



ADAPTIVE MICROFLUIDIC - AND NANO - ENABLED SMART SYSTEMS FOR WATER QUALITY SENSING

# Mid-Period Report on Dissemination and Exploitation

Deliverable D6.3



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# Executive summary

This deliverable describes the dissemination activities carried out during the first 18 months of PROTEUS project, including publications in journals and conferences, demonstrations, and presentations at water related events and fora. Moreover, the deliverable addresses PROTEUS exploitation activities. The deliverable also provides a report on health and environmental risks that can arise from using carbon nanotube technologies.



# 1 Introduction

Water management requires massive, low-cost monitoring means coping with differentiated and evolving requirements. However, the majority of multifunctional water sensors only supports predefined goals hindering interoperability, with a high cost, impeding large scale deployments.

Addressing this, PROTEUS aims at offering x10 reduction in both size and unit function cost compared to state of the art. To this end, an increased number of functions will be integrated at a reduced cost and PROTEUS will deliver a reconfigurable nano-enabled sensor platform for cognitive water quality monitoring. Innovative embedded software will provide reconfigurability of the sensing board to support several differentiated applicative goals while cognitive capabilities will manage evolving requirements during exploitation. Energy autonomy will be made by harvesting several sources of energy. In addition, low cost of additional sensing components will enable redundancy increasing life span of the systems.

This document describes the dissemination and exploitation carried out in the PROTEUS project during the first 18 months, including publications in journals and conferences, demonstrations, presentations at water related events and fora, organisation of workshops, as well as training activities. It identifies the major dissemination fora and depicts the plans and actions for communicating the PROTEUS concepts and results into these fora preparing the exploitation phase. It gives an update of the exploitation plan based on consortium members experience and foreseen opportunities for PROTEUS outcomes. The deliverable also provides a report on health and environmental risks that can arise from using carbon nanotube (CNT) technology.

The deliverable is organized as follows: Section 2 presents in detail the dissemination strategy including standardisation plan, the various communication tools, activities in social networks and external events, scientific publications and networking. Section 3 provides details on exploitation activities. Section 4 presents the health and environmental risks from the use of CNT technology. Finally, conclusions are drawn in Section 5.



# 2 Dissemination Activities and their Impact

## 2.1 Overall strategy

### 2.1.1 Objectives

Contrary to the energy market that has been able to show significant changes and evolution due to the adoption of ground-breaking technologies, the water sector keeps seeking for reliable and cost-effective solutions to enhance its network monitoring capabilities. Even if the concept of “Smart water” has existed for years, its digital transformation is still at its early stage.

PROTEUS solution will bring a significant breakthrough in the water field by integrating some new technologies and concepts to easily monitor and manage rain & storm events, drink and waste water networks. To support this technology adoption, the consortium designed and set up, from project kick-off, a dissemination and exploitation strategy based on the innovation process. It first aims at creating and maintaining awareness about PROTEUS outcomes and then at preparing their exploitation and diffusion, as depicted in Figure 1.

