Supplementary Information (SI) for Analyst.

Supporting Information

Rapid detection of capsaicinoids via Bi_2S_3/rGO/MWCNT-COOH-modified screen-printed electrodes coupled with glass bead-assisted ultrasonic extraction

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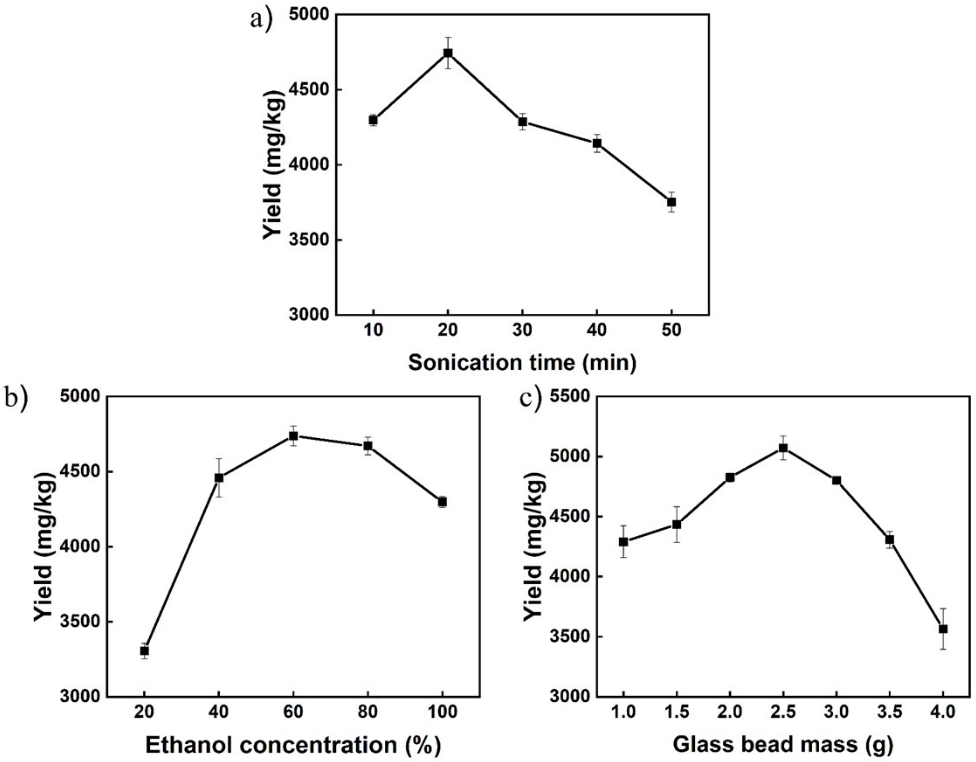

Fig. S1 Effects of (a) sonication time, (b) ethanol concentration, and (c) glass bead quality on the extraction yield of capsaicinoids.

The standard curves for capsaicin and dihydrocapsaicin, based on the peak areas of the standard samples, are provided. These curves are used to establish the relationship between the peak area and concentration of capsaicin and dihydrocapsaicin. The concentration of the target substance in the sample is then determined by comparing the sample's peak area to the standard curve. Once the mass concentration of the target substance in the sample is determined, the mass fraction can be calculated.

