks, such as clay and mineral masks, have seen limited appeal among users due to the difficulty of application and removal. Therefore, researchers have examined peel-off facial masks made from either natural or synthetic polymer that overcome these limitations (3).

A peel-off facial mask is a gel-based cosmetic product that is applied to the face and allowed to dry, forming a thin, transparent, and elastic film. This film exhibits a high occlusive capacity, providing it to be easily peeled off or removed from the skin without leaving any residue behind (4, 5). This formulation offers moisturizing properties and enhances the

effect of active substances on the epithelium due to the occlusive effect induced by layers of plastic polymers (5). Peel-off gel is a successful technique for incorporating active ingredients into a plastic film, resulting in numerous advantageous effects including reducing wrinkles, combating aging and acne, lightening the skin tone, and moisturizing the skin (5). Additionally, peel-off gels are beneficial for pore shrinkage, deep pore cleansing, and removal of skin debris (1, 4, 6).

Torch ginger, scientifically known as *Etlingera elatior* (Jack) R. M. Smith, is a plant of the Zingiberaceae family. It is extensively grown in several South-East Asia countries (7). The inflorescences of torch ginger are characterized by their large spear-like shape and covering with attractive bracts that can be white, pink, or red in color. It has been observed that the biological activities of *E. elatior* leaves and flowers include anticancer, antityrosinase, cytotoxicity, antioxidant, and antibacterial and antifungal properties (7-8). In recent study conducted by Sinsuebpol et al. (2023), it was found that the extract of torch ginger flowers exhibited significant levels of phenolic compounds, flavonoid, especially chlorogenic acid (9). It also demonstrated a number of cosmeceutical properties, including antioxidant, antityrosinase, anticollagenase, and inductive collagen production from fibroblast. In addition, our previous work showed that differences in the antioxidant activity were associated with different levels of total phenolic compound and chlorogenic acid produced from extracts of white, red and pink torch ginger flower. The findings indicated that the red flower extract possess the highest antioxidant activity, with the lowest concentration to inhibit 50% of free radical activity (IC50). The extract from the white and pink flowers also showed antioxidant activity, but to a lesser extent than the red flower (9). Hence, the extracted red torch ginger flower has the potential to be developed into a facial peel-off mask gel for that can slow down the aging process and minimize the production of wrinkles.

Numerous researchers have been conducted on peel-off facial masks that contain natural antioxidants. These mask formulations are made with a film-forming agent and alcohol, which helps speed up the drying process (3). Nevertheless, the use of skin products that do not contain alcohol has become more popular because of possible risks related to alcohol poisoning, as well as the negative impact on the dissolution of skin lipids, dehydration, and reduction of sebum, which can result in severe cases of eczema (10). Therefore, this study aimed to develop and characterize a peel-off facial mask gel that is alcohol-free and contains red torch ginger flower extract with a short drying period.

MATERIALS AND METHODS

Materials

Etlingera elatior (red flower) were gathered from Pathum Thani province, Thailand. 2,2-diphenyl-1-picrylhydrazyl (DPPH), gallic acid were acquired from Sigma-Aldrich (St. Louis, MO, USA). Polyvinyl alcohol (PVA), hydroxypropymethylcellulose (HPMC), phenoxyethanol SA, glycerin, polyethylene glycol 400 (PEG 400) were purchased from Chemipan.Corp (Bangkok, Thailand)

Torch ginger flower extraction

The extraction of the red torch ginger flower was conducted using the method outlined by Sinsuebpol et al., 2023 (9). In brief, flower bracts were taken out, cleaned, and allowed to dry for 18 hours at 50 °C in a hot air oven. The dried bracts were pulverized using a herbal blender (Spring Green Evolution, Thailand) and the resulting powder was extracted with 50% ethanol (v/v) for 24 hours. The resultant extract was then filtered through Whatman No. 1 filter paper. The extraction method was repeated for three times and all extracted fragments were combined. The extraction was concentrated using a rotary evaporator and subsequently subjected to freeze-drying for a duration of 24 hours. The powder of torch ginger flower extract (TFEs) was subsequently collected.

