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Effect of extraction pH and temperature on the physicochemical properties and pectin yield from mango peel (*Mangifera indica* L.)

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ABSTRACT

Objective: To evaluate the effect of the pH and extraction temperature on the pectin yield from mango (*Mangifera indica*) peel, cultivar Banilejo, and its physicochemical properties.

Design/methodology/approach: Pectin extraction was done by acid hydrolysis, using hydrochloric acid and ethyl alcohol to precipitate and purify. A randomized design with a factorial arrangement was used, evaluating the effect of pH (1.5, 2.0, and 2.5) and temperature (70, 80, and 90 °C) on the yield and quality of pectin. Quality was determined by measuring pH, viscosity and moisture content, ash, methoxyls, and esterification degree. Their means were compared using Tukey's test at 95% confidence.

Results: The best results were obtained at pH 2.0 and 80 °C, reporting an 18.159% yield, 6.766% moisture, 2.630% ash, 0.085 Pa.s of viscosity, 26.307% methoxyl, and 64.753% esterification.

Study limitations/implications: The different treatment combinations demonstrate that pH, ash, methoxyl content, and esterification degree vary as a function of the assessed pH and extraction temperatures; while viscosity, moisture and yield were not influenced by these variables.

Findings/conclusions: It is concluded that mango peel is a viable source to obtain quality pectin.

Keywords: Pectin, *Mangifera indica*, pH, extraction temperature, peel.

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INTRODUCTION

The Dominican Republic stands out for its geo-climatic conditions, suitable for fruit trees production. According to the FAO (2020a) mango continues to be one of the most traded tropical fruits worldwide and in 2019, along with guava and mangosteen, accounting for over a quarter of the total world tropical fruit trade. By 2029, global mango, mangosteens, and guavas production is expected to reach 72.8 million tons, annually increasing by 2.9% over the next decade (FAO, 2020b).

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Mango industrialization residues can be utilized. The peel accounts for 7 to 24% (wet basis) of the total weight of these fruits, according to the variety. Most of the peels are disposed of or used for animal feed. However, these constitute a potential source of pectins and polyphenols with market value and are therefore, exploitable (López, Sañudo, Aguilar, Rodríguez and Contreras, 2011; cited by Martínez, 2018).

Pectins are heterogeneous polysaccharides capable of absorbing large amounts of water and gelling, found in most vegetables and fruits, and in greater proportion in peels; specifically, in the primary cell walls and the middle lamella (Chasquibol, Arroyo and Morales, 2008; Picot-Allain, Ramasawmy, and Emmambux, 2020). Pectins production from fruits waste constitutes a reasonable and promising strategy to obtain natural biopolymers while contributing to recycling and promoting sustainable development (Dranca and Oroian, 2018).

There are different techniques for pectins extraction from plant tissues, either using physicochemical or enzymatic procedures. Their yield depends on the operating conditions, such as temperature extraction, extraction time, pH extraction (Pagan and Ibarz, 1999), extraction solvents, and adding chelating agents that help to release pectins from cell walls (Aldana, Aguilar, Contreras, Villaruel, Nevárez, 2011). According to Púa, Barreto, Vallejo, and Ariza (2015), the most influential factor for pectins extraction is temperature, followed by time, whose variations considerably alter yield.

Therefore, the objective of this research was to evaluate the effect of pH and temperature extraction on the physicochemical properties and yield of pectins obtained from mango (*Mangifera indica* L.) peel, cultivar Banilejo.

MATERIALS AND METHODS

This research was conducted at ISA University, Santiago de los Caballeros, Dominican Republic.

Raw material

The main raw material was mango peels, cultivar Banilejo, acquired from local suppliers.

Experimental design

A completely randomized design, in a factorial arrangement, was used to evaluate the effect of pH (1.5, 2.0, and 2.5) and temperature (70, 80, and 90 °C) extraction on the yield and physicochemical properties of pectins. Nine treatments and three replicates were analyzed, a total of 27 experimental units.

Description of the operations

For the extraction of pectin, the methodology described by Cabarcas, Guerra, and Henao (2012) was followed and included the improvement recommendations stated at the end of their research. Mango peels were weighed, washed, and dried in a Quincy Lab Model 30 convection oven at 60 °C to a 10% moisture maximum. They were then crushed in an HC-500 multifunctional grain mill and sieved on a U.S.A. Standard Test Sieve ASTM E-11 106 μm. Five parts of acidulated water, prepared with 37% pure hydrochloric acid, were added to adjust the pH levels. The acid hydrolysis process was then initiated by placing the treatments in a Thermo Scientific model 2835 water bath with manual agitation, for 20 minutes. The resulting material was filtered through a cloth strainer and manually squeezed (pressing through the strainer) to separate the liquid from the solid material. The pectic solution was rapidly cooled (<25 °C to minimize pectins degradation); then 95 % ethanol, corresponding to twice its volume, was added to induce pectin precipitation. It was left to stand for 60 minutes, then filtered and washed with 50% ethanol to eliminate the chlorides present. It was then placed in the drying oven at 60 °C until constant weight. The dried pectin was crushed, sieved, and analyzed.

