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Recovery of Beneficial Compounds from Fruit Waste for Application to Potential Skincare Products

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Pectin was adopted as a natural ingredient in cosmetic formulations due to its moisturizing, stabilizing, binding, viscosity-controlling, and gel-forming properties. Pectin extracted from fruit peels has the potential to become another environmentally friendly alternative to commercial pectin. This study used pectin extracted from red dragon fruit peels and durian rinds, tropical fruit waste sources, to prepare skincare lotion formulations. Also, betalains recovered from red dragon fruit were applied to those formulas as a colorant and active ingredient. The developed lotions were evaluated for pH, viscosity, spreadability, and stability. Red dragon fruit peel pectin and encapsulated betalains-based formula exhibited suitable properties and indicated antioxidant and antimicrobial activity through tests. The results provided a basis for selecting and designing vegan skincare products based on fruit waste-derived compounds.

1. Introduction

Food waste, produced from one-third of the global food (equivalent to 1.3 Gt) thrown away yearly, has been recognized as a significant international problem threatening the long-term viability of food supply chains (Mohd Basri et al., 2021). For Sustainable Development Goals by 2030, the United Nations calls for halving global per capita food waste (retail and consumer levels) (FAO, 2020). Fruit and vegetable waste, a significant contributor to global food waste, is generated in large quantities, particularly in open markets. Since the fruit and vegetable value chain generates significant waste, valorization strategies have been developed to convert these wastes into value-added products. Large quantities of fruit and vegetable waste commonly come from peels and seeds, which are promising sources for valorization. These wastes contain high concentrations of bioactive ingredients such as antioxidants (polyphenols and fibers), pigments and flavour compounds, proteins, essential oils, enzymes, and fibers (Trigo et al., 2020). Many research groups have approached the potential components obtained via the valorization and applied them to food processing or nutrition. Pectin from fruit peels is one such common component. It is a natural and versatile polysaccharide that has been of interest in the food, pharmaceutical, and cosmetic industries for decades due to its health benefits and properties of thickening, gelling, and emulsifying (Li et al., 2021). The food applications of pectin extracted from fruit peels have been demonstrated in many food models, for example, red dragon fruit peels fruit applied in jam and gummy candy (Dang-Bao et al., 2023), banana and papaya peel pectin developed in yogurt formulation (Mada et al., 2022), pectin from watermelon and pomegranate peels employed as an emulsifier in ice cream (Fırat et al., 2023), etc. While pectin has a long history of use in food products, its applications in cosmetics have only recently been explored. Lupi et al. (2015) mentioned that the predisposition to gel in suitable conditions of low methoxyl pectin room temperature could make it particularly suitable for cosmetic systems, preventing damage to thermolabile products. They intensively investigated the rheological characterization of low methoxyl pectin gels. They also proposed simulating model systems for the potential cosmetic applications of pectin. Chuenkaek and Kobayashi (2024) developed a pectin-based moisturizing film, a mixture of plasticizer (Glycerol or Polyethylene Glycol) and Citrus Waste pectin, showing potential cosmetics applications. These films demonstrated high-quality

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moisturizing properties with a strong skin affinity and water retention. They exhibited increased flexibility, high water retention, and enhanced affinity for water, which protects the skin against dryness.

Red dragon fruit and durian are tropical fruits grown in large quantities in Vietnam, containing many inedible parts (rinds and seeds) that contribute to waste issues. This work proposed the application of pectin extracted from red dragon fruit peels and durian rinds as thickers to skincare gel lotion formulation. Also, betalains extracted from red dragon fruit peels were encapsulated successfully by microcrystalline cellulose (Dang-Bao et al., 2023). As inherited from that result, encapsulated betalains were applied as bioactive ingredients for these lotions in this study. The pectin-based lotions were evaluated for pH, spreadability, viscosity, moisture absorption property, and gel stability. In addition, antioxidant and antimicrobial tests were conducted. To the best of our knowledge, no study has used pectin recovered from red dragon fruit peels and durian rinds to prepare lotions. It is highlighted that the lotions were supplemented with betalains, also extracted from red dragon fruit peels.

2. Methodology

2.1 Pectin Recovery from Fruit Waste

Fruit waste, including fresh red dragon fruit peels and durian rinds, was collected from local wholesaler fruit stores and juice and smoothie stores in Ho Chi Minh City, Vietnam. Pectin extraction of red dragon fruit peel and durian rinds was based on previous work with slight modification (Tran et al., 2022).

