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RESEARCH ON THE ISOLATION AND MODIFICATION OF STARCHES FROM UNCONVENTIONAL SOURCES AND APPLICATION ORIENTATIONS

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INTRODUCTION

Starch is an essential carbohydrate source, playing a dominant role in providing energy for bodily metabolic activities. The hydrolysis of starch by digestive enzymes generates glucose; however, a specific starch fraction known as resistant starch (RS) remains resilient against enzymatic breakdown in the small intestine. Instead of being absorbed into the bloodstream, RS translocates to the colon, where it undergoes fermentation by the gut microbiota, mimicking the behavior of soluble dietary fiber. Consuming RS offers various biological benefits, including stabilizing blood glucose levels, improving digestive health, and supporting the prevention of metabolic disorders.

With projections indicating that over 643 million adults globally will be living with diabetes by 2030, the demand for nutritional interventions in treatment management is rising sharply. This trend drives the development of health-protective food products with a low glycemic index (GI). Within this context, resistant starch (RS) has emerged as a crucial functional ingredient due to its distinct bio-functional mechanisms, playing a key role in improving overall health and preventing metabolic complications.

Since the natural content of RS is typically low and thermally unstable, the application of modification techniques has become a pivotal strategy to optimize RS yield and overcome the inherent drawbacks of native starch. Physical approaches (such as heat-moisture treatment - HMT, and annealing - ANN), chemical methods (etherification, esterification, cross-linking), and enzymatic modifications are being widely implemented. The core objective of these processes is to restructure the starch molecules, creating physical or chemical barriers that enhance resistance against digestion by the gastrointestinal enzyme system.

In the context of global food security, current research is shifting focus toward alternative starch sources to reduce reliance on conventional commodities like corn and cassava. Vietnam, benefiting from a tropical climate, possesses abundant botanical resources with high potential for resistant starch, notably green bananas and jackfruit seeds. Exploiting these underutilized starch sources not only optimizes the economic value of agricultural produce but also represents a sustainable supply solution for RS,

aligning with domestic production trends.

Driven by societal needs and the demand for next-generation functional foods, the dissertation topic "Research on the isolation and modification of starches from unconventional sources and application orientations", was selected for investigation. The central objective of this doctoral thesis is to study modification methods applied to green banana and jackfruit seed starches to optimize their resistant starch (RS) content. Concurrently, the research focuses on elucidating the structural transformation mechanisms, physicochemical properties, and digestion kinetics of the modified starches. Based on these findings, the thesis evaluates the applicability of the developed starches in noodle manufacturing, aiming to enhance nutritional value and control the glycemic index (GI) for consumers.

To achieve these objectives, the primary research contents of the dissertation include: (1) Isolation and recovery of native starches from green bananas and jackfruit seeds; (2) Modification of the isolated starches to increase resistant starch content using methods such as phosphorylation, hydrothermal treatments (HMT and ANN), and carboxymethylation; (3) Application of the modified starches in noodle processing.

Chapter 1. OVERVIEW OF THE STUDY

1.1. Overview of Starch

Starch is the primary storage polysaccharide in plants, consisting mainly of amylose (20–30%) and amylopectin (70–80%), which are organized into semi-crystalline structures classified as A-, B-, C-, and V-type polymorphs. The proportion and arrangement of these components determine the physicochemical properties, functional characteristics, and digestibility of starch. Based on the rate of enzymatic hydrolysis, starch is categorized into rapidly digestible starch (RDS), slowly digestible starch (SDS), and resistant starch (RS).

1.2. Overview of Resistant Starch (RS)

Resistant starch (RS) is the fraction of starch that resists digestion in the small intestine and undergoes fermentation in the colon, producing beneficial short-chain fatty acids (SCFAs), particularly butyrate. RS is classified into five types (RS1–RS5), among which RS3 (retrograded starch) and RS4 (chemically

modified starch) are of particular interest due to their high thermal and structural stability. Various physical, enzymatic, and chemical modification methods have been employed to increase RS content. Owing to its health benefits, RS has been widely utilized in functional foods and low-glycemic-index products.

1.3. Unconventional starch sources

Alternative starch sources, including green bananas, jackfruit seeds, canna, and legumes, have attracted increasing attention because of their high starch content and potential for RS production. Green banana starch naturally contains high levels of RS and exhibits a B-type crystalline structure, whereas jackfruit seed starch is rich in amylose, making it suitable for RS enhancement. These materials are abundant, inexpensive, and well adapted to Vietnamese agricultural conditions; however, their industrial utilization remains limited.

1.4. Current research on native and modified starches in Vietnam

Research on native and modified starches in Vietnam has expanded considerably since 2005, evolving from a primary focus on cassava starch to include unconventional sources such as green bananas, jackfruit seeds, and legumes. Previous studies have reported RS contents exceeding 40–50% depending on the starch source and modification method. Modified starches have demonstrated potential applications in food, pharmaceutical, and biomaterial industries. Nevertheless, research efforts remain fragmented, highlighting the need for more systematic studies aimed at maximizing the utilization and commercial value of alternative starch resources.

Chapter 2. SUBJECTS, RESEARCH METHODS, AND EXPERIMENTS

2.1. Research subjects

Green bananas (*Musa acuminata*, AAA) harvested in Phu Tho, 80-90 days after flowering. Fresh jackfruit seeds of the Thai jackfruit variety (*Artocarpus heterophyllus* Lam.) harvested in Dong Nai.

2.2. Research methods

2.2.1. Starch isolation and recovery

Green banana starch, jackfruit seed starch, and green banana flour were isolated according to the methods described by Zhang et al. (2005), Thuy et al. (2020), and Agama-Acevedo et al. (2012).

2.2.2. Modification for resistant starch production

Cross-linking modification with STMP/STPP phosphate salts was

performed according to the method of Woo and Seib (2002). Heat-moisture treatment (HMT), annealing (ANN), and carboxymethylation modifications were conducted following the procedures described by Chung et al. (2012), Liu et al. (2016), and Nisit et al. (2013), respectively.

2.2.3. Preparation of noodles containing resistant starch

Wheat flour was blended with resistant starch, extruded into noodle strands using a noodle making machine, and dried at 35°C until the moisture content reached below 10%, followed by packaging and storag.

2.3. Analytical and evaluation methods

2.3.1. Starch isolation and recovery yield

The yield was calculated based on the weight ratio of the recovered starch to the initial raw material.

2.3.2. Determination of basic starch composition

Carbohydrate content was determined using the Ewers method (TCVN 9935:2013). Ash, moisture, protein, lipid, crude fiber contents were analyzed according to TCVN 9939:2013, TCVN 9934:2013, TCVN 8125:2015 (Kjeldahl), TCVN 9938:2013 (Soxhlet), TCVN 5103:1990 (modified Schaven), respectively. Amylose and amylopectin contents were measured by UV-Vis spectrophotometry (JASCO V-670) at 550 nm and 618 nm, respectively.

2.3.3. Digestibility evaluation

The contents of resistant starch (RS), rapidly digestible starch (RDS), and slowly digestible starch (SDS) were determined using the Megazyme Resistant Starch Assay Kit (AOAC Method 2002.02).

2.3.4. In-vitro estimated glycemic index (eGI)

An *in-vitro* enzymatic digestion model was used to simulate starch hydrolysis, from which the hydrolysis index (HI) and estimated glycemic index (eGI) were calculated.

2.3.5. Degree of dubstitution of modified starch

For phosphate starch, the degree of substitution was calculated based on phosphorus content (TCVN 9941:2013). For carboxymethyl starch, the degree of substitution was determined by NaOH titration.

2.3.6. Functional properties of starch

Swelling power (SP) and water solubility index (WSI) were determined by dispersing 1 g of sample in water, heating at 60-90°C,

followed by cooling and centrifugation. The supernatant was dried to measure WSI, while the sediment was used to calculate SP.

Water absorption capacity (WAC) and oil absorption capacity (OAC) were evaluated by mixing the sample with water or oil, equilibrating, centrifuging, and measuring the retained mass.

Freeze-thaw stability was assessed using a 6% (w/v) starch suspension subjected to repeated freezing (-20 °C) and thawing cycles, with syneresis measured after each cycle.

