



Microwave-assisted extraction induced by emulsion breaking based on NADES for elemental determination in edible oils by ICP OES

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Natural Deep Eutectic Solvents (NADES) are low-cost new customizable safe solvents with high extractability.¹ The application of NADES in chemical analysis provides promising results, especially in the development of Green Analytical Chemistry (GAC) methods. In this work, a new Microwave-assisted Extraction Induced by Emulsion Breaking (MW-EIEB) based on Natural Deep Eutectic Solvent was developed aiming at the elemental determination by inductively coupled plasma optical emission spectrometry (ICP OES). NADES composed of carboxylic acids and choline chloride were prepared (microwave heating)² and evaluated for extractability, in addition to molar ratio and water addition. In this approach, the oil samples were emulsified with a surfactant/extractant solution to form a stable water-in-oil emulsion.^{3,4} Further, the emulsion breaking was promoted by microwave heating and the aqueous phase was used in the measurements by ICP OES. Satisfactory results were obtained in the proposed method applied to edible oil samples. The highest recovery range (86–90%) was obtained using the NADES composed of choline chloride, oxalic acid, and water (1:1:4 molar ratio). Also, surfactant type was evaluated, and Triton X-100 (5% v/v) provided recoveries >93% for all analytes (Al, Ca, Cr, Cu, K, Mg, Mn, and Zn). A mixture design was applied to optimize sample and solvent volumes and the optimal condition was provided when using 5.0 mL of sample, 1.7 mL of NADES, and 1.3 mL of Triton X-100. The vortex time, extraction, and MW assisted-emulsion break were optimized by the Box-Behnken design. Optimal results were obtained with 36 s vortexing, 5 min of extraction, and 10 s emulsion breaking. The combination of NADES and MW-assisted EIEB provided high recovery rates, reaching interesting experimental speed and simplicity when compared to conventional methods (e.g., acid digestion). Also, the GAC metrics were complied³ with and the results obtained support the application of NADES as an efficient extractor in methods based on the formation of emulsions. As far as we know, this study is the first work proposing the application of NADES combined with MW radiation in EIEB methods prior to elemental determination by ICP OES.

¹ Liu, Y et al, J. Nat. Prod. 2018, 81, 679.

² Costa, FS et al, J. Food Compos. Anal. 2022, 109, 104510.

³ Valasques GS et al, Appl. Spectrosc. Rev. 2017, 52, 729.

⁴ van Osch DJGP et al, Phys. Chem. Chem. Phys. 2020, 22, 2181.

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