



Phosphorylation to Physicochemical Characteristics (Phosphate Content, Substitution Degree, Resistant Starch, FTIR and Critalinity) of Banggai Yams Starch

Ramadhani Chaniago^{1*}, Asriani Hasanuddin², Abdul Rahim², Darni Lamusu¹

¹ Muhammadiyah University of Luwuk 94711, Indonesia

² Tadulako University, Palu 94105, Indonesia

*Email: idthon86chaniago@gmail.com

Abstract:- One source of starch that is endemic to Banggai Islands Regency, Central Sulawesi, is the Banggai yam plant (*Dioscorea alata* L.). Banggai yam has starch with a high yield and has the potential to be developed through starch processing technology, but it has several weaknesses. If processing continues, a starch modification process is required. Starch phosphorylation is one method of starch modification that produces cross-linked phosphate starch as a functional food ingredient. Phosphorylation reagents can use sodium trimetaphosphate (STMP), sodium tripolyphosphate (STPP), or an STMP/STPP ratio mixture: 99/1. The research objectives are to determine the best concentration of STMP, STPP reagents and a mixture of STMP/STPP ratio: 99/1 in making BYSP and evaluate the physicochemical characteristics of BYSP for functional food ingredients. The first stage was the Synthesis of Banggai Yam Starch Phosphate (BYSP), and the BYSP obtained was then analyzed for its physicochemical properties, including phosphate content and degree of cross-linking substitution, RS content, binding of phosphate functional groups using FTIR, and crystallinity using an X-ray diffractometer. The results of the research were STMP 10% phosphate content 0.274 and DS 0.260; STPP 2% resistant starch 4,450; FTIR BYSP is classified as OH with a frequency area of 3420.41–3419.02 cm⁻¹, CH₃ with a frequency area of 2931.33–2929.74 cm⁻¹ and C=C with a frequency area of 1645.10–1641.84 cm⁻¹; The degree of crystallinity ranges from 12-13%. Moreover, crystallinity using an X-ray diffractometer. The results of the research were STMP 10% phosphate content 0.274 and DS 0.260; STPP 2% resistant starch 4,450; FTIR BYSP is classified as OH with a frequency area of 3420.41–3419.02 cm⁻¹, CH₃ with a frequency area of 2931.33–2929.74 cm⁻¹ and C=C with a frequency area of 1645.10–1641.84 cm⁻¹; The degree of crystallinity ranges from 12-13%. Moreover, crystallinity using an X-ray diffractometer. The results of the research were STMP 10% phosphate content 0.274 and DS 0.260; STPP 2% resistant starch 4,450; FTIR BYSP is classified as OH with a frequency area of 3420.41–3419.02 cm⁻¹, CH₃ with a frequency area of 2931.33–2929.74 cm⁻¹ and C=C with a frequency area of 1645.10–1641.84 cm⁻¹; The degree of crystallinity ranges from 12-13%.

Keywords: phosphorylation; modification; starch; banggai yam.



1. Introduction

Carbohydrates in the form of starch are food reserves in plants and are one of nature's most abundant and widespread elements. One source of starch that is endemic to Banggai Islands Regency, Central Sulawesi, is the Banggai yam plant (*Dioscorea alata* L.).

Banggai yam has starch with a high yield and has the potential to be developed through starch processing technology. Banggai yam starch still needs to be expanded on a household scale, even though it can be developed as a raw material in the food and non-food industries. However, natural Banggai yam starch is less acceptable in processing in the food and non-food industries because it has inappropriate physical, chemical and functional characteristics with standards. Natural starch has poor flowability, does not dissolve in cold water, experiences retrogradation, has a lower viscosity and thickens after heating [1], [2]. Apart from that, natural starch is also not resistant to heat, acid and mechanical processes, one of which is stirring [3], and functional properties are still low [4].

A starch modification process is needed to improve the characteristics and meet starch needs that can be applied in the culinary and non-food industries. Phosphorylation is one method of starch modification that produces cross-linked phosphate starch as a functional food ingredient and edible polymer film. The cross-linking treatment is intended to add chemical bonds to the granules that make up the starch (granule). Cross-linking stabilizes the granules and strengthens the relatively soft starch. Cross-linked starch paste has good flowability, dissolves in cold water, inhibits retrogradation, has a more stable viscosity, is thicker and is less likely to break down with long periods of heat, increased acid content, or vigorous stirring [5].