Determination of total phenolic content

The total phenolic content (TPC) of TFE samples was assessed using the method described by Sinsuebpol et al., 2023 (9). In brief, 20 µL of TFEs was combined mixed with 100 µL of Folin-Ciocalteu reagent (10% v/v) and 80 µL of sodium carbonate

(7.5%w/v) in each well of 96 well plate. The mixture was incubated for 1 hour at room temperature in the dark. The UV absorbance of reaction mixture was detected at 765 nm. Gallic acid was utilized to establish a standard calibration curve for quantifying the total phenolic content and the result was represented as milligram of gallic acid equivalents per gram of TFE powder (mg GAE/g).

concentration. The reaction mixture was incubated for 30 min in the dark at room temperature. The absorbance was measured at a wavelength of 517 nm using a microplate reader (Biohit® 830, Biohit, Helsinki, Finland). The antioxidant activity was quantified by calculating the % inhibition using the following equation;

$$\% \text{ inhibition } = \left[\frac{[\text{Abs control } - \text{Abs blank control}] - [\text{Abs sample or standard } - \text{ Abs blank sample or standard }]}{[\text{Abs control } - \text{Abs blank control}]}\right] \times 100$$

Determination of antioxidant Activity

The DPPH radical scavenging activity was assessed using a method described by Sinsuebpol et al., 2023 (9). Briefly, the TFE concentrated sample was prepared using 50% (v/v) ethanol solution and then diluted to the required concentration using purified water. A TFE sample of various concentrations, standard gallic acid, or purified water (control), each measuring 100 µL, were combined with 100 µL of 0.25 mM DPPH in ethanol. In order to reduce the impact of TFE color on the DPPH reaction, the absorbance of a blank solution (consisting of the sample at a specific concentration mixed with an equal volume of ethanol) was substracted from the absorbance of sample solution. However, the color of TFE is nearly colorless at a very low tested

Preparation of peel-off facial mask formulation

The peel-off facial mask compositions consist of containing polyvinyl alcohol as a film forming agent, hydroxypropyl methylcellulose as a gelling agent and glycerin and/or PEG 400 as plasticizers as indicated in Table 1. The peel-off facial mask gel was prepared by initially preparing a concentrated stock solution of polyvinyl alcohol (PVA) and hydroxypropyl methylcellulose (HPMC). PVA and HPMC stock solutions were aliquoted in volumes that corresponded to the intended concentrations of PVA and HPMC in the formulation, respectively. A homogenous solution was obtained by combining these two stock solutions. Glycerin and PEG 400 were combined and well mixed before being adjusted to the desired weight using purified water.

Table 1 The composition of the peel-off facial mask gel base formulations

Ingredients	Formulation				
	P1	P2	P3	P4	P5
PVA (g)	12	12	12	12	12
HPMC (g)	1.5	1.5	1.5	1.5	1.5
Glycerin (g)	15	5	10	20	-
PEG 400 (g)	5	15	10	-	20
Phenoxyethanol SA (%)	5	5	5	5	5
Purified water (g) qs. to	100	100	100	100	100

Assessment of physical properties of the peel-off facial mask gel

The physical characteristic of peel off mask gel such as color, homogeneity, pH, viscosity, spreadability and film drying time were assessed to selecting the suitable formulation.

- The characteristics of color and homogeneity

The color of the gel was observed within the container. The gel was applied and evenly distributed onto the surface of a glass slide, and its uniformity was observed.

- pH assessment

A digital pH meter was used to measure the pH of peel off mask gel. The values of pH were measured subsequent to immersing the pH meter's probe in gel. The pH was measured three times and the average, and the standard deviation were recorded.

