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# 브로콜리 추출물의 화장품 안정성 평가 연구

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# A Study on the Evaluation of Cosmetic Stability of Broccoli Extracts

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**요 약** : 본 연구는 브로콜리 추출물의 DPPH radical 소거활성을 통한 항산화 활성과 저온(5℃이하), 고온(50℃이상)의 보관 조건을 통해 화장품 제조 시 나타날 수 있는 온도 변화에 따른 항산화력 변화를 비교해보고, 브로콜리 추출물 5%가 함유된 크림을 제조하여 화장품의 제형 안정성, 변색 및 변취, pH, 1차 첩포 테스트를 통해 물리적, 화학적 안정성에 대해 확인하고 하였다. 실험 결과, 브로콜리 추출물 1%의 농도에서 95.5%의 높은 DPPH radical 소거 활성이 확인되었으며, 온도 변화에 따른 보관 조건에도 항산화력은 높에 유지되는 것을 확인되었다. 브로콜리 추출물 5% 함유된 크림에서도 제형의 안정성, 변색 및 변취, pH, 1차 첩포 테스트 모두 안전성과 안정성이 유지되는 것이 확인되었다. 본 연구들을 통해 브로콜리 추출물의 높은 항산화 활성과 온도 변화에 따른 항산화력 유지력을 통해 피부에 안전하고 효과적으로 사용가능한 화장품 소재로써의 가능성이 확인되었다.

*주제어 : 브로콜리, 안전성, 안정성, 천연소재, 화장품*

**Abstract** : This study compared antioxidant activity through DPPH radical scavenging activity of broccoli extract, low temperature (5℃ or less), and high temperature (50℃ or higher) storage conditions. As a result of the experiment, high DPPH radical scavenging activity of 95.5% was confirmed at 1% concentration of broccoli extract, and antioxidant power was maintained at a high level even under storage conditions due to temperature changes. Even cream containing 5% broccoli extract has been confirmed to maintain safety and stability in both preparation stability, discoloration and odor change, pH, and primary adhesion tests. Through these studies, it has been confirmed that broccoli extract is a safe and effective cosmetic material for the skin through high antioxidant activity and antioxidant power maintenance due to temperature changes.

*Keywords : broccoli, stability, safety, natural materials, cosmetic*

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## 1. Introduction

Recently, as more and more modern people try to maintain healthy lives and activities, interest in resource materials that can help them maintain health has increased, and recently, their value as medicinal plants, food, natural materials, and new material extraction materials has been recognized as important [1].

Typically, Reactive oxygenspecies (ROS), which cause inflammation in the body, are known to cause cell damage by being excessively accumulated in the body, and cause aging and disease in the human body[2,3]. We need to ensure that ROS is not excessively generated or the antioxidant system is unbalanced, and it is very important to remove ROS to keep our antioxidant system in vivo normal. Substances with antioxidant activity that remove these ROSs are called antioxidants, and for this purpose, continuous research on antioxidants is needed.

Synthetic antioxidants currently used in cosmetics are known to have antioxidant effects such as Butylated Hydroxytoluene (BHT) and Butylated Hydroxyanisole (BHA). Natural antioxidants are typical such as vitamin C, vitamin E, polyphenol, flavonoids, carotenoids, and Superoxide Dismutase (SOD). However, as synthetic antioxidants are known to cause toxicity when used for a long time[4], research on the development of antioxidants has been actively conducted, and research results on various antioxidants have been reported[5-7].

Broccoli (Brassica dleracea var, italica Plenck) is a vegetable in the cruciferous family. Representative antioxidants of Broccoli are known to contain a large amount of physiologically active substances such as $\beta$-carotens, rutin, tocopherol, ascorbic acid, glutathione, glucosinolates, quercetin, and selenium[8,9]. In particular, Broccoli is known to have a high content of glucoraphanin with excellent anti-microbial and anticancer effects[10]. In broccoli, glucoraphanin is broken down into sulforaphane by myrosinase, which has been reported to inhibit cancer cell proliferation, induce antidote, and inhibit skin damage from ultraviolet rays[11,12].

However, although broccoli is known to have excellent antioxidant power to date, research on the antioxidant activity according to temperature changes and the stability of cosmetics containing broccoli extract is insufficient.

Therefore, in this study, by using the broccoli extract extracted with 70% ethanol to confirm the DPPH radical scavenging activity, and by checking the DPPH radical scavenging activity at low temperature (5℃ or less) and high temperature (50℃ or higher), the stability of the formulation when the broccoli extract is manufactured as an antioxidant according to temperature change and the stability through physical and chemical changes in discoloration and deodorization, pH change, etc., was confirmed to confirm the applicability of cosmetics containing broccoli extract.

## 2. Research Method

### 2.1. Sample preparation

The sample in this study was extracted for 72 hours after drying the broccoli extract, adding 70% ethanol solution 10 times the weight of the sample. In order to separate only the extract, it was filtered using a filter paper, ethanol was removed through concentration under reduced pressure, and the final extract was obtained and used in this experiment.