2.3.7. Physicochemical characterization of starch

Fourier-transform infrared spectroscopy (FTIR, NEXUS 670) was used to analyze starch functional groups in the range of 400-4000 cm^{-1}. X-ray diffraction (XRD, Shimadzu XRD-7000) was applied to determine crystalline structure and relative crystallinity (2θ = 10-50°). Surface morphology was examined using scanning electron microscopy (SEM, JEOL JSM-6510LV).

2.3.8. Cooking properties of noodles

Optimal cooking time (OCT) was determined by boiling noodles until the opaque core disappeared, assessed by pressing strands between two glass plates. Water absorption (DMWA) was calculated from the weight difference before and after cooking at optimal time, followed by cooling and surface drying. Cooking loss (DML) was measured by evaporating the cooking water to constant weight to quantify solid loss. Noodle breakage rate was determined by counting broken strands relative to the total number after cooking

2.4. Statistical analysis

Experiments were performed in triplicate, and the results are expressed as mean ± standard error. Experimental data were processed using Microsoft Excel and subjected to statistical analysis via one-way Analysis of Variance (ANOVA).

Chapter 3. RESULTS AND DISCUSSION

3.1. Starch isolation and recovery from uncommon sources

*3.1.2. Effect of steeping temperatur*e

A steeping temperature of 6°C resulted in the highest starch isolation and recovery yields, as well as the maximum carbohydrate content, for both green bananas (14,73% and 85,31%, respectively) and jackfruit seeds (25,21% and 80,47%, respectively). Both the yield and carbohydrate content

decreased as the temperature rose to 25-30°C; therefore, 6°C was determined to be the optimum steeping temperature for starch recovery from these two raw materials.

Table 3.1. Effect of steeping temperature on the isolation and recovery process of starches from green bananas and jackfruit seeds

Sources	*Temperature (ºC)*	*Yields (%)*	*Carbohydrate (%)*	*Protein (%)*
Green bananas	6	14,73 ± 0,95	85,31 ± 2,58	0,15 ± 0,004
	25	12,89 ± 0,63	71,25 ± 2,15	0,11 ± 0,004
	30	9,57 ± 0,13	62,71 ± 2,21	0,08 ± 0,003
Jackfruit seeds	6	25,21 ± 1,52	80,47 ± 1,82	0,58 ± 0,08
	25	16,32 ± 0,31	65,17 ± 2,02	0,55 ± 0,07
	30	10,87 ± 0,17	56,74 ± 2,11	0,51 ± 0,08

3.1.2. Effect of steeping time

Table 3.2. Effect of steeping time on the isolation and recovery process of starches from green bananas and jackfruit seeds

Sources	*Time (hours)*	*Yields (%)*	*Carbohydrate (%)*	*Protein (%)*
Green bananas	8	11,26 ± 0,46	76,83 ± 2,31	0,23 ± 0,005
	12	14,73 ± 0,95	85,31 ± 2,58	0,15 ± 0,004
	16	14,21 ± 0,26	80,57 ± 2,63	0,16 ± 0,005
	24	12,93 ± 0,32	73,35 ± 2,26	0,19 ± 0,006
Jackfruit seeds	8	25,21 ± 1,52	80,47 ± 1,82	0,58 ± 0,08
	12	26,87 ± 1,24	82,07 ± 1,73	0,55 ± 0,07
	16	21,55 ± 1,03	76,31 ± 1,95	0,59 ± 0,08
	24	18,14 ± 0,77	70,14 ± 1,84	0,62 ± 0,06

A steeping time of 12 hours resulted in the highest starch isolation and recovery yields, as well as the maximum carbohydrate content, for both green bananas (14.73% and 85.31%, respectively) and jackfruit seeds (26.87% and 82.07%, respectively). Extending the steeping duration beyond 12 hours led to a reduction in both the recovery yield and carbohydrate content; therefore, 12 hours was determined to be the optimum steeping time for the starch isolation process from these two raw materials.

3.1.3. Effect of steeping agents

The addition of $NaHSO_3$ or NaOH during the steeping process enhanced the starch recovery yield and carbohydrate content, while simultaneously reducing the protein content compared to the water-steeped control. $NaHSO_3$ was selected as the optimum chemical agent for starch recovery from green bananas and jackfruit seeds due to its high efficiency and suitability for food applications.

Table 3.3. Effect of steeping agents on the isolation and recovery process of starches from green bananas and jackfruit seeds

Sources	*Agents*	*Yields (%)*	*Carbohydrate (%)*	*Protein (%)*
Green bananas	H_2O	11,26 ± 0,46	76,83 ± 2,31	0,23 ± 0,005
	$NaHSO_3$ 0,1%	12,82 ± 0,73	80,35 ± 2,53	0,18 ± 0,006
	$NaHSO_3$ 0,25%	14,73 ± 0,95	85,31 ± 2,58	0,15 ± 0,004
	$NaHSO_3$ 0,5%	14,28 ± 0,72	89,41 ± 2,33	0,09 ± 0,004
	NaOH 0,25%	14,85 ± 0,77	88,27 ± 2,35	0,09 ± 0,004
Jackfruit seeds	H_2O	14,13 ± 1,27	70,42 ± 2,03	0,72 ± 0,07
	$NaHSO_3$ 0,1%	18,51 ± 1,61	74,14 ± 1,84	0,69 ± 0,06
	$NaHSO_3$ 0,25%	21,94 ± 1,35	77,02 ± 1,98	0,62 ± 0,07
	$NaHSO_3$ 0,5%	25,21 ± 1,52	80,47 ± 1,82	0,58 ± 0,08
	NaOH 0,25%	27,02 ± 1,21	80,59 ± 2,05	0,60 ± 0,05

3.1.4. Chemical composition of starches from unconventional source

Table 3.4. Proximate composition of raw materials and native starches

No.	Parameter	Jackfruit seeds	Green bananas	Jackfruit seed starch	Banana starch
1	Moisture (%)	50,61 ± 2,73	66,64 ± 2,35	6,25 ± 0,24	6,57 ± 0,21
2	Ash (%)	1,53 ± 0,02	1,01 ± 0,02	1,52 ± 0,25	1,16 ± 0,005
3	Crude fiber (%)	2,25 ± 0,11	2,55 ± 0,10	0,71 ± 0,21	0,31 ± 0,009
4	Protein (%)	5,67 ± 0,14	2,24 ± 0,09	0,58 ± 0,08	0,15± 0,004
5	Lipid (%)	1,36 ± 0,03	1,18 ± 0,04	0,19 ± 0,03	0,11 ± 0,03
6	Carbohydrate (%)	35,14 ± 2,84	23,28 ± 2,71	80,47 ± 1,82	85,31 ± 2,58
7	Other components (%)	~ 3,44	~ 3,10	-	-
7	Amylose (%)	-	-	23,65 ± 0,18	11,27 ± 0,40
8	Amylopectin (%)	-	-	76,35 ± 0,18	88,73 ± 0,40

Jackfruit seed starch and green banana starch were recovered with yields of 25,21% and 14,73%, respectively. Both starches exhibited high carbohydrate contents (80,47-85,31%) and low impurity levels (less than 1%). Jackfruit seed starch contained 23,65% amylose, whereas green banana starch comprised 88,73% amylopectin. Both starch types possessed a resistant starch (RS) content of approximately 25%, demonstrating strong potential for applications in health-promoting food products.

3.1.5. Structural characteristics of starch

SEM images (x500, x1500) show that jackfruit seed starch is spherical or semi-oval in shape, with a smooth surface and separated granules; while green banana starch is elongated oval or rod-shaped, with an intact structure. The granules of banana starch are larger than those of jackfruit seeds. XRD patterns show that green banana starch has a B-type crystalline structure (2θ ≈ 15°, 17°, 22-24°), while jackfruit seed starch has an A-type structure (2θ ≈

15°, 17-18°, 23°). The relative crystallinity is 25,38% (banana) and 28,77% (jackfruit seed), reflecting differences in amylose/amylopectin structure, respectively. The FTIR spectra of green banana starch and jackfruit seeds both showed characteristic polysaccharide peaks, including the valence vibrations of the O-H group (3600-3000 cm^{-1}), C-H bond (2930-2880 cm^{-1}), C-O and C-C bonds (1200-800 cm^{-1}), C-O-H group, CH_2 (1350-1342 cm^{-1}) and C-H bond in the glucose unit (998-702 cm^{-1}).

