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CRUDE POLYSACCHARIDES AND PHENOLIC COMPOUNDS FROM MANGOSTEEN PEELS: CO-EXTRACTION, CHARACTERIZATION AND APPLICATION IN FOOD PROCESSING

Major: Food Technology
Major code: 9540101

PHD THESIS SUMMARY

HO CHI MINH CITY 2025

The thesis was completed in **University of Technology – VNU-HCM**

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Independent examiner 1:
Independent examiner 2:

Examiner 1:
Examiner 2:
Examiner 3:

The thesis will be defened before thesis committee at
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On……...

The thesis information can be looked at following libraries:

- General Science Library Ho Chi Minh city
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LIST OF PUBLICATION

International journal paper

1. Ma, N. B., Ton, N. M. N., & Le, N. L. (2024). Co-optimization of polysaccharides and polyphenols extraction from mangosteen peels using ultrasound-microwave assisted extraction (UMAE) and enzyme-ultrasound assisted extraction (EUAE) and their characterization. *Journal of Food Measurement and Characterization*, *18*(8), 6379-6393. SCIE, Q2, IF = 3.0
2. Ma, N. B., Tien, N. N. T., Vu, L. T. K., Ton, N. M. N., & Le, N. L. (2024). Comparison on chemical, structural, thermal, functional and antioxidant properties of polysaccharides and phenolics co-extracted from mangosteen (Garcinia mangostana Linn) peels by different eco-friendly hybrid technologies. *Journal of Agriculture and Food Research*, p.101507. Q1, IF = 4.8
3. Ma, N. B., Tran, N. M., Trinh, P. H., Vo, T. D., Vu, L. T. K., Ton, N. M. N., & Le, N. L. (2025). Characterization, sensory evaluation and oxidative stability of reduced-fat mayonnaise formulated with polysaccharides extracted from mangosteen peels as fat replacer. *Journal of Agriculture and Food Research*, *19*, 101747. Q1, IF = 4.8

Project research

1. Co-extraction of polysaccharides and phenolics from fruit peels using green technologies and their applications in development of health-benefit food products. Vietnam National University, Ho Chi Minh City, Vietnam. 2022 -2024. Role: main member

INTRODUCTION

1. Problem statement

Mangosteen has been cultivated and consumed not only in Asia but also throughout the world. The fruit is famous because of its delicious taste and beneficial for human health. Several research shows that its rind contains high amounts of phytochemicals and phenolics coumpounds (xanthones, anthocyanin, tannin, etc.). These compounds have been proven to have some functional properties such as anti-fungal, antioxidant, antibacterial and anti-inflammatory. In addition, its polysaccharides, in particular pectin, can be a good resource for natural stabilizers, thickeners, gelling agents, stabilizers, emulsifiers and fat-substitutes in the food industry. But mangosteen peel becomes waste after being consumed which contribute to the environment issue in Vietnam. Based on these reasons, the mangosteen pericarps (MPs) will be chosen as the raw materials for the extraction process and the physical, chemical, antioxidant properties and application in foods will be analyzed.

2. Research aims

The aim of this dissertation thesis is to focus on the co-extraction of polysaccharides and phenolic compounds from mangosteen peels, utilizing hybrid green technologies. In addition, the obtained extracts will be characterized and utilized to develop healthier mayonnaise.

3. New contributions of the thesis

- Provide the parameters for the optimization process of co-extracting polysaccharides and phenolics from MPs using different hybrid technologies

- Provide comprehensive characterizations of the obtained polysaccharides and polyphenols to have deeper insights into their potential usages

- Demonstrate potential application of mangosteen peel extracts in the food industry through a deeper understanding of their functional uses.

CHAPTER 1: LITERATURE REVIEW

Garcinia mangostana Linn, commonly grown in Southeast Asia. The total annual production of mangosteen in Southeast Asia was reported to be higher than 700,000 million tons in 2020. The mangosteen pericarp is a by-product that makes up approximately two-thirds of the total fruit weight as agricultural waste. Due to the lack of commercial applications, this waste has received little attention. However, this by-product was long ago used as a traditional medicine as a cure for chronic intestinal catarrh, dysentery, skin infections, and respiratory disorders. In addition, its components like polysaccharides and phenolics, exhibit unique structural characteristics, resulting in diverse health-promoting features. They are also becoming more popular in the development of functional foods and pharmaceuticals due to their non-toxic nature and absence of side effects. However, research on the extraction and functional description of these compounds from mangosteen peels (MPs) is still limited.

For decades, polysaccharides and phenolics have been commonly separated from botanical sources in independent procedures, but the integration of both methodologies in a simultaneous co-extraction can be a promising strategy in the handling of food wastes toward sustainable valorization and eco-friendly scenarios. Additionally, the eco-compatible technologies including microwave, ultrasound, and enzyme are attaining more recognition as a surrogate for traditional methodology by virtue of their competence to promote higher recovery of qualified products with specifically targeted characteristics. These techniques are energy-sparing, solvent-saving, time-thrifty, and cost-effective without modifying the stabilized properties of the extracts. Previous studies affirmed that the combination of enzymatic hydrolysis, sonication, and microwave irradiation have recently emerged as an effective method to improve the yield of polysaccharides from pumpkin, pomelo peels, dragon fruit peels and phenolics from mangosteen. However, there is currently a lack of information regarding the simultaneous extraction of both polysaccharides and phenolics using eco-friendly techniques as well as the comparative analysis of their

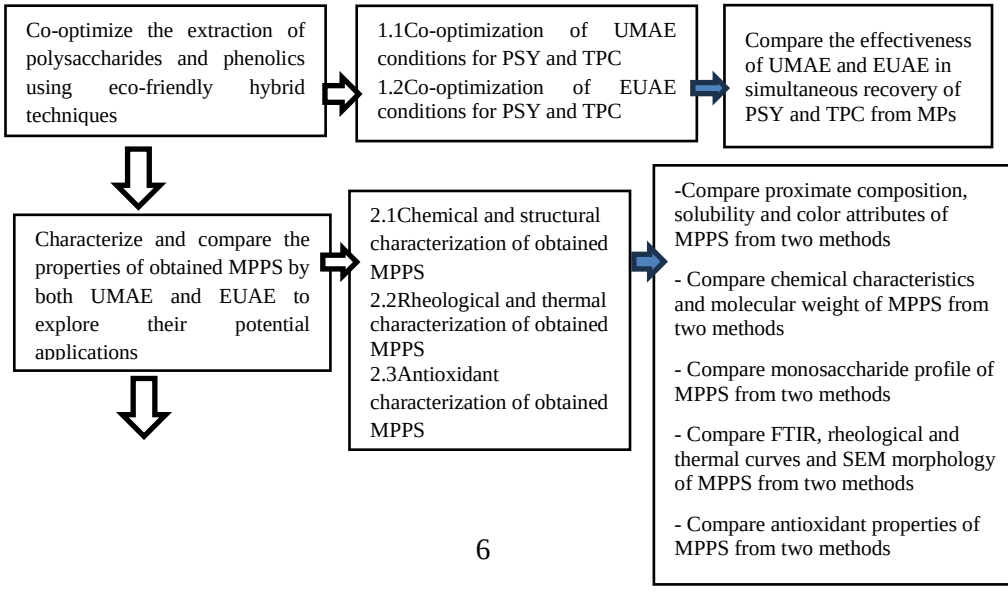
chemical structure and physicochemical characteristics and applications of these compounds in the food sector. Hence, this study will focus on co-extraction of both polysaccharides and phenolics from MPs by using ultrasound-microwave-assisted (UMAE) and enzyme-ultrasound-assisted (EUAE) hybrid extraction techniques. Their physicochemical properties, structural profiles, and antioxidant activities were characterized and compared to have a comprehensive understanding of their structures and functions for exploration of their potential applications. In addition, these compounds was applied to develop low fat mayonnaise formuation.