- Assessment of gel viscosity

The viscosity of peel off mask gel (50 g) was measured using Brookfield DV-II Viscometer with spindle No. S95 rotating at a speed of 10 rpm. The viscosity was measured three times and the average, and the standard deviation were recorded.

- Test for spreadability

The peel off mask gel, weighing 1 g, was applied to the center of the petri dish. A load cell, weighing 150 g, was used to provide pressure on the formulation. The sample was allowed undisturbed for 30 seconds. The gel's diameter was measured in two dimensions: longest diameter and diameter perpendicular to the first line. The spreadability was measured three times and the average and the standard deviation were recorded.

- Film drying time test

This test is to determine the time taken for a film to dry. A uniformly thin layer of the 1 g peel off mask gel was applied over a 2.5 x 7.5 cm glass slide. The glass slide was subjected to the thermal treatment in an oven of 37±2°C, which corresponds to the approximate temperature of the human body. The film forming process was monitored at every 5-minutes intervals, which is at 5, 10, 15 and a maximum allowed period of 20 minutes. The test was conducted in triplicate. At this point, the film should be completely removed off the glass as shown in Figure 1. The results were recorded by identifying the quantify of the films that had dried at that time.

Stability studies

The stability of peel off mask gels containing TFE was studied at both room temperature and 40 ± 2°C over a period of 28 days. The TFE- peel off mask gels were assessed for their physical properties and antioxidant activity at initial, day 14 and day 28.

In order to assess the antioxidant activity of the peel off mask gels containing TFE formulation, a gel sample of 1 g was weighed and then dissolved in 95% v/v ethanol to extract the active ingredient for testing. Distilled water was used to adjust the sample volume to 10 mL. The DPPH assay was performed according to the method described in the previous section.

Statistical analysis

The results were reported in terms of the mean and standard deviations. The mean parameters were analyzed using one-way analysis of variance (ANOVA) and student's t-test to determine the significance different at the level set at 0.05. Statistical analysis was conducted using Microsoft Excel (Redmond, Washington, USA).

RESULTS AND DISCUSSION

Torch ginger (red) flower extract has been reported to have a high total phenolic content (TPC). In agreement with our previous study, Sinsuebpol et al. (2023), the TPC found in TFE, in this study, was 136.66 ± 26.06 mg GAE/g (9). For TFE and gallic acid, the concentration that exhibited the 50% DPPH radical inhibition activity, or IC50, was 76.87 ± 22.47 µg/mL and 8.88 ± 0.06 µg/mL, respectively. This demonstrated that the high TPC identified in TFE resulted in a strong antioxidant activity. The IC50 was used to determine the amount of TFE loading to

Figure 1 The dried film can be entirely removed from the the glass slide without tearing

the formulation. In order to achieve a concentration of 80 µg/mL TFE in peel off mask gel, a minimum 8 mg of TFE needs to be added in to the 100 mL of gel. Nevertheless, according to our recent study, Sinsuebpol et al. (2023), TFE (red) demonstrated additional cosmeceutical benefits such as inhibiting tyrosinase activity, preventing collagen breakdown, and stimulating collagen formation from fibroblasts at higher concentration (9). Therefore, in this investigation, the concentration of TFE in the formulation was varied between 0.05-0.2 mg/mL.

In order to formulate the peel-off mask gel base, a preliminary investigation on PVA and HPMC was conducted to determine the optimal concentration that would result in an effective formulation. The concentration of PVA, a film forming agent, was varied between 2.5 and 12% (w/w). The findings indicated that increasing the percentage of PVA resulted in a higher viscosity of the gel and a shorter drying time. Moreover, a higher concentration of PVA results in a stronger film that can be completely removed off the glass slide without tearing, as seen in Figure 1. This is due to the PVA's ability to improve film thickness and film formation after drying. Therefore, a 12% w/w PVA was selected for further investigation to determine the appropriate percentage of HPMC which was used as gelling agent in the formulation. The HPMC concentration was varied from 0-2% w/w. Increasing the concentration of HPMC leads to an increase in gel viscosity. However, when HPMC concentration exceeds 1.5% w/w, the drying time exceeds 20 minutes (data not shown).