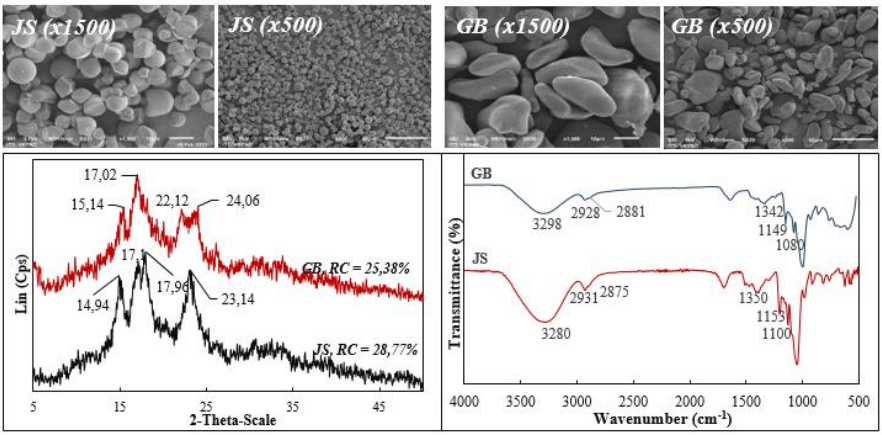

Figure 3.1. SEM images, X-ray diffraction patterns, and FTIR spectra of jackfruit seed starch and green banana starch

3.1.6. Functional properties of starch

Table 3.5. Functional properties of green banana and jackfruit seed starches

Functional properties		*Unit*	*Green bananas starches*	*Jackfruit seeds starches*
SP	60ºC	g/g	2,88 ± 0,23	2,14 ± 0,23
	70ºC		7,62 ± 0,25	6,21 ± 0,15
	80ºC		16,37 ± 0,31	15,11 ± 0,34
	90ºC		24,30 ± 0,31	23,42 ± 0,67
WSI	60ºC	%	4,28 ± 0,26	2,14 ± 0,11
	70ºC		8,69 ± 0,25	8,35 ± 0,17
	80ºC		13,54 ± 0,26	12,86 ± 0,37
	90ºC		20,59 ± 0,22	20,04 ± 0,82
OAC		g/g	1,19 ± 0,013	1,07 ± 0,011
WAC		g/g	1,38 ± 0,012	0,91 ± 0,010
Syneresis	1 cycle	%	16,25 ± 0,11	19,64 ± 0,14
	2 cycle		33,51 ± 0,15	38,36 ± 0,13
	3 cycle		43,34 ± 0,24	48,95 ± 0,28
	4 cycle		51,53 ± 0,14	54,18 ± 0,35

The swelling power (SP) and water solubility index (WSI) of both starches increased progressively with temperature from 60 to 90°C, reaching their maximum values at 24,30 g/g and 20,59% for green banana starch (GB), and 23,42 g/g and 20,04% for jackfruit seed starch (JS), respectively. Green banana starch exhibited higher water and oil absorption capacities (WAC of 1,38 g/g; OAC of 1,19 g/g) compared to those of jackfruit seed starch (WAC of 0,91 g/g; OAC of 1,07 g/g). After four freeze-thaw cycles, the syneresis rate of GB was lower than that of JS (51,53% versus 54,18%), demonstrating superior water retention and better freeze-thaw stability.

Conclusion of Section 3.1

The optimum steeping conditions for starch recovery from green bananas were determined to be 6°C for 12 hours with 0.25% $NaHSO_3$ 0,25%, whereas those for jackfruit seeds were 6°C for 8 hours with 0.5% $NaHSO_3$. Under these conditions, the isolation yields reached 14,73% for green banana starch (GB) and 25,21% for jackfruit seed starch (JS), with corresponding resistant starch (RS) contents of 24,76% and 25,25%. Structurally, GB exhibited a B-type crystalline polymorph, while JS displayed an A-type crystalline pattern; both starches maintained intact granule morphologies. Furthermore, green banana starch demonstrated superior swelling power, water solubility, water absorption, oil absorption, and structural stability compared to jackfruit seed starch.

3.2. Phosphate modification of starch

3.2.1. Effect of polyphosphate salt content

Table 3.6. Effect of polyphosphate salt content on DS of phosphate starch

$m_{polyphosphate}$ (%)	Green banana starch		Jackfruit seed starch	
	P (%)	*DS*	*P (%)*	*DS*
Control	0,0067 ± 0,0041	0,0004 ± 0,0002	0,0103 ± 0,0019	0,0005 ± 0,0001
6	0,1820 ± 0,0024	0,0096 ± 0,0001	0,1597 ± 0,0070	0,0084 ± 0,0004
8	0,2520 ± 0,0054	0,0133 ± 0,0003	0,2183 ± 0,0033	0,0115 ± 0,0002
10	0,3760 ± 0,0036	0,0199 ± 0,0002	0,3260 ± 0,0057	0,0172 ± 0,0003
12	0,4513 ± 0,0039	0,0239 ± 0,0002	0,4097 ± 0,0063	0,0217 ± 0,0003

Increasing the polyphosphate concentration from 6% to 12% increased the bound phosphorus content and degree of substitution (DS), indicating a higher degree of starch phosphorylation. As a result, RS and SDS contents increased, while RDS decreased due to enhanced resistance to enzymatic

hydrolysis. The highest RS contents were achieved at 10% polyphosphate (58.29% for banana starch and 40.27% for jackfruit seed starch). At 12%, RS decreased slightly, likely due to structural disruption and excessive phosphorus content; therefore, 10% polyphosphate was considered the optimal concentration.

Table 3.7. Effect of polyphosphate salt content on the digestibility of banana starch and jackfruit seeds

$m_{polyphosphate}$ (%)	Green banana starch			Jackfruit seed starch		
	RS (%)	*RDS (%)*	*SDS (%)*	*RS (%)*	*RDS (%)*	*SDS (%)*
Control	24,76±0,83	65,05±0,92	10,19±0,56	25,25±1,11	19,71±0,53	55,04±1,92
6	38,70±0,54	51,03±1,55	23,27±1,05	30,45±0,97	13,32±0,25	56,23±1,21
8	40,24±1,05	29,38±0,88	30,38±0,74	36,55±0,45	6,22±0,32	57,23±1,33
10	58,29±0,90	15,78±1,13	25,93±1,06	40,27±0,79	1,20±0,14	58,53±1,27
12	60,26±0,84	11,66±1,15	28,09±1,17	40,34±0,85	1,05±0,19	58,61±1,46

3.2.2. Effect of Na_2SO_4 content

Table 3.8. Effect of Na_2SO_4 content on DS of starch phosphate

m_{Na2SO4} (%)	Green banana starch		Jackfruit seed starch	
	P liên kết (%)	*DS*	*P liên kết (%)*	*DS*
Control	0,0067 ± 0,0041	0,0004 ± 0,0002	0,0103 ± 0,0019	0,0005 ± 0,0001
5	0,2248 ± 0,0012	0,0118 ± 0,0001	0,2160 ± 0,0022	0,0114 ± 0,0001
10	0,3760 ± 0,0036	0,0199 ± 0,0002	0,3260 ± 0,0057	0,0172 ± 0,0003
20	0,3966 ± 0,0057	0,0210 ± 0,0003	0,3594 ± 0,0073	0,0190 ± 0,0004

Table 3.9. Effect of Na_2SO_4 content on the digestibility of banana starch and jackfruit seeds

m_{Na2SO4} (%)	*Green banana starch*			*Jackfruit seed starch*		
	RS (%)	*RDS (%)*	*SDS (%)*	*RS (%)*	*RDS (%)*	*SDS (%)*
Control	24,76 ± 0,83	65,05 ± 0,92	10,19 ± 0,56	25,25 ± 1,11	19,71 ± 0,53	55,04 ± 1,92
5	33,87 ± 1,10	47,67 ± 1,03	18,46 ± 1,30	33,67 ± 0,66	10,29 ± 0,47	56,04 ± 0,97
10	58,29 ± 0,90	15,78 ± 1,13	25,93 ± 1,06	40,27 ± 0,79	1,20 ± 0,14	58,53 ± 1,27
20	59,28 ± 1,04	11,14 ± 0,97	29,58 ± 0,67	41,04 ± 0,57	0,04 ± 0,06	58,92 ± 0,51

Na_2SO_4 acted as an electrolyte that enhanced phosphorylation efficiency. Increasing the Na_2SO_4 concentration from 5% to 10% increased the bound phosphorus content, degree of substitution (DS), RS, and SDS, while reducing RDS, indicating improved resistance to enzymatic hydrolysis. As no significant improvements were observed beyond 10% due to reaction-site saturation, 10% Na_2SO_4 was selected as the optimal concentration.