### 2.2. DPPH radical scavenging activity measurement

To measure the DPPH radical scavenging activity of the broccoli extract, it was diluted to 0.1, 0.25, 0.5, 1%, and then mixed with 180 $\mu$L of a 10 mM DPPH (1,1-diphenyl-2-picryl-hydrazyl) solution dissolved in ethanol in a 96 well plate and 20 $\mu$L of a sample

solution to react at 37° C for 30 min, and then the absorbance was measured at 517 nm. Ascorbic acid was used as a standard material when measuring DPPH radical scavenging ability.

### 2.3. Measurement of DPPH radical scavenging activity capacity according to temperature

The DPPH radical scavenging activity of the broccoli extract was measured in the same manner as 2.1 after storing the broccoli extract additionally at low and high temperatures for 24 hours to confirm the DPPH radical scavenging activity of the broccoli extract according to the temperature change.

### 2.4. Production of cream containing broccoli extract

A cream containing a broccoli extract was prepared with reference to the study of Moon[13]. After measuring the water phase A and the paid phase B, it was heated to 70℃ or higher to dissolve all components, and then the phase B was put into phase A and emulsified at 3,000 RPM for 15 minutes. The C phase was added and emulsified under conditions of 3,000 RPM and 50℃ or less for 10 minutes, stirred, sealed, and allowed to stand for 24 hours, and used in this experime.

### 2.5. Evaluation of the first patch test

The participants in this experiment were conducted on 10 men and women in their 20s and 30s in accordance with the guidelines for the human application test and effectiveness test of cosmetics. It was applied to the inside of the upper thin of the clinical subject for 24 h without coloring or skin damage. After removing the patch, the researcher conducted a visual evaluation of the degree of skin irritation after 24 hours of daily life, and the erythema response was evaluated on a scale of 5 points. The evaluation contents are shown in Table 2.

Table 1. A Cream recipe containing broccoli extract

| No. | Ingredients | Control (%) | Experiment (%) |
|---|---|---|---|
| A | Distilled Water | 69.95 | 64.95 |
| | Glycerine | 15 | 15 |
| | Panthenol | 1 | 1 |
| | EDTA 2Na | 0.05 | 0.05 |
| B | Jojoba Oil | 8 | 8 |
| | Grape seed oil | 2 | 2 |
| | Cetearyl alcohol/Cetearyl glucoside | 1 | 1 |
| | Cetearyl Olivate | 2 | 2 |
| | Cetyl Alcohol | 1 | 1 |
| C | Broccoli Extract | - | 5 |
| | Total | 100 | 100 |

Table 2. Criteria for testing evaluation criteria (erythema)

| | Nothing at all | None. | erythema | weak erythema | Serious erythema |
|---|---|---|---|---|---|
| Value | 5 | 4 | 3 | 2 | 1 |

### 2.6. Stistical processing

This experiment was measured more than three times under the same conditions, and was expressed as mean±standard deviation (Mean ± SD). Statistical processing was analyzed using SPSS Window Version 17.0.

## 3. Results and considerations

### 3.1. DPPH radical scavenging activity

Radical erasing activity was measured using a DPPH solution to determine the electron donating capacity of the broccoli extract (Fig. 1). As a result of this experiment, it was confirmed that ascorbic acid used as a positive control group had a high radical erasing activity of 98.08% at 1% concentration, and when the broccoli extract was treated by concentration, the radical erasing activity increased as the concentration increased to 73.2%, 82.1%, 91%, and 95.5%.

Kim[14] previous study reported 55.56% of water extract and 94.75% of ethanol extract at a concentration of 2,500 μg/mL, which is thought to be very highly correlated with the content of polyphenolic compounds in broccoli extract in Pande & Akoh[15].

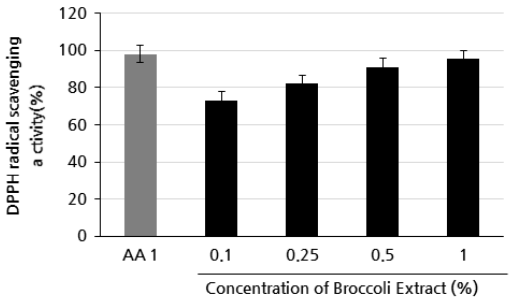


Fig. 1. DPPH radical scavenging activity of broccoli extract (AA: Ascorbic Acid).

### 3.2. Measurement of DPPH radical scavenging activity according to storage temperature changes

Radical scavenging activity was measured using a DPPH solution to check whether the broccoli extract could maintain antioxidant activity at low temperatures (5℃ or less) and high temperatures (50℃ or higher)(Fig. 2).

