Preparation of monoethyl fumarate-based molecularly imprinted polymers and their application as SPE sorbent for enrichment of scopolamine from tropane alkaloids

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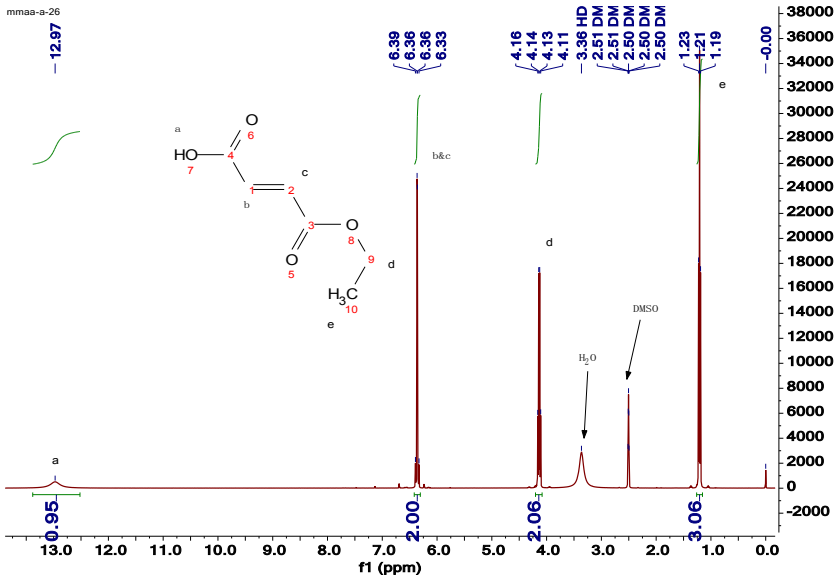
1. ^{1}H NMR spectra

The structure of synthetic functional monomer was identified using ^{1}H NMR spectra. As seen from Fig. S1, ^{1}H NMR (400 MHz, DMSO-d_6) δ: 1.21 (t, J=8 Hz, 3H), 4.14 (dd, J1=4 Hz, J2=8 Hz, 2H), 6.36 (q, J=12Hz, 2H), 12.97 (s, 1H), which proved



that MFMA had been successfully synthesized.

Fig. S1. ^{1}H NMR spectra of MFMA.

2. Method validation

To verify the MISPE system integrated with HPLC, the favorable linear response was achieved within the scope of 8.0–4.0×10^4 μg L^{-1}. with R^2 of 0.9982. Furthermore, limit of detection (LOD), which stands for minimum detectable concentration, was 2.2

μg L^{-1}. Limit of quantification (LOQ) that represents smallest amount of analyte measured with reasonable accuracy was 6.5 μg L^{-1}. To evaluate the precision of the MISPE for scopolamine analysis, three Hindu Datura samples each added with scopolamine (0.1 mg L^{-1}) were researched by MISPE system to obtain the relative standard deviation (RSD) of 2.4%.

In order to further evaluate the MISPE system, the established a non-aqueous SPE method using silica based strong cation exchange (SCX)[37] (SCX-SPE) were applied to study the Hindu Datura and Belladonna samples. The data summarized in Table S1 exhibited that the recovery of MFMA-MISPE method (96.0–106.0%) was higher than that of SCX-SPE method (78.0-85.0%). The above results indicate that the MFMA-MISPE method is more advantageous than the SCX-SPE method in enrichment of scopolamine from plants samples. Therefore, the MFMA-MISPE system integrated with HPLC is more desirable for the extraction and detection of scopolamine in the mixture of tropane alkaloids.

Table S1. Comparison of the MFMA-MISPE method with SCX-SPE method.

Samples	Added (μg L^{-1})	Detected (μg L^{-1})		Recovery (%)		RSD (%)	
		MFMA-MISPE	SCX-SPE	MFMA-MISPE	SCX-SPE	MFMA-MISPE	SCX-SPE
Hindu Datura	-	ND[a]	ND	-	-	-	-
	50.0	51.0	45.0	102.0	85.0	3.4	4.5
	100.0	96.0	80.0	96.0	80.0	2.7	5.2
Belladonna	-	ND	ND	-	-	-	-
	50.0	53.0	41.0	106.0	82.0	4.1	6.7
	100.0	99.0	78.0	99.0	78.0	3.5	5.5

[a] ND is not detected