3.2.3. Effect of reaction temperature

Increasing the reaction temperature from 60°C to 70°C significantly enhanced phosphorylation efficiency, as evidenced by higher bound

phosphorus content and degree of substitution (DS). This resulted in increased RS and SDS contents and decreased RDS, with RS reaching 45.62% for banana starch and 40.27% for jackfruit seed starch. At 80°C, RS decreased slightly and RDS increased due to partial disruption of the starch structure. Therefore, 70°C was identified as the optimal reaction temperature.

Table 3.10. Effect of reaction temperature on DS of starch phosphate

(°C)	Green banana starch		Jackfruit seed starch	
	P (%)	*DS*	*P (%)*	*DS*
Control	0,0067 ± 0,0041	0,0004 ± 0,0002	0,0103 ± 0,0019	0,0005 ± 0,0001
60	0,1627 ± 0,0033	0,0085 ± 0,0002	0,1377 ± 0,0037	0,0072 ± 0,0002
70	0,3760 ± 0,0036	0,0199 ± 0,0002	0,3260 ± 0,0057	0,0172 ± 0,0003
80	0,3420 ± 0,0054	0,0181 ± 0,0003	0,3083 ± 0,0053	0,0163 ± 0,0003

Table 3.11. Effect of reaction temperature on the digestibility of banana starch and jackfruit seeds

(°C)	Green banana starch			Jackfruit seed starch		
	RS (%)	*RDS (%)*	*SDS (%)*	*RS (%)*	*RDS (%)*	*SDS (%)*
Control	24,76±0,83	65,05±0,92	10,19±0,56	25,25±1,11	19,71±0,53	55,04±1,92
60	39,61±0,56	40,36±0,32	20,03±0,81	35,61±0,32	7,33±0,24	57,06±0,27
70	58,29±0,90	15,78±1,13	25,93±1,06	40,27±0,79	1,20±0,14	58,53±1,27
80	49,08±0,38	19,94±0,22	30,98±0,51	37,75±0,38	5,99±0,10	56,26±0,48

3.2.4. Physicochemical characteristics of starch phosphate

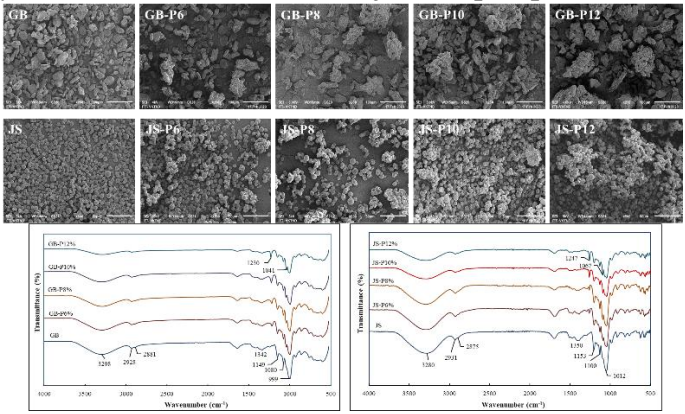

Figure 3.2. SEM images and FTIR spectra of green banana starch and jackfruit seed starch before and after phosphate treatment

SEM images revealed that phosphorylation induced surface roughness, slight deformation of starch granules, and increased intergranular

aggregation. FTIR analysis showed characteristic bands at 1230-1247 cm^{-1} and 1041-1067 cm^{-1}, corresponding to P=O and P–O–C vibrations, confirming the incorporation of phosphate groups into the starch structure. Phosphorylation also altered the crystalline pattern from B- to C-type in green banana starch and from A- to C-type in jackfruit seed starch, accompanied by an increase in crystallinity (from 25,34% to 39,16% and from 28,77% to 35,51%, respectively), indicating enhanced structural order and stability.

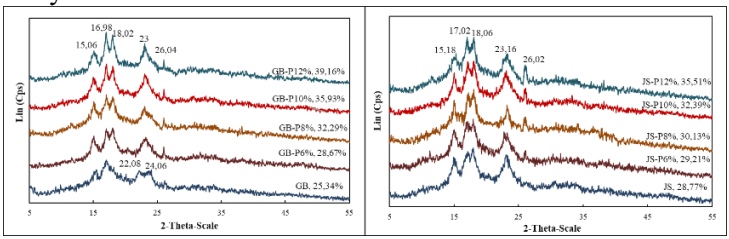

Figure 3.3. X-ray diffraction patterns and crystallinity of green banana starch and jackfruit seed starch before and after phosphate treatment

3.2.5. Functional properties of starch phosphate

Table 3.12. Swelling power (SP) and water solubility index (WSI) of starch phosphate

Starch	*ºC*	*SP/WSI*	*Control*	*P6%*	*P8%*	*P10%*	*P12%*
GB	**60**	SP (g/g)	2,88 ± 0,23	2,35 ± 0,23	2,12 ± 0,19	1,66 ± 0,17	1,47 ± 0,12
		WIS (%)	4,28 ± 0,26	3,89 ± 0,19	3,47 ± 0,14	3,01 ± 0,16	2,86 ± 0,11
	70	SP (g/g)	7,62 ± 0,25	5,41 ± 0,11	5,08 ± 0,14	4,75 ± 0,12	4,59 ± 0,13
		WIS (%)	8,69 ± 0,25	8,12 ± 0,17	6,87 ± 0,14	5,51 ± 0,13	5,23 ± 0,16
	80	SP (g/g)	16,37 ± 0,31	13,56 ± 0,26	12,03 ± 0,33	9,06 ± 0,10	8,41 ± 0,13
		WIS (%)	13,54 ± 0,26	11,35 ± 0,35	10,12 ± 0,37	7,86 ± 0,41	7,08 ± 0,24
	90	SP (g/g)	24,30 ± 0,31	20,41 ± 0,43	17,03 ± 0,32	14,36 ± 0,11	14,10 ± 0,11
		WIS (%)	20,59 ± 0,18	17,48 ± 0,47	16,16 ± 0,58	14,12 ± 0,47	13,78 ± 0,24
JS	**60**	SP (g/g)	2,14 ± 0,23	2,05 ± 0,20	1,84 ± 0,18	1,67 ± 0,21	1,44 ± 0,17
		WIS (%)	2,14 ± 0,11	1,89 ± 0,10	1,58 ± 0,11	1,23 ± 0,13	1,07 ± 0,08
	70	SP (g/g)	6,21 ± 0,15	5,89 ± 0,19	5,56 ± 0,16	5,25 ± 0,17	4,58 ± 0,18
		WIS (%)	8,35 ± 0,17	7,58 ± 0,18	7,03 ± 0,14	5,53 ± 0,16	5,05 ± 0,15
	80	SP (g/g)	15,11 ± 0,34	14,26 ± 0,23	12,03 ± 0,22	10,15 ± 0,19	9,06 ± 0,13
		WIS (%)	16,86 ± 0,37	13,47 ± 0,28	10,81 ± 0,29	9,74 ± 0,18	9,09 ± 0,11
	90	SP (g/g)	23,42 ± 0,67	21,27 ± 0,31	18,75 ± 0,24	15,34 ± 0,19	13,52 ± 0,22
		WIS (%)	20,04 ± 0,82	19,83 ± 0,68	18,28 ± 0,44	16,01 ± 0,35	13,86 ± 0,28
Phosphated starch with STMP/STPP phosphate salt content of 6, 8, 10, and 12% corresponds to P6%, P8%, P10%, and P12%, respectively.							

Phosphorylation decreased the swelling power (SP) and water solubility index (WSI) of starch due to the formation of cross-links between

polymer chains, while water absorption capacity (WAC) and oil absorption capacity (OAC) increased with the degree of phosphorylation. At the optimal phosphate level, WAC and OAC increased from 1.38 to 1.94 g/g and from 1.19 to 1.87 g/g, respectively, for green banana starch, and from 0.91 to 1.48 g/g and from 1.07 to 1.55 g/g, respectively, for jackfruit seed starch. Phosphorylation also significantly improved freeze-thaw stability, reducing water loss after four cycles from 51.53% to 35.02% for green banana starch and from 54.18% to 38.02% for jackfruit seed starch. These results indicate enhanced water retention, improved gel stability, and reduced starch recrystallization.