Variables evaluated

The variables were analyzed at the Food Safety and Industrial Analysis Laboratory (LIAAI) at ISA University. These were: pH (AOAC 943.02, using a Consort R735 pH meter); viscosity (ISI, 2002), assessed with a Brookfield DV2T Viscometer, at 25 °C and 10 RPM and reported in Pa.s); moisture (AOAC 925.09); ash (AOAC 923.03); methoxyl content (Gierschner, 1997); esterification degree (Owens and McCready, 1952); yield: the final weight of the pectin was related with the initial weight of the peel, following the formula:

$$Yield\left(DB\right)=\frac{Pectins\ weight\times 100}{Mango\ \ peels\ weight}$$

Statistical analysis

The data obtained were evaluated by an analysis of variance (one-way ANOVA). Means were separated using Tukey's test ($P<0.05$). Statistix version 8.0 software was employed and the arithmetic means ± standard deviation was used to present the results.

RESULTS AND DISCUSSION

The results of this research are summarized in Tables 1 and 2. Regard the pH of acidified water, the pH of the obtained pectin from mango peel ranged from 3.442 to 3.814, showing significant differences between treatments means (Table 1). Ferreira, Peralta, and Rodríguez (1995) reported the same pattern, where an increase in the pH of pectin was found when using higher pH solutions, which could be because alkalinity is inversely proportional to the level of hydrogen ions: as pH levels increase, H^+ ions decrease, resulting in higher pH (Cabarcas *et al.*, 2012).

The studied temperature variations (70, 80, 90 °C) produced pH from 3.631 to 3.673, with no difference between the treatments means (Table 1). Paredes, Hernández, and Canizares (2015), in their research about pectin extraction from guava hulls at different maturity stages, at 90 and 95 °C, obtained similar values to reported here, corresponding to 4.05-4.25 (green hulls), 3.8-4.37 (pinto hulls) and 3.9-4.1 (ripe hulls), with no significant differences in relation to the assessed extraction temperatures.

The results of pH on the interaction between extraction pH and extraction temperature are shown in Table 2, with values ranging from 3.41 to 3.83, with the treatments being

Table 1. Effect of pH and temperature extraction on the yield and physicochemical properties of pectins from mango (*Mangifera indica* L.) peel cultivar Banilejo.

		pH	**Viscosity (Pa.s)**	**Humidity (%)**	**Ashes (%)**	**Methoxyls (%)**	**Esterification (%)**	**Pectin yield (%)**
pH	1.5	3.442±0.049 c	0.069±0.008 b	6.768±0.681 a	2.880± 0.125a	26.536±0.243 a	66.897±2.235 b	16.188±1.736a
	2.0	3.706±0.046 b	0.080±0.012 ab	6.808±0.389 a	2.744±0.272a	26.434±0.292 a	67.141±2.628 b	17.206±1.131a
	2.5	3.814±0.047 a	0.083±0.013 a	6.574±0.961 a	2.883±0.161a	26.040±0.111 b	70.241±1.160 a	16.142±1.549a
Temperature (°C)	70	3.673±0.148 a	0.073±0.009 a	6.623±0.522 a	3.002±0.194a	26.432±0.306 a	69.012±2.138 a	16.642±1.404 a
	80	3.631±0.171 a	0.080±0.008 a	6.529±0.399 a	2.751±0.132 b	26.226±0.195 a	66.738±2.617 b	16.739±1.382 a
	90	3.658±0.193 a	0.080±0.018 a	6.993±1.003 a	2.754±0.167 b	26.352±0.394 a	68.529±2.534 ab	16.155±1.845 a

Values placed after the±symbol indicate the standard deviation between the means. Different letters in the same column indicate difference ($P<0.05$) between the means of the evaluated treatments.