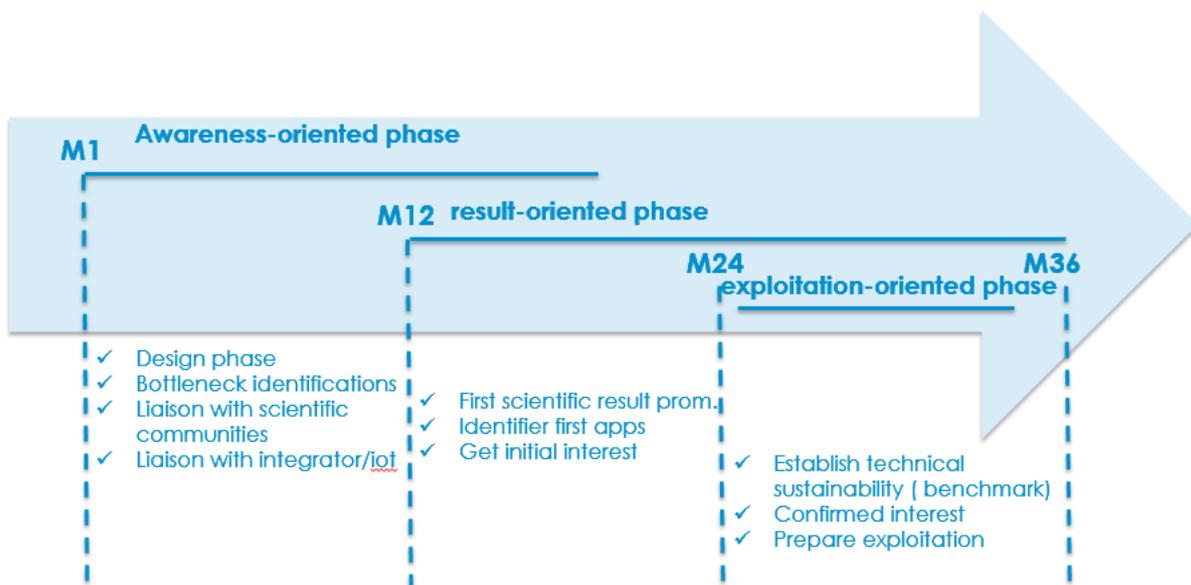


Figure 1 Dissemination and Exploitation Phases



## 2.1.2 Target groups

On hardware and software side, PROTEUS brings new solutions that could be translated in related fields. Even if the Smart Water is the main target market, our strategy strongly involves making other communities aware of the project. Potential users for PROTEUS sensor node or software such as IoT players or Smart systems integrators have a significant role to play, precisely like investors, policy makers, scientific/research communities and the general public also do. This strategy translated into a strong participation to events mixing the three target communities, illustrated in Figure 2, to enhance innovation translation.

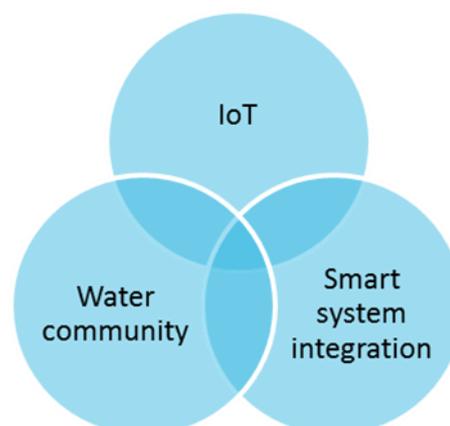


Figure 2 PROTEUS Target Groups

## 2.1.3 Key features in dissemination message

Our dissemination strategy was adapted depending on the targets but below are the key points derived from the features of the PROTEUS solution. We underlined at the same time the technical part with the integration of ground-breaking technologies and the business part with the cost-efficiency and the economic relevance of the solution:

- Using disruptive / advanced technologies reducing cost and size (CNT and MEMS)
- Fully integrated, highly multifunctional sensor chip with MEMS
- Printed carbon nanotubes sensors for multifunctional, highly selective chemical sensing
- Reconfigurable CMOS System on Chip (SoC) for Heterogeneous Integration with CNT and MEMS Sensing Devices
- Predictive, reactive and cognitive functions for reconfigurable and cognitive systems for multifunctional water quality monitoring
- Energy autonomy, with hybrid energy harvester from multiple sources

## 2.1.4 Key impacts in dissemination message

PROTEUS solution benefits from use cases that directly imply specific impact on water networks, may they be for drinking or waste or rain water. We focused in our dissemination message on main concepts such as cost efficiency and quality monitoring, impacting various actors:

- Low distribution cost of the sensor node in its industrial version, enabling it massive deployment for water networks of any kind and size.
- A win-win technology that may benefit every water stakeholders (utilities, public authorities, general public...)
  - Decrease in water costs (Drink & Waste)
    - Minimized water loss in networks
    - Optimized (less expensive) treatment strategies
  - Improved water quality
    - Accurate control for health and safety concerns
    - Reduction of environmental pollutions with enhanced monitoring.



