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Extraction and characterization of pectin from Saba banana [*Musa 'saba'(Musa acuminata* x *Musa balbisiana)*] peel wastes: A preliminary study

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Abstract

Saba banana, *Musa 'saba'(Musa acuminata* x *Musa balbisiana)* peel is an underutilized waste from processed banana manufacturing in the Philippines. The feasibility of saba banana peel pectin for use in food processing was assessed. Pectin from saba banana peel was extracted under two different extraction conditions. Pectin extraction was carried out using hydrochloric acid (0.5N, pH 1.5) and citric acid (0.5N, pH 1.7). Pectin from the acidified fruit peels were extracted at 90±5°C for 1, 2, 3 and 4 hours. Highest pectin yield was obtained using HCl extracted for 4h (17.05% dry basis). Effect of stage of maturity of saba banana on pectin yield was also determined. Higher yield was obtained in saba banana peels at the unripe stage. Chemical characterization of saba banana peel pectins revealed that moisture content of pectin from ripe and unripe saba banana peels were comparable while ash contents were much higher (11.15 and 13.83% respectively) compared with commercial citrus pectin (1.76%). Equivalent weight of pectin from ripe peels (953.89) is near that of commercial citrus pectin (893.00). Methoxyl content and anhydrouronic acid (AUA) values were significantly lower than those obtained from commercial citrus pectin while degree of esterification (DE) did not vary significantly for both ripeness stages compared with commercial citrus pectin. The extracted pectin was used in the processing of strawberry jam in order to assess its potential as a gelling agent. Sensory evaluation using Triangle test was conducted to compare strawberry jam with saba banana pectin and commercial citrus pectin. The samples were evaluated for color, flavour, cloudiness, consistency, mouth feel and overall acceptability. No statistical difference in all the attributes were observed between the two treatments.

Introduction

Bananas are considered as one of the most important tropical fruits in the world market. In the Philippines, saba banana *Musa 'saba'(Musa acuminata* x *Musa balbisiana)* is the most popular cultivar grown domestically which accounts to 39% of total production. Saba banana is most widely processed into chips/crackers which account for 95% of the country's earnings on processed banana (Molina and Roa eds., 2000). Banana fruit peels comprise a significant quantity of wastes produced from saba banana processing, which is equivalent to 40% of the total weight of fresh banana. These peels are just left as solid waste at large expense and are not being used for any other purposes. Thus, the operation of food processing wastes in the industry is now becoming a very serious ecological problem and additional pollution for the environment (Nagarajaiah and Prakash, 2011).

Fruit peels contain some valuable compounds like pectin. Pectin designates those water soluble pectinic acid (colloidal polygalacturonic acids) of varying methyl ester content and degree of neutralization, which are capable of forming gels with sugar and acids under suitable conditions (GITCO, 1999). In the food industry, pectin has a commercial value as gelling agent in food products. Pectin form gels under certain circumstances, the gelling mechanism is highly dependent on the degree of methoxylation (DM). Conventionally, pectin is divided into high methoxy (HM) pectin with DM > 50% and low methoxy (LM) pectin with DM < 50%. Pectin with DM > 50% forms gels in the presence of high sugar concentration, usually sucrose or fructose and low pH; whereas pectin with DM < 50% forms gels in the presence of divalent ions. The viscoelastic properties of pectins are the base of their broad use as a gelling agent and stabilizer in food products (Urias-Orona, 2010). Characterization of pectin is essential to determine its suitability for various food products.

Researches concerning the extraction methods and characterization of fruit pectin have been reported elsewhere (Fissure *et al*., 2009; Kurita *et al*., 2008;

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Mollea *et al*., 2008; Yapo *et al*., 2007). The yield and quality of pectin depends mostly upon the source as well as the method employed for extraction of pectin (Rehman *et al.*, 2004). In the present study, saba banana fruit peel waste was utilized as the source of pectin. It aimed to optimize the acid extraction method in terms of extraction time, type of acid and ripeness stage of the peels; and to chemically characterize the extracted pectin. The extracted pectin was also utilized in strawberry jam processing to determine whether it is comparable in terms of sensory characteristics with the commercially used citrus pectin.

Materials and Methods

Preparation of Saba banana peel powder

Banana peel wastes were soaked in 0.05% sodium metabisulfite for an hour to prevent discoloration, after which were dried in an oven at 55°C for 24 hours. The dried peels were then cooled at ambient temperature and were made into flour using a grinding mill. The powdered banana peels were then stored in polyethylene bags. Figure 1 shows the saba banana fruit and Figure 2 shows the steps for the preparation of the saba banana peel powder.