As a result of this experiment, concentration-dependent DPPH radical scavenging activity was confirmed when the broccoli extract was stored at low temperature and high temperature, and 95.5% of low temperature storage and 97.32% of DPPH radical scavenging activity were confirmed at 1% concentration. In addition, the DPPH radical scavenging activity of broccoli extract did not differ significantly from the room temperature-stored DPPH radical scavenging activity tested in 3.1 of this study.

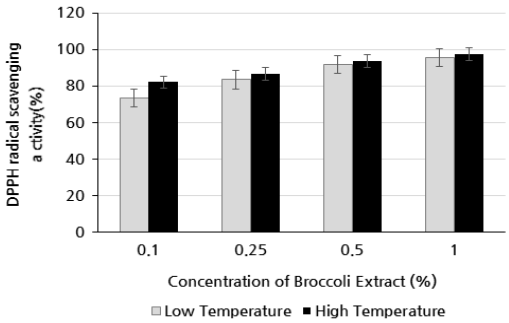


Fig. 2. DPPH radical scavenging activity of broccoli extract according to low temperature (5℃ or less) and high temperature (50℃ or higher) storage changes.

### 3.3. Changes in the stability of cosmetic formulations depending on storage temperature

It was intended to confirm the safety change of cream containing 5% of broccoli extract stored at low temperature (5℃ or less), room temperature (20℃ to 25℃), and high temperature (50℃ or higher)(Fig. 3). As a result of checking all creams manufactured under all storage conditions from day 1 to day 30, no color change was observed in creams containing broccoli extract 30 days after manufacture, and no physical or chemical changes such as

formulation separation or deodorization appeared.


Fig. 3. Results of physical and chemical changes in cream containing broccoli extract.

### 3.4. Change in pH of cosmetics depending on storage temperature

In general, the skin surface has a characteristic of weak acidity with a pH of 4.5 to 5.5, and if the skin becomes alkaline, bacterial propagation and skin resistance may decrease and adversely affect the skin, so it is recommended to use neutral or weak cosmetics, and the pH change should not be large and stable. Through this experiment, cream containing 5% broccoli extract according to changes in storage temperature at low temperature (5℃ or lower), room temperature (20℃ to 25℃), and high temperature (50℃ or higher) measured pH changes under natural light exposure conditions to confirm the stability of the product (Table 3). As a result of the experiment, the pH of the speaker product was confirmed to be low temperature 4.98, room temperature 5.02, and high temperature 5.01, and it was confirmed that the cosmetic formulations according to low temperature, room temperature, and high temperature storage were all stable in terms of pH as the differences were insufficient on the 10th, 20th, and 30th days.

### 3.5. Results of primary patch test according to storage temperature

Table 4 shows the results of the first patch experiment to evaluate the skin safety of the cream containing broccoli extract. As a result of this experiment, it was confirmed that A. cream containing no broccoli extract, B. cream containing low-temperature (5℃ or less), C. cream containing room temperature (20℃ to 25℃) storage broccoli extract, and D. cream containing high-temperature (50℃ or higher) storage broccoli extract did not show erythema reaction in the skin. After that, it was confirmed that skin erythema reaction did not appear in all items even in the result of 24 h of leaving the patch removed. These results confirmed that the broccoli extract was non-polar in the skin through the first patch test, and the safety of the skin was confirmed.

Table 3. Change in pH of cosmetics depending on storage temperature

| Day | Low Temperature | Room Temperature | High Temperature |
|---|---|---|---|
| 1 | 4.98 | 5.02 | 5.01 |
| 10 | 5.03 | 5.05 | 4.89 |
| 20 | 5.01 | 5.04 | 5.02 |
| 30 | 5.03 | 5.05 | 5.04 |

## 4. Conclusion

This study attempted to confirm the possibility of using broccoli extract as a cosmetic material by confirming the safety of formulation, discoloration and deodorization, pH and changes in antioxidant activity and temperature storage of broccoli extract. As a result of the experiment, it was confirmed that the broccoli extract showed stable and high DPPH radical scavenging activity not only at room temperature (20℃ to 25℃) but also at low temperature (5℃ or lower) and high temperature (50℃ or higher), and that the stability of the formulation, discoloration and deodorization, and pH were all maintained after manufacturing the cream containing 5% of the broccoli extract according to the change in storage temperature change. Finally, as the first patch test confirmed the safety of broccoli extract to the skin, antioxidant activity can be maintained even if temperature changes occur during the manufacturing process, and the possibility of using it as a safe cosmetic material for the skin was confirmed.

## Acknowledgement

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Table 4. Results of the first patch test for broccoli extract

| Time | A | | B | | C | | D | |
|---|---|---|---|---|---|---|---|---|
| No. | 24 h | 48 h | 24 h | 48 h | 24 h | 48 h | 24 h | 48 h |
| 1 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 2 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 3 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 4 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 6 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 7 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 8 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 9 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| 10 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |
| Mean | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 |

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