Nanosensor Technologies for Monitoring - materials and methods

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Conference with Posters and Exhibition

Organised by the Automation and Analytical Management Group
Royal Society of Chemistry

A one day meeting on
Wednesday 7th November 2012

At The Royal Society of Chemistry,
Burlington House,
Piccadilly, London W1J 0BA

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Nanosensor Technologies for Monitoring - materials and methods

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Wednesday 7th November 2012
at The Royal Society of Chemistry, Burlington House, London

09:15 - 09:45 Registration and Coffee

Session 1: Overview & Synthesis

Chairperson: **Krishna Persaud,** University of Manchester, Manchester, UK

09:45 New Methods of Bioanalysis using Functionalised Nanoparticles and SERS
Duncan Graham
University of Strathclyde, Glasgow, UK

10:15 Potenital Hazards and Benefits of Nanomaterials
Lang Tran
IOM, Edinburgh, UK

10:45 Applications of Nanomedicine in Diabetes
John Pickup
King's College London, London, UK

11:15 Coffee

11:45 Automatic Synthesis of MIP Nanoparticles ("plastic antibodies")
Sergey Piletsky
University of Cranfield, Cranfield, UK

12:05 Solid-phase Synthesis of Molecularly Imprinted Nanoparticles
Alessandro Poma
University of Cranfield, Cranfield, UK

12:25 Nanomaterials Employed in Flexible Organic Field-Effect Transistors for Food Freshness Monitoring Applications
Maria Daniela Angione
University of Manchester, Manchester, UK

12:45 Lunch - Exhibition & Posters

Session 2: Monitoring Applications

Chairperson: **Chris Walton,** University of Cranfield, Cranfield UK

14:15 Mapping the Intestinal pH of *C.elegans* using Ratiometric Extended Dynamic Range pH-Sensitive Nanosensors
Veeren Chauhan
University of Nottingham, Nottingham, UK

14:35 Self-Assembled Odorant Binding Proteins as Biosensors for the Detection of Odorants and Pheromones
Elena Tuccori
University of Manchester, Manchester, UK

14:55 Nanotechnology-Based Sensors for Air Pollution Monitoring in Confined Spaces: A Real Industrial Approach
Alejandro Alija
Ingenieros Asesores - Envira, Asturias, Spain

15:15 Tea / Coffee - Exhibition & Posters

15:45 Hybrid Composites of Polyaniline/Modified Carbon Nanoparticles Employed as Highly Sensitive Ammonia Sensors on Flexible Substrates
Ehsan Danesh
University of Manchester, Manchester, UK

16:05 Sensing Atomic-Scale Electric Fields by Self-Organised Lipid Layers on Hg
Shahrzad Mohamadi
University of Leeds, Leeds, UK

16:25 The Detection of Solvent Dyes in Hydrocarbon Fuels Using Surface Enhanced Raman Spectroscopy
Kelly Whittingham
University of Lincoln, Lincoln, UK

16:45 Exhibitor Presentation

17:00 Concluding Remarks and End of Conference

ABSTRACTS

New Methods of Bioanalysis using Functionalised Nanoparticles and SERS

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ABSTRACT

Functionalized nanoparticles have been used in a number of different studies including detection of DNA at ultra low levels, immuno histochemistry and more recently as substrates for surface enhanced resonance Raman scattering (SERRS) based imaging approaches. The advantages of using metallic nanoparticles are that they are very bright in terms of their optical characteristics and also if functionalized in a particular manner to provide a SERRS response give a unique vibrational fingerprint. Here we present the functionalisation of gold and silver nanoparticles in such a way that the enhancement effect can be greatly increased through biological recognition and as such effectively turns on the SERRS effect. This process can give rise to exquisite selectivity in terms of the interaction of the nanoparticles, especially when DNA hybridizations are used and single base mismatches can be analyzed at room temperature. Dye oligonucleotide silver nanoparticles (DOSN) have also been used to detect double stranded DNA through triplex formation to switch on the SERRS and a distance relationship between nanoparticles and SERRS response established for the first time. In an advancement of this approach functionalized nanoparticles have also been used as imaging agents for single cells and when functionalized with an appropriate antibody can give back information on the expression of specific receptors on cell surfaces as well as sub-cellular compartmentalization information. Finally in moving away from the *in vitro* applications the functionalized nanoparticles can be modified in such a way that they are active *in vivo* and preliminary data relating to *in vivo* studies of imaging and therapeutic uses of functionalized SERRS active nanoparticles will also be presented. This presentation covers the full range of design, the optical properties and finally the biological properties of functionalized nanoparticles in relation to specific disease states.

