

The Effect of Anthocyanin Pigment Extracted from Roselle (*Hibiscus sabdariffa*) Flower on the Quality Attributes of Manufactured Ice Cream

Zahraa Hussein Jabr¹, Adnan Wahab Habib Al-Mudhaffar²

Department of Food Science, College of Agriculture, University of Kufa (¹, ²)

1E- mail: zahraah alkarawi@student.uokufa.edu.iq.com

2E- mail: adnan.almudhafar@uokufa.edu.iq

Abstract

This study aimed to extract anthocyanin pigment from roselle (*Hibiscus sabdariffa*) using two different solvents: ethanol with hydrochloric acid and distilled water, and to evaluate the yield of the pigment. The study also investigated the effect of adding anthocyanin at different concentrations (0.1, 0.2, 0.3, and 0.5) % on the quality attributes of ice cream. Results showed that the anthocyanin yield using ethanol with hydrochloric acid and distilled water was 546.20 mg/100 g and 450.33 mg/100 g of roselle powder, respectively. The maximum absorption wavelength of anthocyanin was recorded at 516 nm.

Upon incorporating the pigment into ice cream, the treatment P4 (0.3% concentration) exhibited the best characteristics, with total solids and titratable acidity reaching 37.2% and 0.180%, respectively. Additionally, the pH value decreased to 5.50 compared to the control sample. Total solids and acidity increased with higher pigment concentrations.

Regarding physical properties, the overrun percentage in treatment T4 reached 70.73%. The viscosity before and after aging was recorded as 8.75 and 9.18 g/cm³s, respectively. The melting rate was also evaluated.

Keywords: Roselle plant, anthocyanin pigment, ice cream, quality attributes.

Introduction

Hibiscus (commonly known as Karkadeh) is a plant that contains a wide range of bioactive compounds, including phenolic acids, flavonoids, phenols, carotenoids, glycosides, and other functional constituents, making it highly valuable in functional food applications (1). The functional food market is witnessing continuous growth due to increasing consumer interest in products that combine health benefits with sensory attributes such as flavor, taste, and appearance. Among these products, edible flowers rich in anthocyanin compounds—such as hibiscus flowers—stand out (2). Hibiscus flowers are characterized by their bright red calyces, which are rich in

vibrant anthocyanins (3). The anthocyanin present in hibiscus is a type of polyphenol in the form of glycosides, composed of a sugar moiety and an organic moiety, with the latter responsible for the coloration (4). The primary anthocyanin found in hibiscus is cyanidin-3-glucoside (5). Anthocyanin colors vary from red, orange, purple, and blue to even black, making them ideal natural pigments for use in food products, as visual appearance is a key factor influencing consumer preferences. In recent years, global interest has grown in using plant extracts proven to enhance immunity and promote human health, owing to their high concentrations of phytochemicals (6). One of the food products that can be

enriched with these compounds is ice cream—a frozen dairy product composed of two phases: a continuous phase containing sugars, proteins, salts, and water, and a dispersed phase consisting of ice crystals, air bubbles, and partially aggregated fat globules (7). Ice cream is a widely favored food product across the world due to its delicious taste, nutritional value, and low cost. Its diverse forms and unique flavors make it appealing to all age groups. In recent years, research interest has increased in using ice cream as a vehicle for delivering healthy and beneficial ingredients to consumers, along with reducing sugar and fat levels that negatively affect health. This has led to a growing interest in utilizing plant extracts as natural additives in ice cream as alternatives to synthetic additives (8).

Materials and Methods

Raw Materials

Fresh hibiscus flowers were obtained from the Al-Musayyib region in Baghdad, while the dried flowers were purchased from local markets. The flowers were thoroughly washed to extract the pigment from them.

Methods

Alcoholic Extraction of the Pigment:

The hibiscus pigment was extracted following the method described by (9). A total of 50 grams of dried hibiscus flowers were soaked in 500 mL of 96% ethanol containing 3 mL of hydrochloric acid (1.5N).

Aqueous Extraction of the Pigment:

Anthocyanin pigment was extracted from hibiscus flowers according to the method described by (10). Ten grams of the flowers were soaked in 100 mL of distilled water for 24 hours. The extract was then filtered and

concentrated using a vacuum oven at 45°C to obtain a dry powder.