Table 2 The physical properties of peel-off mask gel base

Ingredients	Formulation				
	P1	P2	P3	P4	P5
Physical Characteristics	Transparent gel with a distinct PVA odor and foam that formed on the surface while being prepared				
pH	5.30 ± 0.21	5.53 ± 0.04	5.44 ± 0.12	5.29 ± 0.08	5.39 ± 0.06
Viscosity (x1000Cps)	13.58 ± 0.08*	16.73 ± 0.05*	15.21 ± 0.03*	15.90 ± 0.13*	17.87 ± 0.07*
Spreadability (cm)	2.62 ± 0.18	2.58 ± 0.22	2.70 ± 0.10	2.77 ± 0.04	2.67 ± 0.11
Drying time (mins), (**)	20.00 (3)	20.00 (3)	20.00 (3)	20.00 (3)	15.00 (2) 20.00 (1)

* Denoted significant difference between the formulation and the other four formulations.

** the term "Number in blanket" refers to quantity of films that have completed dry within a specified time.

The peel off mask gel formulation used in this study consisted of PVA (12% w/w) and HPMC (1.5% w/w). Furthermore, the study examined the influence of the glycerin to PEG400 ratio, which was used as a plasticizer, on the gel physical properties. The results of this investigation are presented in Table 2. The variation in the concentration of glycerin and PEG 400 in the peel-off mask gel formulation had a significant impact on the viscosity value (p-value < 0.05) observe between different formulations. The formulations using high quantities of PEG 400 exhibited higher viscosity values. Among the formulation tested, formulation P5, which contained PEG400 (20%w/w) as a sole plasticizer exhibited the maximum viscosity and the fastest drying time. Therefore, formulation P5 was selected as the appropriate peel-off mask gel base to loading the active torch ginger (red) flower extract (TFE).

The peel-off mask gel base (formulation P5) was loaded with torch ginger flower extract (TFE) at concentrations of 0, 50, 100 and 200 mg/100 mL. These formulations were named Blank, TFE-50-POM, TFE-100-POM, TFE-200-POM, respectively. Figure 2 displays the physical appearance of the developed formulations while Table 3 provided a comprehensive physical properties overview of them. The addition of TFE to the peel-off mask gel base resulted in an altered appearance of the formulation from the clear gel to pale orange gel. The increase concentration of TFE resulted in a gel that exhibited a darker orange color and a lower pH, primarily due to the presence of the acid component of TFE, such as chlorogenic acid (9, 11). Furthermore, an increased TFE content leads to a decrease in the viscosity of peel-off mask gel, while it does not have an impact on spreadability. This phenomenon is