Table 3.13. Water and oil absorption capacity of starch phosphate

Starch	*OAC/WAC*	*Control*	*P6%*	*P8%*	*P10%*	*P12%*
GB	OAC (g/g)	1,19	1,23	1,55	1,82	1,87
	WAC (g/g)	1,38	1,45	1,59	1,88	1,94
JS	OAC (g/g)	1,07	1,15	1,32	1,51	1,55
	WAC (g/g)	0,91	1,06	1,21	1,42	1,48
Phosphated starch with STMP/STPP phosphate salt content of 6, 8, 10, and 12% corresponds to P6%, P8%, P10%, and P12%, respectively.						

Table 3.14. Freeze-thaw stability of starch phosphate

Starch	*Cycles*	*Syneresis (%)*				
		Control	*P6%*	*P8%*	*P10%*	*P12%*
GB	1	16,25 ± 0,11	14,24 ± 0,13	12,05 ± 0,14	10,05 ± 0,14	9,56 ± 0,18
	2	33,51 ± 0,15	27,37 ± 0,15	22,26 ± 0,16	17,51 ± 0,17	16,37 ± 0,14
	3	43,34 ± 0,24	35,41 ± 0,19	31,74 ± 0,21	25,84 ± 0,19	28,73 ± 0,19
	4	51,53 ± 0,14	44,63 ± 0,21	40,85 ± 0,19	32,24 ± 0,21	35,02 ± 0,20
JS	1	19,64 ± 0,14	14,58 ± 0,18	12,25 ± 0,17	10,17 ± 0,16	11,36 ± 0,14
	2	38,36 ± 0,13	30,25 ± 0,19	25,14 ± 0,18	21,55 ± 0,16	19,47 ± 0,15
	3	48,95 ± 0,28	37,61 ± 0,18	32,75 ± 0,19	29,34 ± 0,23	27,14 ± 0,13
	4	54,18 ± 0,35	45,69 ± 0,24	40,12 ± 0,21	36,27 ± 0,25	38,02 ± 0,17
Phosphated starch with STMP/STPP phosphate salt content of 6, 8, 10, and 12% corresponds to P6%, P8%, P10%, and P12%, respectively.						

3.2.6. Phosphating of green banana flour

Table 3.15. DS and digestibility of green banana starch (GB) and green banana flour (BF) treated by phosphorylation.

Sample	**P (%)**	**DS (x10^{-3})**	**RS (%)**	**RDS (%)**	**SDS (%)**
GB	0,0012 ± 0,0001	0,061 ± 0,0065	24,76 ± 0,83	65,05 ± 0,92	10,19 ± 0,56
BF	0,0017 ± 0,0002	0,087 ± 0,0089	27,42 ± 0,95	45,12 ± 1,11	27,46 ± 0,87
GB-PP	0,264 ± 0,0139	13,92 ± 0,5874	40,24 ± 1,05	29,38 ± 0,88	30,38 ± 0,74
BF-PP	0,298 ± 0,0158	15,75 ± 0,5441	49,63 ± 1,13	20,29 ± 1,07	30,08 ± 1,01

Phosphate addition increased the bound phosphorus content (0,264-0,298%) and degree of substitution (DS, 13,92-15,75.10^{-3}), leading to a

significant increase in RS content from 24,76% to 40,24% in green banana starch and from 27,42% to 49,63% in green banana flour. The higher RS content observed in phosphate-treated green banana flour (BF-PP) compared with phosphate-treated starch (GB-PP) was attributed to the presence of protein and dietary fiber, which contributed to the formation of a more resistant matrix.

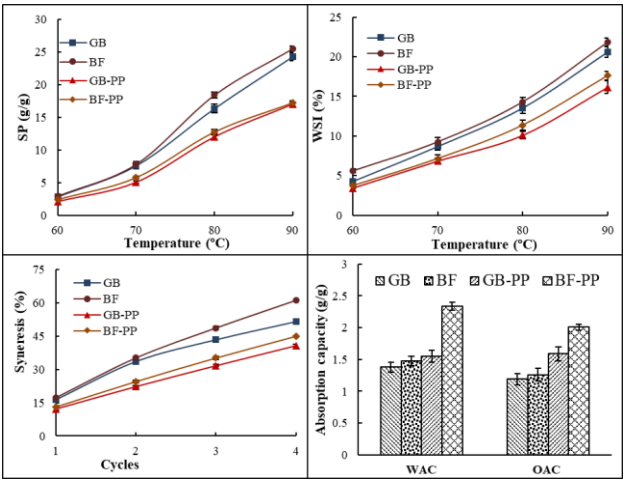

Figure 3.4. Functional properties of green banana starch phosphate and green banana flour phosphate

Swelling power (SP) increased with temperature; however, at 90°C the native samples showed higher SP values (24-26 g/g) than the phosphorylated samples (17 g/g), reflecting enhanced structural stability after modification. The water solubility index (WSI) decreased after phosphorylation, while freeze-thaw stability improved markedly, with BF-PP and GB-PP exhibiting lower water separation after four cycles than the native samples. In addition, water absorption capacity (WAC) and oil absorption capacity (OAC) increased following phosphorylation, with BF-PP showing the highest values.

SEM images revealed surface erosion and deformation of starch granules after phosphorylation. FTIR spectra displayed characteristic P=O and P–O–C bands (1219-1235 cm^{-1}), confirming the incorporation of phosphate groups into the starch structure. XRD analysis showed that native green banana starch exhibited a B-type crystalline pattern, whereas green banana flour displayed a mixed A/B-type pattern. Phosphorylation reduced

crystallinity to 32,29% in starch and 20,25% in flour, indicating an increase in the amorphous region due to structural modification.

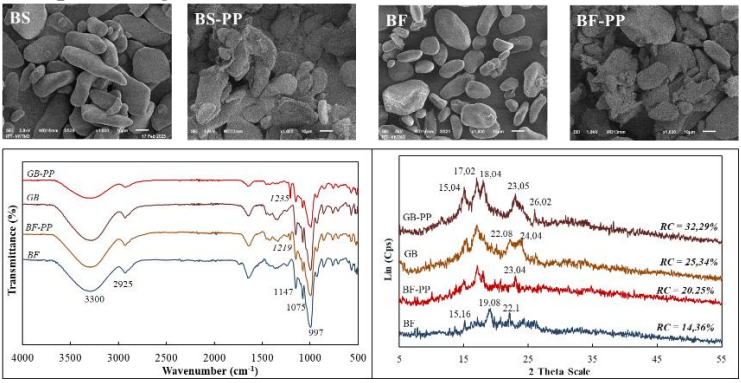

Figure 3.5. SEM images, X-ray diffraction patterns, and FTIR spectra of green banana starch phosphate and green banana flour phosphate

Conclusion of Section 3.2

Phosphorylation was optimized at 10% STMP/STPP, 10% Na_2SO_4, 70°C, and 1 h, yielding RS contents of 58,29% for green banana starch and 40,27% for jackfruit seed starch. For green banana flour, the highest RS content (49,63%) was obtained at 8% STMP/STPP. Phosphorylation reduced swelling power (SP) and water solubility index (WSI), while enhancing water and oil absorption capacities as well as freeze-thaw stability.

3.3. Modification of starch by hydrothermal treatment

3.3.1. Digestibility of hydrothermally modified starch

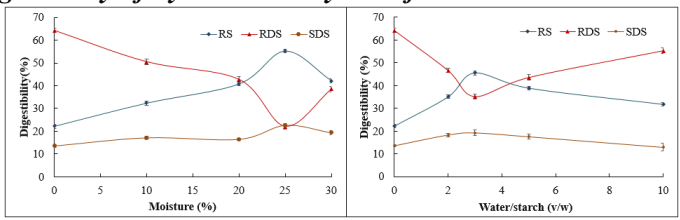

Figure 3.6. Effect of starch moisture and water content during HMT and ANN treatment on the digestibility of green banana starch

Hydrothermal treatments increased RS and decreased RDS by promoting the formation of a more stable crystalline structure. Under HMT, RS reached a maximum of 55,22% at 25% moisture content, whereas ANN produced a lower maximum RS value of 45,58%. Beyond the optimal

moisture level, RS decreased due to structural disruption. SDS showed only minor changes in both treatments. Overall, HMT was more effective than ANN in increasing RS content and reducing starch digestibility.