Potential Hazards & Benefits Of Nanomaterials

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ABSTRACT

Exposure to nanomaterials in sufficient dose may cause adverse effects in the body. The challenge for Nanotoxicology is to determine which physico-chemical characteristics of the nanomaterials drive their toxicity. This information would help in setting regulations for a more robust control of exposure and also inform the production of future nanomaterials that are 'safe by design'. While engineered nanomaterials may be exposed to workers and consumers inadvertently, in nanomedicine, nanoparticles (also known as nano delivery system) are used for targeted detection and drug delivery at the sites of the lesions. Here, the priorities are to deliver the drug at the right place, in sufficient amount, without side effects. In both cases, an understanding on how nanomaterials interact with the biological milieu is essential.

In this presentation, we will summarise the current knowledge on the potential hazard on nanomaterials and also their potential benefit in nanomedicine.

Applications of Nanomedicine in Diabetes

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ABSTRACT

Diabetes is a serious world health problem. Opportunities for applications of nanomedicine in diabetes are numerous.

In type 1 diabetes, improved insulin replacement is urgently needed, but transplantation of islet cells is not routine, limited by poor availability of human islets and post-transplant immune rejection. Longer survival of transplanted islets might be ensured by nano-encapsulation which protects islets from early cell death caused by the inflammatory in vivo environment and isolates against later immune rejection. We found allogeneic transplantation of mouse islets encapsulated with nanofilms made by layer-by-layer application of polysaccharides improved survival compared to naked islets, when transplanted into diabetic animals. In type 2 diabetes, early introduction of insulin is often resisted, but switching to insulin might be encouraged by non-injectable nano-formulations of oral insulin, where layer-by-layer nano-encapsulation of the protein may also be a suitable formulation.

In both types of diabetes, more stable and accurate continuous glucose monitoring is needed. We are researching nanosecond fluorescence lifetime photonics as a glucose detection technology. Mutants of glucose/galactose-binding protein (GBP) have been engineered and labelled with the fluorophore, badan. Glucose binding causes conformational change in the GBP and increased fluorescence lifetime and intensity. Prototype fibre-optic glucose sensors for subcutaneous implantation, based on GBP, have been constructed and tested. GBP-badan microvesicles have been fabricated and might be implanted eventually in skin as a 'smart tattoo'-type of non-invasive glucose sensor.

Automatic Synthesis of MIP Nanoparticles (“plastic antibodies”)

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Elena Piletska, Isabel Perez De Vargas Sansalvador,
Francesco Canfarotta and Michael J. Whitcombe
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ABSTRACT

Molecularly Imprinted Polymers (MIPs) are generic alternatives to antibodies and natural receptors in diagnostics and in separation. Here we report an efficient and flexible method for automatic synthesis of MIP nanoparticles using a solid-phasephotoreactor. Our approach requires a column-cartridge with an immobilised template docked into a thermostatic computer-controllable UV photoreactor, thereby allowing controlled manufacturing of affinity nanoparticles with narrow size distributions in the range 30-400 nm. We demonstrate the synthesisof water-soluble affinity nanoparticles for various targets such as melamine, vancomycin, drugs of abuse, peptides and proteins with minimal manual intervention and short reaction-cycle times. The affinity of all synthesised nanoparticles is at the nanomolar level which makes them suitable for practical applications in assays, sensors and in affinity chromatography. The MIP nanoparticles can be easily decorated with fluorescent, magnetic or electroactive labels for diagnostic applications. With this new development in MIP synthesis we foresee a time when the application of natural antibodies in diagnostics would be challenged by appearance of new sensor devices and assays thatutilize stable and inexpensive “plastic antibodies” with integrated recognition and signalling functionalities.

