Electronic Supplementary Material (ESI) for Analytical Methods.

Supporting Information:

Cloud-point extraction associated to voltammetry: Preconcentration and elimination of sample matrix for trace determination of methyl parathion in honey

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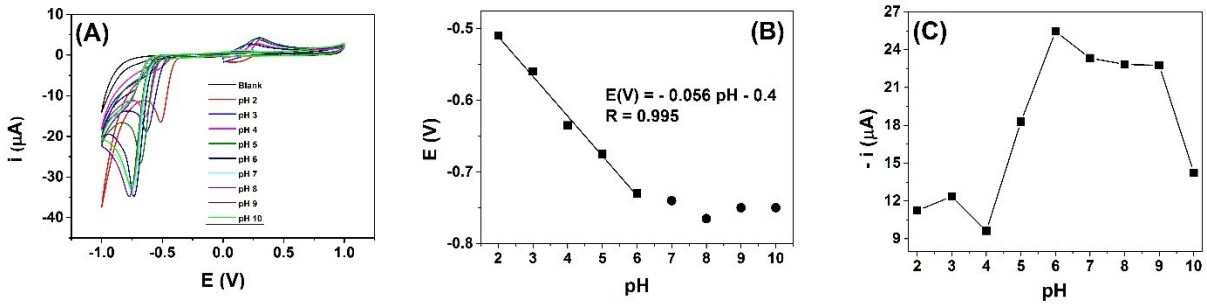

Fig. ESI 1(A) Cyclic voltammograms for 100 μmol L^{-1} methyl parathion in BR buffer solutions (pH range 2.0 to 10.0) over H-BDDE. **(B)** Plot of the dependence of E (V) *vs.* pH and respective slope of the linear regression equation. **(C)** Plot of the dependence of i(μA) *vs.* pH. Scan rate = 50 mVs^{-1}.

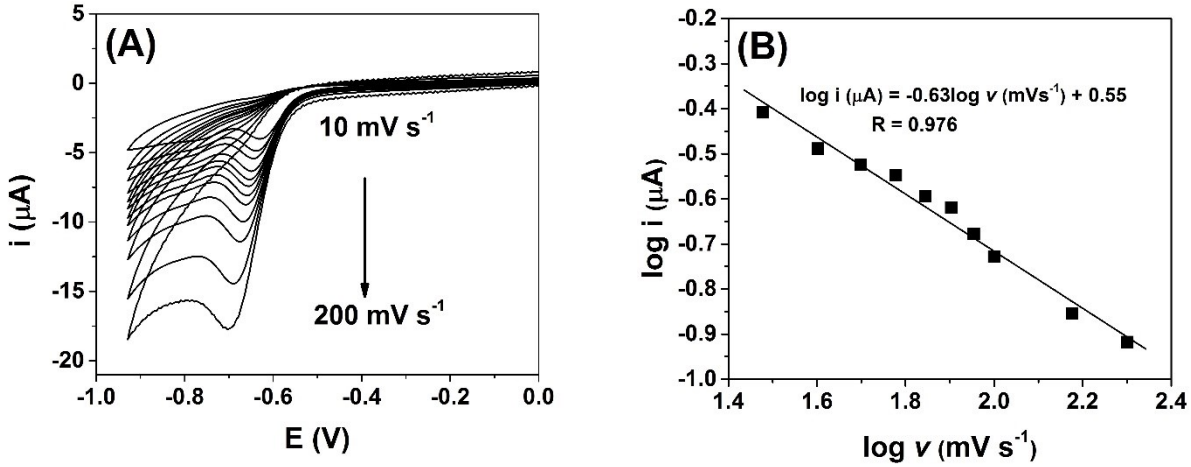

Fig. ESI 2(A) Cyclic voltammograms of 50 μmol L^{-1} MP on H-BDDE at different scan rates (10 to 200 mV s^{-1}). **(B)** Plot of the dependence of log i *vs.* log *v* and respective linear regression equation. Supporting Electrolyte: BR buffer pH 6.