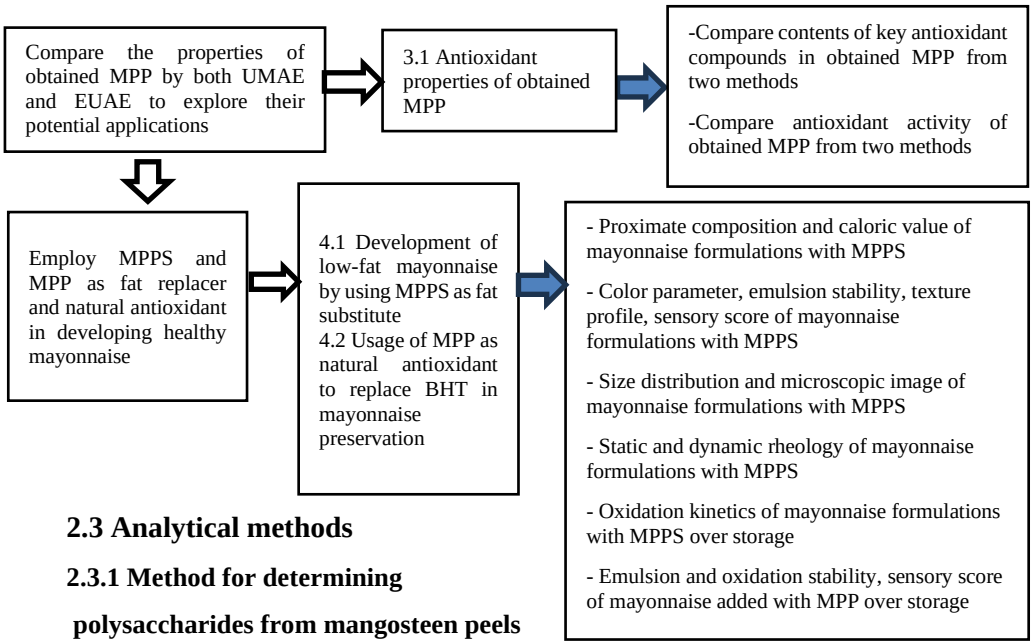
CHAPTER 2 EXPERIMENTAL

2.1 Materials

The mangosteen peels (MPs) were obtained from an organic farm located in Ben Tre Province, Vietnam. The moisture of MPs was less than 10%. MPs were pulverized and sieved at the size of 250 µm. The fine powder was stored in desiccator and used within a month. Mayonnaise ingredients including soybean oil, eggs, xanthan gum, sugar, salt, apple vinegar, mustard was purchased in Vietnam.

2.2 Research diagram





2.3 Analytical methods

2.3.1 Method for determining polysaccharides from mangosteen peels

- Moisture content, lipid contents, protein contents, ash, carbohydrate

- Determination of monosaccharide profile of polysaccharide

-The equivalent weight, methoxyl content, degree of esterification and anhydrouronic acid content, molecular weight of MPPS

- The color, surface morphology, rheological properties, thermal analysis of MPPS: FTIR, DSC

- The antioxidant properties of polysaccharides

2.3.2 Method for determining phenolics from mangosteen peels

- Determination of TPC, TFC, DPPH and anthocyanin content

- Determination of α-, β- mangostin contents.

2.3.3 Method for determining mayonnaise production

- The moisture, ash, lipid, protein, carbohydrate and caloric value

- The particle size distribution of mayonnaise samples, color of mayonnaise

- The texture, rheological properties of mayonnaise

- Other experiments: emulsion stability of mayonnaise, determination of peroxide value, sensory evaluation.

CHAPER 3: RESULTS AND DISSCUSSIONS

3.2 Co-optimization of polysaccharides and phenolics using eco-friendly hybrid techniques: UMAE, EUAE

Table 3.2 Experimental and predicted results for the optimization of PSY and TPC with the UMAE process

Run	X_1	X_2	X_3	PSY (%)		TPC (mg GAE/g DW)	
				Exp.	Pred.	Exp.	Pred.
1	40	30	10	10.72 ± 0.38	10.65	99.66 ± 2.08	100.05
2	10	50	30	11.88 ± 0.48	11.99	64.97 ± 0.17	66.03
3	40	50	20	17.62 ± 0.26	18.27	107.15 ± 1.22	109.54
4	40	50	20	17.18 ± 0.21	18.27	107.89 ± 3.30	109.54
5	40	50	20	17.35 ± 0.91	18.27	109.16 ± 2.32	109.54
6	10	30	20	13.63 ± 0.69	13.39	93.02 ± 0.49	92.88
7	40	70	10	18.30 ± 1.22	18.17	90.99 ± 1.55	91.92
8	70	30	20	10.33 ± 0.29	10.51	98.35 ± 0.79	99.03
9	70	50	30	11.67 ± 0.85	11.35	81.23 ± 0.99	81.48
10	10	70	20	14.55 ± 0.96	14.37	91.21 ± 1.29	90.53
11	40	50	20	18.70 ± 2.83	18.27	105.86 ± 3.17	109.54
12	70	50	10	14.12 ± 0.81	14.01	105.30 ± 1.75	104.24
13	40	70	30	11.53 ± 1.32	11.61	83.19 ± 0.49	82.80
14	40	50	20	20.50 ± 1.65	18.27	117.66 ± 1.38	109.54
15	70	70	20	16.13 ± 0.38	16.37	113.71 ± 1.96	113.84
16	10	50	10	13.93 ± 0.10	14.25	90.48 ± 2.23	90.23
17	40	30	30	12.15 ± 0.57	12.29	63.14 ± 1.93	62.21

X_1: Time of sonication (min); X_2: Temperature of sonication (^oC); X_3: Time of microwave irradiation (min)

Table 3.2 presents the run of the Box-Behnken design for the optimization of UMAE process. The highest PSY and TPC were observed in the number 14.

Table 3.3 and 3.4 present the results of the analysis of variance (ANOVA) for the regression models of PSY and TPC. Both models were significant ($p < 0.001$) while their lack-of-fit values were insignificant ($p > 0.05$). This demonstrates that the suggested models fitted well. Additionally, there were high degrees of

correlation between factors and responses, as the values of R^2 for PSY and TPC were 0.9486 and 0.9762, respectively. It indicates that only 5.14% and 2.38% of the total variation were undetermined, respectively for PSY and TPC.

Table 3.3 Analysis of variance for the fitted models of the responses PSY and TPC for the UMAE process

Source	Sum of Squares	Degree of freedom	Mean square	F-value	p-value
PSY					
Model	148.98	9	16.55	14.36	0.0010
X_1	0.3785	1	0.3785	0.3282	0.5846
X_2	23.39	1	23.39	20.29	0.0028
X_3	12.10	1	12.10	10.50	0.0143
X_1X_2	5.95	1	5.95	5.16	0.0573
X_1X_3	0.0400	1	0.0400	0.0347	0.8575
X_2X_3	16.81	1	16.81	14.58	0.0066
X_1^2	25.12	1	25.12	21.78	0.0023
X_2^2	19.78	1	19.78	17.16	0.0043
X_3^2	36.09	1	36.09	31.30	0.0008
Residual	8.07	7	1.15		
Lake of fit	0.4566	3	0.1522	0.0800	0.9675
R^2		0.9486			
Adj. R^2		0.8825			
Pred. R^2		0.8777			
Adeq. Precision	9.42				
C.V (%)	7.29				

X_1: Time of sonication (min); X_2: Temperature of sonication (^oC); X_3: Time of microwave irradiation (min)

Table 3.3 and 3.4 also highlights the statistical significance of the linear, quadratic and interaction effects of variables in the models through their p-values. The p-value is a statistical measure that quantifies the significance of each coefficient, providing insights into the relationship between the variables. As the p-value diminishes, the coefficient's relevance increases.