Extraction process optimization of pectin from banana peels

Homogenized banana peel powder were added to each of 0.50 N HCl, pH 1.5 and 0.50 N citric acid, pH 1.7. These were then heated with continuous stirring at 90±5°C in a stirring hot plate for 1, 2, 3 and 4 hours. The solution was then cooled and filtered through an ordinary screen with 1-mm mesh size with two-layer cheesecloth. The filtrate was collected then added with twice its volume of absolute ethanol. The precipitates were filtered through a miracloth. The residue was oven dried for 2 days at 55°C and then weighed. The pectin yield was calculated using the equation:

$$\text{Pectin yield (\%)db} = P/Bi \times 100$$

where p = extracted pectin in gram
Bi = weight of alcohol-insoluble-residue (AIR) in gram

Optimization of ripeness stage of banana peels was also performed using banana peels from ripe and unripe bananas. Pectin was extracted using 0.5N HCl, pH 1.5, for 3 h. The extracted pectin from ripe and unripe banana peels were analyzed for their chemical characteristics.

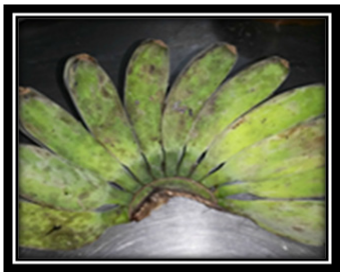

Figure 1. Saba banana [*Musa 'saba' (Musa acuminata x Musa balbisiana)*] fruit

Figure 2. Saba banana peel powder preparation. (a) oven-drying of banana peels (b) banana peels after drying (c) banana peels after grinding

Characterization of pectin

Moisture content determination

One gram of pectin sample was weighed, ground to pass 80-mesh screen, and placed into a metal dish (5cm in diameter with cover). The sample was dried in an oven for 5 hours at 100°C, cooled in a dessicator and then weighed. This sample was not used for subsequent measurement, as pectin tends to degrade after exposure to the atmosphere. One percent was added to the percent moisture observed to obtain agreement with the Fischer method (Johnson, 1945). The moisture content was determined using the equation:

$$\text{Moisture content (\%)} = \frac{\text{weight of residue} \times 100}{\text{Weight of sample}}$$

Ash content determination

One to two grams of pectin was ground to pass 80-mesh screen and placed into tared crucibles then ignited in a furnace for 3-4 hours at 600°C. For the determination of the alkalinity of the ash, ash was dissolved in 25 mL of 0.1 N HCl, heated gently to boiling and then cooled. It was then titrated with 0.1 N NaOH using phenolphthalein as an indicator. (The normality of HCl and NaOH used should be the same or else it will carry out a blank titration using 25 mL of the HCl used) (Owens *et al*., 1952; Gee *et al*., 1958). The ash content was determined using the formula below:

$$\text{Ash content (\%)} = \frac{\text{weight of ash} \times 100}{\text{weight of pectin}}$$

For the following analyses, the standard methods by Owen *et al*. (1952) were employed:

Equivalent weight

Equivalent weight was used for calculating the anhydrouronic acid content and the degree of esterification. It was determined by titration with NaOH (Titration A) to pH 7.5 using phenol red or Hinton's indicator. Equivalent weight was calculated using the equation:

$$\text{Equivalent weight (EW)} = \frac{\text{weight of sample (g) x 1000}}{\text{mL of alkali x N of alkali}}$$

Methoxyl content

The methoxyl content or degree of esterification is an important factor in controlling the setting time of pectins, the sensitivity to polyvalent cations, and their usefulness in the preparation of low solid gels, films, and fibers. It is determined by the saponification of the pectin and titration of the liberated carboxyl group.

To the neutral solution titrated for equivalent weight containing 0.5g of pectic substance, 25mL of 0.25N NaOH was added, shaken thoroughly and was allowed to stand for 30 minutes at room temperature in a flask with stopper. A 25mL portion of 0.25N HCl (or an amount equivalent to the base added) was added and was titrated with 0.1N NaOH to the same endpoint as the previous one (Titration B). The methoxyl content was calculated using the equation:

$$\text{Methoxyl content (\%)} = \frac{\text{mL alkali x N alkali x 3.1}}{\text{Weight of sample (g)}}$$

Anhydrouronic acid (AUA)

Pectin, which is a partly esterified polygalacturonide, contains 10% or more of organic material composed of arabinose, galactose and other sugars. Estimation of anhydrouronic acid content is essential to determine the purity, degree of esterification, and in evaluating the physical properties of pectin.