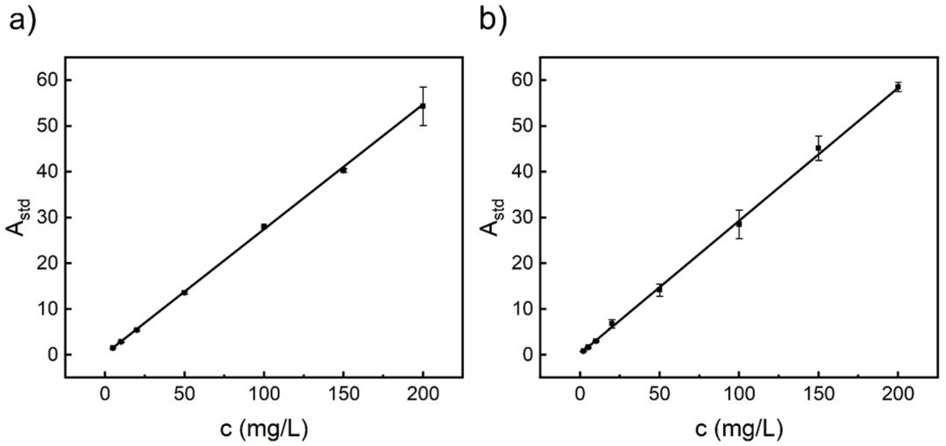

Fig. S2: Relationship between Standard Peak Area (A_{std}) and Mass Concentration (c) of (a) Capsaicin and (b) Dihydrocapsaicin.

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After extraction, the glass beads were separated from the extract by filtration and subjected to ultrasonic cleaning using deionized water and anhydrous ethanol (two cycles each) to remove residual pepper solids and organic solvents. The cleaned beads were then dried at 60 °C for 4 hours until completely moisture-free. The regenerated beads were reused under identical extraction conditions, and the capsaicinoids yield was recorded after each cycle to evaluate the effect of reuse on extraction efficiency.

To further assess structural stability, the morphology of the glass beads before use and after five reuse cycles was compared by optical microscopy.

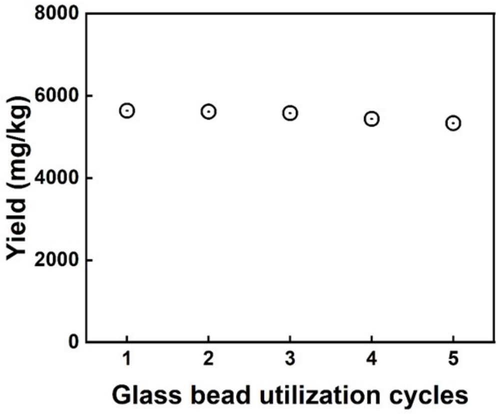

Fig. S3 Effect of glass bead reuse cycles on the extraction efficiency of capsaicinoids.

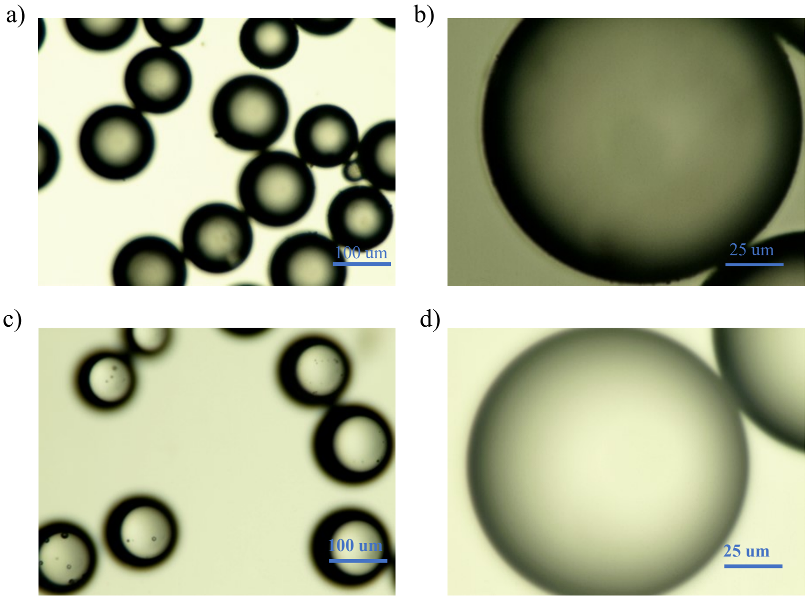

Fig. S4 Optical microscopy images comparing the morphology of glass beads before and after repeated use.(a) Overall morphology before the first use; (b) enlarged view of (a); (c) overall morphology after five cycles of reuse; (d) enlarged view of (c).

