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A Green In-Syringe Microextraction Approach Using Deep Eutectic Solvents for the Determination of Pesticides in Environmental Water by GC-MS

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INTRODUCTION & AIM

The pervasive presence of pesticide residues in aquatic environments poses significant risks to both ecosystems and human health. In response to the growing need for environmentally sustainable analytical techniques, we present a novel in-syringe dispersive liquid-liquid microextraction (IS-DLLME) method utilizing a natural deep eutectic solvent (DES) for the extraction of selected pesticides from water samples.

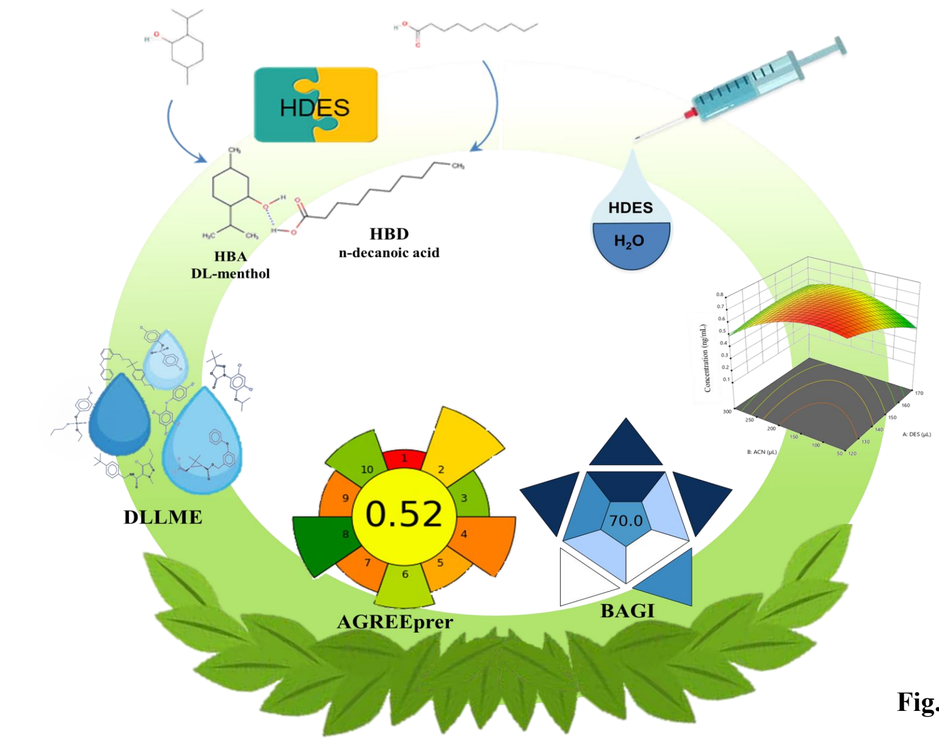

Fig. 1. Graphical abstract

METHOD

In this study, the DES was synthesized from decanoic acid as hydrogen bond acceptor and menthol as hydrogen bond donor, offering a biodegradable and non-volatile alternative to traditional organic solvents. The microextraction was carried out inside a standard cheap glass syringe available, eliminating the need for additional equipment and minimizing solvent consumption. Following extraction, analytes were separated and quantified using gas chromatography-mass spectrometry (GC-MS). A schematic illustration of the IS-DLLME-DES method for extracting pesticides from a water sample is shown in Fig. 2.

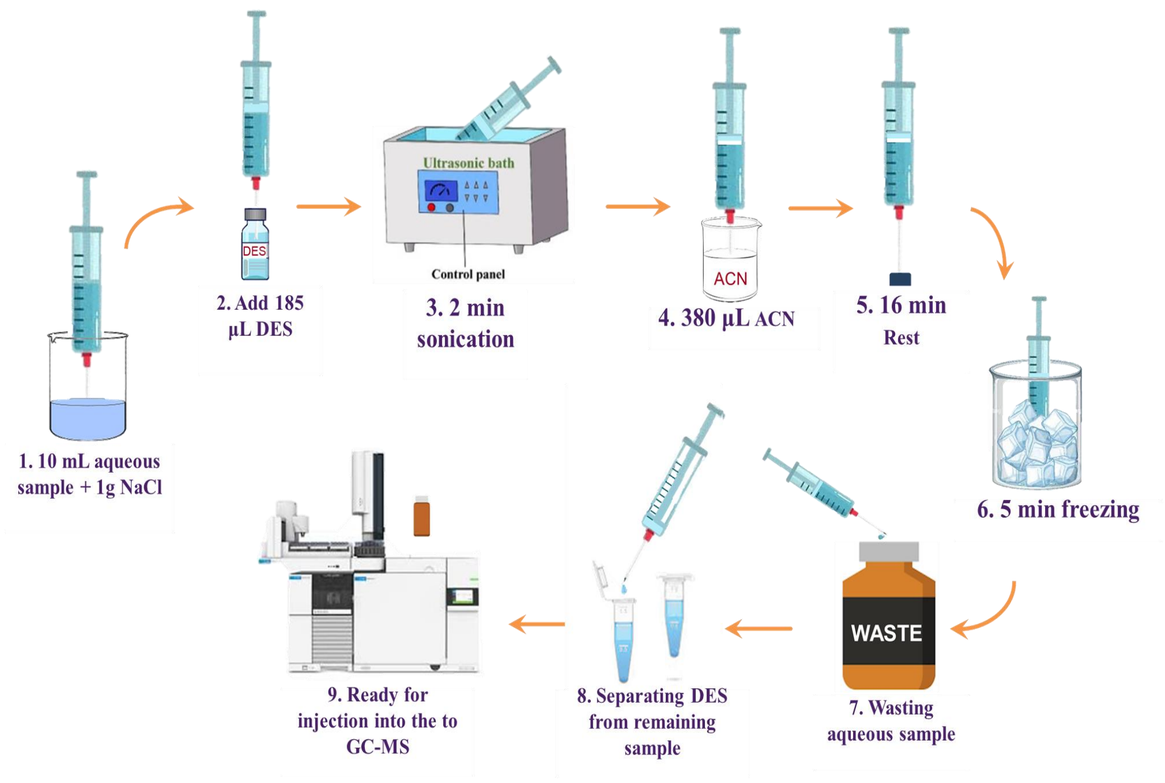

Fig. 2. Graphical scheme of the IS-DLLME-DES method, illustrating the complete workflow from sample preparation and extraction using a deep eutectic solvent to analysis by GC–MS.