Making use of the equivalent weight, methoxyl content and the alkalinity of the ash data, anhydrouronic acid was calculated from the expression given below.

$$\text{AUA (\%)} = \frac{176 \text{ x } 100}{z}$$

where 176 is the molecular weight of AUA

$$z = \frac{\text{weight of sample (mg)}}{\text{meq Titration A + meq Titration B}}$$

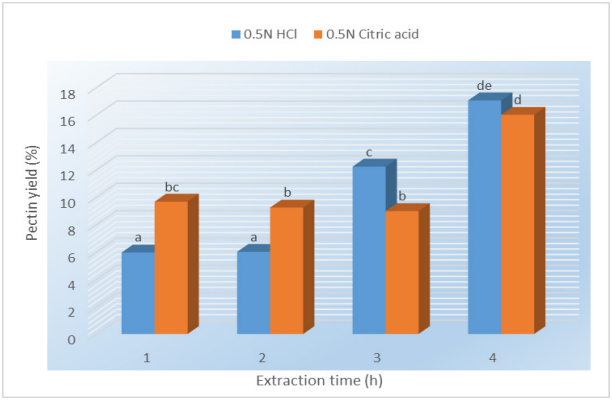

Figure 3. Pectin yield obtained using 0.5 N HCl and 0.5N Citric acid at different extraction times. (Values with the same letter symbols are not significantly different based on DNMRT at 5% level of significance)

Degree of esterification

The degree of esterification (DE) of pectin was determined according to the formula below (Shaha *et al.*, 2013):

$$\text{DE (\%)} = \frac{176 \text{ x MeC(\%) x } 100}{31 \text{ x AUA (\%)}}$$

Comparison by sensory evaluation of extracted pectin from saba banana peels and commercial citrus pectin as gelling agent in strawberry jam

Strawberry jam processing

To compare the extracted saba banana peel pectin from commercially used citrus pectin as a gelling agent in a food product, these were utilized in strawberry jam processing. Fresh strawberries were washed thoroughly and chopped into small pieces. The pulp was then weighed and was cooked in a stainless kettle with constant stirring to avoid scorching. Sugar and pectin were added and boiled vigorously with constant stirring. Citric acid was added when the mixture was thick.

Sensory evaluation

Triangle test was performed to determine significant difference of the treatment from the control. The treatment was that with saba banana peel pectin while the control was that with commercial citrus pectin. The samples were served to 15 panel judges. The data was subjected to Analysis of Variance. Dunnet's Single Range Test was used to determine significant differences among treatments.

Results and Discussion

Extraction process optimization for pectin revealed that hydrochloric acid is more efficient in extracting pectin compared with citric acid starting

Table 1. Yield and chemical characteristics of pectin extracted from ripe and unripe saba banana peels

Characteristics	Ripeness stage of banana peel		Commercial Citrus Pectin
	Ripe	Unripe	
Pectin yield, %	11.87[a]	16.54[b]	-
Moisture content (MC), %	10.00[a]	14.13[a]	12.03[a]
Ash, %	11.15[a]	13.83[b]	1.76[c]
Equivalent weight (EW)	953.89[a]	1503.16[b]	893[a]
Methoxyl content (MeC), %	6.40[a]	5.25[b]	9.09[c]
Anhydrouronic acid (AUA), %	57.32[a]	39.68[b]	74.29[c]
Degree of esterification (DE), %	63.37[a]	75.03[a]	69.48[a]

Means in a row followed by the same letter are not significantly different based on DNMRT at 5% significance level.

at 3h extraction time (Figure 2). At 4h extraction time, the amount of pectin extracted using both acids increased drastically though HCl still yielded higher amount but the yield does not differ significantly with citric acid. Attri and Maini (1996) also obtained higher yield of pectin from galgal (Citrus pseudolimon) peels when HCl was used compared to citric acid or tartaric acid. This is due to the ionic strength of HCl which is higher than weak acids such as citric. Given the same concentration, higher ionic strength acids have a higher capability to precipitate pectin due to their higher affinity for cations such as Ca^{2+} which stabilizes the pectin molecule. Thus, given enough time, higher amount of pectin can be precipitated out. As seen in Figure 3, at 3 h extraction time, significantly higher amount of pectin was extracted compared with those obtained at 1 and 2 h. At 4 h, pectin yield using both acids did not vary significantly. The amounts extracted were however higher compared with those at 3 h. Thus, extraction time at 4 h using 0.5N HCl is considered optimum for saba banana peel pectin extraction.

The ripeness stage of saba banana peels can also affect the pectin yield as the amounts of pectins, hemicelluloses, celluloses and lignin vary as the fruit matures. Pectin yield was observed to be higher at the unripe stage as compared with the ripe stage (Table 1). Pectin generally increase at first as the fruit peel becomes more tender, making the connections between pectins and other cellular compounds more fragile, thus making the pectin more available for extraction. However, over ripening of banana peels may result to a decrease in yield due to the degradation of pectin under the action of enzymes, such as polygalacturonase, pectin methyl esterase or pectate lyase (Emaga *et al*., 2007).