Table 2. Effect of the interaction of pH and temperature extraction on the yield and physicochemical properties of pectins from mango (*Mangifera indica* L.) peel cultivar Banilejo.

pH	**Temperature (°C)**	**pH**	**Viscosity (Pa.s)**	**Humidity (%)**	**Ashes (%)**	**Methoxyls (%)**	**Esterification (%)**	**Pectin yield (%)**
1.5	70	3.490±0.062 c	0.072±0.012 a	6.452±0.382 a	2.913±0.158abc	26.493±0.313 ab	68.980±2.304 ab	16.559±0.982 a
	80	3.427±0.021 c	0.073±0.003 a	6.677±0.446 a	2.850±0.173abc	26.370±0.092 ab	66.030±2.018 ab	16.128±1.366 a
	90	3.410±0.010 c	0.062±0.002 a	7.160±1.058 a	2.877±0.063abc	26.743±0.144 a	65.680±0.858 b	15.877±2.976 a
2.0	70	3.723±0.045 ab	0.071±0.004 a	7.180±0.127 a	3.043±0.273ab	26.640±0.305 a	67.247±1.519 ab	16.371±0.431 a
	80	3.667±0.051 b	0.085±0.005 a	6.766±0.269 a	2.630±0.035bc	26.307±0.131 ab	64.753±1.799 b	18.159±0.571 a
	90	3.727±0.021 ab	0.085±0.020 a	6.477±0.374 a	2.560±0.121c	26.357±0.367 ab	69.423±2.387 ab	17.086±1.476 a
2.5	70	3.807±0.032 a	0.075±0.014 a	6.237±0.443 a	3.050±0.180a	26.163±0.065 ab	70.810±1.065 a	16.996±2.535 a
	80	3.800±0.078 a	0.081±0.010 a	6.142±0.159 a	2.773±0.029abc	26.000±0.092 b	69.430±1.595 ab	15.930±0.935 a
	90	3.837±0.021 a	0.091±0.013 a	7.341±1.464 a	2.827±0.078abc	25.957±0.032 b	70.483±0.383 a	15.501±0.719 a

Values placed after the ± symbol indicate the standard deviation between the means. Different letters in the same column indicate difference ($P<0.05$) between the means of the evaluated treatments.

statistically different. The highest value (3.83) is above that reported by Beltrán, Díaz and Sáenz (2011), for commercial pectin derived from citrus products (3.5).

As for the viscosity of mango peel pectins, it is affected by variation pH extraction levels, reporting values between 0.069 and 0.082 Pa.s. This shows a clear tendency to decrease as pH is lowered, which is consistent with the research by Alfonso (2010) about the rheological behavior of pectins, where treatments at pH 1.2 and 70 °C resulted in 0.026-0.032 Pa.s, while treatments with higher pH showed higher values.

The viscosity of mango peel pectin was not affected by the variation of the studied temperatures nor with the pH-temperature interaction, so these results will not be discussed.

Tables 1 and 2 show that the independent factors and their interaction do not influence the moisture content of mango peel pectins. Pectin is a substance with a high-water retention capacity, so moisture content is an important parameter for its commercialization, whose maximum limit is officially set at 12% (Food Chemical Codex, 2003). The moisture results in the present research are within the established limits and are similar to those obtained by Ferreira (2007) in pectin from tropical fruits (1.10 to 5.63%); Cabarcas *et al.* (2012) in pectin from banana peels (1-12%) and 11.04 and 2.10% by Vásquez *et al.* (2008), when using pH 2 and 3, respectively.

Another evaluated response variable was the ash content of the pectin. Table 1 shows that, by modifying the pH of the solution, pectin with 2.74 to 2.88% ash was obtained, with no significant differences between their means. These values are similar to those reported by Normah and Hasnah (2000) and Corona, Díaz, Páez, Ferrer, Mármol and Ramones (2012), 3.40% in kiwifruit and 2.04% in parchita peel, respectively.

The extraction temperature influenced the ash content of the studied pectin, between 2.75% to 3.00%. These results differ from those reported by Lliuyacc (2018) in pectin from Serrano tumbo peel, where despite finding no significant differences in the assessed temperatures (60 °C, 64 °C, 70 °C, 76 °C, and 80 °C), found the highest ash content between 76 °C to 80 °C, the lowest at 60 °C.

The interaction between pH and extraction temperature (Table 2) also affected the ash content of pectin, with values from 2.56 to 3.05%; being similar to those obtained by Cabarcas *et al.* (2012) in banana peel pectin (0.9-3.5%) and different from those reported by Lliuyacc (2018), which range from 5.755 to 23.772%.

Methoxyl values were affected by the extraction pH, ranging from 26.04% to 26.54%. It was observed that the lower the pH, the higher the methoxyl content, which differs from the findings of Vásquez *et al.* (2008), where methoxyl content decreased with decreasing pH, showing 1.47% at pH 2.0 (66% lower compared to that obtained at pH 3.0). These data are consistent with the findings of Pagan and Ibarz (1999), who observed that high temperatures and low pH seem to favor pectin demethylation.

