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**VORTEX-ASSISTED LIQUID-LIQUID MICROEXTRACTION COMBINED WITH HPLC-MS
FOR THE DETERMINATION OF PESTICIDES IN WATER**T. M. Y. Ferraz¹, I. Martins², L. Cavalcanti³, E.C. Albuquerque Júnior⁴, P.T.S. Silva¹¹Embrapa Semiárido, Petrolina - PE, Brasil²*Instituto Federal de Educação, Ciência e Tecnologia do Sertão Pernambucano, Petrolina-PE, Brazil*³Universidade do vale do São Francisco, Juazeiro – BA, Brazil⁴ Instituto Tecnológico do Estado de PernambucoPaula.silva@embrapa.br

The extensive use of pesticides on agricultural plantations can lead to the accumulation of these compounds in soil and water. Extraction and analytical methods have been developed to and determine the concentrations of such compounds in these matrices. In the literature, the most widely employed extraction methods are liquid-liquid extraction and solid phase extraction. However, these methods are labor intensive and use large amounts of organic solvents.¹ Therefore, an innovating techniques denominated vortex-assisted liquid-liquid microextraction (VALLME) has piqued the interest of researchers due to the reduction in the amount of solvent, low cost, easy operation, good recovery and pre-concentration of the analyte and fast extraction.¹ This method is based on the partition of analytes between two immiscible liquid phases: an aqueous phase and organic phase in the case of vortex dispersion.¹ Thus, the aim of the present study was to develop and validate the VALLME technique and analysis, employing LC-(ESI)MS in the determination of 10 pesticides in water. The following were the optimized conditions for the extraction of the pesticides using VALLME: sample volume: 10 mL; extractor solvent volume: 0.5 mL of dichloromethane; vortex extraction time: 3min; and 3.5 g of NaCl). The extracts were analyzed using LC-(ESI)MS for water samples. All compounds studied (acetamiprid, carbendazim, carbofuran, dimethoate, imidacloprid, ioxynil, metalaxyl, methomyl, paclobutrazol and thiamethoxam) exhibited good linearity between 2 and 50 ug L⁻¹, with r² ≥ 0.99 and the limits of quantitation < 0.07 ug L⁻¹. Spiked water samples (0.4 and 5 ug L⁻¹) showed recovery results ranging from 95.4 to 110% and relative standard deviation < 5%. Pesticides were found in the 16 of the 28 water samples from drainage ditches in agricultural areas, with concentrations ranging from 0.08 to 50.28 ug L⁻¹. Therefore, the proposed method is efficient for the routine analysis of water samples.

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