The phosphorylation process is the insertion of phosphate compounds, which produce starch with different properties, such as modification of solubility, pulp properties and morphology [6]. Starch phosphates are grouped into two classes: starch phosphate monoesters and starch phosphate diesters (cross-linked starch) [7]. Phosphorylation reagents can use sodium trimetaphosphate (STMP), sodium tripolyphosphate (STPP), a mixture of STMP/STPP ratio: 99/1, epichlorohydrin and phosphorus oxychloride [8]. Previous study says that integrating phosphate in starch molecules implies that the retrogradation process is hindered and has resilience to heat, acid-base and water. Phosphorylation of Banggai yam starch was carried out according to the technique proposed by gredients and have the potential to become edible polymer films.



2. Methods

Time and place

The research was carried out from July to September 2023. Banggai Natural Yam Starch (BNYS) is made in Luwuk, Banggai Regency. Phosphorylation of Banggai yam starch or Banggai yam Starch Phosphate (BYSP) was carried out at the Chemistry Research Laboratory, FMIPA, Tadulako University; FTIR and Crystallinity at the LTTP Integrated Laboratory, Gajah Mada University, Yogyakarta; Degree of Substitution Test at the Food Technology and Agricultural Products Testing Laboratory, Gajah Mada University, Yogyakarta; Phosphate Test at PT. Saraswanti Indo Genetech Bogor; Resistant Starch Test at the Department of Food Science and Technology Laboratory, Bogor Agricultural Institute.

Material

Banggai yam are obtained from the Banggai Islands Regency. The materials used in making BYSP include natural Banggai yam starch, distilled water, 3% NaOH, STMP, STPP, HCl, and 95% ethanol.

Method

Synthesis of Banggai Yam Starch Phosphate (BYSP)

Phosphorylation of Banggai yam starch was carried out according to the technique proposed by [9] with slight modifications. The suspension comprised of Banggai yam starch (100 g) and distilled water (225 mL) was agitated with a magnetic agitator for one hour at room temperature. After that, the pH of the suspension was adjusted and maintained at pH 10.5 by adding 5% NaOH while continuing to stir, then adding a). STMP 2, 6, 10% b). STPP 2, 6, 10% and c). mixture of STMP/STPP ratio: 99/1 with concentrations of 2, 6, 10% (w/w) for each phosphorylation reagent. The suspension was stirred for 45 minutes at 40°C while maintaining a pH of 10.5. After the phosphorylation is complete, add 0.5 N HCl to the suspension to pH 4.5 to stop the reaction. The following process is deposition and washing with distilled water three times and ethanol once, then drying with a cabinet drier at 50°C for 12 hours until the water content is 10-12%, crushed, and filtered with a 100mesh sieve. Phosphorylation results produce phosphoric Banggai yam starch (BYSP) at various concentrations of STMP, STPP reagents and the ratio STMP/STPP: 99/1.

Physicochemical and Functional Characterization of Banggai Phosphate yam Starch (BYSP)

The phosphate-bearing Banggai yam starch obtained was then analyzed for its physicochemical analysis, including phosphate content and degree of cross-linking substitution, RS content, binding of phosphate functional groups using Fourier transform infrared (FT-IR) spectra, and crystallinity using an X-ray diffractometer. The data processing



results for all parameters obtained BYSP, which can be used as a functional culinary constituent and an edible polymer film.

3. Results and Discussion

Phosphate Content and Degree of Substitution (DS)