The studied temperature levels resulted in pectins with values between 26.226 and 26.432% of methoxyl, no significant differences between the means are observed. As the extraction temperature increases, the methoxyl content decreases. Lliuyacc (2018) indicates that this may be because increasing the time and extraction temperature increases yield, but the methoxyls percentage reduces due to increased esters hydrolysis in the methoxylated carboxyl groups and directly relates to the pectins quality.

By evaluating the interaction between pH and extraction temperature, methoxyl values ranging from 25.957 to 26.743% were found (in the pH 1.5 treatment at 90 °C), showing statistical differences. These findings are concurring with those by Alfonso (2010), who showed that the temperature-pH interaction produces a higher percentage when the temperature is at maximum and pH at minimum. The results in this research were superior to those obtained by Cabarcas *et al.* (2012) and Vásquez *et al.* (2008) in banana peel pectin and to those obtained by Ferreira (2007) in tropical fruit pectins, being these of low methoxyl in their respective research.

Table 1 also presents the effect of the pH of the solution on the pectin's esterification degree (ED), where values from 66.897 to 70.241% were observed, with a significant difference between the evaluated means. These results show that increasing the pH produces an increase in ED, which is consistent with the values obtained by Flores, Mariños, Rodríguez, and Rodríguez (2014), who, by increasing the pH from 2 to 3, obtained a 53.8 to 65% increase in ED. They also concur with the data obtained by Rodríguez and Rodríguez (2018) in their research on the concentration of citric acid in the quality of pectin's, where, when using 0.2% obtained 86.51% ED, while when using high concentrations, the results were lower.

The ED of mango peel pectins is also affected by the extraction temperature, resulting in values between 66.738 and 69.012%. The pectin with the highest ED was the one extracted at 70 °C, probably because moderate temperatures allow reaching higher esterification degrees.

The results of the ED in relation to the pH-extraction temperature interaction are shown in Table 2, with values ranging from 64.75 to 70.81%, the treatments being statistically different. These data are lower than those reported by Ferreira (2007) for tropical fruit pectins (84.2-93.5%). Except for the treatments corresponding to pH 2, by combining low pH values and temperatures, better results are obtained. This is consistent with the research by Flores *et al.* (2014), whereby using pH between 1 and 1.5 and temperature between 70 and 80 °C, the ED was higher than 80%, concluding that low pH and moderate temperatures are needed to achieve high ED, indicating good pectin quality (Baltazar, Carbajal, Baca and Salvador, 2013).

The yield of mango peel pectins was not affected by the pH or extraction temperature (Table 1), nor by the combination of these factors (Table 2). Regard the pH, the values were between 16.142 and 17.206%. There are divergences because of the effect of the extraction pH on the results of the final pectin, since research, such as that of Flores *et al.* (2014) showed a tendency that at constant extraction times, the decrease in pH produces an increase in the yield of extracted pectin. Still, Lliuyacc (2018) reports that the higher the pH, the higher the yield. In the present research, the highest yield was obtained at pH 2.

The evaluated temperatures resulted in yields between 16.155 and 16.739%, without significant changes; being numerically higher the one obtained at 80 °C. This concurs with that reported by Flores *et al.* (2014), in French lemon peel, who obtained the best values at temperatures between 73 and 80 °C. According to Púa *et al.* (2015), the most influential factor in pectin extraction is temperature, since high temperatures increase the hydrolysis of protopectin (insoluble) bonds, converting it into water-soluble pectin.

Regard the interaction between pH and extraction temperature, yields from 15.501 to 18.159% were found. Although the means are statistically equal, the treatment with the highest yield (18.159%) was the one extracted at pH 2.0 and 80 °C. These results are close to the highest levels of commercial pectin extraction, such as that from sunflower, with 25% obtained by Rojas, Perea, and Stashenko (2008), cited by Baltazar *et al.* (2013). Similarly, Cabarcas *et al.* (2012) obtained a maximum yield on a dry basis of 23.06% when extracting pectin from banana peels at pH 1.5 and 80 °C and its minimum yield at a temperature of 73 °C and pH 2.7.

CONCLUSIONS

The results show that pH, ash, methoxyl content, and esterification degree vary according to the applied pH and extraction temperatures, while viscosity, moisture, and yield are not influenced by these variables. The best results were obtained when using the pH*temperature combination of 2.0*80 °C. No difference was observed between the yields of the evaluated treatments, but numerically, the highest yield was obtained when using the pH*temperature combination 2.0*80 °C, which corresponds to the intermediate assessed values.

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