Moisture content of the banana peel pectins did not vary significantly with the commercial citrus pectin. Low moisture content is necessary for pectin for safe storage as well as to inhibit the growth of microorganisms that can affect the quality due to the production of pectinase enzymes (Muhmadzadeh *et al.*, 2010). Lower ash content was also obtained from unripe peels which implies that it can form better gels than that from the ripe peels, the maximum limit though, for good quality gel is 10% (Norazelina *et al*., 2011), which is lower than that of unripe peels (11.15%). Commercial citrus pectin has a very low ash content (1.76%) compared with the banana peel pectin Thus, the gel quality that will be produced from these pectins would vary, with banana peel pectin expected to be of lower quality. This could however be improved thru the extraction procedure, if a more efficient acid extraction will be employed to chelate more Ca^{2+} which contributes to majority of the ash content. Equivalent weight of pectins is another indicator of its jelly-forming ability, with high molecular weight pectins having better ability (Vaclavik and Christian, 2008). Pectin from unripe bananas peels has higher equivalent weight (Table 1).

Pectin, which is a partly esterified polygalacturonide, contains 10% or more of organic materials composed of arabinose, galactose and other sugars. AUA (%) is essential to determine the purity and degree of esterification and to evaluate physical properties (Ranganna, 1986). The higher galacturonic acid and lower ash content are the two criteria governing its purity (Hwang *et al.*, 1992). The purity of the pectin obtained from ripe banana peels is higher compared with that from unripe peels as indicated by its higher %AUA and lower ash content. Though the yield from unripe peels is high, the purity is however low, thus further purification is needed to obtain a higher quality pectin. Anhydrouronic acid content indicates the purity of extracted pectin if it is not less than 65% (Food Chemical Codex, 1996). AUA content of less than 65% may indicate impurities due to the presence of proteins, starch and sugars in the precipitated pectin (Norazelina and Nazarrudin, 2012). The pectins from saba banana peels have low purity compared with commercial citrus pectin with AUA greater than 65%. The value

Table 2. Sensory evaluation using Triangle test of strawberry jam with pectin from saba banana peels and commercial citrus pectin.

Quality traits	Calculated F-value
Color	4.0469
Flavor	4.3200
Cloudiness	3.5626
Consistency	3.0681
Mouthfeel	3.3116
Overall acceptability	0.4118

Tabular F-value = 4.60

obtained for AUA of pectin from ripe saba banana peels is in close agreement with that obtained by Madhav and Pushpalatha (2002) from banana peels (53%). Likewise, the value obtained by the same authors for methoxyl content was also in close agreement (7.03%) with that of the ripe banana peels (6.40%). Methoxyl content of extracted pectin vary from 0.2-12% depending on the source and mode of extraction (Aina *et al.*, 2012). The methoxyl contents of the extracted pectin pectin fall within this range. Methoxyl content of commercial pectins generally varies from 8-11% and can form high sugar gels (>65% sugar). On the other hand, low methoxyl pectins (less than 7.0%) can form gels with lower concentrations of sugars. Methoxyl content also influence the dispersability of pectin in water, higher methoxyl content being more readily dispersible in water than that with less than 7.0% methoxyl content (Rouse *et al*., 1962).

Degree of esterification (DE) values obtained were also within the range of 60-90% which is generally found in tissues (Shaha et al., 2013). Pectins could be classified as rapid-set (DE >72%) and slow-set (DE 58-65%), which describes the rate of gel formation (Shaha *et al.*, 2013). Pectin from ripe peels can be classified as slow-set while that from unripe peels is rapid-set. In terms of methoxyl content, AUA and DE, pectin from ripe saba banana peels is comparable with commercial citrus pectin.

The extracted saba banana peel pectin was applied to strawberry jam as a gelling agent. Sensory evaluation was conducted to compare the product with saba banana peel pectin and with commercial citrus pectin. In Table 2, the calculated F-values for all traits are less than the tabular value which indicates that all quality trait ratings were not significantly different from each other. In this product, the properties imparted by saba banana peel pectin such as color and flavour were not distinguishable as compared with that of commercial citrus pectin. Likewise, the textural properties such as consistency and mouth feel were the same for both pectins. This shows possible commercial use of the extracted pectin from saba banana peel in food processing.

Conclusion

Extraction of saba banana peel obtained highest yield using 0.5N HCl for 4 h at 90°C. Higher pectin yield was obtained from unripe saba banana peels. Chemical characteristics such as moisture content, degree of esterification and equivalent weight of pectin from ripe saba banana peels were similar with commercial citrus pectin. When applied to strawberry jam as gelling agent, no significant difference between commercial citrus pectin in terms of sensory properties were detected. Saba banana peel can be a potential source of pectin for food applications.

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