3.3.2. Physicochemical characteristics of hydrothermally modified starch

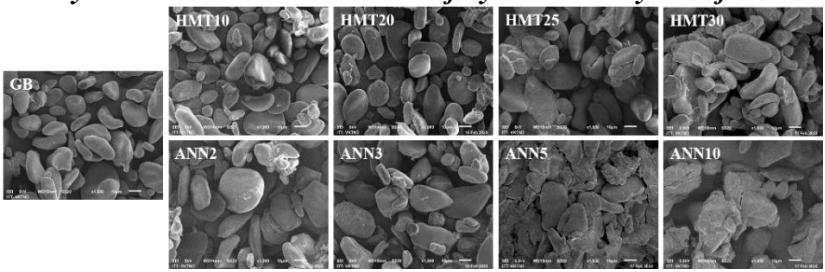

Figure 3.7. SEM images of green banana starch treated with HMT and ANN

SEM micrographs showed that HMT caused only minor morphological changes, whereas ANN induced greater granule deformation and structural disruption, particularly at higher moisture levels. This difference reflects the limited water availability during HMT, which mainly promotes internal reorganization, while excess water during ANN enhances granule swelling.

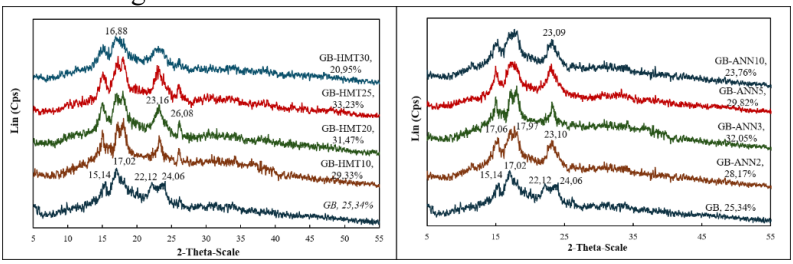

Figure 3.8. X-ray diffraction patterns and crystallinity of green banana starch treated with HMT and ANN

XRD analysis revealed a transition from a B-type to a C-type crystalline pattern after both treatments. Relative crystallinity increased to an optimum level (approximately 25% moisture for HMT and 3 mL/g for ANN) before declining under more severe conditions due to disruption of the crystalline regions. These results indicate that moderate treatment promotes crystalline reorganization, whereas excessive moisture increases the amorphous fraction.

3.3.3. Functional properties of hydrothermally modified starch

Table 3.16. Swelling power and solubility of heat-moisture starch (HMT)

(ºC)	*SP/WSI*	*Control*	*HMT10*	*HMT20*	*HMT25*	*HMT30*
60	SP (g/g)	2,88 ± 0,23	2,50 ± 0,24	2,31 ± 0,25	2,14 ± 0,23	1,88 ± 0,16
	WIS (%)	4,28 ± 0,26	4,02 ± 0,22	3,90 ± 0,22	3,37 ± 0,25	3,16 ± 0,20
70	SP (g/g)	7,62 ± 0,25	7,09 ± 0,23	6,27 ± 0,24	5,74 ± 0,20	4,96 ± 0,19
	WIS (%)	8,69 ± 0,25	8,06 ± 0,22	7,31 ± 0,26	5,28 ± 0,23	5,10 ± 0,20
80	SP (g/g)	16,37 ± 0,31	14,27 ± 0,24	12,61 ± 0,23	10,52 ± 0,24	9,54 ± 0,19
	WIS (%)	13,54 ± 0,26	11,33 ± 0,22	10,30 ± 0,21	8,92 ± 0,26	8,07 ± 0,20
90	SP (g/g)	24,30 ± 0,31	22,47 ± 0,38	19,33 ± 0,29	15,30 ± 0,26	12,64 ± 0,21
	WIS (%)	20,59 ± 0,18	18,26 ± 0,25	16,59 ± 0,22	14,02 ± 0,26	13,45 ± 0,24

HMT starch at moisture levels of 10, 20, 25, and 30% corresponds to HMT10, HMT20, HMT25, and HMT30, respectively.

Table 3.17. Swelling power and solubility of annealing starch (ANN)

(ºC)	*SP/WSI*	*Control*	*ANN2*	*ANN3*	*ANN5*	*ANN10*
60	SP (g/g)	2,88 ± 0,23	2,59 ± 0,28	2,23 ± 0,19	2,13 ± 0,19	2,03 ± 0,20
	WIS (%)	4,28 ± 0,26	3,56 ± 0,24	3,10 ± 0,23	3,09 ± 0,25	2,91 ± 0,26
70	SP (g/g)	7,62 ± 0,25	6,03 ± 0,18	5,63 ± 0,24	5,29 ± 0,22	5,09 ± 0,23
	WIS (%)	8,69 ± 0,25	6,28 ± 0,27	5,73 ± 0,25	5,52 ± 0,23	5,37 ± 0,31
80	SP (g/g)	16,37 ± 0,31	12,32 ± 0,26	10,59 ± 0,23	10,10 ± 0,23	9,89 ± 0,22
	WIS (%)	13,54 ± 0,26	11,31 ± 0,26	9,50 ± 0,24	9,29 ± 0,23	9,03 ± 0,19
90	SP (g/g)	24,30 ± 0,31	17,72 ± 0,21	15,73 ± 0,28	15,00 ± 0,22	14,80 ± 0,26
	WIS (%)	20,59 ± 0,18	15,75 ± 0,22	12,71 ± 0,25	12,11 ± 0,22	11,88 ± 0,24

ANN starch was treated with added water ratios of 2, 3, 5, and 10 mL/g starch, corresponding to ANN2, ANN3, ANN5, and ANN10, respectively.

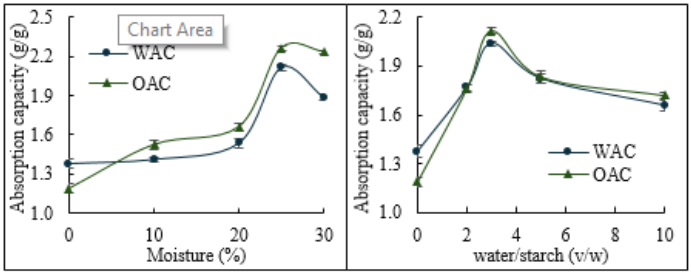

Figure 3.9. Water and oil absorption capacity of HMT and ANN starches

Table 3.18. Freeze-thaw stability of heat-moisture starch (HMT)

Cylces	*Syneresis (%)*				
	Control	*HMT10*	*HMT20*	*HMT25*	*HMT30*
1	16,25 ± 0,11	14,76 ± 0,16	13,45 ± 0,16	10,26 ± 0,17	12,60 ± 0,21
2	33,51 ± 0,15	28,49 ± 0,12	24,12 ± 0,20	19,57 ± 0,16	22,32 ± 0,19
3	43,34 ± 0,24	38,57 ± 0,29	35,49 ± 0,25	27,76 ± 0,27	31,98 ± 0,23
4	51,53 ± 0,14	47,05 ± 0,18	42,20 ± 0,20	35,23 ± 0,20	38,30 ± 0,25

HMT starch at moisture levels of 10, 20, 25, and 30% corresponds to HMT10, HMT20, HMT25, and HMT30, respectively.

Table 3.19. Freeze-thaw stability of annealing starch (ANN)

vCylces	*Syneresis (%)*				
	Control	***ANN2***	***ANN3***	***ANN5***	***ANN10***
1	16,25 ± 0,11	14,22 ± 0,21	11,29 ± 0,25	13,87 ± 0,21	15,52 ± 0,24
2	33,51 ± 0,15	27,76 ± 0,21	22,12 ± 0,19	25,94 ± 0,30	26,50 ± 0,24
3	43,34 ± 0,24	38,55 ± 0,29	33,73 ± 0,21	36,21 ± 0,20	38,12 ± 0,28
4	51,53 ± 0,14	45,62 ± 0,26	40,30 ± 0,20	43,58 ± 0,16	45,23 ± 0,20
ANN starch was treated with added water ratios of 2, 3, 5, and 10 mL/g starch, corresponding to ANN2, ANN3, ANN5, and ANN10, respectively.					