Solid-phase Synthesis of Molecularly Imprinted Nanoparticles ("Artificial Antibodies")

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ABSTRACT

Molecular recognition processes have a fundamental role in *in vitro* diagnostics, for which the estimated market size is $44 billions. Most of these devices rely on antibodies, which exhibit several disadvantages such as high costs, low stability and poor compatibility with microfabrication. Molecularly Imprinted Polymers (MIPs) are generic alternatives to antibodies in diagnostics and separation.

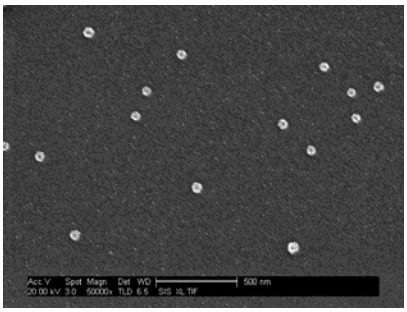

Figure 1. SEM image of automatically produced MIP NPs.

Here we report an efficient and flexible method to synthesise MIP nanoparticles using a solid-phase automated photoreactor. Our approach requires a column-cartridge with an immobilised template docked into a thermostatic computer-controllable UV photoreactor, thereby allowing controlled manufacturing of affinity nanoparticles with narrow size distributions. We demonstrate the synthesis of MIP nanoparticles with nanomolar affinity for various targets such as melamine, vancomycin, peptides and proteins, with minimal manual intervention and short reaction-cycle times. The solid-phase photoreactor developed in Cranfield is capable of producing 20 mg of "artificial antibodies" with size 30-400 nm per day(**Figure 1**).The performance of MIP nanoparticles is superior to those of polyclonal and even monoclonal antibodies which makes them suitable for practical applications in assays, sensors, and affinity chromatography.Furthermore, MIP nanoparticles imprinted for enzymes exhibited biological activity,making them a potential replacement for therapeutic antibodies.With this new development in MIP synthesis we foresee a time when the application of natural antibodies in diagnostics would be challenged by new sensor devices and assays that utilize inexpensive "artificial antibodies" with integrated recognition and signalling functionalities.

Nanomaterials Employed In Flexible Organic Field-Effect Transistors For Food Freshness Monitoring Applications

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ABSTRACT

Organic Field-Effect Transistors (OFETs) have already proven, in recent years, to satisfy the needs for low-cost, lightweight, and flexible electronic applications. However, realizing a low-power, flexible OFET is still an issue to be solved. The operation at low voltages may in fact allow the use of these OFETs in digital logic circuit applications like flexible displays [1], large area sensors [2] and radio frequency identification [3].

Here we present a low-cost Organic Field-Effect Transistor realized on a flexible substrate, using as the gate dielectric Poly(Vinyl Alcohol) (PVA), as the organic semiconductor Poly(3-Hexylthiophene) (P3HT), and as the gate material a nano-emulsion of polyaniline (PANI) in water. The structure used for this device is a planar-electrode configuration, but the device is realized in such a way that the gate material is the outer layer, resulting in a bottom-contact top-gate structure. Additional advantages are low-cost processes, compatibility with flexible substrates and relatively high dielectric constant for the PVA. The choice of nanodispersed PANI as gate material resides in its good conductivity and in its well known properties of being highly sensitive towards analytes such as ammonia. The good electrical performance obtained, opens the way for the employment of this device in gas sensing measurements of ammonia, that can be used in food freshness monitoring applications. For example amines are well known to be potential markers of spoilage for meat and fish and low cost sensors are desirable to track food quality. The high sensitivity of nanodispersed PANI towards ammonia, combined with the gate enhancement property of the field-effect transistor, make the device promising for gas sensing applications.

This work was supported by FlexSMELL-FP7-PEOPLE-ITN-2008-238454.