## 2.1.5 Open Source and Standards

A central aim of the consortium is to benefit the European community. As such, some of the project partners are either using Open Source code to produce the content of their deliverables or are contributing their deliverables to the Open Source communities. Alternatively, some of the partners are contributing to Standards, may they be open standards or others. A key principle of the project open source strategy is to offer the project results in a business friendly way i.e. in a way that makes it possible for third-party European enterprises (i.e. enterprises outside the consortium) to benefit from the project results. To this end, the partners commit to choosing a business friendly license (MPL, LGPL...) for the project open source results.

The PROTEUS project aims at initiating and contributing to pre-standardisation activities so as to early on support the fast uptake of the solution by the end-users and stakeholders. To achieve these goals several challenges must be addressed. Standardization of semantic approaches and development of labelling strategies for water monitoring relating ontologies will need to take place. This could be envisioned as a follow-up of ongoing activities within ETSI SmartM2M<sup>1</sup>, currently targeting home energy management.

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<sup>1</sup> <https://portal.etsi.org/TBSiteMap/SmartM2M/SmartM2MToR.aspx>



## 2.2 Communication tools

PROTEUS project designed from its beginning a professional toolkit for consortium members to enhance dissemination and prepare exploitation. Each member play a key role because the consortium represents the whole value chain of water monitoring and this variety of profile grants us access to a variety of dissemination targets. The project branding participates in PROTEUS awareness by creating an unified graphical appearance to exchange with other projects, stakeholders... It also gives tips for quality management by implementing key data about versioning, authoring...

### 2.2.1 Public website

The official website was launched on February 10th, 2015 using the Wordpress CMS and integrating Google analytics to follow up its audience. The website address is "http://www.proteus-sensor.eu/". A screenshot of the website's home page is found in Figure 3.

#### 2.2.1.1 Content

According to the wordpress administration website, the website of Proteus comprises on June 30<sup>th</sup>, 2016:

- 17 main pages presenting the project, consortium, news, events and outcomes
- 25 posts highlighting members participation and special events

A special page has been set up for events, to announce partner's participation in conferences and water related events, to disseminate PROTEUS and to enhance networking and linking between people interested in PROTEUS outcomes.



Figure 3 PROTEUS website

#### 2.2.1.2 Audience

The Google analytics tool has been set on the website to establish a global overview of the audience. All figures are based on the half-project period, from February 1<sup>st</sup>, 2015 to June 30<sup>th</sup>, 2016.

The website registered more than 5000 sessions, representing around 4500 different users. Among the whole audience, 631 are regular visitors and represents the core basis for dissemination. The website referencing appears to have improved as we registered a long-term decrease of the bounce rate: this



indicator goes from 91% to 77% between June 2015 and June 2016, as depicted in Figure 4. It is confirmed by the increasing browsing time which is currently higher than one minute.

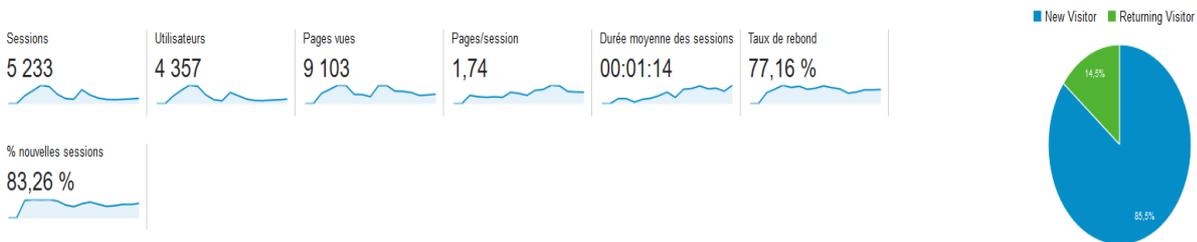


Figure 4 Website: Audience overview (Source: Google analytics – Data from 01/02/2015 to 30/06/2016)

Because our targets are not large public at this step, the analysis of visitors' behaviours is quite promising in terms of quality. This first period has enabled to create a settled basis of faithful public that will be able to pass on information and disseminate the project.