Table 3.4 Analysis of variance for the fitted models of the responses PSY àd TPC for the UMAE process (continued)

Source	Sum of Squares	Degree of freedom	Mean square	F-value	p-value
TPC					
Model	3838.37	9	426.49	31.96	< 0.0001
X_1	433.80	1	433.80	32.50	0.0007
X_2	77.69	1	77.69	5.82	0.0466
X_3	1102.15	1	1102.15	82.58	< 0.0001
X_1X_2	73.70	1	73.70	5.52	0.0511
X_1X_3	0.5184	1	0.5184	0.0388	0.8494
X_2X_3	206.21	1	206.21	15.45	0.0057
X_1^2	89.51	1	89.51	6.71	0.0360
X_2^2	144.62	1	144.62	10.84	0.0133
X_3^2	1590.93	1	1590.93	119.21	< 0.0001
Lake of fit	5.37	3	1.79	0.0813	0.9668
R^2	0.9762				
Adj. R^2	0.9457				
Pred. R^2	0.9432				
Adeq. Precision	18.43				

X_1: Time of sonication (min); X_2: Temperature of sonication (^oC); X_3: Time of microwave irradiation (min)

Regarding PSY, the p-values indicate that the variables X_2 and X_3 influenced the response by their both linear (X_2, X_3) and quadratic effects (X_2^2, X_3^2) while the variable X_1 only caused the quadratic effect (X_1^2). Among the interaction effects between two factors, the interaction X_2X_3 was significant at $p < 0.05$ while X_1X_2 was significant at $p < 0.1$ and X_2X_3 was insignificant ($p > 0.1$).

Similarly, Table 3.4, regarding TPC, all factors had significant linear and quadratic effects (X_1, X_2, X_3, X_2X_3, X_1^2, X_2^2, X_3^2) at $p < 0.05$ while the effects of the interactions had a similar trend with those of PSY.

Table 3.5 Experimental and predicted results for the optimization of PSY and TPC with the EUAE process

					PSY (%)		TPC (mg GAE/g DW)	
Run	**X_4**	**X_5**	**X_6**	**X_7**	**Exp.**	**Pred.**	**Exp.**	**Pred.**
1	30	6	50	30	19.00 ± 0.44	19.18	81.64 ± 0.55	82.91
2	60	5	50	30	31.20 ± 2.11	30.49	105.21 ± 0.78	105.61
3	30	5	40	30	22.75 ± 1.25	22.62	58.61 ± 0.66	58.55
4	90	6	50	30	18.17 ± 0.25	17.96	72.26 ± 0.40	71.26
5	60	4	40	30	17.85 ± 0.48	17.41	72.34 ± 0.60	71.29
6	60	6	50	50	15.65 ± 0.46	15.65	82.93 ± 0.48	82.38
7	60	5	60	10	18.38 ± 0.69	18.50	61.94 ± 0.79	60.27
8	30	4	50	30	22.38 ± 1.04	22.75	101.47 ± 1.23	102.57
9	60	5	40	10	18.27 ± 1.56	18.76	56.93 ± 3.68	56.59
10	60	6	60	30	15.95 ± 3.51	16.04	48.24 ± 1.56	49.63
11	30	5	60	30	20.70 ± 0.51	20.49	60.38 ± 0.30	58.71
12	60	5	40	50	16.30 ± 0.96	16.33	53.54 ± 1.62	55.31
13	90	5	60	30	20.78 ± 3.49	21.11	52.92 ± 1.56	52.54
14	60	5	50	30	31.05 ± 1.75	30.49	104.78 ± 4.50	105.61
15	60	4	50	10	19.00 ± 1.39	19.20	108.89 ± 2.04	109.00
16	30	5	50	10	20.71 ± 1.68	20.23	75.12 ± 1.31	75.36
17	90	5	40	30	16.67 ± 0.81	17.08	47.84 ± 3.19	49.07
18	60	4	50	50	18.10 ± 1.19	18.00	97.32 ± 0.79	96.43
19	90	5	50	50	16.42 ± 0.35	16.55	64.29 ± 0.78	64.39
20	60	5	60	50	18.83 ± 1.08	18.50	54.83 ± 0.64	55.27
21	60	5	50	30	29.67 ± 0.55	30.49	106.98 ± 3.66	105.61
22	60	6	40	30	15.40 ± 0.44	15.05	53.59 ± 0.32	52.04
23	60	4	60	30	18.33 ± 0.58	18.33	75.46 ± 2.45	77.34
24	90	5	50	10	23.71 ± 0.50	23.08	92.81 ± 0.60	94.02
25	30	5	50	50	24.05 ± 0.71	24.33	99.58 ± 1.31	98.71
26	90	4	50	30	19.08 ± 0.81	19.05	99.72 ± 3.40	98.56
27	60	6	50	10	16.58 ±0.88	16.88	75.64 ± 0.60	76.09
28	60	5	50	30	30.62 ± 0.75	30.49	102.86 ± 1.25	105.61
29	60	5	50	30	29.92 ± 1.27	30.49	108.21 ± 0.79	105.61

X_4: incubation time (min); X_5: pH; X_6: incubation temperature; X_7: sonication time (min)

Table 3.5 illustrates the run matrix of the Box-Behnken design for EUAE with 29 runs, containing their experimental and predicted PSY and TPC values. The PSY data ranged from 15.65% to 31.2 %, while TPC ranged from 47.84 to 108.89 mg GAE/g DW. The highest PSY value at run number 28.

Table 3.6 Analysis of variance (ANOVA) for the fitted models of the response PSY and TPC for the EUAE process

Source	Sum of Squares	DF	Mean square	F-value	p-value
PSY					
Model	700.83	14	50.06	178.35	< 0.0001
X_4	18.15	1	18.15	64.68	< 0.0001
X_5	16.31	1	16.31	58.11	< 0.0001
X_6	2.74	1	2.74	9.75	0.0075
X_7	4.44	1	4.44	15.82	0.0014
X_4X_5	1.53	1	1.53	5.43	0.0352
X_4X_6	9.49	1	9.49	33.80	< 0.0001
X_4X_7	28.25	1	28.25	100.64	< 0.0001
X_5X_6	0.0012	1	0.0012	0.0044	0.9483
X_5 X_7	0.0002	1	0.0002	0.0008	0.9778
X_6X_7	1.46	1	1.46	5.22	0.0385
X_4^2	82.72	1	82.72	294.69	< 0.0001
X_5^2	335.07	1	335.07	1193.76	< 0.0001
X_6^2	282.32	1	282.32	1005.81	< 0.0001
X_7^2	223.77	1	223.77	797.23	< 0.0001
Lack of fit	2.10	10	0.2098	0.4580	0.8556
Pure error	1.83	4	0.4580		
Cor total	704.76	28			
R^2	0.9944				
Adj. R^2	0.9888				
Pred. R^2	0.9788				
Adeq. Precision	40.54				

X_4: incubation time(min); X_5: pH; X_6: incubation temperature; X_7: sonication time (min), DF: degree of freedom

Table 3.6 and 3.7 also displays that the adequate precision measures for both PSY and TPC were much greater than 4, and their coefficients of variation were sufficiently low. Therefore, it can be concluded that there were strong correlations between the responses and studied independent variables.