For red dragon fruit peels, some pretreatment steps (washing, cutting, and blanching) were required to obtain 2–3 cm pieces. Red dragon fruit peels were blended with the same weight of 96 % ethanol to obtain a pulp. The pulp extraction process was then carried out in a thermostat water bath, maintaining the temperature at 45 °C for 1 h. The mixture was filtered and pressed to obtain a red-colored solution and a solid waste. The solution was concentrated to remove solvents to form betalain extract (BE extract). The extract was encapsulated by microcrystalline cellulose (MCC) or rice bran (RB) and freeze-dried to obtain red powders as pigments (encapsulated BE). The solid waste of the filter press process was dried to obtain a dry matter for pectin extraction. Pectin extraction was carried out in 0.1 M citric acid solution with the solid-to-liquid ratio (S:L) of 1:40 (w/v) at 85 °C for 2 h in a thermostat water bath. The mixture was filtered, and the filtrate was precipitated by 95 % ethanol. The precipitate was filtered and washed with 70 % ethanol. The resulting pectin, called RDF pectin, was dried at 55 °C overnight.

Some pretreatment steps (washing, cutting, and drying) were conducted to obtain dried white chunks for durian rinds. The white chunks were ground into powder for pectin extraction. The pectin extractions were carried out in a water bath at 80 °C thermostat for 1 h with S:L of 1:20 (w/v) using 0.5 M citric acid solution. The following steps are the same as extracting red dragon fruit peel to gain pectin, which is called DR pectin.

The main functional groups present in pectin were determined by Fourier-Transform Infrared spectroscopy (FTIR) with wavelengths in the range 4,000–400 cm^{-1}. The degree of esterification (DE) of Pectin was determined by the titrimetric method of Food Chemical Codex. The viscosity of pectin solutions was measured at different concentrations (1.0–5.0 % (w/v)).

2.2 Pectin gel lotion preparation

Table 1 shows pectin gel lotion formulas based on Guide Formulations of Skin Care, Make-Up, and Hair Care (Adeka Europe Gmbh, 2017) with slight modifications. The formula composition includes three phases - A, B, and C. Phase A was prepared by mixing pectin, glycerin, and distilled water.

Table 1: Pectin gel lotion formulation

Phase	Ingredient	wt %	Function	Supplier/country
A	Distilled water	87.5	-	Viet Nam
	Pectin	1.5	Thickener	-
	Glycerin	1.9	Humectant	Malaysia
B	Butylene glycol	5.0	Humectant	China
	Caprylyl glycol	0.5	Humectant and preservative	UK
	Xanthan gum	0.2	Adhesion	China
C	Betalains in glycerin (1/24 g/mL)	1.2	Active ingredient	-
	Retinol in glycerin (1/24 g/mL)	1.2	Active ingredient	China
	Leuconostoc/Radish root ferment filtrate	1.0	Antimicrobial	US

The pectin investigated was commercial pectin, RDF pectin, and DR pectin. Phase B was prepared separately by mixing butylene glycol, caprylyl glycol, and xanthan gum with a stirrer. Phase C contains betalains and retinol in glycerin and Leuconostoc/Radish root ferment filtrate. Three types of betalains investigated in phase C were betalains extract, MCC-encapsulated powder, and RB-encapsulated powder. In this study, nine different lotion formulations were manufactured. Phase A was heated to 75–80 °C with a magnetic stirrer. Phase B was added to phase A heating, mixed at 1,500 rpm until homogenous, and then cooled down to room temperature. Phase C was added to the mixtures and stirred at 1,500 rpm until a uniform solution was obtained. Citrate buffer was added to adjust pH ≥ 4. The resulting lotions were poured into sterilized bottles for the next analysis.

2.3 Evaluation of Lotions

Appearance: In this test, the lotions were visually inspected for color, clarity, and homogeneity.
pH: The pH of formulas was measured by Laqua pH meter (Horiba, Japan) at 25 °C .
Viscosity: Viscosity was measured by DV1 Viscometer (Brookfield Ametek) at 20 rpm, 25 °C . The results were expressed in cP (centipoise).
Spreadability: One drop of each formula was dropped on the center of a glass slide. Another glass slide of the same length was placed above that slide, followed by a mass of 10 g. The lotion is sandwiched between the two glass slides and spread at a certain distance. The diameter of the spreading round was recorded.
Storage Stability: Prepared lotions were kept in closed Falcon tubes and were stored at three different conditions: 4 °C (fridge), 27 °C (atmosphere, 67–81 % relative humidity, daylight), and 40 °C (incubator) for 15 days. Any change in color and phase separation was observed and recorded visually.
Antioxidant activity: The antioxidant activity of lotions was evaluated using a slightly modified DPPH method (Swastawati et al., 2023). 24 mg of DPPH were dissolved in 100 mL of methanol to make a stock solution. DPPH stock solution was filtered using methanol to yield a usable mixture with an absorbance of 0.973 at 517 nm. 3 mL DPPH workable solutions were mixed with 100 μL lotion in a test tube. 3 mL of DPPH solution in 100 μL of methanol is given as a standard. The tubes were put in the dark for 30 min. The absorbance was determined at 517 nm. The antioxidant percentage was calculated as Eq(1):