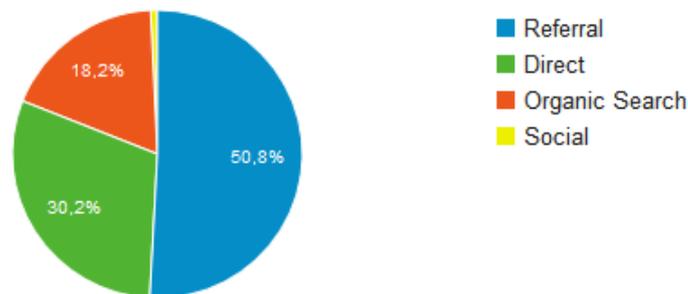


Figure 5 Website: Traffic sources (Source: Google analytics – Data from 01/02/2015 to 30/06/2016)

The audience acquisition comes mainly from referrals such as links from other websites or links in emails. Currently, about 110 active links to the address [www.proteus-sensor.eu](http://www.proteus-sensor.eu) are found on Google. One third of our visitors accesses the website by direct way, typing directly the website address in their browser. Visits from search engines remain non-prevailing and visits from social networks appear marginal. The geographical origin of the audience is global (Figure 6), with about 40% coming from Europe. Northern America represents one fourth but with very high bounce rate, search engines robots may explain that situation. France is the first European country followed by Portugal and United Kingdom.

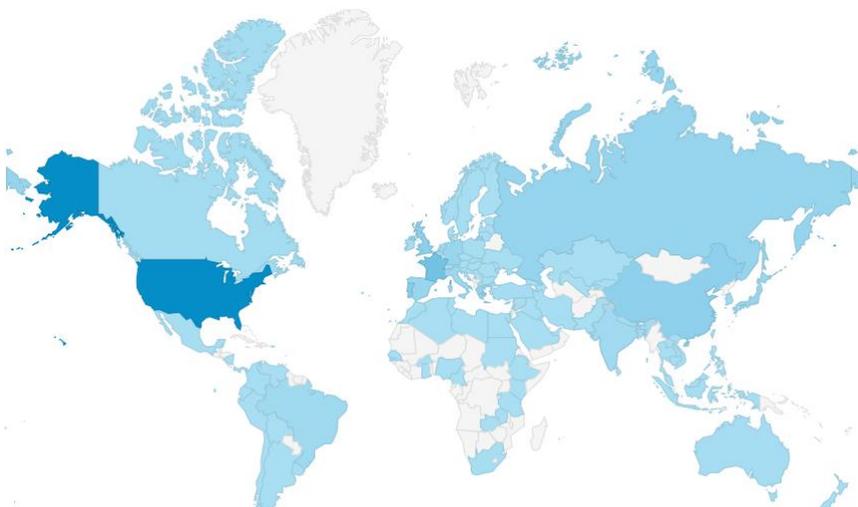


Figure 6 Website: Geographical distribution of visitors (Source: Google analytics – Data from 01/02/2015 to 30/06/2016)



## 2.2.2 Printed materials

The PROTEUS project benefits from a set of marketing materials to give support to the dissemination. An initial design was proposed at the beginning of the project and further developed in different supports and sizes.

### 2.2.2.1 Leaflets

The PROTEUS project provides two versions of the official project leaflet (Figure 7) gathering key information on objectives, use-case presentation, outcomes and contacts. An update has been published in February 2016 with double-sided printing and insertion of QR code with full contacts details and social networks. These leaflets are mainly used as visit cards as well as distributed in conferences during exchanges with contacts or future potential partners. At this stage, 1000 units of this new flyer have been given during events on the last semester.