Preparation of Dairy Ice Cream with Hibiscus Pigment

Dairy ice cream was prepared using 118g whole milk powder, 129g table sugar, 3g vanilla, 753g distilled water, and 0.3g a stabilizer, with the addition of hibiscus pigment powder at concentrations of 0.1%, 0.2%, 0.3%, and 0.5%, alongside a control sample without pigment. The experiments were conducted in triplicate and included chemical, physical, P1: Treatment 1 – Control (no dye), P2: Treatment with hibiscus dye at 0.1% concentration, P3: Treatment with dye at 0.2% concentration, P4: Treatment with dye at 0.3% concentration, P5: Treatment with dye at 0.5% concentration.

Chemical Analyses

Total Solids Content:

The percentage of total solids in the ice cream mixtures was determined following the method described in (13).

Titrateable Acidity:

Titrateable acidity was measured as a percentage of lactic acid according to the method described in (14).

pH Measurement:

The pH of the ice cream mixtures was measured using a pH meter in accordance with the method outlined in (15).

Physical Analyses

Yield Percentage:

The yield percentage of the final ice cream product was determined gravimetrically according to the method in (12), to evaluate production efficiency.

Melting Rate:

The melting rate of the ice cream was estimated according to the procedure outlined in (17).

Statistical Analysis

Data were statistically analyzed using a Completely Randomized Design (CRD) and factorial experiment design. The Least Significant Difference (L.S.D) test and Duncan's Multiple Range Test were applied to compare means at a significance level of ($P < 0.05$). Analysis was performed using GenStat software version 12.1 (2009).

Results and Discussion

Yield of Extracted Pigment from Hibiscus

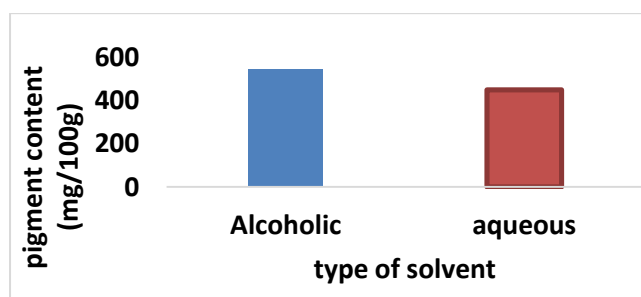


Figure (1) Yield of aqueous and alcoholic extraction of the Roselle dye.

The chemical composition of dairy ice cream mixtures prepared by adding Roselle dye

Percentage of total solids

The results in Figure (2) indicated that the percentage of total solids increased in the mixtures to which Roselle dye was added, compared to the control treatment (P1), which was 36.03%. The highest percentage was observed in treatment P5, reaching 37.7%.

As shown in Figure (1), the alcoholic extract yielded a higher anthocyanin content (546.20 mg/100 g) compared to the aqueous extract (450.33 mg/100 g), confirming the efficiency of alcohol in extracting bioactive compounds (19). The variations in anthocyanin content can be attributed to several factors, including cultivar, environmental conditions, harvest season, and storage (20). Additionally, pH plays a crucial role in the stability of anthocyanins (21). A study conducted by (22) reported that anthocyanin concentration extracted with distilled water reached 520.33 mg/g, while the concentration in the alcoholic extract was 470 mg/g.

This increase is explained by the presence of various organic compounds in Roselle, which contribute to the rise in the percentage of total solids when added. Furthermore, (23) reported that adding different concentrations of Roselle dye to ice creams raised the percentage of total solids.

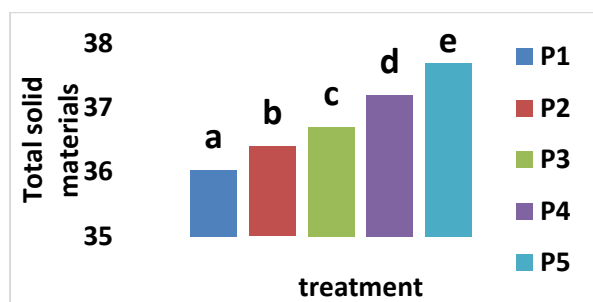


Figure (2) Total Solids of Ice

Different letters indicate significant differences at ($P < 0.05$).