$$\%\ of\ antioxidant\ activity = [(Ac - As)/Ac] \times 100 \quad (1)$$

where: Ac: Control reaction absorbance; As: Testing lotion sample absorbance.
Antimicrobial activity: The antibacterial activity against *S. aureus* and the antifungal activity against *C. albicans* of lotions were Standard Test Methods for Evaluation of Inactivators of Antimicrobial Agents (ASTM 1054, ASTM 2315) with the addition of CLSI M26-A to suit the test conditions. The discs filled with lotions are placed on the Mueller Hinton medium inoculated with *S. aureus* or *C. albicans*, and then incubated for 24 h at 37 °C. The formula Eq(2) was used to compute the antimicrobial percentage:

$$\%\ of\ antimicrobial\ activity = (1 - Na/Nc) \times 100 \quad (2)$$

where: Nc: the concentrations of microbes growing on the plates of the control sample; Na: the concentrations of microbes growing on the plates of the lotion sample (CFU/mL).
All results are given as average values ± standard deviations from at least three independent experiments.

3. Results and discussion

3.1 Properties of Pectin

Pectin was recovered from red dragon fruit peels in 19.8 % yield with 56.8 % of DE, which is consistent with our previous work (Dang-Bao et al., 2023), while the pectin extraction of durian rinds offered 23.5 % yield and 30.4 % of DE. Accordingly, DR is categorized as low methoxyl pectin, and RDF is high methoxyl one. Figure 1 shows the FTIR spectra of DR, RDF, and commercial pectin (CM pectin), indicating their structure similarity. A broad absorption band around 3,400 cm^{-1} was attributed to the stretching of O-H groups, and an absorption band at 2,900 cm^{-1} was due to the C-H stretching of CH_2 groups. The absorption bands in the wavelengths 800–1,200 cm^{-1} are the fingerprint regions of the typical pectin polymers. The absorption bands at 1,630–1,650 cm^{-1} and 1,730–1,760 cm^{-1} indicated the free and esterified carboxyl groups.
The viscosity of pectin solution of different concentrations (1.0–5.0 % w/v) is shown in Figure 2. The viscosity of the pectin solution increased with increasing pectin concentration, which was possibly due to shortening intermolecular distances between pectin molecules, increasing the number of hydrogen bonds with the hydroxyl group of pectin and the distortion in the velocity pattern of the liquid by hydrated molecules of pectin at an increasing concentration. The increase in pectin concentration and viscosity was reported in previous studies by other authors (Jong et al., 2023). DR pectin solutions have a lower viscosity than DRF pectin solutions.

