

ACID AND ENZYMATIC EXTRACTION OF PECTIN FROM MACAÚBA PULP CAKE

Robert W. R. Silva¹, Carlos H. S. R. Campos¹, Guilherme A. Silva, Mariana A. E. Silva¹, Simone P. Favaro²,
Alessandra C. Vilaça¹, Enio N. O. Junior¹, Ana M. Oliveira^{1*}

¹Department of Chemistry, Biotechnology, and Bioprocess Engineering, Federal University of São João del-Rei, Ouro Branco, Brazil.

²Empresa Brasileira de Pesquisa Agropecuária - Embrapa, PqEB, W3 Norte - Asa Norte, Brasília, DF, Brazil.

*amaria@ufsj.edu.br

ABSTRACT

Macauba (*Acrocomia aculeata*) is an emergent oleaginous crop of palm tree from South America that presents a high potential for the sustainable development of new food, pharmaceutical and domestic products. The pulp is an edible part of the macauba fruit and it has high-quality oil content, rich in unsaturated fatty acids, such as oleic and linoleic acids. Pulp cake is a byproduct from macauba pulp after oil extraction and it is mainly composed of hemicellulose, pectin and cellulose. The purpose of this study was to investigate the extraction of pectin from macaúba fruit pulp cake by using hydrochloric acid and enzyme (pectinase). Pectin extraction was carried out with variable pH (2.0 – 4.0) and temperature (50 – 80°C) for hydrochloric acid and enzyme mass (0.02 – 0.06g) and time (30 – 90 min) for pectinase, by using a central composite rotatable design. The pectin yields were 7.37 to 12.10% (m/m) for hydrochloric acid and 8.72 to 12.16% (m/m) for enzymatic extraction. The pectin purities were 23.66 to 49.12% (m/m) for hydrochloric acid and 8.72 to 12.69% (m/m) for enzymatic extraction. Macauba pulp cake emerges as an alternative source of pectin and more studies should be developed to characterize pectin samples extracted from this source.

Keywords: Experimental design. Pectin. Galacturonic acid.

1 INTRODUCTION

The macauba palm (*Acrocomia aculeata*) is a species native from Americas, with widespread distribution throughout the Brazilian cerrado. Its product, known as macauba nut, consists of several parts, including husk, pulp, endocarp, and kernel. The plant has drawn interest due to its economic potential in the production of high-value oils, marketed in various sectors. When the oil from the fruit pulp is extracted, cake is generated as a co-product, which, due to its high levels of fiber, proteins, minerals and lipids, is currently used as animal feed¹. However, previous studies showed that it is possible to get pectin from macauba pulp cake².

Pectin is a complex of polysaccharides with wide application in the food, pharmaceutical, and cosmetic industries. It is found in the cell wall of plants, exhibits properties such as stabilizing, texturizing, and thickening, being used in a variety of products such as juices, jams, ice creams, tablets, moisturizers, and tonics. Despite its widespread presence in plant tissues, commercial sources of pectin are generally limited to orange peel or apple pomace, due to requirements regarding its purity and gel-forming capacity. The aim of this study was optimize the pectin extraction process by acid and enzymatic means and compare the purity of the two methods.

2 MATERIAL & METHODS

To optimize the extractions, 2² factorial designs were used with 3 central points. In acid extraction, the variables evaluated in the process were extraction pH (2, 3 and 4), adjusted with 0.02 mol L⁻¹ hydrochloric acid solution, and temperature reaction (70, 80 and 90 °C). In enzymatic extraction, it was evaluated mass of Sigma-Aldrich pectinase enzyme (EC 3.2.1.15) (0.02, 0.04 and 0.06 g) and reaction time (40, 60 and 80 min). For extraction, 6.0 g of macerated pulp cake will be added to 100 mL of buffer solution or acid solution and it occurred according to the parameters studied in the experimental planning. After extraction, the supernatant was separated from the solid part by centrifugation. The supernatant was treated with ethanol to precipitate the pectin, which was then collected, lyophilized and weighted to determine extraction yield. In addition, the concentration of galacturonic acid was determined. Statistica® software 5.0 was used to data processing.