The pH value of dairy ice cream with roselle extract:

The results of the study, presented in Figure (3), showed that the pH values of the ice cream containing roselle extract in the different treatments exhibited significant differences at ($P < 0.05$). The pH values decreased with the increase in the level of addition, ranging between 6.62 and 5.37,

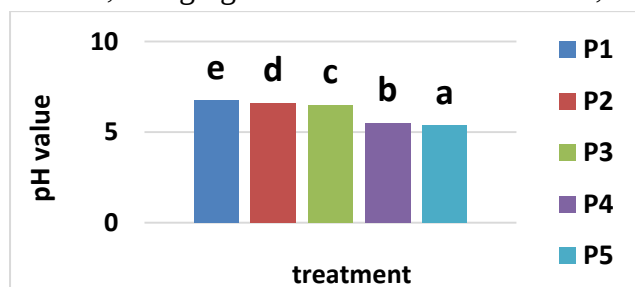


Figure (3): pH Value of Dairy Ice Cream

Different letters indicate significant differences at ($P < 0.05$).

Titrateable Acidity Percentage of Dairy Ice Cream with Added Roselle Extract

The results shown in Figure (4) indicate the effect of adding roselle extract on the titrateable acidity of the ice cream. A significant increase in titrateable acidity values was observed in the treatments, ranging between 0.17–0.195%, compared to the control treatment (P1), which

Cream Added with Roselle Dye compared to the control treatment (P1), which recorded a value of 6.75. Reference (24) confirmed that pH plays an important role in the development of flavor in dairy products. Reference (25) indicated that the decrease in pH values is attributed to the presence of organic acids in the roselle extract, in addition to its content of citric, malic, and ascorbic acids. Furthermore, Reference (25) found that the pH values of ice cream containing roselle extracts decreased with increasing concentrations.

recorded a value of 0.16%. Reference (26) stated that the increase in titrateable acidity was associated with the decrease in pH of the ice cream, due to the presence of organic acids in the roselle extract (25). Moreover, Reference (25) concluded that the titrateable acidity of ice cream containing roselle extracts increased with higher addition levels.

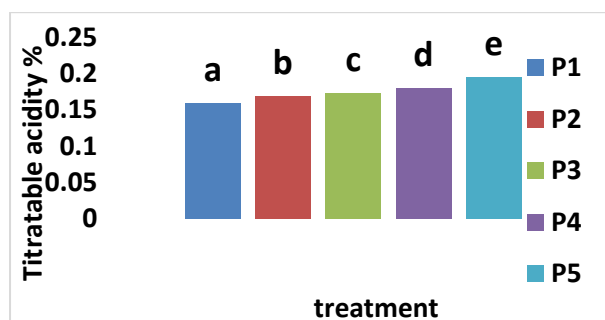


Figure (4): Titrateable Acidity Values of Dairy Ice Cream with Added Roselle Extract

Different letters indicate significant differences at ($P < 0.05$).

Physical Properties of Dairy Ice Cream Produced with Added Roselle Extract

Overrun Percentage

Overrun is a measure that reflects the increase in the volume of ice cream and is considered one of the key factors determining the structure of the final product. The incorporation of air into the product gives it a light and pleasant texture and directly affects the melting rate and shape retention during melting. The results indicate that the addition of roselle extract had an effect on the overrun

percentage of the ice cream, with the percentage increasing as the concentration of the added extract increased (25).

The results shown in Figure (5) revealed significant differences among the treatments, as the overrun percentage increased with the rise in roselle extract concentration, ranging between 64.5% and 72.96%, compared to the control treatment (P1), which recorded 62.60%. Reference (25) confirmed that the overrun percentage increased with higher concentrations of roselle extract added to the ice cream.

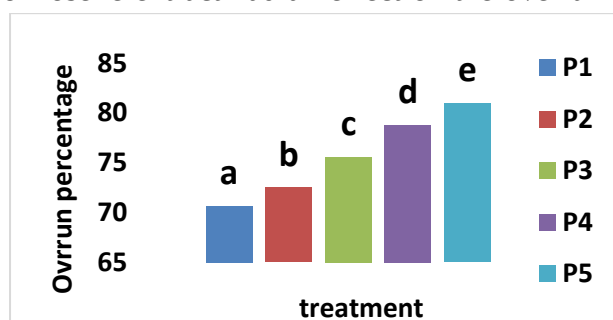


Figure (5): Overrun Percentage in Dairy Ice Cream with Added Roselle Extract

Different letters indicate significant differences at ($P < 0.05$).

Melting

The results in Table (2) indicate the effect of adding roselle extract on the melting percentage of dairy ice cream, as it contributed to a decrease in melting at 25°C over different time intervals (30, 60, and 90 minutes). After

Percentage

30 minutes at 25°C, the melting percentages were (12.2%, 12.6%, 11.3%, and 10.0%) for treatments P2, P3, P4, and P5 respectively, compared to 14.5% in the control treatment P1.