The phosphate content test was carried out to evaluate the phosphate of Banggai yam starch (BYSP) given the phosphorylation method (STMP, STPP, and STMP/STPP). The results of the BYSP phosphate level test are presented in Figure 1, which shows that giving 10% STMP gives the highest value (0.274) while giving 2% STPP gives the smallest value (0.097). This research shows that the greater the percentage or ratio of phosphorylation, the higher the BYSP phosphate value. The higher the amount of phosphate content, the higher the cross-linking carried out, and when used as a reactant, the quantity of phosphate groups incorporated in starch is higher, and they are included in the granules [10]. The results of this study were higher than several previous studies, such as tapioca starch 0.155-0.237% [11], on yam, banana, corn, cassava and breadfruit starch with phosphate content ranging from 0.037–0.084% [12], in hosting phosphate flour ranges between 0.023-0.077% (bk) [13] and phosphate in modified tapioca/cassava starch in the range of 0.032-0.076% [14]. The phosphorus content in phosphorylated Natan starch ranges from 0.067 to 0.220 [13]. The phosphate level in BYSP increases with the concentration of each phosphorylation because the higher the reagent concentration, the higher the concentration of available phosphate salts, resulting in the possibility of phosphate group substitution. The phosphorus groups bind only starch molecular chains, not in her molecular chains [15]. Apart from that, increasing the percentage of STMP and STPP increases the kinetic energy of the particles so that the reaction takes place more quickly and produces more varied products [16]. If starch modification is carried out exclusively with STMP, the phosphorus concentration should not exceed 0.4%. However, when the procedure is carried out by combining STMP/STPP, the limit is 10 times greater (0.4%) [17].

The degree of substitution (DS) is carried out to determine the number of functional groups that can give rise to OH- groups in BYSP. The results of the Degree of Substitution (DS) BYSP test are presented in Figure 1, which shows that giving 10% STMP gives the highest value (0.260) for DS BYSP while giving 2% STMP gives the smallest value (0.140) for DS BYSP. This is higher when compared to previous research, such as palm phosphate starch with DS values ranging from 0.0143-0.0617 [18]; phosphate tapioca starch with DS values ranging from 0.00814-0.0121 [11]; in phosphate palm starch with DS values ranging from 0.040-0.140 [19]. This research also shows that the higher the percentage or ratio of STMP or STMP/STPP given, the higher the DS BYSP value, while the higher the STPP percentage or ratio, the lower the level of DS BYSP, although it is not significant. The STMP concentration



of 10% BYSP is reactive and expands, so the OH groups are easily separated from the starch molecules. The ratio of STMP and STPP concentrations is estimated to influence the starch modification process [16]. The DS value in this study can be used as a functional food. It has a DS value < 1 (0.140-0.260), whereas if the DS value is > 1 , it can be directed to non-food products because the human body cannot digest them [20].

