



Review

Hyphenated analytical techniques for multidimensional characterisation of submicron particles: A review

Gaëtane Lespes*, Julien Gigault

Université de Pau et des Pays de l'Adour (UPPA)/CNRS, Laboratoire de Chimie Analytique Bio-Inorganique et Environnement (LCABIE), UMR IPREM 5254, Technopôle Hélioparc, Av. Du président Angot, 64000 Pau, France

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ABSTRACT

The stakes concerning the characterisation of particles ranged in the size from 1 to 1000 nm, namely submicron particles, are today more and more important. Because of the variety of particles even inside a given sample in terms of dimension, mass, charge or chemical composition a characterisation as complete as possible is needed. The possibility of obtaining a multidimensional information by relevant analytical methods is then of the greatest interest. One very interesting strategy consists in using hyphenated techniques, which are intrinsically capable to provide rapidly and accurately such information. This paper summarises the different hyphenated techniques that can be used to characterise submicron particles and is focussed on their main applications to illustrate their current and potential uses. In order to have a relevant overview various on-line separation techniques are considered in a comparative way. In the same way various on-line detectors are then presented. Finally the concepts of multidetector and multidimensional analysis are discussed and their interest showed through different typical examples of hyphenated techniques illustrating submicron particle characterisation in fields of applications such as environmental and nanomaterial sciences.

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Gaëtane Lespes has a PhD in Physical Chemistry (spectroscopy). She is professor of Analytical Chemistry at the University of Pau, LCABIE (France). She works on the development of analytical strategies for trace element speciation and fractionation. She is especially interested by colloidal transport of trace elements and characterisation of colloidal carriers (natural as well as manufactured nanomaterials). Her investigations are focused on hyphenated techniques involving Field Flow Fractionation (FFF), light scattering and atomic mass spectrometry.



Julien Gigault has a master in Environmental Chemistry. During his PhD in Analytical Chemistry and Environment at the University of Pau, LCABIE (France), his work has been focused on Field Flow Fractionation (FFF) combined to static and dynamic light scattering for the characterisation of manufactured nanomaterials and the study of their environmental behaviour. He has a special interest for single- and multi-walled carbon nanotubes (SWCNT and MWCNT).

Abbreviations: A4F, asymmetrical flow field-flow fractionation; CE, capillary electrophoresis; CHDF, capillary hydrodynamic chromatography; CNT, carbon nanotubes; CZE, capillary zone electrophoresis; DAD, diode array detector; DEP, dielectrophoresis; DLS, dynamic light scattering; EI-FFF, electrical field-flow fractionation; ENM, engineered nanomaterials; ETAAS, electrothermal atomic absorption spectrometry; FFF, field-flow fractionation; FI-FFF, flow field-flow fractionation; GC-PFPD, gas chromatography-pulsed flame photometric detector; HDC, hydrodynamic chromatography; ICP-AES, inductively coupled plasma- atomic emission spectrometry; ICP-MS, inductively coupled plasma- mass spectrometry; L, channel length; LC, liquid chromatography; LIBD, laser-induced breakdown detection; LS, light scattering; MALS, multi-angle light scattering; MALLS, multi-angle laser-light scattering; MS, mass spectrometry; NP, nanoparticles; PCS, photon correlation spectroscopy; QUELS, quasi elastic light scattering; RI, refractive index detector; RSD, relative standard deviation; Sd-FFF, sedimentation field-flow fractionation; SEC, size exclusion chromatography; SEM, scanning electron microscopy; SLS, static light scattering; SAXS, small-angle X-ray scattering; TEM, transmission electron microscopy; Th-FFF, thermal field-flow fractionation; UV-vis, ultraviolet-visible detector.

* Corresponding author. Tel.: +33 5 59 40 76 71.

E-mail address: gaetane.lespes@univ-pau.fr (G. Lespes).

Nomenclature

C	concentration
D	diffusion coefficient
d	diameter
d_h	hydrodynamic diameter
d_g	gyration diameter
dn	refractive index
F	external physical force applied in FFF
k	Boltzman's constant
L	length
M_w	weight-average molar mass
r_g	gyration radius
rms	root-mean-square
S_d	selectivity
T	temperature
t_R	retention time

Greek letters

ε_λ	molar extinction
λ	wavelength
μ_{ep}	electrophoretic mobility
η	viscosity
ρ_p	particle density
ξ	Zêta potential

1. Introduction

For the last 30 years submicron particles have given rise to a great deal of interest leading to a lot of works and publications. This field of study is very large. So it is important to agree the different definitions or expressions that can be found in the referenced literature because different terms are used. Complementary it is also important to understand why submicron particle characterisation has attracted so much attention.