RESULTS & DISCUSSION

There were three influential factors which were optimized by CCD to maximize the performance and extraction efficiency (Fig. 2). The method demonstrated excellent analytical performance, with extraction recoveries ranging from 103% to 142%, relative standard deviations ranging from 0.59% to 16.79%, and limits of detection is 0.33 ng/mL range (Table 1). The linearity was confirmed over a broad concentration range, and matrix effects were negligible. Application to environmental water samples confirmed the method's robustness and applicability to real-world analysis. The greenness of method was calculated using AGREEperp and BAGI (Fig. 4).

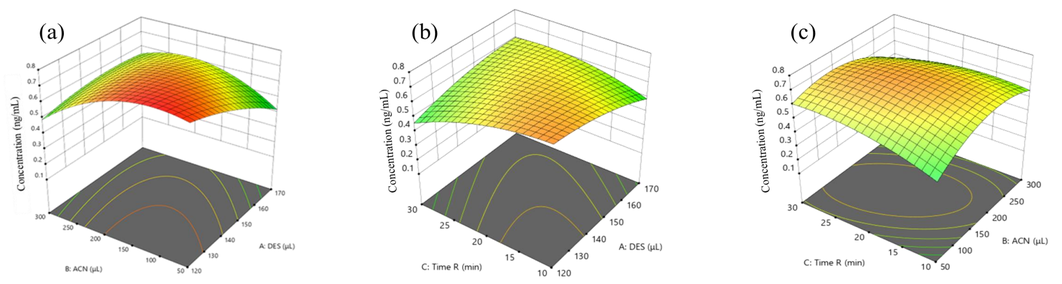

Fig. 3. Response surface plots displaying the desirability functions obtained from the CCD optimization of the IS-DLLME-DES method.

Table 1 Analytical figures of merit of proposed method for the target pesticides.

Pesticides	LOD (ng/mL)	LOQ (ng/mL)	Linear rang (ng/mL)	R^2	Spike level (ng/mL)	Recovery (ng/mL)	Inter-day RSD%	Intra-day RSD%
Ovex	0.33	1	1-20	0.9822	2 4 12	127 110 103	9.98 11 1.95	6.86 4.01 1.37
Oxadiazon	0.33	1	1-20	0.9921	2 4 12	131 107 103	2.94 7.13 1.62	6.76 3.84 1.36
Tetrasul	0.33	1	1-20	0.9842	2 4 12	134 108 104	14.79 10.87 2.46	6.49 3.88 1.36
Sulprophos	0.33	1	1-20	0.9903	2 4 12	142 111 104	5.45 4.91 2.57	6.89 3.86 1.37
Tebufenpyrad	0.33	1	1-20	0.9955	2 4 12	125 112 103	16.75 8.66 0.59	7.26 3.85 1.37
Permenthrin	0.33	1	1-20	0.994	2 4 12	126 106 103	13.80 6.42 2.40	7.11 4.03 1.38
Etofenprox	0.33	1	1-20	0.9909	2 4 12	123 110 103	12.44 3.49 1.44	7.19 3.84 1.37

* Preconcentration factor = [Sample volume (10 ml)/Extracted volume (300 µl)] = 33.33.

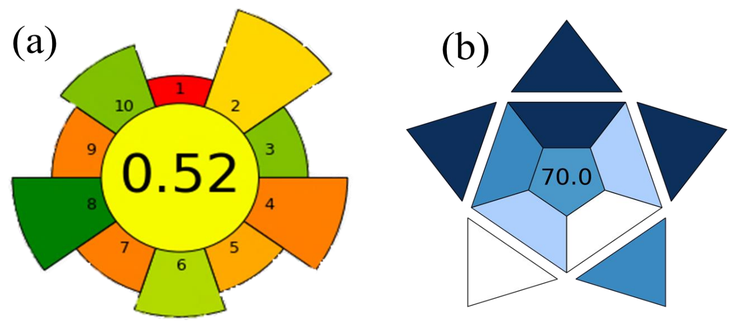

Fig. 4. Schematic overview of the greenness assessment for the IS-DLLME-DES method based on the (a) AGREEprep and (b) BAGI metric, summarizing the environmental performance across various criteria.

CONCLUSION

This work highlights the potential of combining IS-DLLME with DESs for rapid and sustainable monitoring of trace contaminants in water. The integration of green chemistry principles into sample preparation not only enhances analytical performance but also supports safer and more eco-conscious laboratory practices.

FUTURE WORK / REFERENCES

Future studies will focus on extending this green and efficient method to the analysis of other aqueous matrices, particularly groundwater.

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2. Hoseinipour, P., Daryanavard, S.M., Memar, M., and Naccarato, A., *Development of ultrasound-assisted dispersive liquid–liquid microextraction based on solidification of floating organic droplets and deep eutectic solvents for multi-class pesticide analysis in agricultural waters.* Microchemical Journal, 2025. **212**: p. 113404.

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