Concerning PSY, all variables exerted considerable influences ($p < 0.05$) at their linear (X_4, X_5, X_6 and X_7) and quadratic (X_4^2, X_5^2, X_6^2, X_7^2) levels. The interaction coefficients of X_4X_5, X_4X_6, X_4X_7, X_6X_7 indicated remarkable effects ($p < 0.05$) while other interaction coefficients (X_5X_6 and X_5X_7) of the model were not significant ($p > 0.05$).

Table 3. 1 Analysis of variance (ANOVA) for the fitted models of the response PSY and TPC for the EUAE process (continued)

Source	Sum of Squares	DF	Mean square	F-value	p-value
TPC					
Model	12665.02	14	904.64	278.73	< 0.0001
X_4	183.77	1	183.77	56.62	< 0.0001
X_5	1654.40	1	1654.40	509.75	< 0.0001
X_6	9.94	1	9.94	3.06	0.1020
X_7	29.58	1	29.58	9.11	0.0092
X_4X_5	14.55	1	14.55	4.48	0.0526
X_4X_6	2.74	1	2.74	0.8439	0.3738
X_4X_7	701.72	1	701.72	216.21	< 0.0001
X_5X_6	17.94	1	17.94	5.53	0.0339
$X_5 X_7$	88.92	1	88.92	27.40	0.0001
X_6X_7	3.46	1	3.46	1.07	0.3194
X_4^2	984.44	1	984.44	303.32	< 0.0001
X_5^2	129.14	1	129.14	39.79	< 0.0001
X_6^2	9650.55	1	9650.55	2973.48	< 0.0001
X_7^2	671.14	1	671.14	206.79	< 0.0001
Lake of fit	28.39	10	2.84	0.6661	0.7262
Pure error	17.05	4	4.26		
Cor total	12710.46	28			
R^2	0.9964				
Adj. R^2	0.9929				
Pred. R^2	0.9850				
Adeq. Precision	46.26				

X_4: incubation time(min); X_5: pH; X_6: incubation temperature; X_7: sonication time (min), DF: degree of freedom

Regarding TPC, considerable influences ($p < 0.05$) were observed for all variables at their linear (X_4, X_5 and X_7) and quadratic (X_4^2, X_5^2, X_6^2, X_7^2) levels, except X_6. Moreover, the interaction coefficients such as X_4X_7, X_5X_6 and X_5X_7 had significant effects at $p < 0.05$ while X_4X_5 showed its significant influence at $p < 0.1$ and X_4X_6 and X_6X_7 were considered not significant ($p > 0.1$).

Concerning the UMAE method, to validate the prediction of these models, the extraction process was replicated three times under the optimum condition, resulting in 18.40% for PSY and 104.17 mg GAE/g DW for TPC.

In the case of EUAE, upon replicating the experimental optimal condition three times, the obtained data included the PSY of 32.86% and TPC of 111.17 mg GAE/g DW, which closely aligned with the predicted data and hence confirmed them.

3.3 Characterize and compare the properties of polysaccharides extracted by both UMAE and EUAE to explore their potential applications

Figure 3.6 introduces the visual and scanning electron images of polysaccharides extracted from MPs. CP had a bright-yellow color while both PS-UMAE and PS-EUAE were dark yellow to brown. On the other hand, the chromatographic profiles of polysaccharides extracted from MPs as a function of elution volume are exhibited in Figure 3.7 and their molecular weights are presented in Table 3.9

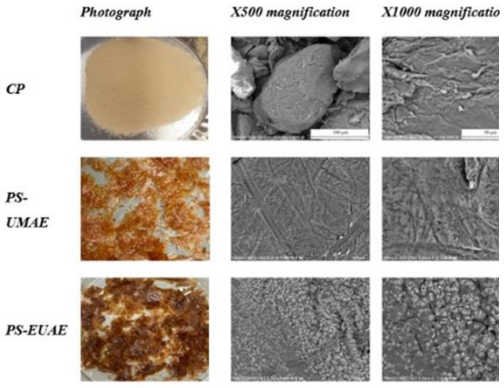


Figure 3.6 Photographs and surface morphologies of commercial pectin and MPPS

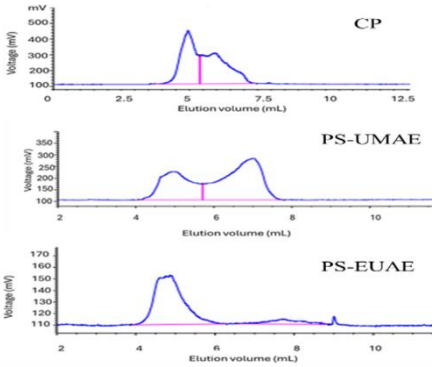


Figure 3.7 Elution profile of commercial pectin and MPPS

Table 3.9 showed that the ash and lipid contents of CP was similar to those of PS-UMAE (3.00 and 1.25%, respectively), but a bit lower than those of PS-EUAE (3.73 and 2.32%, respectively). The highest content of protein was observed in CP followed by PS-EUAE and PS-UMAE. There was no significant difference in carbohydrate content between CP and PS-EUAE, which was a bit lower than that of PS-UMAE. The highest value was observed for PS-EUAE followed by CP and PS-UMAE.

Table 3.9 proximate compositions, solubility, and color analysis of commercial pectin and polysaccharides derived from mangosteen peels

Characteristics	CP	PS-UMAE	PS-EUAE
Moisture content (%)	10.07 ± 0.51^{a}	8.87 ± 0.15^{b}	9.59 ± 0.19^{a}
Ash content (%, db)	2.90 ± 0.36^{b}	3.00 ± 0.20^{b}	3.73 ± 0.42^{a}
Protein content (%, db)	2.63 ± 0.01^{a}	0.82 ± 0.05^{c}	1.31 ± 0.09^{b}
Lipid content (%, db)	1.05 ± 0.18^{b}	1.25 ± 0.13^{b}	2.32 ± 0.20^{a}
Carbohydrate (%, db)	93.23 ± 0.34^{b}	94.82 ± 0.21^{a}	92.53 ± 0.30^{b}
Solubility (%)	73.33 ± 2.89^{b}	69.33 ± 2.31^{b}	91.33 ± 1.16^{a}
L*	89.39 ± 0.97^{a}	36.22 ± 0.12^{b}	30.20 ± 0.18^{c}
a^{*}	24.91 ± 2.25^{c}	53.86 ± 0.32^{b}	58.69 ± 0.20^{a}
b^{*}	16.26 ± 0.41^{c}	21.75 ± 0.15^{a}	21.15 ± 0.34^{b}
H^{*}_{ab}	33.22 ± 1.64^{a}	21.99 ± 0.23^{b}	19.82 ± 0.35^{c}
C^{*}	29.75 ± 2.11^{c}	58.08 ± 0.26^{b}	62.39 ± 0.14^{a}

CP, commercial pectin; PS-UMAE, obtained polysaccharides by ultrasound-microwave assisted extraction; PS-EUAE, obtained polysaccharides by enzyme-ultrasound assisted extraction; db, dry weight basis.Different letters refer to significant differences among samples in the same row ($p < 0.05$).

It demonstrates the color attributes of polysaccharides extracted from MPs. The L^{*} and H^{*}_{ab} values of the extracted polysaccharides were 30.20-36.22 and 19.82-21.99, respectively, which was lower than those of CP (89.39 and 33.22, respectively).

