EXTRACTION OF PECTIN FROM MACAÚBA PULP CAKE (*ACROCOMIA ACULEATA*) BY ACETIC AND CITRIC ACID: A COMPARATIVE STUDY

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ABSTRACT

Pectin is an important polymer for the industrial sector composed of chains of galacturonic acid and methoxylated galacturonic acid, frequently employed as a stabilizer, emulsifier and gelling agent. The purpose of this study was to investigate the extraction of pectin from macaúba fruit pulp cake by using acetic acid and citric acid. Pectin extraction was carried out with variable pH (2.0 - 3.5) for acetic acid, pH (2.2 – 3.8) for citric acid and temperature (70 - 90ºC) by using a central composite rotatable design. The pectin yields were 5.44 to 9.40% (m/m) for acetic acid and 4.76 to 11.55% (m/m) for citric acid. The pectin purities were 19.03 to 80.36% (m/m) for acetic acid and 45.41 to 66.19% (m/m) for citric acid. Commercial citrus pectin has an average of purity of 50% (m/m) and the pectin from macaúba fruit pulp cake could be another candidate for commercial pectin.

Keywords: Pectin. Extraction. Citric acid. Acetic acid. Macauba.

1 INTRODUCTION

In the food industry, pectin is a high-value functional component frequently employed as an emulsifier, gelling agent and stabilizer[1]. Based on the degree of esterification (DE) of pectin, the polymer is categorized into high methoxy pectin (DE > 50%) and low methoxy pectin (DE < 50%).[2] The chemical properties of pectins are related to their different DE. Citrus and apple residue is the primary source of pectin in the market. According to Data Bridge Market Research, the global pectin market was USD 1,062.27 million in 2022 and there is a forecast of an increase of USD 1,866.52 million for the year 2030. Therefore, it is important to expand the source of pectin. The component of pectin depends on the extraction conditions and plant source; for that reason, it is important to investigate another source of pectin and the objective of this work was to compare acid extraction of pectin from macaúba pulp cake by using acetic (AA) and citric acid (CA).

2 MATERIAL & METHODS

Extractions of pectin were carried out in a 125mL Erlenmeyer flask, containing 100mL acid solution consisting of 6% (m/v) of macaúba pulp cake in different pH and temperatures. Pectin extraction was carried out with variable pH (2.0 - 3.5) and temperature (70 - 90ºC) according to the experimental design shown in Table 1. The results of analysis of variance (ANOVA) and figures were obtained by using the software Statistica[3].

Table 1- The matrix of the central composite rotatable design with the real and coded values (in parenthesis) for the responses pectin yields extracted by acetic and citric acid

Run	pH	Temperature (ºC)	Pectin yield % (m/m)		Pectin purity % (m/m)	
			Acetic acid	Citric acid	Acetic acid	Citric acid
1	2.5[a] 2.2[b] (-1)	70 (-1)	0	8.03	0	45.41
2	2.5[a] 2.2[b] (-1)	90 (+1)	9.40	11.55	80.36	59.03
3	3.5[a] 3.8[b] (+1)	70 (-1)	6.76	8.81	19.03	66.19
4	3.5[a] 3.8[b] (+1)	90 (+1)	5.44	4.76	43.31	53.56
5	3 (0)	80 (0)	6.99	9.03	50.19	-
6	3 (0)	80 (0)	6.63	7.69	-	-
7	3 (0)	80 (0)	5.79	10.3	-	59.45

[a]Acetic acid [b]Citric acid

The yields of pectins were determined according to equation (1), where *m* is the lyophilized pectin mass (g) and M is the pulp mass of macaúba (g).

$$R = \frac{m}{M}\, x100\% \quad (1)$$

The galacturonic acid content of pectins were determined according to the method described by Bitter and Muir.[4] Five runs were selected for the determination of galacturonic acid from each acid extraction of pectin. The galacturonic acid contents were expressed as a percentage (g of galacturonic acid/100 g of pectin). Table 1 shows the purity of the extractions.