[1] G. H. Gelinck, et al., “Flexible active-matrix displays and shift registers based on solution-processed organic transistors,” Nature Mater., vol. 3, no. 2, pp. 106–110, Feb. 2004.
[2] M. E. Roberts, A. N. Sokolov, and Z. Bao, “Material and device considerations for organic thin-film transistor sensor,” J. Mater. Chem., vol. 19, no. 21, pp. 3351–3363, 2009.
[3] E. Cantore, et al., “A 13.56-MHz RFID system based on organic transponders,” IEEE J. Solid State Circuits, vol. 42, no. 1, pp. 84–92, Jan. 2007.

Mapping the Intestinal pH of *C.elegans* Using Ratiometric Extended Dynamic Range pH-Sensitive Nanosensors

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ABSTRACT

Caenorhabditiselegans is a non-parasitic soil nematode which has traditionally been used to model complex biological processes such as aging, genetics, and drug response. More recently, the *C.elegans* intestinal tract has been identified as a suitable model to discover new targets and therapeutics for acid suppression in mammals, to overcome diseases such as gastro-oesophageal reflux disease or Zollinger–Ellison syndrome.

Extended dynamic range pH-sensitive ratiometric nanosensorscapable of accurately mapping the full physiological pH range, from pH 4.0 to 7.5, have been developed and used to characterise, in real-time, the pH of the intestinal lumen of live *C.elegans* nematodes. Nanosensors, with a diameter of 40 nm, were prepared by immobilising pH-sensitive fluorophores, carboxyfluorescein (CF) and Oregon Green® (OG) in a 1:1 ratio, and a reference fluorophore 5-(and-6)-carboxytetramethylrhodamine (TAMRA) in apolyacrylamide matrix. Accurate ratiometric pH measurements were determined by calculating the fluorescence ratio between the pH-sensitive and reference fluorophores (λ 524 nm/λ 594 nm).

The pH-sensitive nanosensors were incubated with *C.elegans* prior to fluorescence microscopy imaging. Nanosensors were calibrated with McIlvaine pH buffer solutions ranging from pH 3.5 to 8.0andidentical imaging parameters were used to determine the intestinal Ph *of C.elegans*. The pH of the *C.elegans* pharynx, intestine and rectum was found to be 5.65 ± 0.68, 4.56 ± 0.63, and 4.82 ± 0.85, respectively.

Mapping the pH throughout the *C.elegans* alimentary system, a teach stage of its life cycle, could enhance the understanding of disease progression and the roles new and existing therapeutics play in acid suppression.

Self Assembled Odorant Binding Proteins As Biosensors For The Detection Of Odorants And Pheromones

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ABSTRACT

Odorant binding proteins are soluble proteins found in vertebrates and invertebrates and thought have important roles in odorant transport. We investigated the possibility of using odorant binding proteins (OBPs) for the detection of food odorants in both vapour and liquid phase. OBPs combined with different kinds of transducers can be employed in several practical applications. We prepared recombinant proteins from a selection of verterbrate and invertebrate OBPs and investigated their sensing characteristics using different types of transducer platforms. Our strategy was to consider the proteins as nanoparticles that could be self assembled on to a selected transducer platform. Target analytes were pyrazines which are highly important components of a variety of food odours and the human nose has very good sensitivity (low odour thresholds) to these compounds.

Recombinant OBPs from insects and vertebrates were immobilised on mass transducers (quartz microbalance) using a self assembled monolayer based on immobilisation of thioctic acid on a gold surface. These were used as gas sensors and a change in frequency was recorded when the sensors were exposed to analyte vapour.

Capacitative sensors were also constructed using immobilised porcine OBPs. The biosensor was constructed by immobilising pOBPm2 on screen printed gold electrodes using self assembled monolayers (SAMs) of thioctic acid or by entrapment in an agarose-hydrogel.

Both immobilisation methods were suitable for measurements in liquid phase, with the sensor showing gradual reduction of the initial capacitance of the biosensor with increasing concentrations of the analyte (2-isobutyl-3-methoxypyrazine). The results demonstrated that OBPs can be used as the sensitive layer for the detection of chemical molecules with a detection limits in the order of *parts per billion* for high affinity volatile compounds and lower than 1 µM in the case of capacitive liquid assays.