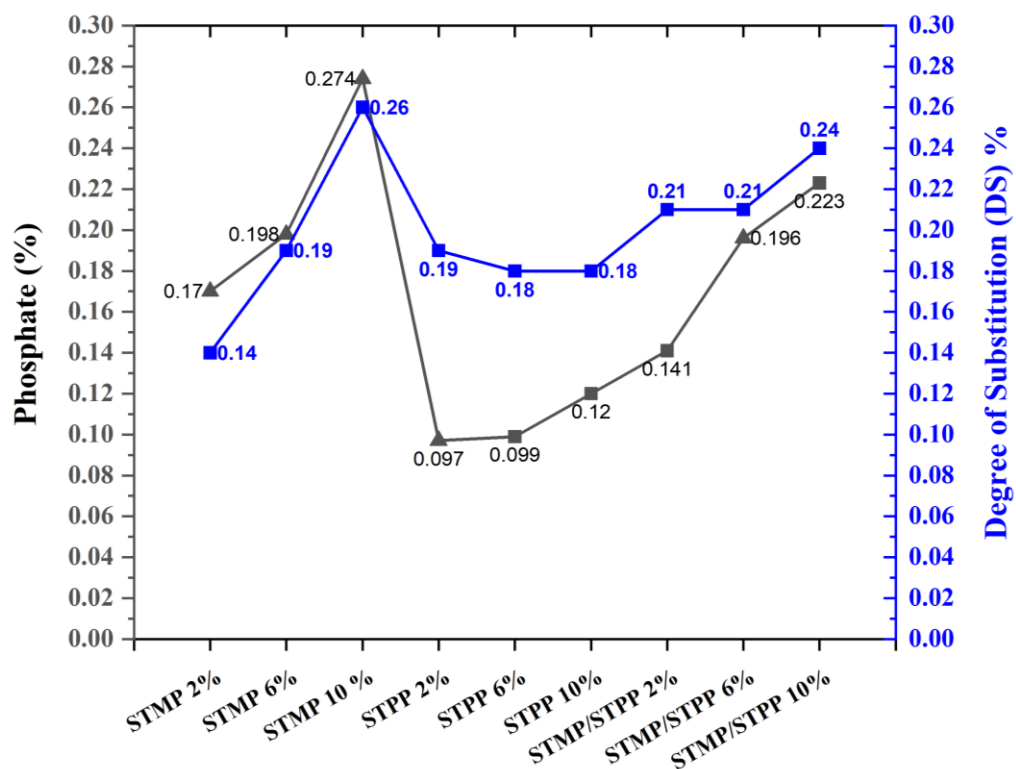


Figure 1. Phosphate levels and degree of substitution (DS) in BYSP

Resistant Starch

Resistant starch (RS) cannot be digested because the starch fraction is partially fermented in the large intestine to form short-chain fatty acids [21], [22]. Type 4 RS starch has been chemically altered, making it challenging to digest through oxidation, etherification, esterification, or irradiation with γ -rays [23]. Resistant starch has properties and functions similar to dietary fibre as a functional food ingredient. Resistant starch is essential to the body's physiological functions, such as reducing the glycemic index, cholesterol and the risk of colon cancer [24]. This RS level test was carried out to evaluate the RS level in BYSP. The results of the BYSP RS concentration test are presented in Figure 2, which shows that giving 2% STPP gives the highest value (4,450) for BYSP Hospital and giving 10% STMP/STPP gives the smallest value (2,810) for BYSP Hospital. The RS value in this study is still smaller when compared to the RS value of rice starch phosphorylation, which is around 42.32–47.44% [24];



RS value of modified cassava flour ranges from 2.8-12.5% [25], and RS value of red bean flour is 5.59-12.08% using a combination method of steaming, microwave oven, autoclave and cooling [26]. The following are the daily consumption standards for resistant starch according to several developed countries, such as China 18 g/day [27]; India 10 g/day [28]; European Union 3-4 g/day [29]; UK 2.76 g/day [30]; Australia 5-7 g/day [31]; Sweden 3.2 g/day [29]; Italy 7.2-9.2 g/day [32]; New Zealand 5.2-8.5 g/day [33].