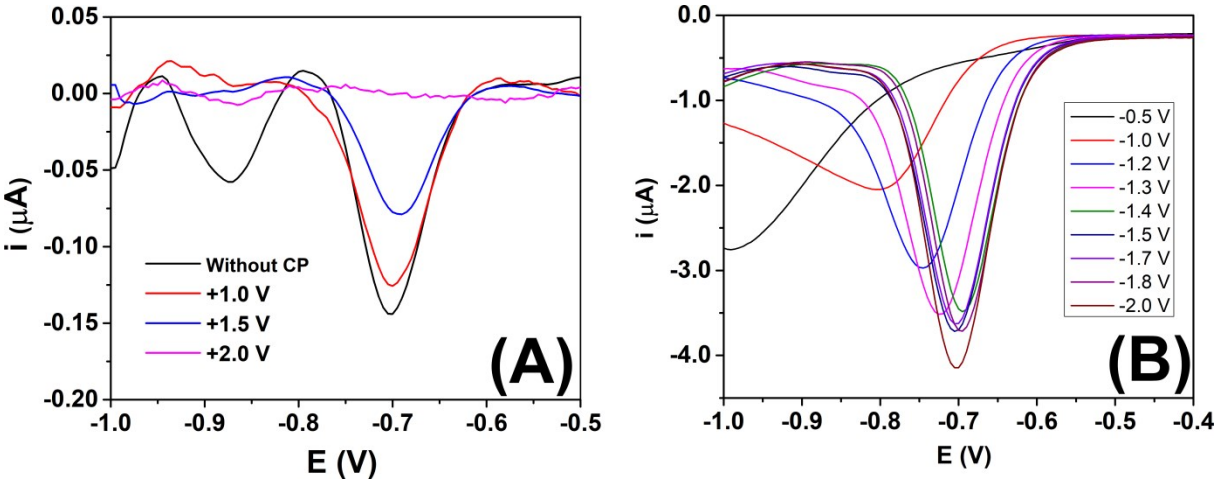

Fig. ESI 3(A) SWV blank in BR buffer pH 6 (after the scan of a 5.0 µmol L^{-1} MP) without and after apply conditioning potentials (+1.0 to +2.0 V) on BDDE. (B) SWV curves in the presence of 5.0 µmol L^{-1} MP and BR buffer pH 6 after the application of activation potential (-0.5 V to -2.0 V) in BDDE. SWV conditions: Frequency: 30 Hz; Amplitude: 50 mV; Step potential: 7 mV; Conditioning potential **(B)**: +2.0 V (for 15 s); Stirring rate: 2000 rpm.

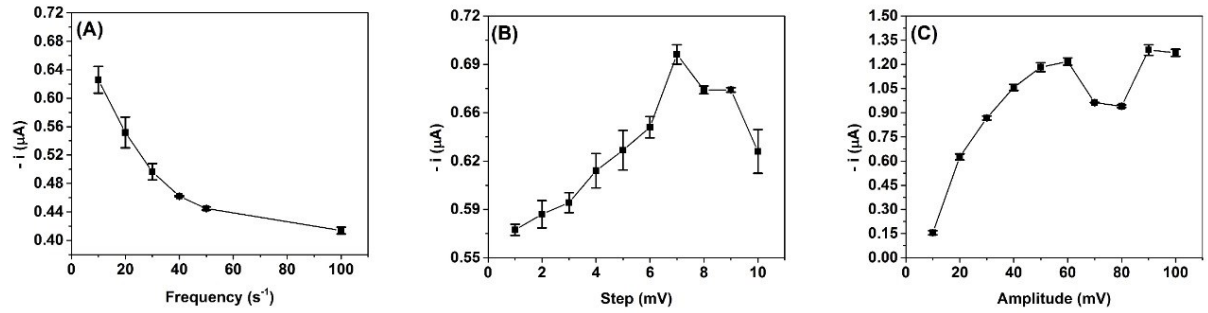

Fig. ESI 4 Variation of peak currents (± SD, n= 3) from SWV curves measured in the presence of 5 µmol L^{-1} MP and BR buffer pH 6 as function of frequency **(A)**, step **(B)** and amplitude **(C)**. Cleaning potential: +2.0 V (for 15 s), Activation potential: −2.0 V (for 15 s); Equilibration time: 15 s; Stirring rate: 2000 rpm.