Acknowledgement: This work was supported by FLEXSMELL Gas Sensors on Flexible Substrates for Wireless Applications FP7-PEOPLE-ITN-2008-238454

Nanotechnology-Based Sensors For Air Pollution Monitoring In Confined Spaces: A Real Industrial Approach

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ABSTRACT

Emissions of air pollutants derive from almost all economic and societal activities. The majority of greenhouse gas emissions (GHG), acidifying substances, tropospheric ozone precursor emissions and material input caused by the life-cycles of activities related to consumption can be allocated to the main consumption areas of eating and drinking, housing, industry infrastructures, and mobility. Detecting pollutants in the air and determining polluted areas using an air monitoring system is important as the initial process of common air quality improvement techniques such as source control, improved ventilation, and air cleaning. In paper, we present different applications of nanotechnology-based sensors for air pollution monitoring in confined spaces, such an underground parking garage and a waste water treatment plant, that can be applied for gathering continuous data and controlling indoor environments. This work demonstrates how nanosensor technologies are ready for real applications in indoor atmospheric monitoring. Beyond this, the combination of both solid state sensors based on metal oxide semiconductor nanostructured materials (*nMOS*) and modern electrochemical cells joint in a single platform provide many advantages over simple-one-technology traditional sensor platforms. This multi-technology sensing platform offers high sensitivity to target gases, long term stability and very high response times. Besides it will also integrate wireless communications, internal data logging over an expandable flash memory, I/O analog and digital signals and alarms via cell phone.

Hybrid Composites of Polyaniline/Modified Carbon Nanoparticles Employed as Highly Sensitive Ammonia Sensors on Flexible Substrates

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ABSTRACT

Fabrication of low-cost and low-power chemical sensors on flexible substrates is aimed to open new applications in environmental monitoring and smart food packaging.Polyaniline (PANI), known for its excellent gas sensing properties, reversibly interactswith ammonia causing a significant decrease in its conductivity. By utilizing composites of PANI and carbon-based materials enhanced sensitivity toward amines has been observed. However, lack of solution processability has limited implementation of these materials in conventional flexible sensor fabrication methods.Here, we have prepared hybrid carbon/PANIcomposite NH_3 sensors using low-cost solution deposition methods. Sulfosuccinic acid, a multifunctional dopant, has been used to make electrically conducting polyaniline solutions in aprotic solvents, and these were characterised by UV-Vis spectroscopy.Hybrid composites were made by simply mixing surface modified carbon nanoparticles(20 wt.%) intodoped PANI solution.Chemiresistors were fabricatedonPEN and Kapton® flexible substrates incorporatinggold and silver interdigitated electrodes (20 µm gap), using drop casting and spin coating methods.The response of the sensor towards NH_3 vapour concentrations in the range of 200 ppb to 2 ppmwas measured and compared to that of a conventional MOS ammonia sensor.The effect of different substrates, electrode materials and deposition methods used were investigated. Successful fabrication of a low-power sensor with repeatable and reproducible response with sensitivities upto 5 times higher than MOS sensors,together with response and recovery times in minutes was achieved. The excellent performance parameters make these flexible hybridnanocomposite materials,attractive candidates for replacing conventional MOS sensors for low-power, real-time ammonia sensing applications.

(This work was supported by FlexSMELL-FP7-PEOPLE-ITN-2008-238454.)

Sensing Atomic-Scale Electric Fields By Self-Organised Lipid Layers On Hg

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ABSTRACT

The behaviour of the phospholipid monolayer on the surface of Hg deposited on microfabricated Pt electrode surface in response to potential changes has been studied using rapid cyclic voltammetry at $40Vs^{-1}$ ramp rates over a potential range from -0.4V to – 1.2V. The monolayers exhibit pronounced potential induced phase transitions characterized by two capacitance current peaks in real time. The peaks correspond to the redistribution of charges with the monolayer interface. Interactions of drug molecules and other compounds with the monolayer are demonstrated by recording changes in the capacitance current peaks in real time. Testing of various compounds has shown that depending on the structure of the compound, the presence of any side chains, or alterations to the shape of molecules, the sensitivity to that compound changes and therefore a range of detection limits of compounds has been observed. These compounds include steroids, polycyclic aromatic hydrocarbons, antipsychotics, tricyclic antidepressants, and others. Interaction does not rely simply on compound hydrophobicity; there are trends dependent on compound structure. The changes in phase transitions due to the interactions can also be used to identify a putative toxic contaminant in the environment. This can be applied specifically in toxicity sensing for water companies to assess exposure of hazardous substances. Furthermore, this nanotechnology based biosensor allows for early detection of toxic compounds in the water supply. The measurements are made in real time which means that potential toxic compounds are detected rapidly <10 minutes per assay. This technology will contribute greatly to environment safety and health.