HMT and ANN significantly reduced swelling power (SP) and water solubility index (WSI), while enhancing water absorption capacity (WAC) and oil absorption capacity (OAC). The highest WAC and OAC values were obtained under optimal conditions (~25% moisture for HMT and ~3 mL/g for ANN), reflecting structural reorganization and increased RS formation. HMT generally produced more moderate and stable changes than ANN.

Freeze-thaw stability was markedly improved after both treatments, as evidenced by reduced syneresis. The lowest syneresis values were observed for HMT25 (~35,23%) and ANN3 (~40,30%), indicating enhanced water retention through a strengthened amylose network and hydrogen bonding. Overall, HMT was more effective than ANN in improving starch stability.

Conclusion of Section 3.3

Both HMT and ANN increased resistant starch (RS) content and reduced rapidly digestible starch (RDS), with optimal conditions identified at 25% moisture for HMT and 3 mL/g for ANN. The treatments transformed the crystalline structure from B-type to C-type and increased relative crystallinity, with ANN inducing greater structural changes. Functional properties, including SP, WSI, WAC, OAC, and freeze-thaw stability, were also improved. Overall, HMT provided a better balance between structural modification and functional performance, making it more suitable for functional food applications.

3.4. Carboxymethylation of starch

3.4.1. Digestibility of carboxymethyl starch

DS increased with NaOH concentration, peaking at ~0.57 at 10% before declining at 12%. RS showed a similar trend, reaching its maximum at 10%, while RDS decreased and SDS slightly increased before dropping. This is because NaOH promotes swelling and carboxymethylation, enhancing

resistance to enzymatic hydrolysis. Therefore, 10% NaOH was optimal.

Table 3.20. Effect of NaOH content on the digestibility and substitution degree of jackfruit seed starch

m_{NaOH} (%)	*DS (%)*	*RS (%)*	*RDS (%)*	*SDS (%)*
Control	-	15,18 ± 1,11	29,64 ± 0,53	55,18 ± 1,92
5	0,284 ± 0,006	29,31 ± 1,02	16,72 ± 0,45	53,97 ± 1,44
7	0,436 ± 0,005	33,14 ± 0,96	12,45 ± 0,38	54,41 ± 1,71
10	0,563 ± 0,009	39,67 ± 1,07	2,21 ± 0,03	58,12 ± 1,63
12	0,492 ± 0,007	35,44 ± 0,85	10,32 ± 0,09	54,24 ± 1,58

3.4.2. Physicochemical characteristics of carboxymethyl starch

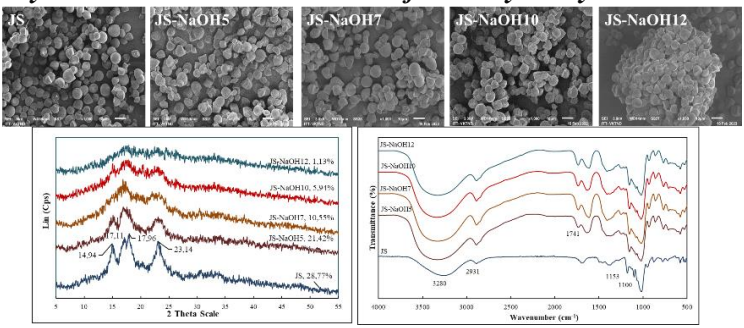

Figure 3.10. SEM images, X-ray diffraction patterns, and FTIR spectra of carboxymethyl starch

Carboxymethylation significantly modified the structure of jackfruit seed starch. SEM images showed progressive surface roughness, porosity, cracking, and erosion of starch granules with increasing NaOH concentration. XRD analysis revealed a transformation from an A-type to a C-type crystalline pattern, accompanied by a marked reduction in crystallinity (from 28.77% to 1.13%), indicating expansion of the amorphous regions. FTIR spectra displayed characteristic bands of carbonyl (C=O, 1741 cm^{-1}) and carboxylate ($–COO^–$, 1600-1650 cm^{-1}) groups, confirming the successful incorporation of carboxymethyl groups. The increasing intensity of these bands with higher NaOH concentrations further demonstrated enhanced modification efficiency.

3.4.3. Functional properties of carboxymethyl starch

Carboxymethylation significantly enhanced the hydration and functional properties of jackfruit seed starch. Swelling power (SP), water solubility index (WSI), water absorption capacity (WAC), and oil absorption

capacity (OAC) increased after modification, reaching maximum values at 10% NaOH. These improvements were attributed to the introduction of carboxymethyl groups (–CH_2COO^-), which increased hydrophilicity and expanded the amorphous regions of the starch granules. However, a further increase in NaOH concentration to 12% slightly reduced these properties

In addition, carboxymethylated starch exhibited lower syneresis than native starch during freeze-thaw cycles, demonstrating improved freeze–thaw stability. The enhanced stability was associated with the greater water-binding capacity of carboxymethyl groups, which promoted water retention and minimized phase separation.

Table 3.21. Swelling power and solubility of carboxymethyl starch

(ºC)	*SP/WSI*	*Control*	*JS-NaOH5*	*JS-NaOH7*	*JS-NaOH10*	*JS-NaOH12*
60	SP (g/g)	5,14 ± 0,23	6,34 ± 0,19	7,89 ± 0,21	8,87 ± 0,21	8,43 ± 0,18
	WIS (%)	2,14 ± 0,11	3,05 ± 0,13	4,83 ± 0,16	6,31 ± 0,15	6,07 ± 0,08
70	SP (g/g)	6,21 ± 0,15	10,39 ± 0,21	15,55 ± 0,17	20,12 ± 0,18	19,74 ± 0,19
	WIS (%)	8,35 ± 0,17	11,44 ± 0,19	13,69 ± 0,19	16,58 ± 0,21	15,95 ± 0,19
80	SP (g/g)	15,11 ± 0,34	19,47 ± 0,31	22,32 ± 0,28	25,74 ± 0,32	23,68 ± 0,28
	WIS (%)	12,86 ± 0,37	19,53 ± 0,31	22,21 ± 0,28	27,76 ± 0,33	26,89 ± 0,29
90	SP (g/g)	23,42 ± 0,67	27,24 ± 0,57	31,13 ± 0,62	34,69 ± 0,61	31,34 ± 0,58
	WIS (%)	20,04 ± 0,82	29,58 ± 0,77	33,61 ± 0,75	36,86 ± 0,81	34,76 ± 0,78

Table 3.22. Water and oil absorption capacity of carboxymethyl starch

OAC/WAC	*Control*	*JS-NaOH5*	*JS-NaOH7*	*JS-NaOH10*	*JS-NaOH12*
OAC (g/g)	1,07 ± 0,011	1,22 ± 0,013	1,34 ± 0,015	1,55 ± 0,014	1,43 ± 0,012
WAC (g/g)	0,91 ± 0,010	1,01 ± 0,011	1,18 ± 0,014	1,37 ± 0,013	1,33 ± 0,013

Table 3.23. Freeze-thaw stability of carboxymethyl starch

Cycles	*Syneresis (%)*				
	Control	*JS-NaOH5*	*JS-NaOH7*	*JS-NaOH10*	*JS-NaOH12*
1	19,64 ± 0,14	17,39 ± 0,17	15,68 ± 0,19	12,05 ± 0,13	13,75 ± 0,12
2	38,36 ± 0,13	32,75 ± 0,16	27,04 ± 0,19	24,47 ± 0,18	22,21 ± 0,15
3	48,95 ± 0,28	40,41 ± 0,23	35,67 ± 0,25	31,42 ± 0,31	33,27 ± 0,22
4	54,18 ± 0,35	48,54 ± 0,29	43,39 ± 0,31	38,77 ± 0,32	40,68 ± 0,34

Conclusion of Section 3.4

NaOH concentration strongly influenced the carboxymethylation of jackfruit seed starch. A 10% NaOH treatment produced the highest DS, RS content, and functional properties, whereas excessive alkali (12%) reduced these characteristics. Carboxymethylation decreased crystallinity and enhanced hydration, with 10% NaOH identified as the optimal condition for food applications.