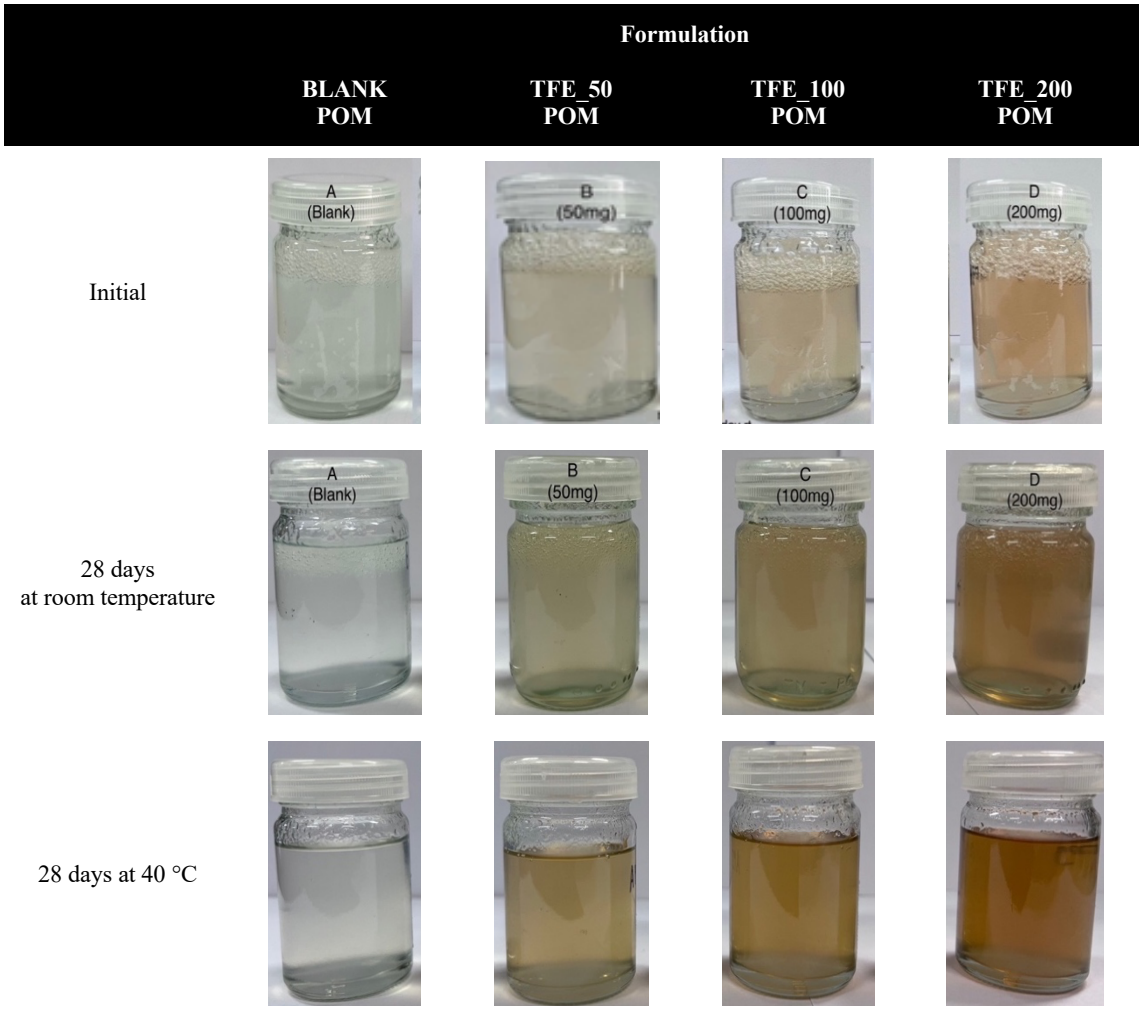

Figure 2 The physical appearance of peel-off mask gel containing TFE at the initial and 28 days after storage at room temperature and 40 °C.

explained by the formation of complexes between the polysaccharide (HPMC) and the acid component present in TFE, such as gallic acid or primarily chlorogenic acid. This interaction leads to decrease in the viscosity of the polymer, as revealed in the study conducted by Tudorache et al. (2020) (12). The addition of TFE to the formulation resulted in a longer drying time to 20 minutes of all tested samples, which however considered acceptable. Figure 3 illustrated the percentage of antioxidant activity of all formulations. Peel-off mask gel's antioxidant activity correlated with the quantity of TFE added; TFE-200-POM displayed the maximum antioxidant activity at 78.43 ± 2.71% while Blank also showed weak antioxidant activity, at 34.30 ± 1.87 %. Several research have revealed similar weak antioxidant activity results for blank formulation containing HPMC or PVA (13, 14). In addition, KIMA, the manufacturer of HPMC, reveal that several studies have demonstrated the antioxidant characteristics of HPMC. HPMC included hydroxyl groups that exhibit high reactivity and can easily undergo reactions with free radicals (15).