3 RESULTS & DISCUSSION

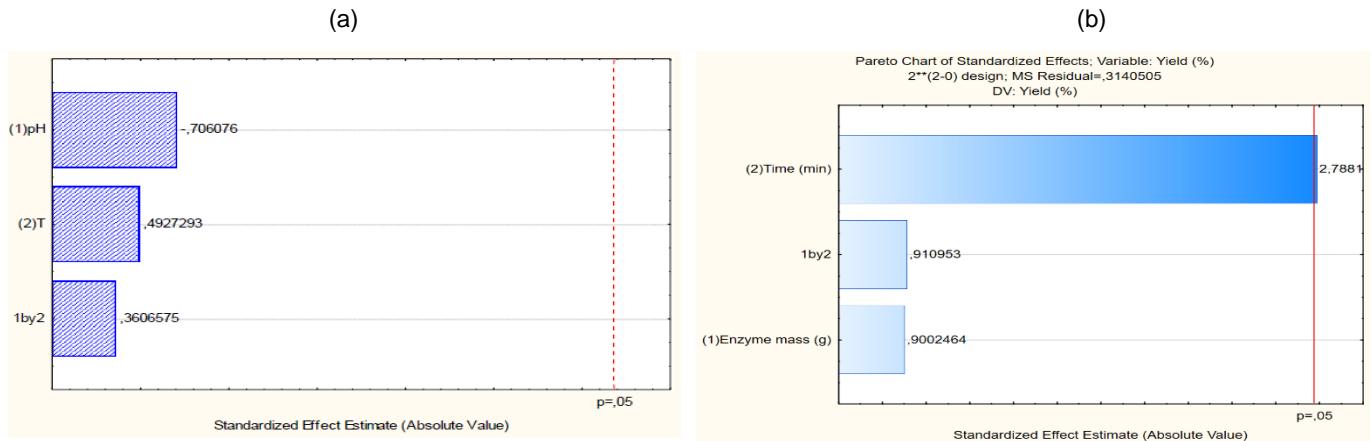
The experiments of acid and enzymatic hydrolysis were conducted following the factorial design outlined in Table 1. Through the tables, it can be observed that the highest yield for acid hydrolysis was achieved in experiment 6 (pH 3.0 and temperature 65°C), where the low pH of the solution combined with the high reaction temperature led to greater pectin release. As for enzymatic hydrolysis, the highest yield was obtained in experiment 4, with conditions of 0,06 g of pectinase enzyme in 90 minutes of extraction. It is noted that the yield increases with the increase in enzyme mass used and extraction time.

Table 1 Experimental conditions of design experimental used in acid and enzymatic extraction of macauba and yield galacturonic acid concentration obtained

Experiment	Acid Extraction				Enzymatic Extraction			
	pH	Temperature (°C)	Yield (m/m %)	Galacturonic acid (%)	Enzyme mass (g)	Time (min)	Yield (m/m %)	Galacturonic acid (%)
1	4	80	9.05	38.12	0.02	30	5.13	8.72
2	4	50	7.37	23.66	0.02	90	6.18	12.69
3	2	80	9.73	43.03	0.06	30	5.12	9.61
4	2	50	9.47	33.90	0.06	90	7.20	11.04
5	3	65	11.37	48.78	0.04	60	5.99	12.16
6	3	65	12.10	44.07	0.04	60	6.77	11.61
7	3	65	10.74	49.12	0.04	60	6.94	11.58

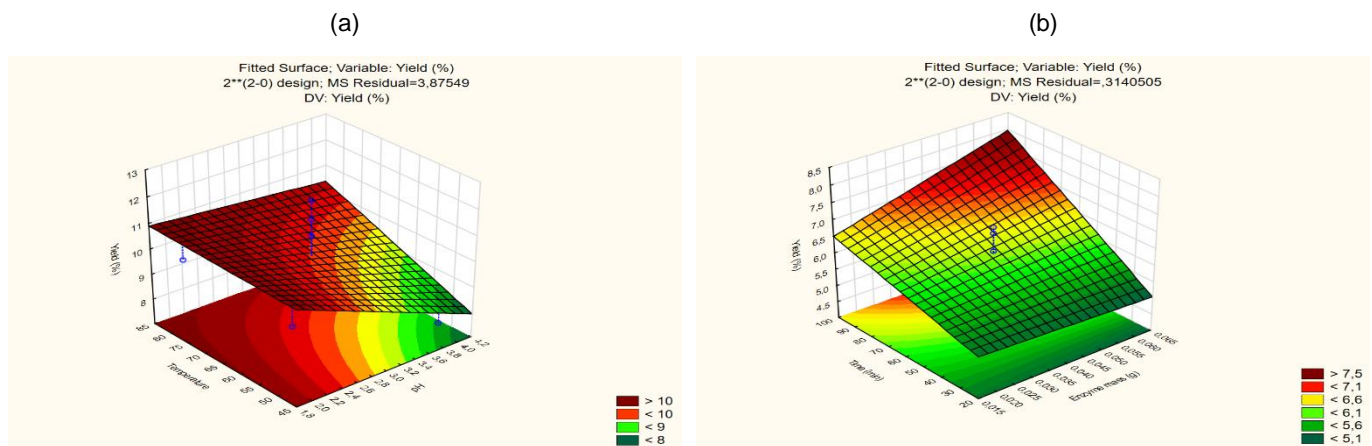
In Figure 1 and 2 Pareto diagrams for the experimental designs are shown.