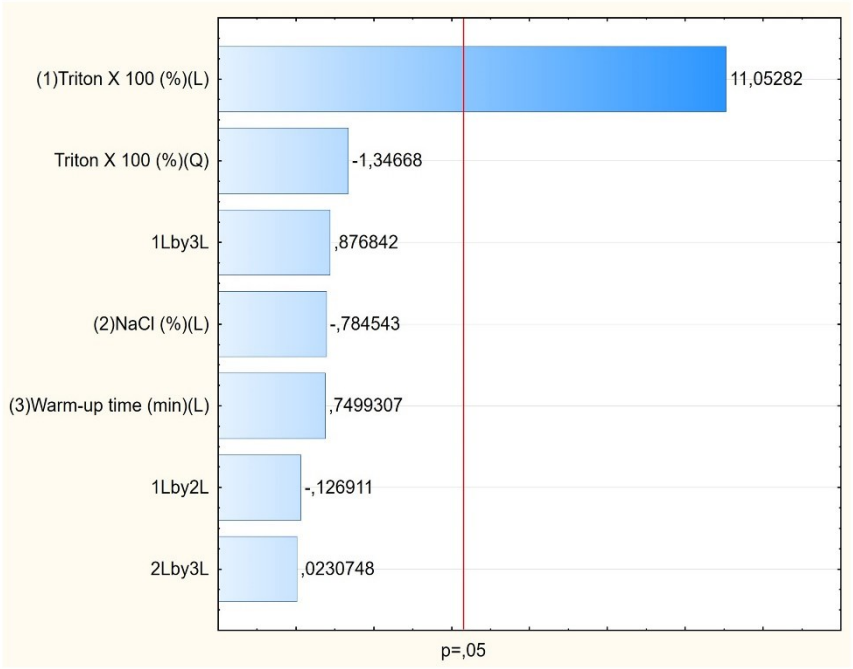

Fig. ESI 5 Pareto chart of the variable effects based on the data in Table 1 for preconcentration of methyl paration.

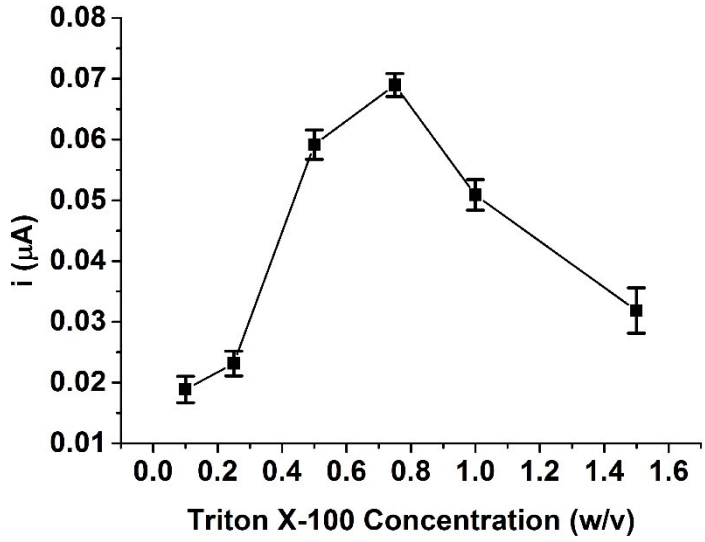

Fig. ESI 6 Optimization of Triton X–100 concentration and its analytical MP response on BDD. Optimized conditions: 0.1 mol L−1 Britton-Robinson (BR) buffer pH 6.0; Frequency: 30 Hz; Amplitude: 50 mV; Step potential: 7 mV; Conditioning potential: +2.0 V (for 15 s), Activation potential: −2.0 V (for 15 s); equilibration time: 15 s and stirring rate: 2000 rpm.

Table ESI 1. Optimization of the SWV parameters for the determination of MP using the cathodically pre-treated boron-doped electrode.

Parameters	Studied range	Optimized value
Stirring rate (rpm)	250 to 2500	2000
Conditioning (Cleaning) potential (V)	+1.0 to +2.0	+2.0
Conditioning (Cleaning) time (s)	5 to 30	15
Activation potential (V)	−0.5 to −2.0	−2.0
Activation time (s)	15 to 180	15
Equilibration time (s)	15 to 30	15
Step (mV)	1 to 10	7
Amplitude (mV)	10 to 100	50
Frequency (Hz)	10 to 100	30

Table ESI 2. Analytical characteristics of the method for determination of MP using cathodically pre-treated boron-doped electrode.

Analytical parameters	Obtained Value
Sensitivity (μA μmol^{-1} L)	0.154
Limit of detection (μmol L^{-1})	0.03
Limit of quantification (μmol L^{-1})	0.1
Linear range (μmol L^{-1})	0.5 to 16
R	0.998
Repeatability (n = 15 for 0.5 μmol L^{-1})	3.9 %
Repeatability (n = 15 for 5 μmol L^{-1})	1.4 %
Inter-day (n = 3)	5.2 %
Intra-day (n = 3)	3.6 %
Inter-electrode (n = 2)	5.0 %