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WATER, METHANOL AND ACETONE MIXTURE FOR OPTIMIZED EXTRACTION OF PHENOLIC COMPOUNDS FROM PALM FLOUR USING A SIMPLEX CENTROID MIXTURE DESIGN

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ABSTRACT

There is a diversity of phenolic compounds in plant foods, being divided between phenolic acids (hydroxybenzoic acids - gallic, protocatechuic, *p*-hydroxybenzoic, vanillic and syringic acids - and hydroxycinnamic acids - *p*-coumaric, ferulic, caffeic, sinapic, chlorogenic and cinnamic acids), flavonoids (flavonols, flavones, catechins, flavanones, anthocyanidins and isoflavonoids) and non-flavonoids (stilbenes, chalcones, coumarins, lignans and tannins). These compounds have a wide range of structural forms (aromatic rings and organic carboxylic acid), with functional chemical groups (hydroxyls), distinct polarities and a wide range of combinations, resulting in compounds called polyphenols. These compounds are found in conjugated polymeric through esterification or etherification to macro components or in free form. The free form is called total soluble phenolic compounds (TSPC). Identifying the ideal extracting solution is of great relevance to extracting, identifying, and quantifying these polyphenolic compounds (TSPC) with bioactive properties. The most used solvents are methanol, ethanol, acetone, water, and propanol, among others, in isolation or combined. This work aimed to apply a simplex-center mixing design to select the ideal extracting solution for the palm flour (*Opuntia ficus-Indica* (L.) Miller) obtained by microwave drying (720 W, up to lower moisture 15 %). The solvents studied were water, methanol, and acetone. Extracting solutions were added to flour at 1:9 (w/v) and kept under continuous agitation at 250 rpm at 20°C for 16 hours, followed by centrifugation (2500 *g* for 10 min) and with10 mL final volume adjustment. The Folin-Ciocalteu spectrophotometric method (a mixture of phosphomolybdate and phosphotungstate) was used to determine the absorbance (in 750 nm) of the samples, being performed in quadruplicate. The absorbance results of the tests ranged from 0.103 ± 0.007 and 0.448 ± 0.009, and the largest absorbances match the best extractions. This condition was verified in greater intensity through the binary components between water and acetone (β_{13} = 0.7894, p-value = 0.002) and methanol and acetone (β_{23} = 0, 4157, p-value = 0.045). Regarding pseudo components, the greatest effect was observed by methanol (β_2 = 0.4166, p-value <0.001), followed by water (β_1 = 0.3654, p-value <0.001) and acetone (β_3 = 0, 1245, p-value = 0.014). Experimental results were explained by a R^2 = 0.8852, p-value = 0.002 and F_{cal}/F_{tab} ratio of 3.28. Experimental optimization indicated the desirability of 1.00 and the proper mixture for TSPC extraction was 0.5 water, 0.1667 methanol and 0.3333 acetone. It was observed that acetone extracted lower TSPC contents in relation to methanol, indicating the need for greater acetone concentration in the extracting solution. When evaluating the validity of the predictive mathematical model, the predicted value was 0.448 and the experimental value at the optimized point was 0.421 ± 0.013, generating a relative deviation of 6.41 %. Thus, it is concluded that the applied experimental design is an important tool to optimize TSPC extraction, enabling the saving of chemical reagents and favoring the quantification of these phytochemical compounds.

Keywords: Desirability, extraction, optimization, phytochemicals.

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