Turbulence-assisted high throughput liquid-liquid extraction in microfluidics and Ni(OH)₂ nanoparticles for electrochemical determination of monoethylene glycol traces in natural gas condensate

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This supplementary material contemplates information on:

1. **Cyclic voltammograms**

2. **Traditional shake-flask liquid-liquid extractions**

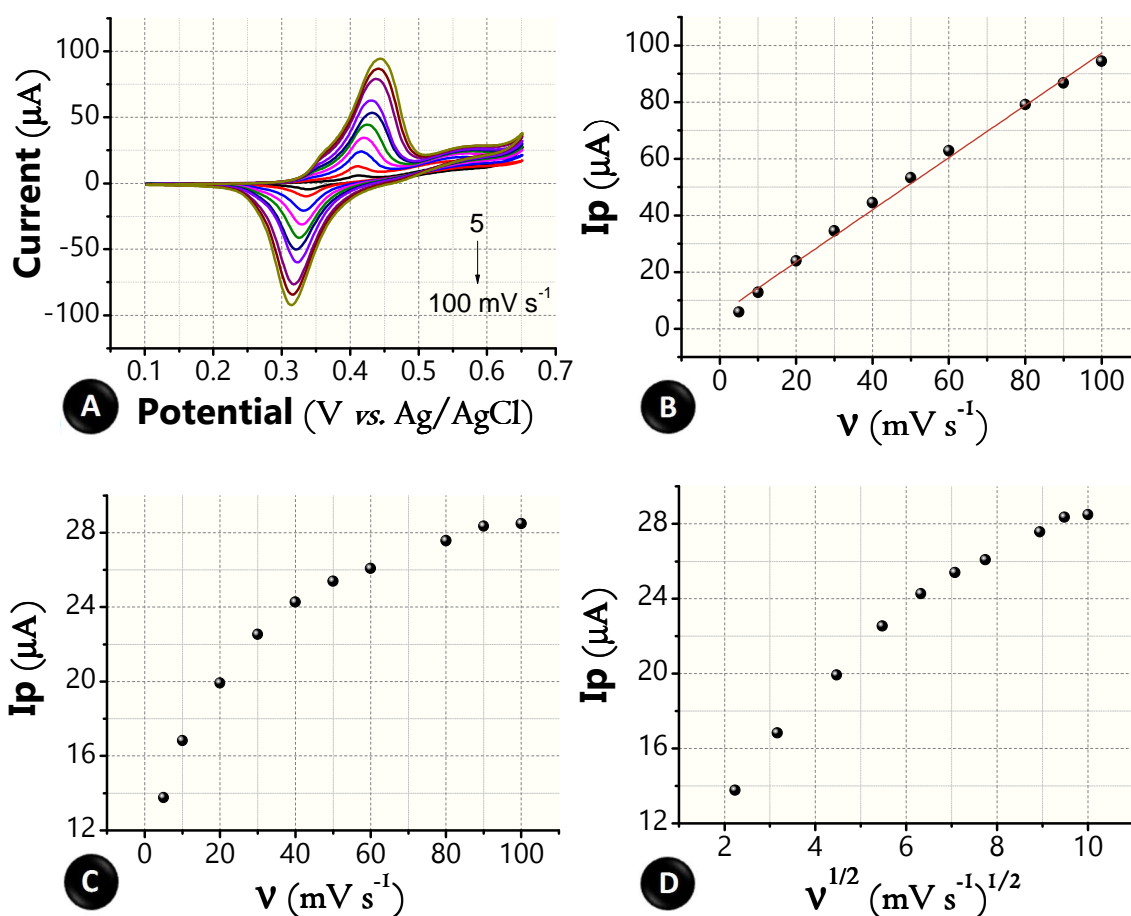3. **Calculation of the Reynolds number**

1. Cyclic voltammograms

Figure S1. Voltammograms obtained for MEG 6.0 mmol L^{-1} at different scan rates (ν) from 5 up to 100 mV s^{-1} **(A)**. Anodic peak current (Ip) at +430.0 mV as a function of ν for Ni(OH)$_2$/NiOOH process **(b)**. Ip at +580.0 mV as a function of ν **(c)** and $ν^{1/2}$ **(d)** for MEG oxidation reaction.

2. Traditional shake-flask liquid-liquid extractions

The procedure adopted by Petrobras for MEG extraction from organic NGC samples to aqueous phase is based on traditional shake-flask technique. In this method, 100.0 mL of NGC sample and, then, 50.0 mL of acetic acid solution (0.4 mol L^{-1}) are added to a separation funnel that is subsequently shaken. The separation of the phases takes approximately 10 min because of the formation of an emulsion between the phases. After phase separation, the aqueous phase is collected in a 250.0 mL volumetric flask. These steps are repeated twice (the same 100.0 mL of NGC is processed in this batch extraction), resulting in 150.0 mL of aqueous extract. Finally, the volume of the flask is filled with water obtaining a final solution with MEG concentration diluted about 1.7 times from original NGC sample. In this case, the MEG content was attained by iodometric titration using a Petrobras' routine.

In order to preconcentrate MEG in aqueous phase, some adaptations were done in traditional shake-flask method. Initially, 30.0 mL of NGC sample and 1.0 mL of aqueous phase, which consisted of a solution of 0.1 mol L^{-1} Na$_2$SO$_4$, are added to a separation funnel. The use of this salt solution was intended to inhibit the formation of emulsion during the vigorous manual shaking of the funnel.[1] Then, the aqueous phase is filtered in syringe filter (Millex®, PTFE hydrophobic,

0.22 μm, from Merck) to remove any remaining organic phase. These steps are repeated three times using 1.0 mL of aqueous phase and, succeeding, another extraction step is accomplished with 1.5 mL of such phase resulting in 4.5 mL of aqueous extract. Finally, 0.5 mL of NaOH 1.0 mol L^{-1} is added to adjust the pH at 10, which is necessary for the electro-chemical measurements. Next, the volume is completed to 5.0 mL in a volumetric flask. This procedure allowed the pre-concentration of MEG in the aqueous phase by a factor of 6 times.

3. Calculation of the Reynolds number

Reynolds number (**Re**) was calculated according to equation: **Re = ρvd/μ**, where **ρ**, **μ**, **v**, and **d** mean the density and dynamic viscosity of the fluid, the average fluid velocity, and the channel diameter, respectively.[2,3] We considered the fluids composed of hexane and water 50.0% v/v at 20°C, with average **ρ** of 0.828 g m^{-3} and **μ** of 0.661 m Pa s^{-1}. The diameter of the mixing microchannel was measured as the maximum value of the wrapped structures, namely, 568.04 μm. The average velocity of the solutions was determined by dividing the applied flow rate by the cross section area of the channel.

4. References

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(2) Pritchard. P. J. *Fox and McDonald's – Introduction to fluid mechanics*, 8th ed.; John Wiley & Sons, Inc. 2011, pp 875–3.
(3) Kumaran, V.; Bandaru, P. Ultra-fast microfluidic mixing by soft-wall turbulence, *Chem. Eng. Sci.* **2016**, 149, 156–168.