Table 3.10 Chemical compositions and molecular weight of commercial pectin and polysaccharides derived from mangosteen peels

Characteristics	CP	PS-UMAE	PS-EUAE
Methoxyl content (%)	5.46 ± 0.21^{b}	6.41 ± 0.72^{a}	1.22 ± 0.04^{c}
Anhydrouronic acid content (%)	63.83 ± 2.85^{b}	69.23 ± 2.03^{a}	14.55 ± 1.13^{c}
Degree of esterification (%)	48.55 ± 1.34^{b}	52.46 ± 4.41^{a}	47.72 ± 2.91^{b}
Equivalent weight (g mol^{-1})	537.1 ± 32^{b}	537.1 ± 32^{b}	2333 ± 289^{a}
Molecular weight - peak 1 (10^{5} Da)	11.85	11.66	13.24
Molecular weight - peak 2 (10^{5} Da)	2.42	0.42	0.12

CP, commercial pectin; PS-UMAE, obtained polysaccharides by ultrasound-microwave assisted extraction; PS-EUAE, obtained polysaccharides by enzyme-ultrasound assisted extraction.

Different letters refer to significant differences among samples in the same row ($p < 0.05$).

As shown in Table 3.10, methoxyl content could indicate the quality of pectin due to the

change of texture and pectin gel formed. The highest methoxyl content was observed for PS-UMAE (6.41%), followed by CP (5.46%), and PS-EUAE (1.22%). On the other hand, the anhydrouronic acid content, reflecting the purity of extracted pectin, the anhydrouronic acid content of PS-UMAE was 69.23%, which was qualified with the criterion, while PS-EUAE had lower anhydrouronic acid content (14.55%). The low anhydrouronic acid content in PS-EUAE could indicate that its chemistry was distinguished with that of pectin due to enzymatic hydrolysis.

Table 3.11 Monosaccharide composition (g/100 g sample) of commercial pectin and polysaccharides derived from mangosteen peels

Characteristics	CP	PS-UMAE	PS-EUAE
Rhamnose	1.11 ± 0.02^{a}	1.14 ± 0.03^{a}	1.05 ± 0.01^{b}
Arabinose	1.41 ± 0.00^{c}	7.87 ± 0.13^{a}	4.80 ± 0.04^{b}
Xylose	0.27 ± 0.01^{c}	0.70 ± 0.01^{a}	0.55 ± 0.01^{b}
Mannose	0.33 ± 0.02^{b}	0.81 ± 0.002^{a}	0.81 ± 0.01^{a}
Glucose	1.81 ± 0.09^{b}	2.59 ± 0.03^{a}	2.70 ± 0.18^{a}
Galactose	4.25 ± 0.16^{a}	2.59 ± 0.14^{b}	1.39 ± 0.2^{c}

CP, commercial pectin; PS-UMAE, obtained polysaccharides by ultrasound-microwave assisted extraction; PS-EUAE, obtained polysaccharides by enzyme-ultrasound assisted extraction.

Different letters refer to significant differences among samples in the same row ($p < 0.05$).

Table 3.11 demonstrates the monosaccharide profiles of polysaccharide extracted from MPs. Six monosaccharides including rhamnose, arabinose, xylose, mannose, glucose, and galactose were detected in all CP, PS-UMAE, and PS-EUAE. The galactose content of CP was 4.25 g/ 100 g sample, which was significantly higher than that of PS-UMAE (2.59 g/ 100 g sample) and PS-EUAE (1.39 g/ 100 g sample). However, CP contained lower levels of the other five monosaccharides as compared to those of the extracted polysaccharides which comprised of 1.05 – 1.14 g of rhamnose, 4.80 – 7.87 g of arabinose, 0.55 – 0.70 g of xylose, 0.81 g of mannose, 2.59 – 2.70 g of glucose, 1.39 – 2.59 g of galactose over 100 g of samples.

Table 3.12 provides the parameters of the Power law models for the rheological behavior of the samples. The *K* value, which refers to the flow consistency index, of the CP was significantly higher than those of PS-UMAE and PS-EUAE, which were consistent with the trends in Figure 3.8

Table 3.12 Power law model parameters of commercial pectin and polysaccharides derived from mangosteen peels

Sample	K (Pa.s^n)	n	R^2
CP	1.19 ± 0.02^b	0.83 ± 0.01^a	> 0.999
PS-UMAE	0.021 ± 0.004^a	0.83 ±0.04^a	> 0.998
PS-EUAE	0.012 ± 0.003^a	0.89 ± 0.04^a	> 0.998

Different letters refer to significant differences among samples in the same column ($p < 0.05$).

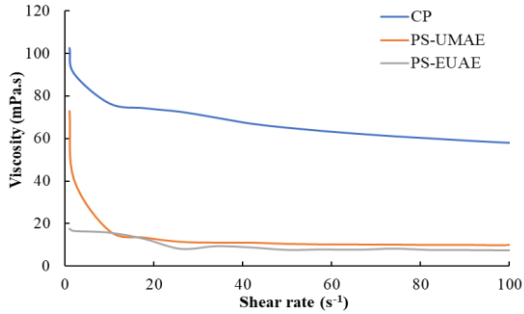


Figure 3.8 Viscosity curves of commercial pectin and polysaccharides derived from mangosteen peels.

the viscosity was in the order of CP > PS-UMAE > PS-EUAE. The viscosity of all samples remarkably lessened along with a boosting shear rate from 1 to 100 s^{-1}.

The *n* values (flow behavior index) of three samples were not significantly different and lower than 1, indicating the pseudoplastic fluids. This flow behavior was commonly observed for polysaccharides extracted from plants.

Thermal properties of polysaccharides extracted from MPs are described as DSC thermograms in Figure 3.9. To be more specific, the first endothermic peak of CP was identified at 112.4°C, while this data was shifted to around 143.5-143.7°C for PS-UMAE and PS-EUAE. In essence, this endothermic peak reflects the energy absorbed by the sample as it undergoes a phase transition. The differences in the temperatures of endothermic peak among the samples could be explained by the variations in the molecular structures like the degree of polymerization, branching patterns and intermolecular interactions, as well as extraction techniques. Another endothermic peak of all CP and extracted polysaccharides was qualified at around 205.5°C, which could be attributed to cleavage of glycosidic bonds or conformational change in galacturonan rings.

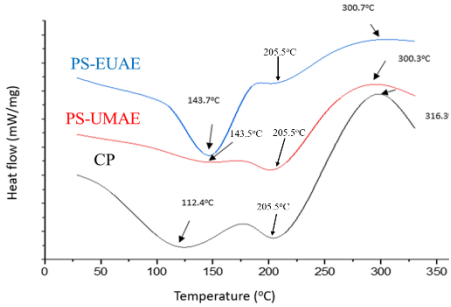


Figure 3.9 DSC thermograms of commercial pectin and MPPS

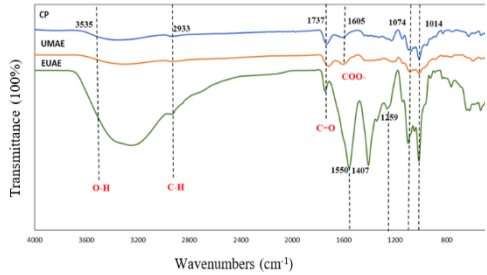


Figure 3.10 FTIR spectra of commercial pectin and MPPS

Regarding FTIR, the distinctive peaks observed at 1737 and 1605 cm^{-1} in the spectra of UMAE polysaccharides and CP, which were absent in that of EUAE one, serve as crucial signals representing the DE (degree of esterification) value of pectin sample. The DE value of CP seemed to be higher than that of UMAE polysaccharides, where the ratio of the peak at 1737 cm^{-1} (C=O stretching) over the peak at 1605 cm^{-1} (asymmetrical stretching of COO^-) of the former was larger than that of the latter. However, both of them could be considered high DE, exceeding 50% [203]. In contrast, the EUAE polysaccharide exhibited two distinct peaks in this area, including 1550 cm^{-1} for carbonyl groups, and 1407 cm^{-1} for COO^- symmetric stretching. These peaks may be associated with the complex network structure of rhamnogalacturonan, homogalacturonan and polygalacturonic acid.