After 60 minutes, the melting percentages were (38.4%, 37.6%, 32.8%, and 28.5%) for P2, P3, P4, and P5 respectively, while the control treatment (P1) recorded 40.0%.

At the 90-minute mark, the melting percentages were (78.7%, 76.0%, 72.4%, and 69.8%) for P2, P3, P4, and P5 respectively, while the control treatment P1 showed 80.6%.

The increase in anthocyanin concentration contributes to the retention of a greater amount of air within the ice cream, thereby reducing the melting rate. In addition, anthocyanins interact with proteins, forming a large and stable network (27). Reference (26) also noted that anthocyanin pigments extracted from black carrots contributed to a reduction in melting rates as their concentration increased.

Table (1): Melting Percentage in Dairy Ice Cream with Added Roselle Extract

treatments	Melting time (minutes)		
	30	60	90
P1	10.50 %	30.00 %	70.60%
P2	10.20 %	29.40 %	69.20 %
P3	9.60 %	27.60 %	67.00 %
P4	9.30 %	24.80 %	65.40 %
P5	9.00 %	22.50 %	63.80 %

Conclusion:

The extraction of anthocyanin from roselle (*Hibiscus sabdariffa*) using different solvents demonstrated significant differences in yield, with ethanol and hydrochloric acid producing a higher yield compared to distilled water. The anthocyanin pigment showed optimal absorption at a specific wavelength, indicating its stability and suitability for food applications. Incorporating anthocyanin into ice cream at varying concentrations resulted in notable improvements in quality attributes,

including enhanced total solids, titratable acidity, and pH adjustments. The addition of anthocyanin also positively influenced the physical properties of the ice cream, such as overrun percentage and viscosity, while the melting rate remained stable. These findings suggest that anthocyanin extracted from roselle can be effectively used to improve the sensory and textural properties of ice cream, offering a natural source of color and functional benefits.

References

Akbaba, E., & Karataş, D. (2023). Phytochemicals of *Hibiscus sabdariffa* with Therapeutic Potential against SARS-CoV-2: A Molecular Docking Study. *Journal of the Institute of Science and Technology*, 13(2), 872-888.

-1
-2Teixeira, M., Tao, W., Fernandes, A., Faria, A., Ferreira, I. M., He, J., ... & Oliveira, H. (2023). Anthocyanin-rich edible flowers, current understanding of a potential new trend in dietary patterns. *Trends in Food Science & Technology*.

- 3Saha, S., Singh, J., Paul, A., Sarkar, R., Khan, Z., & Banerjee, K. (2020). Anthocyanin profiling using UV-vis spectroscopy and liquid chromatography mass spectrometry. *Journal of AOAC International*, 103(1), 23-39.
- 4Brooks, M. S. L., & Celli, G. B. (Eds.). (2019). *Anthocyanins from natural sources: Exploiting targeted delivery for improved health*. Royal Society of Chemistry. Wallace, T.C ;
- 5Abubakar, J., Edo, G., & Aydinlik, N. P. (2021). Phytochemical and GCMS analysis on the ethanol extract of *Foeniculum Vulgare* and *Petroselinum crispum* leaves. *International Journal of Chemistry and Technology*, 5(2), 117-124 .
- 6Mattioli, R., Francioso, A., Mosca, L., & Silva, P. (2020). Anthocyanins: A comprehensive review of their chemical properties and health effects on cardiovascular and neurodegenerative diseases. *Molecules*, 25(17), 3809.
- 7Lawes, A., Davies, C., & Breitweiser, C. (2023). *Natural Sugar Substitutes in Sugar Free Ice Cream* .
- 8Singo, T. M., & Beswa, D. (2019). Effect of roselle extracts on the selected quality characteristics of ice cream. *International Journal of Food Properties*, 22(1), 42-53.
- 9Du, C. T., & Francis, F. J. (1973). Anthocyanins of roselle (*Hibiscus sabdariffa*, L.). *Journal of Food Science*, 38(5), 810-812 .
- 10Akinnibosun, F. I., Akinnibosun, H. A., & Ogedegbe, D. (2009). Investigation on the antibacterial activity of the aqueous and ethanolic extracts of the leaves of *Boerhavia diffusa* L. *Science World Journal*, 4.(2)
- 11Hosseini, S., Gharachorloo, M., Ghiassi-Tarzi, B., & Ghavami, M. (2016). Evaluation the organic acids ability for extraction of anthocyanins and phenolic compounds from different sources and their degradation kinetics during cold storage. *Polish Journal of Food and Nutrition Sciences*, 66(4) .(
- 12Sleem, Riyadh Mohammad. 1986. *Dairy Ice Creams*. Dar Al-Kutub for Printing and Publishing. University of Mosul. Republic of Iraq .
- 13A.O.A.C .2014. *Association of Official Analytical Chemists. Official Method of Analysis*, 18th ed., Washington, USA.
- 14Ling , E.R.1963. *A text book of dairy chemistry*. Vol.2, practical, 3rd Ed.Chapman and Hall Limited, London.
- 15Monrroy, M., García, E., Ríos, K., & García, J. R. (2017). Extraction and physicochemical characterization of mucilage from *Opuntia cochenillifera* (L.) Miller. *Journal of Chemistry*, 2017(1), 4301901.
- 16Arbuckle, W.S. 1986. *Ice cream*. 4th ed. The AVI publishing CO .Inc. Westport . Conn. 483 p .
- 17Buck, J.S.; C.E.Walker and Picrce, M.M. 1986. Evaluation of sucrose esters in ice cream. *J. of Food Sci. , 51 : 2*
- 18Ahmed, A. S., & Ibtisam, E. M. E. Z. 2015. Microbiological and sensory properties of low fat ice cream from camel milk using natural additives. *Annals. Food Science and Technology*, 16236-2
- 19Tshimenga, K., Mukuna, A., Kabele, J., & Mputu, J. N. (2018). Evaluation of antifungal activity of *Moringa oleifera* and *Jatropha curcas* extracts as a natural fungicide against adults of *Acanthoscelides Obtectus*. *International Journal of Engineering and Applied Sciences*, 5(7), 257194 .