After 28 days of storage at room temperature and 40 °C for the physical stability study, Figure 2 and Table 3 summarize the formulation's appearance and physical properties compared with the initial point. At 28 days of storage, all formulations became darker. Higher temperature (40 °C) had a greater impact on darker gel as seen in Figure 2. Table 3 summarizes the gel's physical properties at different storage times and temperatures. The viscosity of Blank POM significantly decreased at 28 days after

Table 3 The physical properties of the peel-off mask gel containing TFE formulations at initial and after stored at room temperature and 40 °C for 28 days

	Initial	RT		40°C	
		14 days	28 days	14 days	28 days
pH					
BLANK_POM	5.58 ± 0.00	5.68±0.08	5.55±0.14	5.51±0.18	5.43±0.23
TFE_50_POM	5.45 ± 0.01[a]	5.52±0.06	5.50±0.10	5.24±0.12	5.34±0.21
TFE_100_POM	5.14 ± 0.00 [a,b]	5.38±0.05	5.33±0.18	5.26±0.16	5.25±0.21
TFE_200_POM	5.12 ± 0.00 [a]	5.20±0.07	5.17±0.12	5.03±0.13	5.03±0.14
Viscosity (x1000 cps)					
BLANK_POM	23.76 ±0.96	22.81±2.79	20.52±2.80	20.24±2.77[c]	14.73±1.99[c]
TFE_50_POM	21.53±0.10 [a]	20.60±1.79	21.98±1.07	20.81±3.65[c]	19.02±1.53[c]
TFE_100_POM	21.01±0.24 [a,b]	22.37±2.34	21.77±0.64	21.64±1.60	20.25±1.15
TFE_200_POM	20.86±0.27 [a]	22.30±0.34	20.62±1.74	19.99±2.80	21.78±1.96
Spreadability (cm)					
BLANK_POM	2.78 ± 0.05	2.67±0.16	2.60±0.33	2.67±0.08	2.83±0.13
TFE_50_POM	2.84 ± 0.31	2.62±0.21	2.72±0.28	3.05±0.25	2.52±0.28
TFE_100_POM	2.72 ± 0.15	2.73±0.19	2.50±0.33	2.57±0.13	2.57±0.13
TFE_200_POM	2.78 ± 0.24	2.58±0.19	2.72±0.16	2.65±0.28	2.35±0.32
Drying time (min)					
BLANK_POM	15.00 (2) 20.00 (1)	15.00 (3)	15.00 (3)	15.00 (3)	15.00 (3)
TFE_50_POM	20.00 (3)	15.00 (3)	15.00 (3)	15.00 (3)	15.00 (3)
TFE_100_POM	20.00 (3)	15.00 (3)	15.00 (3)	15.00 (3)	15.00 (3)
TFE_200_POM	20.00 (3)	15.00 (3)	15.00 (3)	15.00 (3)	15.00 (3)

[a] denoted the different significant value of Blank and peel-off mask gel containing TFE.

[b] denoted the different significant value to TFE_50_POM.

[c] denoted the different significant value of initial value and the value after storage.