3 RESULTS & DISCUSSION

Referring to the acetic acid extraction batches, the highest yield (9.4%) of pectin was obtained in assay 2 (pH 2.5 and 90ºC) and the lowest yield (0%) was obtained in assay 1 (pH 2.5 and 70ºC), both at the same pH and different temperatures. It was observed no pectin extraction at pH 2.5 and 70ºC, however, there was pectin extraction (6.76%) at pH 3.5 and 70ºC (Table 1 and Figure 1C). This assay was repeated and the same result was observed. The yields observed in the triplicate of central point assays (pH=3.0 and 80ºC) were relatively close, obtaining an average yield equivalent to 6.47%. The most statistically significant effects were temperature ($p<0.05$) and the combination of temperature and pH ($p<0.05$), since, according to the Pareto's chart (Figure 1A), these effects are significant.

Regarding to the pectin purity extracted by acetic acid, the highest purity (80.36%) of pectin was obtained in assay 2 (pH 2.5 and 90ºC), the same conditions for the highest yield (9.4%). The lowest purity (19.03%) was obtained at pH 3.5 and 70ºC. These results showed that the high yield and purity of pectin is dependent of the combination of low pH 2.5 and high temperature 90ºC. In the batches 3 and 4, at the same pH 3.5 and temperatures 70ºC and 90ºC, pectin yield is quite similar, however, pectin purities are 19.03 and 43.31%, respectively. This result, reinforces that low temperature can influence the degree of purity, since the assays 3 and 4 were subjected to the same pH 3.5 and different temperatures.

