OPTIMIZATION OF THE PECTIN EXTRACTION PROCESS FROM RENEWABLE RESOURCES

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Introduction

The use of biodegradable polymeric materials, which are decomposed by microorganisms or the action of sunlight, has received increasing attention in the development of research that enables their use from renewable resources. These substances have a series of applications in the most diverse industrial sectors and have significant scientific interest in the areas of food, cosmetics, textiles, pharmaceuticals and more recently in bioengineering. Among the many biopolymers found in nature and with applications in industrial segments, polysaccharides stand out, such as cellulose, pectin, chitin, chitosan and corn starch.[1]. This work aims to optimize the pectin extraction process, using more economically viable extractive methods and obtaining better quality pectin with physical properties. chemical and functional components compatible with their real application.

Material and Methods

Pectin was extracted from citrus fruits purchased randomly from grocery stores and fruit and vegetable stores in the cities of Rio de Janeiro and Niterói, using citric acid. Since a series of factors interfere with the extractive processes, a response surface analysis methodology was used using the central composite arrangement (Central Composite Design –CCD) as experimental planning for data collection. Therefore, 3 independent variables regarding pectin extraction were evaluated: pH, citric acid concentration and reaction time, keeping the temperature at $90 \pm 5^{\circ}C$. The variables were analyzed at five levels (upper limit (+1), lower limit (-1), axial points (+α and –α) and central points (0)) evaluating the extraction yield, the galacturonic acid content and the degree of esterification (dependent variables) of the pectin obtained by titration and infrared spectroscopic analysis methods. The results were also subjected to statistical analysis using Statistica software version 14.0 (Statistic Inc., USA) to evaluate the factors with the greatest effect on yield and type of pectin.[2]

Results and Discussion

The results found for pectin yield showed a wide variation, however, it is interesting to note that they were all equivalent to the experimental design, which leads us to believe in the possibility of using citrus fruit peels as biological waste to obtain pectin from organic acids. Even so, in order to minimize errors inherent to the process, these results were subjected to a statistical analysis of variance, individually for each fruit. The results obtained from the statistical analysis for the 3 fruits generated an R2 with an acceptable value for approximating the data for all samples. The Pareto Chart demonstrates that the independent variables pH (1) and citric acid concentration (2) had a positive effect on yield. This can also be observed in the linear effect (L) of these variables. The same behavior can be observed for the pH-concentration (1Lby2L) and pH-time (1Lby3L) relationships, with values always $p<0.05$. As it does not present a p-value below 0.05, it can be stated that the independent variable time (3) did not generate a significant effect on performance, just as the quadratic effects (Q) also did not present a significant effect. These relationships could also be verified in the response surface graph, in which it is possible to observe the growth in yield according to higher pH levels and citric acid concentration.

Although some findings are differents with most of the literature, such as the values found by Fernandes and collaborators (2014)[3], when extracting pectins from carrot peels, in which they obtained the time variable with a positive linear effect on the yield and pH as a negative linear effect; It is important to note that the pH range worked was only in the acidic range (1.3 to 2.7), different from what was proposed in this experimental design. Pereira and collaborators (2016)[4] extracted pectin from pomegranate peels with citric acid, using pH, temperature and extraction time as independent variables, and also identified a negative linear effect of pH in relation to yield; concluding that extreme conditions of the tested variables result in better yield and lower methoxylation content. More recently, the study carried out by Muñoz-Almagro and colleagues (2021)[5] found considerably higher yields when extracting pectin from wild fruits with citric acid compared to enzymatic extraction; 8.9% and 3.9% respectively, in pectin obtained from strawberries. It is possible to observe, therefore, that there really are considerable variations in the yield of pectin extraction processes, which impacts the viability of an industrial-scale process. It is important to point out that higher pH values (pH > 5) are less explored in acid extraction. Furthermore, there is the possibility of the interaction of citric acid with the ethanol used to separate the pectin gel, indicating that precipitation or formation of interferents occurred during extraction, resulting in a possible Fischer Esterification.[6] The methoxyl content behaves very differently with values between 1.55%-9.92% and analyzed in a regression model all presented results with an acceptable R2. It is important to consider that MeO% is not a single factor to characterize the composition of the pectin obtained, but may be correlated with the degree of esterification. It is interesting to note which factors will impact the result obtained, as this can be a useful parameter to optimize obtaining pectins of different degrees of esterification.[7]

Conclusion

It was possible to extract pectin from citrus fruit peels using an organic acid, however the evaluation of the parameters of this extraction requires further tests in order to establish which parameters significantly interfere with the process yield. pH appears to be the determining factor as an independent variable for the yield of the pectin extraction process and it should be between 1.3-2.7 for best results, which can be corroborated by scientific literature.

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