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The redox process follows the Randles–Sevcik equation:

$$I_p = (2.69 \times 10^5) n^{3/2} A D^{1/2} C v^{1/2}$$

where I_p represents the peak current (A) during the redox process; n represents the number of electron transfers, where it is 1; A represents the effective surface area of the electrode (cm^2); D represents the diffusion coefficient (cm2/s), and in a 5 mM $[Fe(CN)_6]^{3-/4-}$ solution, the diffusion coefficient D is 7.6×10-6 cm^2/s; C represents the concentration of $[Fe(CN)_6]^{3-/4-}$ (mol/cm^3); ν represents the scan rate (V/s).
Based on the slope of the linear fitting in the insets of Fig. S5 a–d, the effective electrochemical surface areas of the electrodes were calculated using the Randles–Sevcik equation.

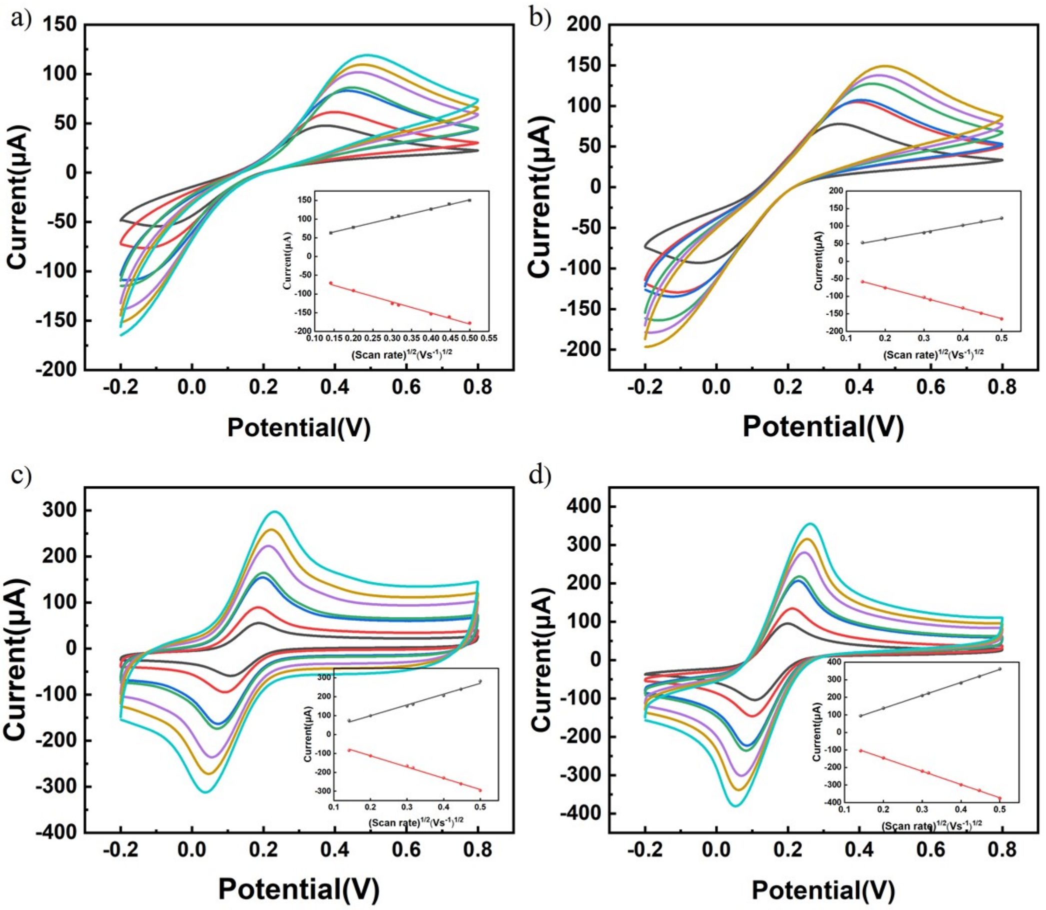

Fig. S5 Cyclic voltammograms of (a) SPE, (b) Bi_2S_3/SPE, (c) rGO/MWCNT-COOH/SPE, and (d) Bi_2S_3/rGO/MWCNT-COOH/SPE in 5.0 mM $[Fe(CN)_6]^{3-/4-}$ (0.1 M KCl) at varying scan rates. The inset shows the linear relationship between the peak current and the square root of the scan rate, indicating a diffusion-controlled process