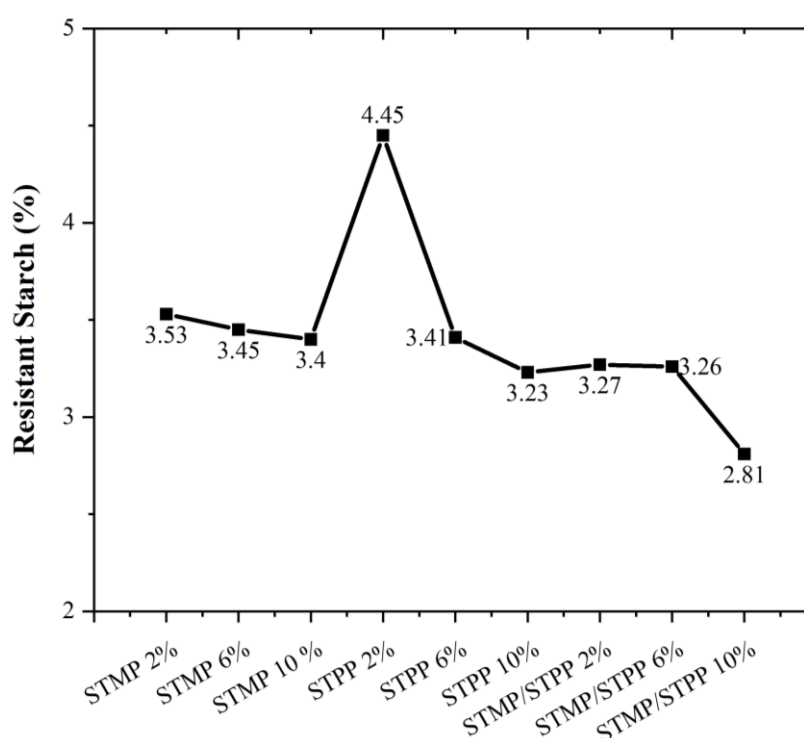


Figure 2. BYSP Resistant Starch

FTIR Spectra

Primary functional groups in nine starch samples were identified using FTIR testing. An FTIR spectrometer with a frequency range of $500\text{--}4000\text{ cm}^{-1}$ was used to determine the phosphorylation pattern in BYSP. Phosphate groups are hypothesized to bind to the OH groups of the C2, C3, or C6 atoms of starch molecules. Suppose this group interacts with the ester group; the resulting FTIR spectrum absorption band changes. The nine FT-IR spectra, as seen in Figure 3, were comparable, suggesting that the functional groups in the nine starch samples were identical. The main differences between the polymorphs are observed in the intensities of two spectral regions: the OH vibration region, $3650\text{--}3000\text{ cm}^{-1}$, and the central adsorption band region, $1200\text{--}800\text{ cm}^{-1}$ [34]. The FTIR spectrum of BYSP is presented in Figure 3, which shows a characteristic peak ranging from $3420.41\text{--}3419.02\text{ cm}^{-1}$ belonging to the hydroxyl



group (-OH) [35]. Absorption at wave numbers ranging from 2931.33–2929.74 cm^{-1} shows the stretching vibration of the alkane CH group (-CH₃) [36]. Absorption at wave numbers ranging from 1645.10–1641.84 cm^{-1} due to the presence of C=C groups, which means there is stretching vibration of bound water molecules [37]. The BYSP absorption band ranges from 1018–1022 cm^{-1} . These peaks can be attributed to the vibration/solvation of the C-OH bond and changes from amorphous to semi-crystalline [38]. According to [39], the area between 1200 cm^{-1} and 1000 cm^{-1} is where starch molecules exhibit their major absorbance, which is mostly caused by C-O, C-C, and C-O-H stretching and bending.

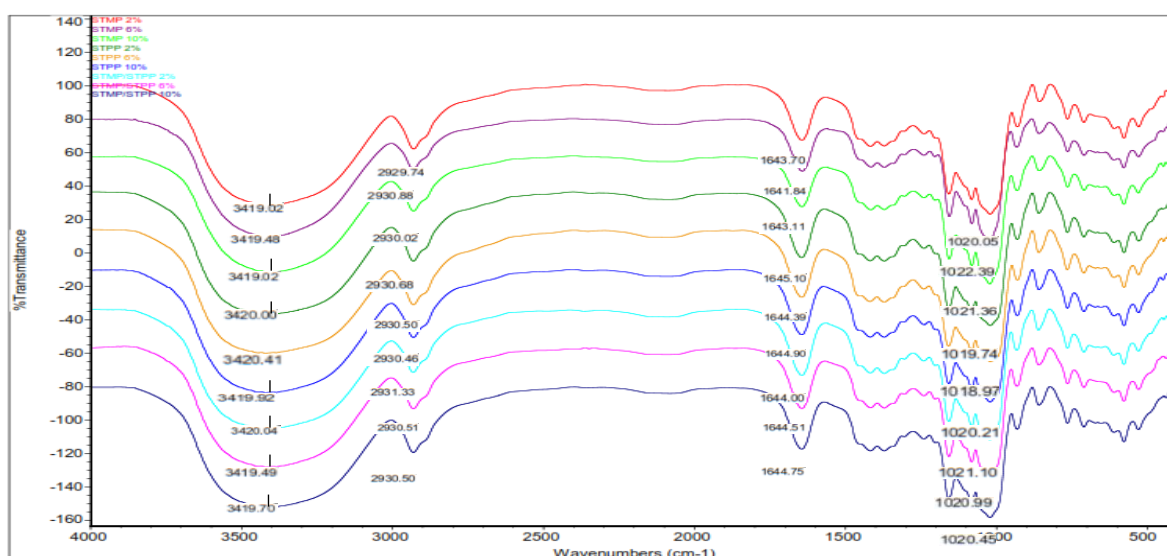


Figure 3. FTIR spectrum of BYSP

Crystallinity test using X-ray diffraction (XRD)

XRD tests determine lattice characteristics and estimate the crystalline size [40] and the sample's ratio of crystalline and amorphous parts [41]. The starch produced in this study is included in type B starch because it is included in the tuber group. Type A starch is found in grains (cereals), while tubers and amylose-rich starches include type B starch. Legume plants have a hybrid of kinds A and B called type C [42]. The value of the degree of crystallinity using XRD BYSP can be seen in Table 1.