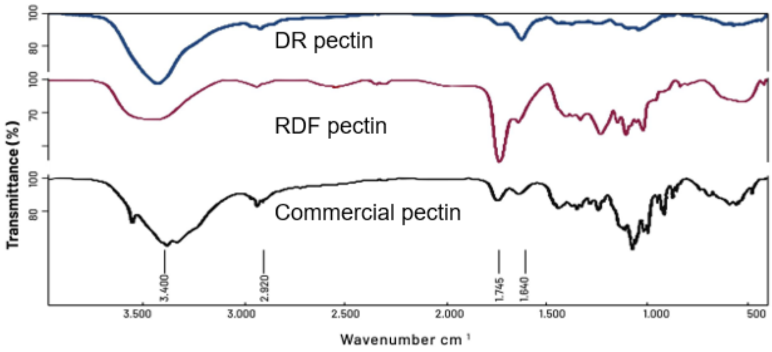

Figure 1: FT-IR spectra of DR pectin, RDF pectin, and commercial pectin

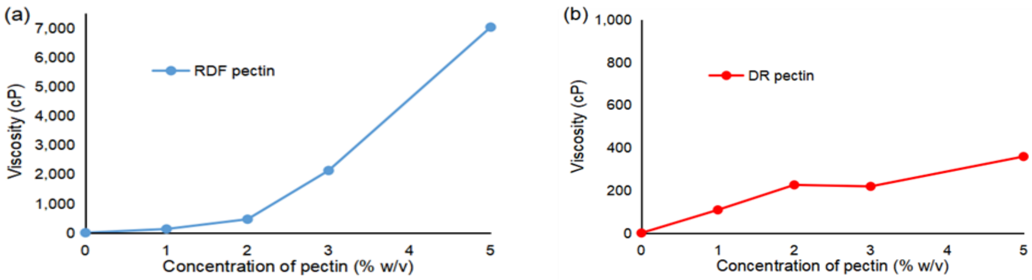

Figure 2: Effect of concentration on the viscosities of a) RDF pectin solution and b) DR pectin solutions

3.2 Characterization of lotions

Based on Adeka's guide formulations, the pectin gel lotion is supposed to be an anti-aging gel lotion that contains antioxidants and cell-communicating compounds. Retinol and betalains might help fade post-breakout marks and promote younger-looking skin. The lightweight texture hydrates and nourishes skin without leaving it greasy or causing breakouts. Pectin acts as a thickener and can be combined with polyols and surfactants. Leuconostoc/Radish Root Ferment Filtrate is used in cosmetics as an antimicrobial ingredient to prevent the product from contamination and alter its effectiveness. As seen in Figure 3a–3c, the formulas of RDF and commercial pectin are pink, while the ones of DR are brown, indicating that the color of lotions was affected by the color of pectin types. Compared to RDF-based ones, lotions using commercial pectin are more apparent. The pink color of lotions prepared with betalain extract is lighter than the one using encapsulated betalains. The texture of all samples is homogenous.

The formulations were evaluated through various parameters, such as appearance, pH, viscosity, spreadability, and stability. The results are given in Table 2. Because lotions contain retinol for anti-aging purposes, the pH range recommended for retinol products is 4–6. The best pH for the skin varies from 4 to 7, and the pH of all formulas in this study was around 4–4.5, indicating that the samples tested were safe to use as an anti-aging lotion and proper for oily skin.

All lotions have low viscosities (< 500 cP), which would be proper for skin absorption and comfort. The lotion's viscosity is acceptable since it meets the viscosity limit for lotions, which is lower than 30,000 cP (Anggelina et al., 2021). The viscosity of RDF pectin gel lotions is higher than that of others. This is beneficial for RDF pectin gel lotions when the higher viscosity of the lotion reveals more protection from microbial growth. The viscosity of RDF and DR pectin-based lotions are consistent with pectin solutions in the same concentration (Figure 2).

An ideal topical gel should spread well when applied or rubbed on the skin surface. Lotions containing RDP pectin and encapsulated BE exhibited the highest spreadability, followed by the ones prepared by commercial pectin and MCC-coated betalains. This shows that adding color powders and high methoxyl pectin causes the gel in the formulation to become thinner. It is assumed that these three formulations, which have higher dispersion, can generate comfort when used on the skin because they do not cause stickiness. Some scientists state that a lotion with a larger diameter has good spreadability (Soliman et al., 2010).

Results of stability testing are shown in Table 2 and Figure 3. Three RDF pectin-based lotions remained gel stable for 15 days in atmosphere conditions even though their color faded. CM pectin-based ones were also decolored after 15 days and slightly phase-separated after 12 days. DR pectin-based ones were phase-separated and discolored after 2 days of observation. A similar situation happened under warm conditions at 40 °C, in which DR pectin-based ones were phase-separated and decolored after 1 day, and two other types of lotions containing commercial pectin and RDF pectin retained their color for 5 days. As demonstrated in previous works, the color stability of encapsulated BE was improved more than that of the extract (Dang-Bao et al., 2023). Encapsulated betalains are also known not to maintain durability in environments with high water activity under air effects. Applying betalains to a cream-based matrix would be better than lotion. All lotions showed their stability at low temperatures. Low-temperature storage is recommended for these high-water-content products.

