



Área: ANA

## EVALUATION OF VA-MSPD FOR THE PESTICIDES DETERMINATION IN RAW AND COOKED TAPIOCA GUM SAMPLES

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### Highlights

VA-MSPD in combination with GC-MS was developed for pesticides determination in tapioca samples; The method was validated; The next step will be the optimization of the method in cooked tapioca gum samples.

### Abstract

Pesticides are synthetic chemical substances widely used in Brazil and worldwide to control agricultural pests, may leave residues on foods and their derivatives, such as cassava and, in addition, tapioca gum<sup>1 2</sup>. However, the country still lacks specific legislation for regulating these residues, which makes the development and application of reliable analytical methods essential to ensure food safety. In this context, vortex-assisted matrix solid-phase dispersion (VA-MSPD)<sup>3</sup>, in combination with gas chromatography–mass spectrometry (GC-MS), was evaluated for the determination of seven pesticides in raw tapioca gum samples. The method was evaluated through solid support optimization, where the reused C18 sorbent provided recovery rates between 68.3% and 122.8%, with RSD < 10% for the most analytes. Once the method presented an acceptable recovery range, it was validated according to INMETRO and ANVISA parameters. The method showed limits of quantification between 0.005 and 0.01 mg kg<sup>-1</sup>, calibration curves with correlation coefficients above 0.9997, and accuracy and precision assessed in terms of repeatability and intermediate precision, with recoveries ranging from 60.7% to 122.8% and RSD ≤ 12 for most analytes. Additionally, robustness was evaluated by applying the method to other cassava-derived food products, such as flour, farofa, sour starch, and granulated tapioca samples, with recoveries within the acceptable range. This demonstrates that, beyond all the advantages of the sample preparation technique, the method is also robust. Finally, the VA-MSPD was applied in different raw tapioca gum samples purchased from local markets in the cities of Rio Grande – RS and São Vicente – SP. However, none of the pesticides studied were detected in the analyzed samples. Even so, the method showed significant advantages compared to conventional extraction techniques, with key benefits including operational simplicity, significant reduction in solvent consumption, lower waste generation, and robustness, confirming its applicability not only to tapioca gum but also to other solid and/or semi-solid cassava-derived products. The next phase of this study is to reproduce this method in the cooked tapioca gum samples, and after that, it will also be validated. Thus, it will be possible to state that this is the first application of VA-MSPD in raw and cooked tapioca gum samples.

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