3.5. Application of resistant starch in noodle processing

3.5.1. Digestibility of noodle

Table 3.24. The digestibility of noodles containing resistant starch

RS substitution level (%)	*Green banana starch phosphate (GB-PP)*			*Green banana flour phosphate (BF-PP)*		
	RS (%)	*RDS (%)*	*SDS (%)*	*RS (%)*	*RDS (%)*	*SDS (%)*
0	19,55	57,76	22,69	19,55	57,76	22,69
10	23,44	52,13	24,43	24,89	50,28	24,82
20	26,56	48,22	25,22	29,90	45,18	24,92
30	31,40	42,39	26,28	35,07	39,21	25,72
40	40,98	36,90	22,12	44,70	34,61	20,69

Substitution of wheat flour with GB-PP or BF-PP (0-40%) significantly increased resistant starch (RS) content, reaching 40,98% and 44,70%, respectively, while reducing rapidly digestible starch (RDS). SDS increased slightly at 10-30% substitution but declined at 40%. At equivalent substitution levels, BF-PP noodles consistently exhibited higher RS and lower RDS contents than GB-PP noodles.

3.5.2. In-vitro estimated glycemic index (eGI) of noodle

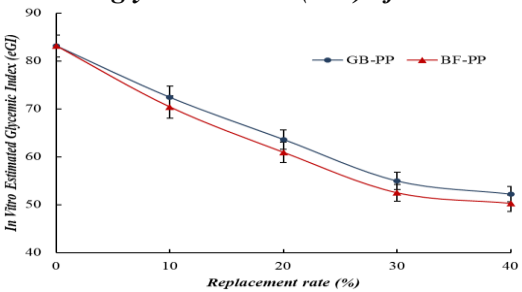

Figure 3.11. Estimated glycemic index of noodle containing resistant starch

The estimated glycemic index (eGI) of the noodles decreased with increasing levels of GB-PP and BF-PP incorporation. The control sample exhibited the highest eGI (83,11), whereas formulations containing 30-40% starch phosphate showed the lowest values (<55). At equivalent substitution levels, BF-PP resulted in lower eGI values than GB-PP, indicating a greater capacity to reduce starch digestibility.

3.5.3. Cooking properties of noodles

Incorporation of GB-PP and BF-PP increased the optimal cooking time (OCT), dry matter water absorption (DMWA), cooking loss (DML), and breakage rate (BR) of the noodles relative to the control. As the substitution level increased from 10% to 40%, OCT and DMWA rose markedly, likely

due to the enhanced structural stability and water-holding capacity imparted by hydrophilic phosphate groups. DML increased slightly, whereas BR increased substantially, particularly in BF-PP-containing formulations. Based on cooking quality and structural integrity, the optimal substitution level was determined to be 30% for GB-PP and below 20% for BF-PP.

Table 3.25. Cooking properties of noodles containing green banana starch phosphate

Sample	***OCT, s***	***DMWA (%)***	***Breakage rate (%)***	***DML (%)***
TB0	310 ± 9,03	122,48 ± 2,52	0,28 ± 0,05	1,39 ± 0,04
TB10 (GB-PP)	326 ± 8,60	143,94 ± 2,58	1,28 ± 0,09	1,56 ± 0,03
TB20 (GB-PP)	354 ± 7,59	162,17 ± 2,73	3,29 ± 0,16	1,75 ± 0,03
TB30 (GB-PP)	395 ± 7,72	192,58 ± 4,96	6,25 ± 0,20	1,94 ± 0,04
TB40 (GB-PP)	487 ± 6,55	217,45 ± 3,70	10,13 ± 0,32	2,18 ± 0,04

Table 3.26. Cooking properties of noodles containing green banana flour phosphate

Sample	***OCT, s***	***DMWA (%)***	***Breakage rate (%)***	***DML (%)***
TB0	310 ± 9,03	122,48 ± 2,52	0,28 ± 0,05	1,39 ± 0,04
TB10 (BF-PP)	336 ± 6,68	135,07 ± 2,93	1,63 ± 0,05	1,68 ± 0,03
TB20 (BF-PP)	366 ± 6,55	152,21 ± 2,80	8,54 ± 0,22	1,86 ± 0,04
TB30 (BF-PP)	415 ± 9,53	179,06 ± 5,27	16,28 ± 0,23	2,28 ± 0,05
TB40 (BF-PP)	505 ± 7,85	200,02 ± 2,65	21,26 ± 0,31	2,18 ± 0,04

Figure 3.12. Noodle containing resistant starch (green banana flour phosphate and green banana starch phosphate)

Conclusion of Section 3.4

Incorporation of GB-PP and BF-PP improved the nutritional quality of noodles by increasing RS content and reducing RDS and eGI. BF-PP exhibited greater efficacy than GB-PP in enhancing starch digestibility characteristics. Nevertheless, excessive substitution adversely affected cooking and structural properties; thus, the optimal substitution levels were 30% for GB-PP and <20% for BF-PP.

CONCLUSIONS

Through this study, the following major findings were obtained:

1. Green banana starch and jackfruit seed starch were isolated with yields of 14,73% and 25,21%, respectively. Both starches exhibited relatively high resistant starch (RS) contents (24,76-25,25%), intact granular structures, and characteristic crystalline patterns (type B for green banana starch and type A for jackfruit seed starch). Swelling power and solubility increased with temperature, with jackfruit seed starch showing greater swelling capacity.

2. Several modification techniques were successfully applied to enhance the RS content and functional properties of the starches:

- Phosphorylation under optimal conditions (10% Na_2SO_4, 10% STMP/STPP, 70°C, 1 h) increased the RS content to 58,29% in green banana starch and 40,27% in jackfruit seed starch. The treatment transformed the crystalline structure to type C, increased crystallinity, improved oil absorption and freeze-thaw stability, and reduced swelling power and solubility. For green banana flour, phosphorylation with 8% polyphosphate salt resulted in an RS content of 49,63%.

- Hydrothermal treatment (HMT) and annealing (ANN) increased the RS content and crystallinity of green banana starch. HMT produced a higher RS content (55.22%) and provided a more favorable balance between structural and functional properties than ANN.

- Carboxymethylation of jackfruit seed starch was optimal at 10% NaOH, yielding a degree of substitution of 0.563 and an RS content of 39.67%. The modification enhanced swelling power, solubility, water and oil absorption capacities, and freeze–thaw stability, while reducing starch crystallinity.

3. Partial replacement of wheat flour with phosphorylated green banana starch (GB-PP) and phosphorylated green banana flour (BF-PP) increased the RS content and reduced the rapidly digestible starch fraction and estimated glycemic index (eGI) of noodles. However, the substitutions also increased cooking time, water absorption, cooking loss, and breakage rate. The optimal replacement level was determined to be 30% for GB-PP and <20% for BF-PP to maintain the desired structure and cookability.

RECOMMENDATIONS

- Continue research on other modification methods such as enzymatic modification, ultrasonic treatment, microwave treatment, or a combination of methods to increase the content of resistant starch and improve the properties of modified starch.

- Expand research on the application of resistant starch from green bananas and jackfruit seeds in other food products such as snacks, pasta, rice noodles, baked goods, or nutritional bars to effectively exploit the nutritional value and application potential of RS-rich preparations from these raw materials.

NEW CONTRIBUTIONS OF THE THESIS

- Chemical modification via phosphorylation is a safe and effective approach that significantly enhances the resistant starch (RS) content of green banana starch up to 58,29% and jackfruit seed starch up to 40,27%. When utilizing green banana flour, the RS content can reach 49,63%. The partial substitution of wheat flour with phosphorylated green banana starch or phosphorylated green banana flour in noodle formulations contributes to a substantial reduction in the in vitro estimated glycemic index (eGI), shifting it from a high level (83,11) down to low levels (54,93 for 30% GB-PP substitution and 52,48 for 30% BF-PP substitution).

- Investigation into the mechanism of moisture-driven effects during hydrothermal treatments of green banana starch revealed that heat-moisture treatment (HMT) increases the RS content up to 55,22%, whereas annealing (ANN) achieves 45,58%. In general, the HMT approach superiorly preserves the starch granule structure and controls functional properties, whereas the ANN treatment induces more pronounced structural alterations and physical property changes.

- The carboxymethylation of jackfruit seed starch using NaOH concentrations within the range of 5-10% (w/w, based on starch weight) increases the degree of substitution (DS) from 0,284 to 0,563, contributing to an elevation in RS content from 29,31% to 39,67%. However, further increasing the NaOH concentration causes severe deformation of the starch granules and disrupts the crystalline structure, subsequently resulting in a simultaneous decline in both DS and RS values.

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