# CARBON BLACK BASED ELECTROCHEMICAL TRANSDUCERS FOR MICROFLUIDIC DETECTION

<u>*Flavio Della Pelle*</u><sup>1</sup>, Daniel Rojas<sup>1,2</sup>, Michele Del Carlo<sup>1</sup>, Luis Vázquez<sup>3</sup>, Dario Compagnone<sup>1</sup> and Alberto Escarpa<sup>2,4</sup>

<sup>1</sup> Faculty of Bioscience and Technology for Food, Agriculture and Environment University of Teramo 64100, Teramo (Italy). Fax:(+39) 0861–266942, E-mail: fdellapelle@unite.it.

<sup>2</sup> Department of Analytical Chemistry, Physical Chemistry and Chemical Engineering, Faculty of Sciences, University of Alcalá, E-28871, Alcalá de Henares, Madrid, Spain.

<sup>3</sup> Institute of Materials Science of Madrid (CSIC), C/Sor Juana Inés de la Cruz 3, 28049, Madrid, Spain.

<sup>4</sup> Chemical Research Institute "Andres M. del Rio", University of Alcalá, E-28871, Madrid, Spain.

# 1.- Objectives

This work aims to demonstrate the carbon black nanoparticles (CBNPs) exploitability to realize effective and cheap transducers for the detection at the microscale level. In order to allow the direct detection (without additional transducers) onto the CBNPs surface, as well as the integration in a microchip electrophoresis platform, the transducers have been realized onto poly(methyl methacrylate) (PMMA) supports using a press-transfer technology. Thus, the CBNPs-based transducer fabrication has been optimized and the resulting devices have been deeply characterized, in terms of both surface features and electrochemical performance (outchip and on-chip). Finally, the realized devices have been challenged on board of microfluidic chips for amperometric determination of neurotransmitters and environmental organic contaminants (pesticides), in order to prove the ability to solve real domain analytical issues.

# 2.- Methods

Press-transferred CBNPs transducers fabrication main steps [1]: a) CBNPs dispersion was filtered with an analytical stainless steel 13 mm vacuum filter holder, using a Teflon filter with a pore size of 0.1 mm; b) Teflon filters were cut out in  $13x1 \text{ mm}^2$  pieces to obtain a CB wire; c) the CB wire was placed on the PMMA support; d) the CBNPs was transferred onto the sheet by applying a pressure of  $5.8\pm0.1$  tons for 60 s. The Teflon filter was removed by using tweezers; e) electrical contacts were made by using conductive silver that was later electrically isolated by using insulating paint. The glass chip was fabricated by Micronit Microfluidics (BV, NL) and consisted of a glass plate ( $88x16 \text{ mm}^2$ ) with a four-way injection cross, a 74 mm long separation channel, and side arms measuring 5 mm long. The amperometric detector (end-channel detection) consisted of an Ag/AgCl wire as reference electrode, a platinum wire as counter electrode, and a CBNP transducer as working electrode.



Fig. 1: Press-transferred carbon black nanoparticles film on board of microfluidic chip.

Amperometric detection, electrochemical impedance spectroscopy (EIS) and cyclic voltammetry (CV) were performed by using a Potentiostat Autolab PGSTAT12 from Eco Chemie. A LabSmith HVS448 high-voltage sequencer with eight independent high-voltage channels and programmable sequencing for an entire level of voltage manipulation (Lab-Smith,

Livermore, CA) was used as the voltage source. The CBNPs-based press-transferred transducers were characterized by field-emission scanning electron microscopy (FESEM), atomic force microscopy (AFM), current-sensing atomic force microscopy (CS-AFM), and Raman spectroscopy. While transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS) were used to investigate the CBNPs features.

## 3.- Results

Carbon black nanoparticles (CBNPs) press-transferred film-based transducers revealed that the CBNPs films retaining the nano CB good conductivity, and a significant correlation between the morphology and the resistance were observed. Indeed, the amount of press-transferred CBNPs is the key parameter to obtain films with improved conductivity, which is in good agreement with the electrochemical response. In addition, the conductivity of such optimum films was not only Ohmic; in fact, tunneling/hopping contributions were observed [1]. Definitely, the CBNPs film act as an exclusive electrochemical transducer, resulting to be an elective device to be coupled with microchip-electrophoresis. Thus, the CBNPs film was employed as detector coupled to microfluidic chip confirming remarkable properties, offering low detection potentials, and negligible surface fouling, allowing to achieve enhanced analytical performance compared to commercial graphite electrodes. Finally, these conductive CBNP press-transferred films have been successfully applied for the transduction of target molecules, and excellent electrochemical performances have been obtained, despite the complex chemical media investigated [1-3]. In particular, neurotransmitters and pesticides, have been successfully analysed in model solutions and real samples [1,2]. The proposed devices coupled to microchipelectrophoresis proven to be an excellent approach to carry out a multiresidual screening analysis characterized by rapidity, low sample and reagent consumption, and low waste generation.

### 4.- Conclusions

The affordable press-transfer technology reported here allowed the design and control of the electrical properties of conductive CBNPs films. The obtained results demonstrated both the analytical potential of CBNPs as a novel alternative to other carbon nanomaterials, the solid establishment, and the suitability of the press-transfer technology and open novel avenues in microfluidics and others related micro- and nanochemistry applications. Moreover, in our perception, these results prove that the proposed strategies represent a viable tool for the analysis of complex real samples, for on-site environmental monitoring, and for rapid diagnosis. Moreover, preliminary results obtained by our group, have proved how the CB nano-hybridized with transition metal dichalcogenides (2D-graphene like materials) show unique and useful properties, thus, starting from this promising results one of our research lines, still on course, aims to exploit these features to fabricate new transducers for microchip-electrophoresis.

Keywords: Microchip; Electrophoresis; Nanocarbon; Pesticides.

### **References:**

- [1] Flavio Della Pelle, Louis Vázquez, Michele Del Carlo, Manuel Sergi, Dario Compagnone, Alberto Escarpa, A. *Press-Printed Conductive Carbon Black Nanoparticle Films for Molecular Detection at the Microscale*. Chemistry-A European Journal, 22(36), 12761-12766, 2016.
- [2] Flavio Della Pelle, Michele Del Carlo, Manuel Sergi, Dario Compagnone, Alberto Escarpa. *Press-transferred carbon black nanoparticles on board of microfluidic chips for rapid and sensitive amperometric determination of phenyl carbamate pesticides in environmental samples*. Microchimica Acta, 183(12), 3143-3149, 2016.
- [3] Flavio Della Pelle, Roberto Di Battista, Louis Vázquez, Michele Del Carlo, Manuel Sergi, Datio Compagnone, Alberto Escarpa. *Press-transferred carbon black nanoparticles for class-selective antioxidant detection*. Applied Materials Today, 9, 29-36, 2017.