Table 3.13 Antioxidant properties of commercial pectin and MPPS

Antioxidant	**CP**	**PS-UMAE**	**PS-EUAE**
TPC (mg GAE/g DM)	2.32 ± 0.11^{c}	25.87 ± 0.44^{b}	42.09 ± 0.75^{a}
AnAc (mg TE/g DM)	-	24.89 ± 0.72^{b}	44.64 ± 0.01^{a}
AnCo (mg C3G/ 100 g DM)	-	37.52 ± 0.11^{b}	65.27 ± 0.75^{a}

AnAc, antioxidant activity; AnCo, anthocyanin content; C3G, Cyanidin-3-glucoside; DM, dry matter; GAE, gallic acid equivalent; TE, Trolox equivalent; TPC, total phenolic content. Different letters refer to significant differences among samples in the same row ($p < 0.05$).

Table 3.13 presents the total phenolic content (TPC) of the polysaccharides extracted from MPs. TPC of CP was 2.32 mg GAE/g DM, which was significantly lower than that of PS-UMAE (25.87 mg GAE/g DM) and PS-EUAE (42.09 mg GAE/g DM). The higher TPC in PS-UMAE and PS-EUAE could be explained that the part of phenolic compounds released from mangosteen peels were trapped in polysaccharides during the extraction process. In addition, anthocyanins were undetectable in CP, while PS-UMAE (37.52 mg C3G/100 g DM) had significantly lower anthocyanin content than PS-EUAE (65.27 mg C3G/100 g DM). The presence of phenolics and anthocyanins contributed to the high antioxidant activity (AnAc) with PS-UMAE (24.89 mg TE/ g DM) displaying lower than PS-EUAE (44.64 mg TE/ g DM).

3.4 Compare the properties of phenolics extracted by both UMAE and EUAE to explore their potential applications

Table 3.14 Antioxidant properties of polyphenols derived from mangosteen peels

Antioxidant	PP-UMAE	PP-EUAE
TPC (mg GAE/g DE)	222.65 ± 0.5^{b}	229.57 ± 2.52^{a}
TFC (mg RE/g DE)	78.08 ± 1.07^{a}	61.58 ± 5.96^{b}
AnCo (mg C3G/100 g DE)	107.48 ± 6.43^{a}	94.97 ± 6.04^{a}
α-mangostin (μg/g DE)	46.06 ± 3.07^{b}	69.16 ± 1.71^{a}
γ-mangostin (μg/g DE)	8.68 ± 0.01^{b}	13.34 ± 2.09^{a}
AnAc (mg TE/g DE)	246.11 ± 2.17^{b}	334.56 ± 1.65^{a}

AnAc, antioxidant activity; AnCo, anthocyanin content; C3G, Cyanidin-3-glucoside; DE, dried extract; GAE, gallic acid equivalent; RE, rutin equivalent; TE, Trolox equivalent; TFC, total flavonoid content; TPC, total phenolic content. Different letters refer to significant differences among samples in the same row ($p < 0.05$)

In this study, polyphenols were simultaneously co-extracted from MPs along with polysaccharides and then concentrated into a dense product. Table 3.14 presents the TPC, TFC, AnCo, α- and γ-mangostin contents and AnAc of the extracted phenolics

Table 3.14 also displays the contents of α- and γ- mangostin, two outstanding xanthones in mangosteen peels, The α- and γ- mangostin amounts of PP-EUAE were 69.16 and 13.34 μg/g DE, respectively, which were significantly higher than those of PP-UMAE (46.06 and 8.68 μg/g DE, respectively).

3.5 Employ MPPS and MPP as fat replacer and natural antioxidant in developing healthy mayonnaise

3.5.1 *Development of reduced-fat mayonnaise by using MPPS as fat substitute*

3.5.1.1 *Proximate composition and caloric value of different mayonnaise formulations*

Table 3.15 Proximate composition (g/100g) and caloric value for control and mayonnaise with oil replacement by polysaccharides from mangosteen peels

Parameter	Control	MP25	MP35	MP45	MP55
Moisture (%)	19.23 ± 1.20^{e}	33.13 ± 1.73^{d}	41.76 ± 0.48^{c}	48.13 ± 1.66^{b}	57.49 ± 1.94^{a}
Lipid*	76.34 ± 2.67^{a}	65.70 ± 2.05^{b}	53.00 ± 2.39^{c}	35.45 ± 3.45^{d}	20.77 ± 0.51^{e}
Protein*	2.03 ± 0.17^{ab}	1.72 ± 0.09^{c}	1.87 ± 0.05^{bc}	1.98 ± 0.03^{ab}	2.04 ± 0.05^{a}
Ash*	0.67 ± 0.00^{c}	1.44 ± 0.19^{b}	1.67 ± 0.33^{ab}	1.78 ± 0.39^{ab}	2.17 ± 0.29^{a}
Carbohydrate*	20.97 ± 1.65^{e}	31.14 ± 2.14^{d}	43.47 ± 2.52^{c}	60.79 ± 3.74^{b}	75.18 ± 0.57^{a}
Caloric value (kcal/100 g)	629 ± 15^{a}	468 ± 13^{b}	383 ± 6^{c}	296 ± 6^{d}	205 ± 3^{e}

*Dry basis (%). MP25, MP35, MP45 and MP55: formulation with 25%, 35%, 45% and 55% substitution of oil by MPPS suspension, respectively. Different letters indicate significant differences in the same row ($p< 0.05$).

Table 3.15 illustrated the significant decrease ($p<0.05$) in lipid content of MPPS-incorporated with reductions of 13.9%, 30.6%, 53.6% and 72.8% in MP25, MP35, MP45 and MP55, respectively compared to the control. Consequently, caloric value were also significantly reduced ($p < 0.05$) by 25.6%, 39.1%, 52.9% and 67.4%, as lipid (9kcal/g) is the primary calorie contributor. Acorrding to Codex Alimentarius standards, a product with at least 25% reduction in total caloric value as compared to its original formulation. Thus, all MPPS – incorporated samples can be classified as light mayonnaise.

3.5.1.2 Appearance and micrograph and droplet size distribution of different mayonnaise formulations

Figure 3.11 illustrates the appearance and micrographs of all mayonnaise samples. MP25 and MP35 still kept the similar appearance with the control while

there was obvious difference in the outlook of MP45 and M55, indicating the large variation in their texture.

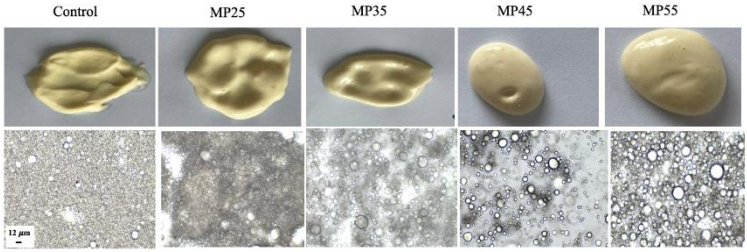


Figure 3.9 Photographs and Microscopic image of different mayonnaise formulations (x 40) scale bar = 12 µm.

The control, MP25 and MP35 displayed relatively uniform oil droplets while their size distribution was broader with various droplet sizes in MP45 and MP55

To evaluate the size distribution of oil droplets in all mayonnaise samples, the Sauter mean diameter, *i.e.* D [3,2], and span values were measured and are presented in Table 3.16. The D [3,2] value increased with higher levels of oil substitution, ranging from 2.81 µm in the control sample (full-fat mayonnaise) to 12.27 µm in MP55. Notably, there were no significant differences among the samples with 35%, 45%, and 55% oil substitution

Table 3. 2 Oil droplet size distribution and power-law model parameters of various mayonnaise formulations.