Figure 1: Pareto diagram for acid hydrolysis (a) and enzymatic hydrolysis (b).



Upon observing the Pareto chart of the experimental design conducted, it was found that none of the studied parameters (pH and temperature) were significant in the acid hydrolysis of macauba cake, at a 95% confidence level, within the range used for each parameter. This fact was confirmed through the response surface (Figure 2). For enzymatic hydrolysis, it was found that time is a significant factor in pectin extraction for the same confidence interval employed. In Figure 3 illustrate the response surfaces according to the variation of the parameters analyzed in the two extraction methods.

Figure 2: Response surface for acid hydrolysis (a) and enzymatic hydrolysis (b).



The galacturonic acid content can divide pectin into different degrees of esterification, with those of high degree of esterification having above 50% of their carboxylic groups esterified. Otherwise, pectin is classified as low degree of esterification. The galacturonic acid content directly affects the characteristics of the obtained pectin and its applications. Therefore, it is highly relevant to determine the galacturonic acid content in pectic samples.

The yields (7.37-12.10% m/m) and purities (23.66-49.12% m/m) of pectins from acid extraction were higher than the yields (23.66-49.12% m/m) and purities (8.72-12.69% m/m) to pectins from enzymatic extraction. These preliminary results showed that the conditions used in the acid extraction were better compared to enzymatic extraction. However, it is important to evaluate other variables in different ranges and their influence in the yield and purity of pectin.

4 CONCLUSION

Based on the results, it is concluded that pH and temperature were not significant in the mass yield of acid hydrolysis of macauba cake with hydrochloric acid in obtaining pectin, within the intervals studied. However, the condition that yielded the highest mass also exhibited the highest content of galacturonic acid. In enzymatic extraction was observed that only time was significative in the mass yield. Regarding the concentration of galacturonic acid, a low concentration was observed in the enzymatic extraction, probably due to the enzyme hydrolysis process. So, the results showed that macauba pulp cake extraction is a relevant role as a potential alternative source for obtaining pectin, standing out as a promising raw material for the industry.

5 REFERENCES

1. GONCALVES, S. A.; VILAÇA, A. C.; ANDRADE, M. H. C.; QUIROGA, F.; LANCETTI, R.; CANALLIS, M. S. B.; RIBOTTA, P. D. J. Food Process. Pres. v. 45, p. 1-14, 2021.
2. VILAÇA, A. C. 124f. Tese (Doutorado em Engenharia Química) –Universidade Federal de Minas Gerais, 2022.
3. MUNHOZ, C. L.; SANJINEZ – ARGANDOÑA, E. J.; SOARES JÚNIOR, M. S. Food Sc. Technology. v. 30, n. 1, p. 1-7, 2010.
4. OLIVEIRA JUNIOR, E. N.; SANTOS, C. D.; ABREU, C. M. P.; CORRÊA, A. D.; SANTOS, J. Z. L. Rev. Bras. Frutic. v. 26, p. 410-413, 2004.
5. MCCREADY, R. M.; MCCOMB, E. A. Anal. Chem. v. 24, n. 12, p. 1986-1988, 1952.
6. BITTER, T.; MUIR, H. M. Anal. Biochem. v. 34, p. 330-334, 1962.

ACKNOWLEDGMENTS

The authors acknowledge FAPEMIG for financial support.