- 20Ahmadiani, N., Robbins, R. J., Collins, T. M., & Giusti, M. M. (2014). Anthocyanins contents, profiles, and color characteristics of red cabbage extracts from different cultivars and maturity stages. *Journal of agricultural and food chemistry*, 62(30), 7524-7531 .
- 21Aqeel, Tahir Sayed Hassan; Al-Barhawi, Najwa Ibrahim. (2021). "Natural Anthocyanin Pigments Competing with Chemical Dyes." *Arab Journal of Scientific Research*, 2(1).
- 22Haza'a, Anwar Ahmed Khalaf. (2022). *Chemophysical Properties of Anthocyanin Pigment Extracted from Different Plant Sources and Its Application in Some Dietary Systems*. Ph.D. Thesis, Tikrit University, College of Agriculture, Department of Food Science.
- 23Bandara, K., & Mahendran, T. (2020). Extraction and utilization of red colour from Roselle (*Hibiscus subdariffa* L.) calyces in ice cream. *Journal of Technology and Value Addition*, 2(2), 97-109 .
- 24Homayouni, A., Azizi, A., Ehsani, M. R., Yarmand, M. S., & Razavi, S. H. (2008). Effect of microencapsulation and resistant starch on the probiotic survival and sensory properties of synbiotic ice cream. *Food chemistry*, 111(1), 50-55 .
- 25Singo, T. M., & Beswa, D. (2019). Effect of roselle extracts on the selected quality characteristics of ice cream. *International Journal of Food Properties*, 22(1), 42-53.
- 26Shamshad, A., Butt, M. S., Nayik, G. A., Al Obaid, S., Ansari, M. J., Karabagias, I. K., ... & Ramniwas, S. (2023). Effect of storage on physicochemical attributes of ice cream enriched with microencapsulated anthocyanins from black carrot. *Food Science & Nutrition*, 11(7), 3976-3988.
- 27Dara, A., Naji-Tabasi, S., Feizy, J., Fooladi, E., & Rafe, A. (2024). Exploring the potential utilization of copigmented barberry anthocyanins in ice cream: Focusing on foaming aspects, and melting attributes. *Current Research in Food Science*, 9, 100811.
- 28Etemadi Razlighi, A., Doroudi, A., Nabeghvatan, N., Jadiri Ghalabi, E., & Smith, A. (2023). Study of the nutritional, antimicrobial and chemical properties of *Hibiscus sabdariffa*: Towards findings novel natural substance for active film. *Journal of Chemical Health Risks*.
- 29Rahman, M. N., Islam, M. N., Mia, M. M., Hossen, S., Dewan, M. F., & Mahomud, M. S. (2024). Fortification of set yoghurts with lemon peel powders: an approach to improve physicochemical, microbiological, textural and sensory properties. *Applied Food Research*, 4(1), 100386.