Samples	D [3,2] (µm)	Span	K (Pa.s^n)	n	R^2
Control	2.81 ± 0.06^c	0.922 ± 0.019^d	142.50 ± 3.25^a	0.167 ± 0.008^c	0.97
MP25	6.82 ± 0.25^b	1.214 ± 0.024^c	60.12 ±3.22^b	0.168 ± 0.005^c	0.97
MP35	11.98 ± 0.79^a	1.332 ± 0.057^c	28.41 ± 0.04^c	0.171 ± 0.001^c	0.99
MP45	10.58 ± 0.34^a	2.094 ± 0.089^b	9.69 ± 0.53^d	0.201 ± 0.012^b	> 0.99
MP55	12.27 ± 1.48^a	2.285 ± 0.002^a	1.83 ± 0.20^e	0.321 ± 0.014^a	> 0.99

MP25, MP35, MP45 and MP55: formulation with 25%, 35%, 45% and 55% substitution of oil by MPPS suspension, respectively.

Different letters indicate significant differences in the same column ($p < 0.05$).

3.5.1.3 Rheological behavior of different mayonnaise formulations

This study utilized steady flow and dynamic viscoelasticity to evaluate the rheology of mayonnaise, which are described in Figure 3.12 and Figure 3.13.

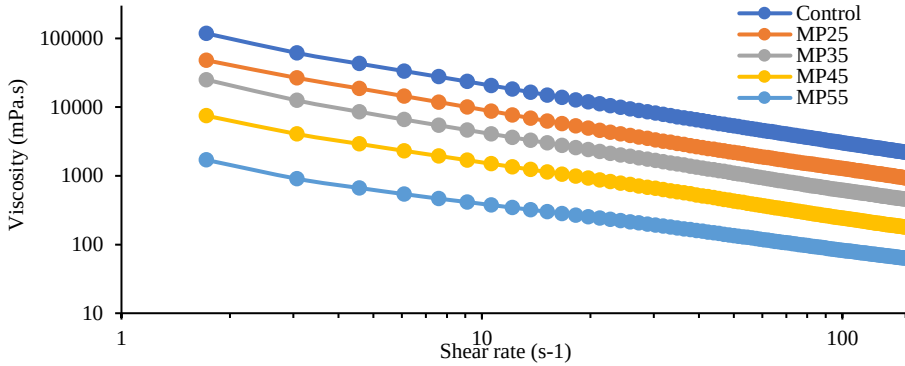


Figure 3. 1 Viscosity curves of control and mayonnaise formulated with mangosteen polysaccharides

Figure 3.12 further illustrates that the control had the highest viscosity at all test shear rates while the substitution of oil in formulation reduced the viscosity.

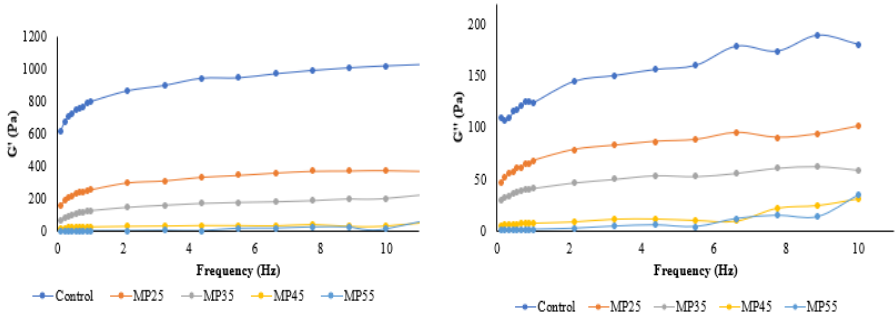


Figure 3. 2 Dynamic mechanical spectra of control mayonnaise and mayonnaise formulated with mangosteen polysaccharides.

The viscoelastic characteristics of mayonnaise samples were further examined using dynamic oscillatory testing, with the corresponding findings illustrated in

Figure 3.13. The control, MP25 and MP35 exhibited similar viscoelastic properties with the storage modulus (G') larger than the loss modulus (G'') in the frequency range of 0.1-10 Hz, indicating their solid-like or weak-gel structure.

3.5.1.5 Sensory characteristics of different mayonnaise formulations

Table 3.18 compares the sensory characteristics of different mayonnaise formulations in terms of appearance, aroma, texture, taste, after taste and overall acceptability

Table 3.18 Sensory score of various mayonnaise formulations

Attributes	**Control**	**MP25**	**MP35**	**MP45**	**MP55**
Appearance	7.83 ± 1.03^{a}	7.92 ± 1.21^{a}	7.37 ± 1.28^{b}	6.38 ± 1.38^{c}	6.45 ± 1.48^{c}
Aroma	7.07 ± 1.18^{ab}	7.40 ± 1.15^{a}	7.38 ± 1.17^{a}	6.92 ± 1.14^{b}	6.70 ± 1.28^{b}
Texture	7.38 ± 1.09^{a}	7.52 ± 1.05^{a}	7.52 ± 1.02^{a}	6.65 ± 1.30^{b}	6.10 ± 1.28^{c}
Taste	7.07 ± 1.25^{b}	7.62 ± 1.09^{a}	7.50 ± 1.27^{ab}	6.52 ± 1.40^{c}	6.20 ± 1.39^{c}
After Taste	7.13 ± 1.24^{a}	7.40 ± 1.37^{a}	7.32 ± 1.26^{a}	6.65 ± 1.13^{b}	6.43 ± 1.39^{b}
Overall Acceptability	7.28 ± 0.98^{a}	7.53 ± 0.89^{a}	7.45 ± 0.96^{a}	6.62 ± 0.98^{b}	6.37 ± 1.01^{b}

Different letters indicate significant differences in the same row ($p<0.05$)

Generally, there are no significant differences ($p > 0.05$) across all five attributes among the control, MP25 and MP35 samples, except the control with a lower taste score and MP35 with a lower appearance score.

3.5.1.6 Oxidative stability of different mayonnaise formulations

Lipid oxidation can lead to the formation of undesirable compounds as well as unpleasant flavors and odors, ultimately reducing the shelf life, safety and consumer acceptance. Table 3.19 presents the level of oxidation in mayonnaise over storage at refrigerated and ambient temperatures through peroxide value (PV).