In environmental sciences the expression “colloidal particles” is the most often used referring more particularly to submicron particles in suspension in aqueous medium. Complementary the term “colloidal” is defined by IUPAC as “a state of subdivision implying that the molecules or polymolecular particles dispersed in a medium have at least in one direction a dimension roughly between 1 nm and 1 μ m” [1]. The expression “environmental particles” is also employed, sometimes without any size precision [2,3]. Accordingly a lot of various natural materials with different physico-chemical natures can be qualified as colloidal particles, e.g. inorganic entities (as oxides, carbonates or clays-based), organic matters (typically humic-like or extra-cellular polymeric substances), living organisms (such as virus or bacteria) or mix particles containing several of these materials as composite or coating-based objects sufficiently stable to be identified in a medium of interest. The increasing attention to natural submicron particles originally comes from an environmental question about contaminant transport in unsaturated and saturated porous media, typically soil or sediment, and their occurrence in groundwater [4]. More specifically colloids are recognised as playing a key role in biogeochemical cycles of the elements, influencing mobility and reactivity especially at the interfaces with living organisms [3,5]. Additionally health impact and ecotoxicology of submicron particles have been also investigated [5,6]. Accordingly the identification of colloidal carriers is of first interest. Generally the characterisation process involves the determination of size distribution, chemical composition and contaminant distribution (the most often concerning trace elements). The knowledge of these parameters is important because they have been found as directly driving mobility, interaction with the surrounding medium

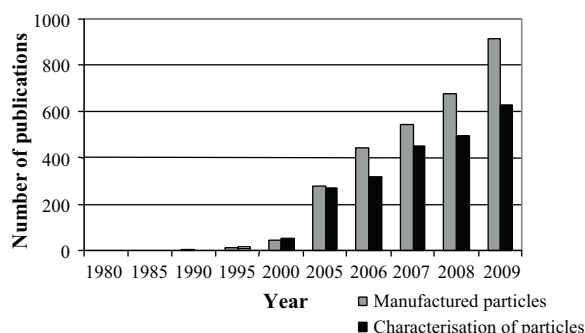


Fig. 1. Evolution of the number of publications (grey and dark bars) dealing with-submicron particles or their characterisation.

and capability of cell internalisation of submicron particles and associated contaminants [5,6].

More recently submicron particles referring to some engineered nanomaterials (ENM) have also held one's attention all the more as nanotechnology is developing. It is especially the case of nanotubes and nanoparticles (NP), which are defined as ranged over 1–100 nm respectively in two and three of their dimensions according to ISO/TS7687:2008-standard. For example carbon nanotubes (CNT, the most known today) as well as polymer-based nanospheres or n-Ag nanoparticles can be quoted to illustrate these definitions. The evaluation of the environmental risk linked to submicron particle release is particularly of high interest because it is recognised depending on their dimensions and shape. More precisely their aspect ratio (ratio between their respective longest and shortest dimensions e.g. length-to-diameter) and their size distribution have to be taken into account. Additionally their behaviour in liquid media is also important to study, i.e. their potentialities of agglomeration and more generally the physical state of any suspension involving them [7–11]. Engineered submicron particle characterisation appears also crucial for the evaluation of the quality of manufactured materials [2]. Indeed nanotubes and nanoparticles can be considered as element units of the matter in a bottom-up approach. Their size and shape determine most of their physico-chemical properties as well as the properties of the final object in which they will be. Their electrical charge is another key parameter. Considering the very various applications of these materials in energy, electronics, mechanics or health, it is then obvious that all these parameters are important to know in material sciences [5,12,13]. However, since engineering process control remains an objective not always well reached in nanotechnology, particle characterisation is needed. As a consequence the number of works devoted to engineered submicron particles and their characterisation has widely increased these last thirty years as illustrated in Fig. 1. It is interesting to notice that this number is inversely proportional to the dimensions of the smallest particles that human being is able to manufacture. Indeed, in the eighties, this smallest size was 1000 nm, while it was 100 nm in the nineties and around some nanometers today. This observation can be considered as a confirmation of the issue existing in particle size control.

Among the different analytical strategies that can be chosen to characterise submicron particles, one interesting approach consists in the hyphenation of an on-line fractionation to one or several complementary detectors. Such approach obviously needs the particles in suspension in a liquid medium. In most of the cases it is not a problem since submicron particles are already in such medium, typically environmental particles from surface and subsurface water. For engineered particles a sample preparation is usually needed. Sometimes this step is still a challenge, like for aqueous CNT suspension [14–16]. However this challenge is not

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