The Detection of Solvent Dyes in Hydrocarbon Fuels using Surface Enhanced Raman Spectroscopy

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ABSTRACT

Solvent dyes are used to mark subsidised fuels such as solvent red dyes used to produce red diesel. These dyes have high solubility in non-polar solvents and poor solubility in water and tend to be anthraquinone (blue dyes) or azo (orange/red dyes) based. Standard methods of analysis include visible spectrophotometry and fluorescence with detection at the parts per million levels. Here we report a simple procedure using a silver colloid in which solvent dyes in hydrocarbon fuels are detected by Surface Enhanced Raman Spectroscopy (SERS). SERS offers detection at the parts per billion level providing a significant advantage over conventional methods of marker analysis. Details of the silver colloid and SERS technique will be presented along with suggestions of how the technique can be employed in a simple disposable sensing format.

POSTER ABSTRACTS

Nanosensors for pH Determination in Individual Cloud Droplets

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POSTER ABSTRACT

Cloud droplets are extremely heterogeneous in nature varying in chemical composition and pH. As yet individual cloud droplets have not been measured directly due to technical challenges. However, the pH in clouds is crucial in determining the pathways and rates of aerosol sulfate formation as well as the dissolution rates of key nutrients such as Fe and P for marine organisms from aerosols in clouds. A recent modelling study suggested that pH in clouds is one of the key uncertainties in climate models.

The use of nanosensors is therefore proposed as a novel analytical chemistry technique to measure the pH of individual cloud droplets. Qualitative measurements of pH can be achieved using fluorescent dyes which switch on or off at sharply defined pH values. A high density of fluorescent dye molecules may be covalently bound to a core nanoparticle platform. Nanosensors will be deposited onto 2D substrates, which can then be incorporated into standard cascade impactors to collect cloud droplets. Using confocal laser scanning microscopy in combination with fluorescence correlation spectroscopy the pH nanosensors will be calibrated in buffer solutions of known pH. Results obtained on real and model individual cloud droplets will then be compared to those predicted by current thermodynamic models.

CITI-SENSE: Development Of A Sensor-Based Citizens' Observatory Community For Improving Quality Of Life In Cities

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POSTER ABSTRACT

Driven by significant technological progress and the resulting increase in measurement accuracy, the use of low-cost sensors for environmental monitoring is currently experiencing rapid growth and stimulating the development of a new and highly active research field revolving around sensor technology.

CITI-SENSE is a recently-funded European project with the main objective to develop "Citizens' Observatories", involving citizens as active partners in environmental monitoring decision-making and empowering them to actively improve their own environment by using the information gathered by low-cost sensors.

In the project, citizens' observatories will be established in 9 cities across Europe (Barcelona, Oslo, Haifa, Vienna, Ostrava and the Silesia region, Beograd, Edinburgh, and Ljubljana) addressing important societal issues as air quality, noise, UV radiation, thermal comfort outdoors and indoor environments in schools.

Multi-parameter atmospheric quality monitoring based on low-cost sensor technologies will be implemented. The monitoring networks will be mainly based on mobile personal sensors. The resulting dense monitoring network (up to 100 participants in each city) will allow us to investigate exposure patterns based on geographical positioning, physical activity and other information. Furthermore, personal information will be provided as feedback to the citizens, and centralized for use by citizen and decision makers contributing to the achievement of community and societal policy objectives.

CITI-SENSE will provide a robust framework to evaluate sensor performance in laboratory and real world conditions and assess their potential for various environmental applications.