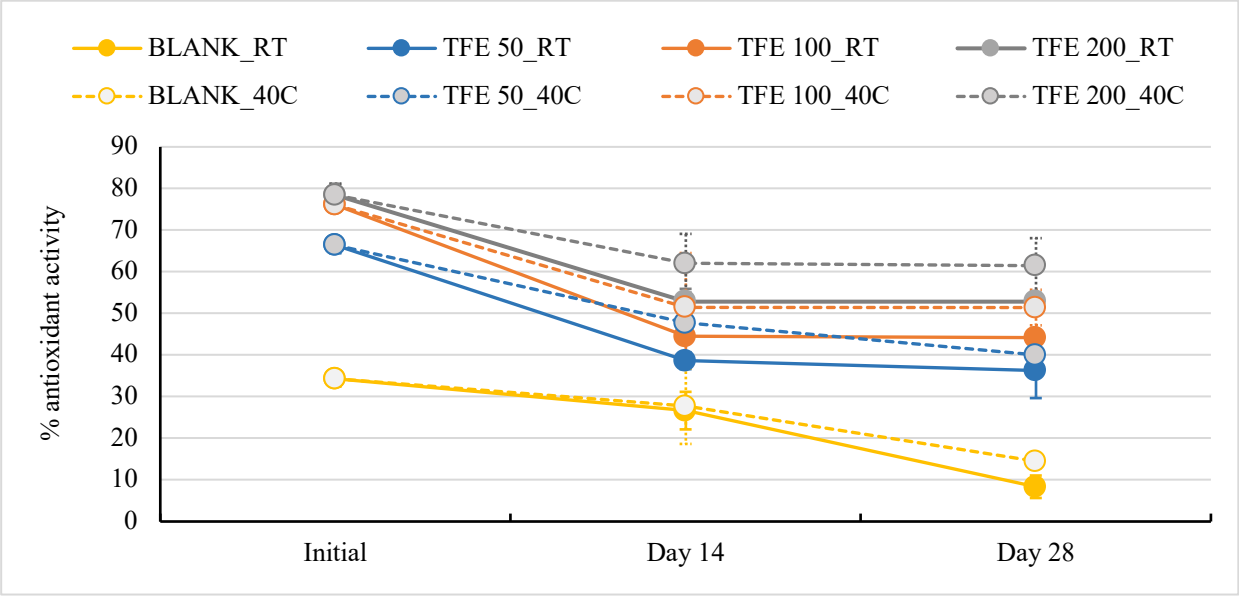

Figure 3 The antioxidant activity of the peel-off mask gel containing TFE formulations at initial and after stored at room temperature and 40 °C for 28 days

storage at 40 °C (p-value < 0.05). However, there was no significant change in viscosity, pH and spreadibility of peel-off mask gel containing TFE at all concentrations when it was stored at room temperature or 40 °C for 28 days (p-value > 0.05). The antioxidant efficacy of all the peel-off mask gel formulations containing TFE decreased after 28 days storage, at both room temperature and 40 °C as depicted in Figure 3. According to Myojin et al. (16) and Lin et al. (17), both the duration of storage and the temperature had an adverse effect on antioxidant activity. The reduction in antioxidant activity can be attributed to a change in the chemical composition of TFE, as revealed by Marsiglia et al study (18) which found a direct correlation between the degradation of TPC and antioxidant activity. Nevertheless, the formulation underwent evaporation of its liquid content leading to a reduction in the drying time from 20 minutes to 15 minutes. Moreover, water loss from the formulation during storage contributed to a more concentrated active ingredient and greater antioxidant activity, which in turn produced the formulation's higher antioxidant activity when stored at 40 °C as compared to the room temperature. In future investigation, to extend the formulation shelf life, the active ingredient must be protected using an appropriate carrier system.

CONCLUSION

A peel-off mask gel incorporating torch ginger flower extract (TFE) with alcohol-free was successfully developed. PVA, HPMC and PEG400 were the primary formulation components. TFE can be loaded to the peel-off mask gel base without any compatibility issue, resulting in a lower pH due to the acidity of chemical composition in TFE. The viscosity of the Blank formulation decreased when stored at high temperature, while the viscosity, pH and spreadibility of peel-off mask gel containing TFE at all concentrations remained unchanged when stored at room temperature or 40 °C for 28 days. Nevertheless, the antioxidant efficacy of all the peel-off mask gel formulations containing TFE decreased after 28 days, whether at toom temperature or at 40 °C. Therefore, the appropriate system has to be used to improve chemical stability of TFE in the formulation; this should be further investigated.

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