Table 3.19 Peroxides value (meq/kg) of various mayonnaise formulations over storage at 4ºC and 28ºC

Samples		**Control**	**MP25**	**MP35**	**MP45**	**MP55**
Temperature	Days					
4ºC	1	0.60 ± 0.00^{aF}	0.43 ± 0.06^{bF}	0.41 ± 0.02^{bF}	0.27 ± 0.06^{cF}	0.20 ± 0.00^{cE}
	7	2.33 ± 0.11^{aE}	1.29 ± 0.10^{bE}	1.11 ± 0.10^{bcE}	0.93 ± 0.12^{cE}	0.73 ± 0.12^{dD}
	13	3.53 ± 0.12^{aD}	3.13 ± 0.12^{bD}	1.40 ± 0.00^{dD}	1.57 ± 0.05^{cD}	0.99 ± 0.02^{eC}
	19	6.60 ± 0.20^{aC}	4.43 ± 0.05^{bC}	3.53 ± 0.11^{cC}	2.43 ± 0.05^{dC}	1.27 ± 0.12^{eB}
	25	7.73 ± 0.46^{aB}	5.13 ± 0.12^{bB}	3.93 ± 0.12^{cB}	2.73 ± 0.12^{dB}	1.49 ± 0.10^{eA}
	31	12.47 ± 0.12^{aA}	5.34 ± 0.06^{bA}	4.60 ± 0.20^{cA}	3.76 ± 0.14^{dA}	1.59 ± 0.02^{eA}
28ºC	1	2.33 ± 0.12^{aF}	1.21 ± 0.02^{bF}	0.87 ± 0.12^{cE}	0.93 ± 0.23^{cF}	0.83 ± 0.05^{cD}
	7	11.53 ± 0.61^{aE}	4.53 ± 0.57^{bE}	3.81 ± 0.02^{cD}	2.13 ± 0.11^{dE}	1.93 ± 0.23^{dC}
	13	19.40 ± 0.40^{aD}	7.87 ± 0.12^{bD}	6.87 ± 0.23^{cC}	4.80 ± 0.35^{dD}	3.13 ± 0.61^{eB}
	19	21.67 ± 0.31^{aC}	9.73 ± 0.23^{bC}	7.47 ± 0.61^{cC}	6.33 ± 0.31^{dC}	3.87 ± 0.23^{eB}
	25	25.07 ± 0.92^{aB}	15.87 ± 0.83^{bB}	14.13 ± 1.40^{cB}	9.93 ± 0.31^{dB}	5.40 ± 0.20^{eA}
	31	40.27 ± 0.46^{aA}	19.87 ± 1.89^{bA}	16.40 ± 0.40^{cA}	12.20 ± 0.20^{dA}	5.60 ± 1.04^{eA}

MP25, MP35, MP45 and MP55: formulation with 25%, 35%, 45% and 55% substitution of oil by MPPS suspension, respectively. $^{a-e}$ Mean values in the same row at same temperature with different letters had significant difference ($p<0.05$). $^{A-E}$ Mean values in the same column at same temperature with different letters had significant difference ($p<0.05$).

The PVs of MP35 were only approximately half as compared to those of the control. A high amount of oil with polyunsaturated fatty acids in the control could provide more double bonds to induce auto-oxidation by free radicals and produce hydroperoxides, leading its high PVs. In addition, the polysaccharides extracted from mangosteen was demonstrated for its antioxidant activity [224], which could slow down the rate of lipid oxidation. Among mayonnaise formulations, MP25 and MP35 could maintain desirable characteristics of full-fat mayonnaise with similar consumer acceptability and better oxidative stability. Therefore, MP35 would be selected for further investigation in Section 3.5.2.

3.5.2 Usage of MPP as natural antioxidant in mayonnaise preservation

3.5.2.1 Oxidative stability of mayonnaise

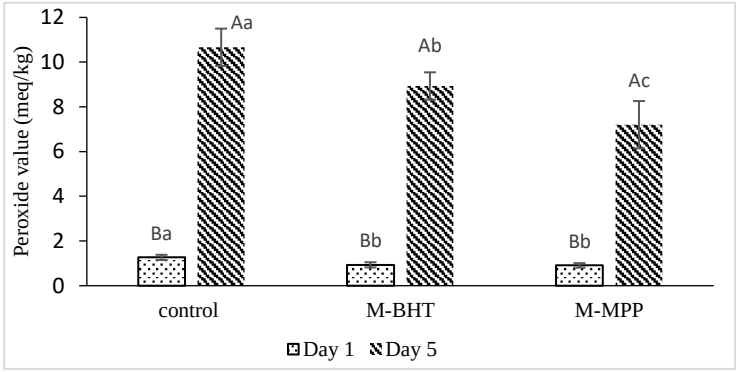


Figure 3. 3 PV of mayonnaise over 5 days of storage at 45°C. Different letters (a, b and c) indicate statistically significant differences ($p<0.05$) among samples on the same day. Different letters (A and B) indicate statistically significant differences ($p<0.05$) within the same sample across different days.

On day 1, the PVs of the mayonnaise with BHT or MPP were not significantly different ($p>0.05$). However, by day 5, M-MPP showed significantly lower PVs ($p<0.05$) than both the control and M-BHT. This difference implied the high antioxidant properties of MPP (as presented in Section 3.4), which effectively slowed down the rate of lipid oxidation

3.5.2.2 Emulsion stability of mayonnaise

Figure 3.15 illustrates the emulsion stability of mayonnaise. The results indicate that the emulsion stability of the control mayonnaise decreased significantly ($p<0.05$) over storage. However, those of M-BHT and M-MPP showed no significant changes ($p>0.05$) from Day 1 to Day 5. Furthermore, the mayonnaise formulation containing MPP exhibited significantly higher ($p<0.05$) emulsion stability than the control and BHT formulations on both Day 1 and Day 5. The better oxidative stability of M-MPP may stabilize its oil-in-water emulsion over storage.

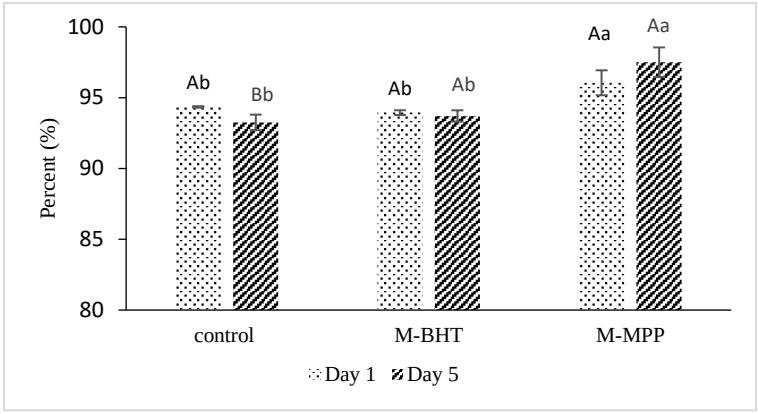


Figure 3. 4. Emulsion stability of mayonnaise over 5 days of storage at 45°C. Different lowercase letters (a, b) indicate statistically significant differences ($p<0.05$) among samples on the same day. Different uppercase letters (A, B) indicate statistically significant differences ($p<0.05$) within the same sample across different days.

CHAPTER 4 CONCLUSION AND RECOMMENDATION

4.1 Conclusion

For the contribution of this study to science, it can be concluded that:

- Polysaccharides and phenolic compounds were co-extracted from mangosteen fruit peels using two integrated methods, i.e. UMAE and EUAE.
- The characteristics of polysaccharides and phenolic compounds simultaneously extracted from mangosteen peels by using UMAE and EUAE were investigated and compared.

For the contribution of this study to practice, it can be concluded that:

- This study provided optimal conditions of UMAE and EUAE to obtain the high yield of polysaccharides and phenolics from mangosteen peels, a byproduct of popular fruit in Vietnam.

- The study also provided comprehensive characterization for the obtained extracts.

- This study also demonstrated the potential of UMAE polysaccharides and phenolics in developing healthier mayonnaise.

4.2 Recommendation

Several research topics can be proposed to enhance the yield and quality of polysaccharide and phenolic compounds, as well as to expand application opportunities:

- Explore the stability of MPPS and MPP derived from UMAE and EUAE over time or under different storage conditions.

- Further investigate other unstudied factors related to the extraction co optimization of polysaccharides and phenolic compounds.

- Conduct comprehensive characterization of the obtained MPP, including its antimicrobial, anti-inflammatory, and anti-cancer properties to explore new applications.

- Compare the properties of polysaccharides and phenolics from individual and simultaneous extraction.

- Apply MPPS and MPP to other foods, such as cream, yogurt and more, especially in products requiring prebiotics to demonstrate the application potential of MPPS from EUAE.