Table 1. BYSP Crystallinity Values

Treatment	Degree of Crystallinity (%)
STMP 2%	12.9
STMP 6%	13.2



STMP 10%	12.7
STPP 2%	13.1
STPP 6%	13.2
STPP 10%	13.1
STMP/STPP 2%	13.4
STMP/STPP 6%	13.4
STMP/STPP 10%	13.3

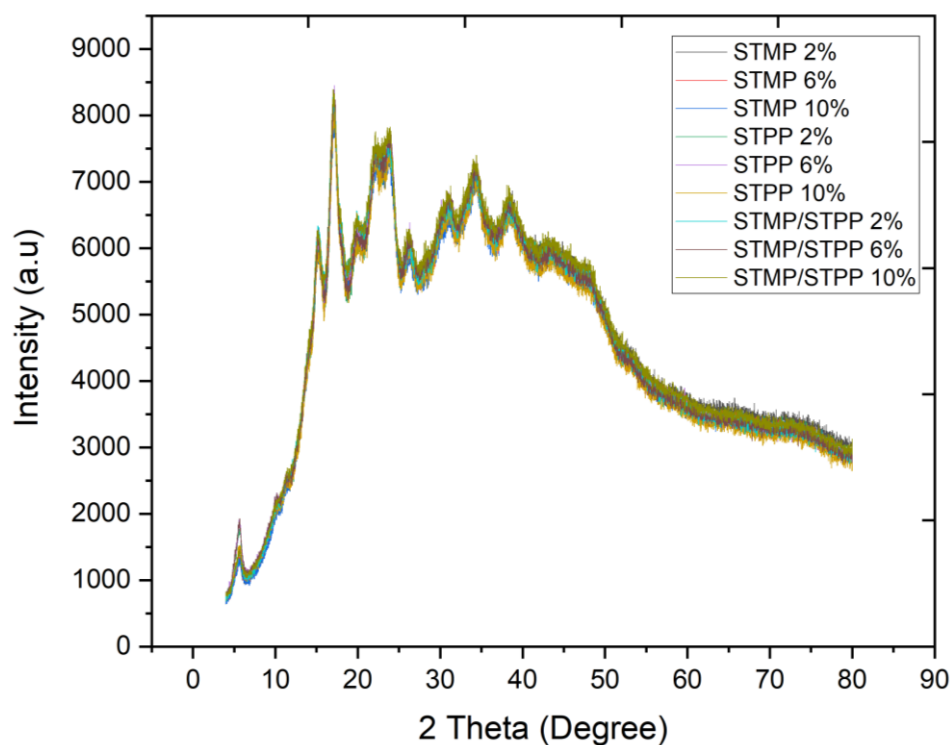


Figure 4. BYSP Crystallinity Results

The results showed that STMP/STPP 2% and STMP/STPP 6% showed the highest degree of BYSP crystallinity compared to other treatments and that the higher the concentration of phosphorylation, the more BYSP crystallinity tended to decrease. The degree of crystallinity in this research is still relatively small compared to research conducted by [14] in tapioca starch with a degree of crystallinity value of 24.79-29.64%. The peak point of the degree of crystallinity in all treatments showed similarities and not much difference, namely at 12° and 13°. This illustrates that phosphorylation has not been able to change the crystalline pattern of



Banggai yam starch because the number of phosphate groups is still tiny. Associated with a decrease in the degree of crystallinity, it can indicate that the area around the crystalline area is decreasing. The reduction in the crystalline area due to the STMP and STTP modification process can cause changes in the crystalline structure of BYSP granules [43]. Additionally, the crystalline region may be reduced due to the breaking of many hydrogen bonds next to the double helix or the reorientation of the double helix structure in the crystalline region [44]. The size of the crystal, the number of crystal parts (which is controlled by the length of the amylopectin chain), the orientation of the double helices inside the crystal, and the level of contact between the double helices are all described by the term "degree of crystallinity" [45].

Conclusion

The results of the research were STMP 10% phosphate content and Degree of Substitution (DS); STPP 2% resistant starch; FTIR BYSP is classified as OH with a frequency area of 3420.41–3419.02 cm⁻¹, CH₃ with a frequency area of 2931.33–2929.74 cm⁻¹ and C=C with a frequency area of 1645.10–1641.84 cm⁻¹; The degree of crystallinity ranges from 12–13%.

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