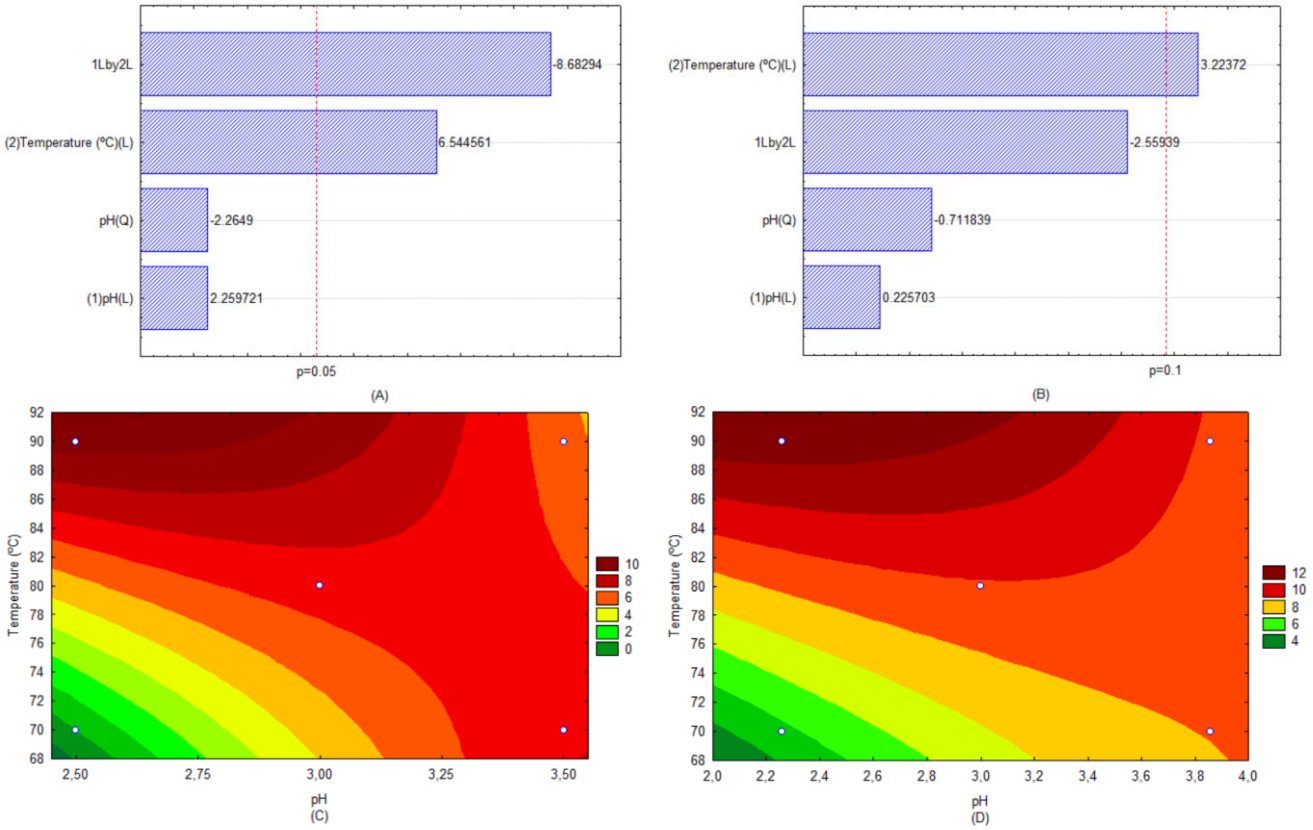

Figure 1 Pareto charts of pectins extracted by acetic acid (A), citric acid (B) and contour diagrams of pectins extracted by acetic acid (C), citric acid (D) for pectins yield as a function of temperature (ºC) and pH.

The pectin yields obtained by citric acid extraction are higher than the pectin extracted by AA (Table 1). The highest yield (11.55%) of pectin by CA was obtained in assay 2 (pH 2.2 and 90ºC) at same conditions of pectin from AA (Figure 1D). The lowest yield of pectin (4.76%) by CA was obtained in assay 4 (pH 3.8 and 90ºC). The assays 2 and 4 were carried out at different pH (2.2 and 3.8) and at the same temperature 90ºC. These results showed that the yields of pectin extracted by CA at high temperature (90ºC) were significatively different depending of pH. On the other hand, the yields (8.03 and 8.81%) of pectin extracted by CA at low temperature (70ºC) and different pH (2.2 and 3.8), respectively, were not significatively different. The effect of temperature in the pectin extraction by CA was positive (3.785) and it was statistically significant at $p<0.1$ (Figure 1B). This result shows that yield of pectin extraction by CA increases with temperature increasing.

The results indicate that sample 3 had the highest galacturonic acid content, with 66.19 g of galacturonic acid per 100 g of pectin, when using citric acid extraction. This experiment was conducted under temperature conditions of 90°C and pH 4 during pectin extraction. Table 1 shows that the yield was 8.81, suggesting that the yield is not directly related to the purity of the pectin.

Regarding to the pectin purity extracted by CA, the highest purity (66.19%) of pectin was obtained in assay 3 (pH 3.8 and 70ºC), and yield (8.1%). In the point of view to reduce the acid and energy consumption, this result is interesting because in this assay was used lower amount of acid and less energy. The lowest purity (45.41%) was obtained at pH 2.2 and 70ºC. These results of purity of pectin extracted by CA are not in accordance to results of purity of pectin extracted by AA.

The highest yields of pectin extraction by AA and CA were obtained in lower pH 2.5 (AA) and 2.2 (CA) and this condition for both acids was significative. Regarding to the range of purity pectin extracted by CA (45.41 to 66.19%) and by AA (19.03 to 80.36%) and the average of purity pectin from AA (48.22%) and from CA (56.73%), more studies are necessary to investigate the effects of pH, acids and temperatures in the yields and purities of pectin.

In a previous study, Vilaça[5] extracted pectin from macaúba pulp cake using citric acid under conditions of pH 2 and 90°C, obtaining a higher yield of 11.61%. Similar result (11.55%) was obtained in the assay 2 (pH 2.2 and 90ºC). Therefore, the results of this study show a remarkable similarity with the results found in the literature. Furthermore, still referring to Vilaça[5], the pectin purity found by CA was 88.85% and by AA 85.42%, which results were higher than results found in this work (66.19%) for CA and (80.36%) for AA. This discrepancy can be attributed to the difference in the batch scales used in the previous study (1000mL), compared to the scale used in the present study (100mL).

4 CONCLUSION

It is concluded that for the extraction of pectin from macaúba pulp cake by using (CA) was 11.55% and (AA) was (9.4%). Highest purities of pectin extracted by CA (66.19%) was at (90°C and pH 3.8) and pectin extracted by AA (80.36%) was at (90°C and pH 2.5). The yields and purities of pectin samples were dependent of pH, temperature and acid type. These finds could corroborate to use pectin from macaúba fruit pulp cake as another candidate for commercial pectin.

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