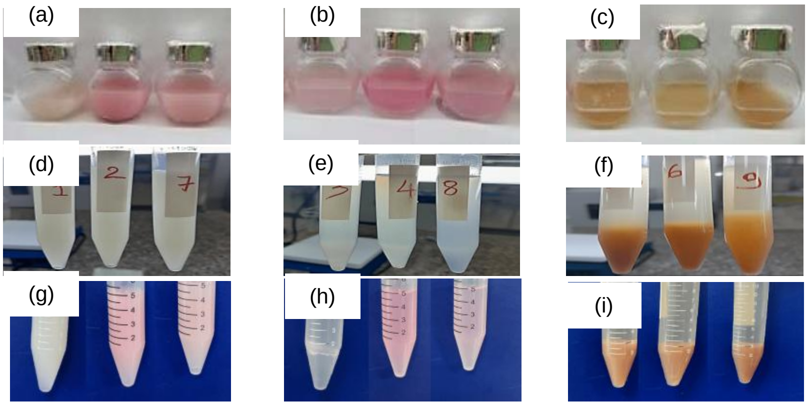

Figure 3: Gel stability observation of lotions at different storages a–c) 4 °C in fridge, d–f) 40 °C in incubator and g–i) Atmosphere 27 °C

Table 2: Characteristics of pectin gel lotions

Formula Pectin	BE	pH	Viscosity (cP)	Spreadability (cm)	Stability (color, phase separation) Atmosphere (27 °C , 15 d)	Incubator (40 °C , 15 d)	Fridge (4 °C , 15 d)
RDF	extract	4.49±0.01	421.80	3.10±0.29	Stable; decolored	Slightly separated, decolored	Stable
RDF	RB	4.53±0.01	308.80	3.60±0.21	Stable; color fades	Slightly separated, decolored	Stable
RDF	MCC	4.51±0.01	364.20	3.40±0.32	Stable; color fades	Slightly separated, decolored	Stable
CM	extract	3.96±0.06	51.63	3.10±0.06	Slightly separated, decolored	Stable, decolored	Stable
CM	RB	4.37±0.46	58.06	3.00±0.06	Slightly separated, color fades	Stable, decolored	Stable
CM	MCC	4.09±0.02	93.02	3.40±0.36	Slightly separated, color fades	Stable, decolored	Stable
DR	extract	4.01±0.01	18.60	3.00±0.20	Unstable, decolored	Unstable, decolored	Stable
DR	RB	4.00±0.01	49.39	3.10±0.15	Unstable, decolored	Unstable, decolored	Stable
DR	MCC	4.02±0.01	30.70	3.30±0.46	Unstable, decolored	Unstable, decolored	Stable

Because DR pectin-based or BE extract-colored formulas are not stable under air and light conditions after 2 days of storage, they would not be proposed for further testing on bioactivity. Among the four remaining lotions, samples with RDF pectin and encapsulated BE showed better stability at atmosphere conditions than others, and they were chosen to test antioxidant and antimicrobial properties. Although contact with the skin is essential for demonstrating moisture retention, this study did not conduct such tests on human skin to avoid ethical issues.

Table 3: Testing antioxidant and antimicrobial activity for pectin gel lotions.

Formula Pectin	BE	Antibacterial capacity (%)	Antifungal capacity (%)	Antioxidant capacity (%)
RDF	RB	> 99.99±0,00	99.97±0,00	20.66±0.00
RDF	MCC	> 99.99±0,00	> 99.99±0,00	32.95±0.01

Retinol was commonly identified in many cosmetic products for slowing the aging process (Zasada et al., 2019), and betalains derived from red dragon fruit peels were evidenced to have antioxidant capacity (Rodriguez et al., 2016). As % DPPH inhibition shown in Table 3, two lotions have higher antioxidant activity than some lotions developed in recent works, such as the activity of fish skin gelatin-based lotions prepared by Swastawati et al. (2023) and similar to skin lotion containing 0.125 % antioxidant extract from coffee pulp developed by Widiputri et al. (2020). The lotion of MCC powder revealed a higher antioxidant capacity than the one of RB powder. Although DPPH inhibition of these two lotions is lower than 50 %, adding more betalain powders can promisingly enhance their activity. Two RDF pectin-based lotions inhibited more than 99 % of the growth of *S. aureus* and *C. albican* (Table 3). This possibility is evident as the antimicrobial properties of betalains (Wijesinghe et al., 2022) and Radish Root Ferment Filtrate (Lee et al., 2020) were confirmed in previous studies, specifically resistance to *S. aureus* and *C. albican*.