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Based on the optimization of modifier loading volume (Fig. S6a), deposition potential (Fig. S6b), and deposition time (Fig. S6c), the optimal parameters of 4.0 µL, –0.1 V, and 300 s were selected for subsequent DPV electrochemical detection.

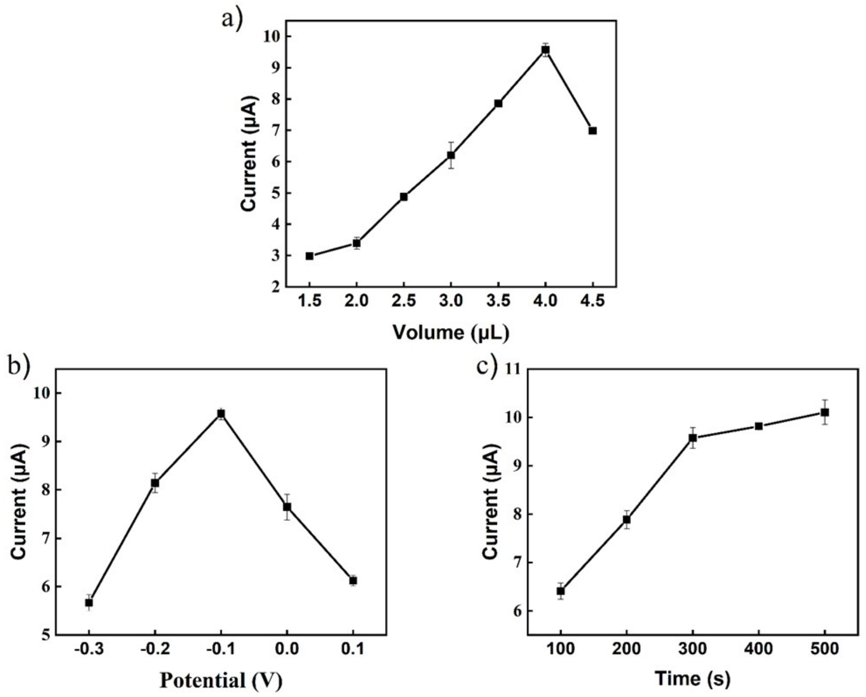

Fig. S6 Effects of (a) the loading volume of Bi_2S_3/rGO/MWCNT-COOH, (b) deposition potential, and (c) deposition time on the current response for capsaicin detection.

Table S1. Comparison of sample pretreatment duration and electrochemical detection performance for capsaicin sensors.

Method	Sample Pre-treatment Time	Linear Range (µM)	LOD	Samples	Reference
Fe^{III}-HMOF-5/SPE	140 min	1–60 µM	0.4 µM	Fresh chili peppers	1
N-doped GrNPs/ePAD	65°C，20min	1–100 µM	0.37 µM	Dried chili samples	2
Ni-CNTs/S-rGO/GCE	90 min	0.01–100 µM	1 nM	Leisure duck neck, beef, chicken feet	3
Stencil-printed graphene electrodes	60 min	1–75 µM	0.1 µM	Homemade extract chili, red pepper sauce	4
This work	**22 min**	**0.05–2000 µM**	**26 nM**	**Dried/fresh chili peppers, chili sauce, chili oil, capsaicin cream**	

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As shown in Table S1, most reported electrochemical sensors for capsaicin suffer from long pretreatment times (up to 140 min) and limited sensitivity. In comparison, our method achieves a wide linear range (0.05–2000 μM), a low LOD (26 nM), and a rapid pretreatment process of just 22 min.

Notes and references

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