EMμ: The Next Generation of Separation Science

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Abstract

The extensive application of monolithic columns for HPLC is severely hindered by a lack of column-to-column reproducibility. EMμ (Electroactive Monolithic μChip) is a new concept that solves the significant reproducibility problems, as well as allowing miniaturization and improving overall efficiency through electrochemically controlled dynamic separations.

This novel μ chip has a micro-structured monolith fabricated from intelligent, electroactive polymer. By application of a specific potential, conducting polymers such as polyaniline (PANI) can be reproducibly grown and readily fine-tuned in terms of porosity, hydrophobicity and ionic capacity. This unique chip provides for an Electroactive Monolithic μ chip capable of multi-dimensional chromatographic separations.

Additionally, EMμ can exploit on-chip electrodes permitting incorporation of contactless conductivity detection (C4D). The monolith microstructuring (provided by templating) will provide reproducibility and improve efficiency by decreasing the A-term of the Van Deemter equation. Furthermore, the use of these intelligent materials will enable gradient control and redox reactions to be exploited during separations of larges biomolecules.

General Concept

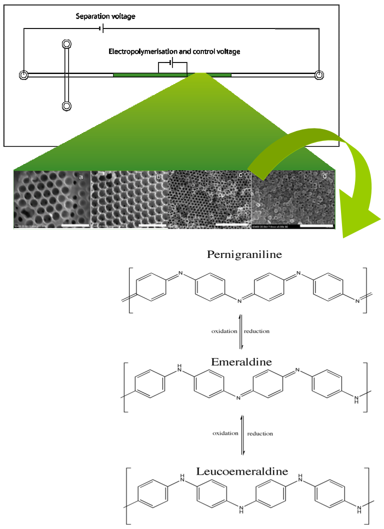

Figure 1: Scheme of the base piece of the microchip .The separation channel containing the electroconducting polymer is illustrated in green, with an insert showing an SEM images of the possible structures that can be fabricated using polystyrene templating. Polyaniline (PANI) is used as the electroconducting polymer and its 3 oxidation states are shown. By electrochemically switching from one oxidation state to another, PANI properties can be tuned as PANI undertakes electronic and conformal mutation.

The microstructured PANI monolith will provide reproducibility as the electrochemical polymer growth is well controlled through templating. Thus no radial heterogeneities are observed once the template can be deposited so that it is defect-free.

As PANI properties such as volume, hydrophilicity, ionic capacity can be tuned by applying a potential onto it. Morphological and chemical control of the PANI monolithic stationary phase before and during separations is enabled by EMμ.

μChip Prototype

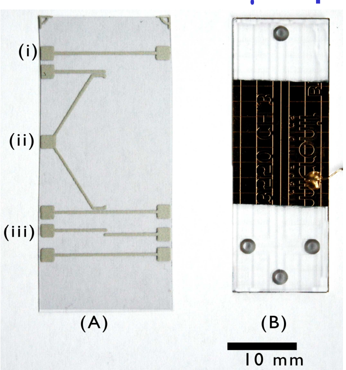

Figure 2: Layout of the screen printed upper piece (A) of the chip containing the reference (i) and auxiliary (ii) electrodes as well as the electrodes for C4D detection (iii).

Layout of the base piece (fabricated in glass) of the chip (B) (channel 110 μm wide and 50 μm deep). The working electrode is a gold sputtered film on the base of the separation channel.

The upper and base piece of the chip are bonded using pressure sensitive adhesive (PSA).

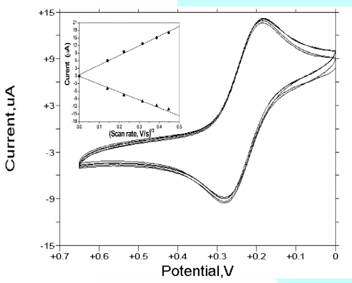

Figure 3: Cyclic voltamogramms (CVs) of ferrocyanide carried out inside the channel (100 mV/s)

The separation channel is a 3-electrode cell suitable for performing electrochemistry. The cell is composed of a reference, auxiliary and working electrode as described in Figure 2 and permits the control of the potential at the working electrode. As shown in Figure 3, ideal electrochemistry for the reversible couple Fe^{2+}/Fe^{3+} is achieved inside the chip separation channel. (peak potential difference of 60 mV)

Electrochemical Monolith fabrication

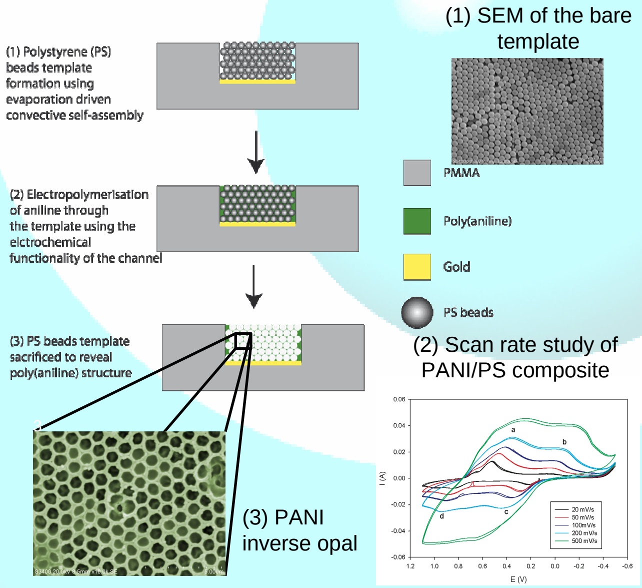

Figure 4: The fabrication of the reproducible microstructured poly(aniline) monolith is a 3 step process.

The choice of electrochemical technique used for polymerisation (potentiostatic or potentiodynamic) had no influence on the final structure.

Conclusion

EMμ provides an alternative to conventional silica and inert polymer monolithic stationary phases as this monolith is reproducible. Studies are underway looking at approaches to improve packing of the PS template. Separations of large biomolecules will de demonstrated in the coming months.

NCSR National Centre for Sensor Research
sfi science foundation ireland
NDP National Development Plan