4. Conclusions

The study attempted to develop skincare lotions using pectin and betalains recovered from fruit waste and evaluated their application for skincare lotions. Among lotion formulations, the ones prepared by red dragon fruit pectin and betalains fulfilled unique quality requirements in gel stability, spreadability, viscosity, and moisture coverage. Compared to DRF pectin gel lotion using RB-coated betalains, the one containing MCC powder indicated higher antioxidant capacity, which could be a potential formula for skincare products. Lotions of RDF pectin and encapsulated betalains demonstrated their activity by inhibiting the growth of *S. aureus* and *C. albicans* at an inhibitory power of > 99 %. This study demonstrated red dragon fruit peel pectin's thickening potential in skincare products, offering more diverse selections for designing vegan cosmetic formulations. The utilization of tropical fruit waste for eco-friendly products aligns with the principles of circular economy and sustainability, offering a valuable solution for fruit waste valorization.

References

Anggelina A., Pringgenies D., Yudiati E., 2021, Potential of Sodium Alginate in Sargassum sp. in Lotion Preparation to Treat Incision Wound in Mice, Biosaintifika: Journal of Biology & Biology Education, 13(1), 99–105.

Dang-Bao T., Tran U.P.N, 2023, Stability Improvement of Betalains Recovered from Red Dragon Fruit Peels (Hylocereus polyrhizus) by Cellulose-Based Encapsulation. Fibers and Polymer, 24, 2683–2696.

Fırat E., Koca N., Kaymak-Ertekin F., 2023, Extraction of pectin from watermelon and pomegranate peels with different methods and its application in ice cream as an emulsifier, Journal of Food Science, 88, 4353–4374.

Lee W.A., Keupp G.M., Brieva, H., Warren M.R., 2010, Compositions containing extracts from radish. US Patent 2010/0129305 A1.

Li D. Q., Li J., Dong H. L, Li X., Zhang J.Q., Ramaswamy S., Xu F., 2021, Pectin in biomedical and drug delivery applications: A review, International Journal of Biological Macromolecules, 185, 49–65.

Lupi F.R., Gabriele D., Seta L., Baldino N., de Cindio B., Marino R., 2015, Rheological investigation of pectin-based emulsion gels for pharmaceutical and cosmetic uses, Rheologica Acta, 54, 41–52.

Mohd Basri M.S., Abdul Karim Shah N.N, Sulaiman A., Mohamed Amin Tawakkal I.S., Mohd Nor M.Z., Ariffin S.H., Abdul Ghani N.H., Mohd Salleh F.S., 2021, Progress in the Valorization of Fruit and Vegetable Wastes: Active Packaging, Biocomposites, By-Products, and Innovative Technologies Used for Bioactive Compound Extraction, Polymers, 13(20), 3503–3540.

Soliman S.M., Abdel Malak N.S., El-Gazayerly O.N., Abdel Rehim A.A., 2010. Formulation of microemulsion gel systems for transdermal delivery of celecoxib: In vitro permeation, anti-inflammatory activity and skin irritation tests, Drug Discover & Therapy, 4, 459–471.

Rodriguez E.B., Vidallon M.L.P., Mendoza D.J.R., Reyes C.T., 2016, Health-promoting bioactivities of betalains from red dragon fruit (Hylocereus polyrhizus (Weber) Britton and Rose) peels as affected by carbohydrate encapsulation, Journal of the Science of Food and Agriculture, 96, 4679–4689.

Mada T., Duraisamy R., Abera A., Guesh F., 2022, Effect of mixed banana and papaya peel pectin on chemical compositions and storage stability of Ethiopian traditional yoghurt (ergo), International Dairy Journal, 131, 105396–105412.

Tran U.P.N., Dang-Bao T., Le P.T.K., Huynh U.D.H., Nguyen T.T.H., Le T.M., 2022, Encapsulation of Betalains Extracted from Red Dragon Fruit Peels by Freeze-drying using Microcrystalline Cellulose and Dragon Fruit Peel Pectin as Wall Materials, Chemical Engineering Transactions, 97, 31–36.

Trigo J.P., Alexandre E.M., Saraiva J.A., Pintado M.E., 2020, High value-added compounds from fruit and vegetable by-products–Characterization, bioactivities, and application in the development of novel food products, Critical Reviews in Food Science and Nutrition, 60, 1388–1416.

Widiputri D., Wijaya S., Kusumocahyo, S., 2020, Development of Skin Lotion Containing Antioxidant Extract from Coffee Pulp and Study on Its Stability, IOP Conference Series: Materials Science and Engineering, 742, 012020-012026.

Wijesinghe V.N., Choo W.S., 2022, Antimicrobial betalains, Journal of Applied Microbiology, 133(6), 3347–3367.

Zasada M., Budzisz E., 2019, Retinoids: active molecules influencing skin structure formation in cosmetic and dermatological treatments, Postepy Dermatol Alergol, 36(4), 392–397.