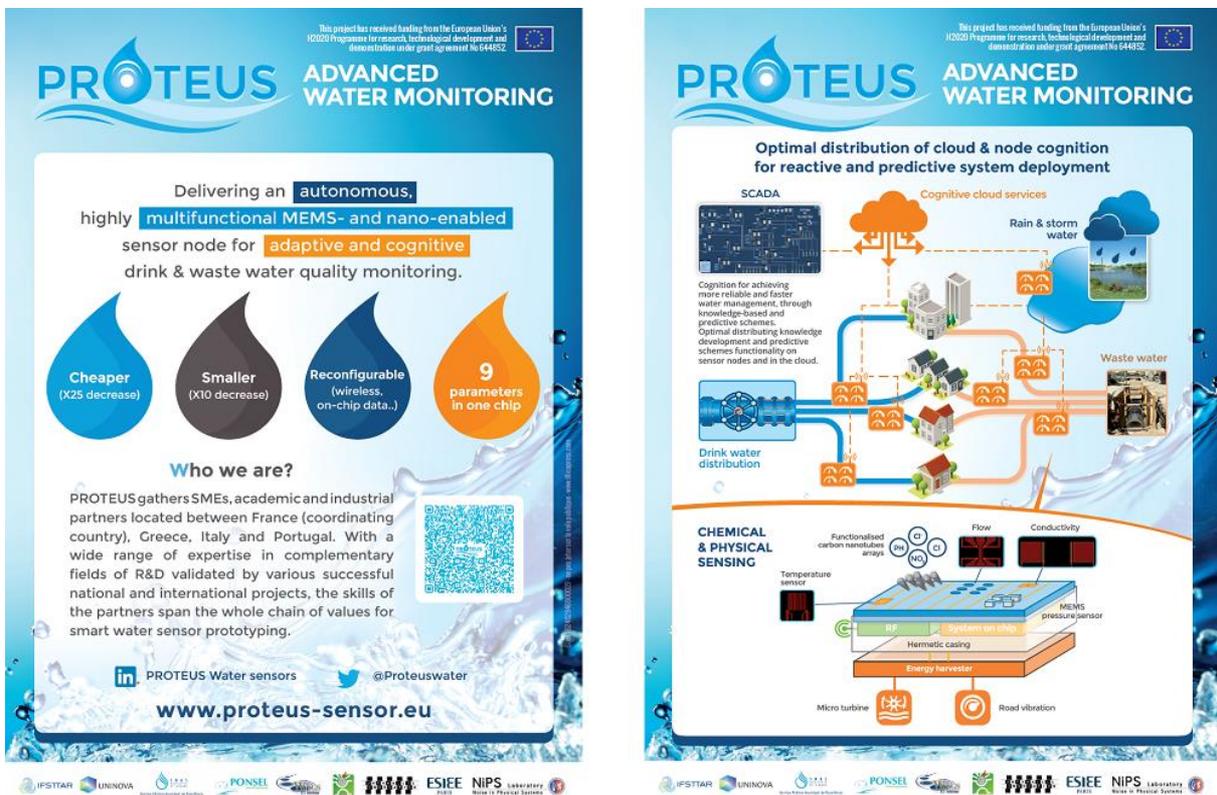


Figure 7 Official Project Leaflet

### 2.2.2.2 Other supports

To facilitate dissemination in conferences and target events, roll-ups and posters have been created. These materials are used on demand depending of the needs of each partner.



## 2.3 Social networks

During the annual assembly, held in Perugia on 2016, January 21<sup>st</sup> to 22<sup>nd</sup>, partners presented their main progress during 2015. This event also officially started project appearance in the social networks such as Twitter [@PROTEUSwater](https://twitter.com/PROTEUSwater).



Figure 8 Social networks: Twitter account overview

Twitter is used as an added channel to disseminate the PROTEUS project. On 2016, June 30<sup>th</sup>, key figures are the following (Figure 8):

- 16 tweets
- 20 follows
- 27 followers

The objective of this media is to get quality referencing on the topics related to the project. Even if the number of followers cannot be compared to website visitors, Twitter enables the project to get in touch with a selective and quality community.

Based on followers' respective audiences, this media actually fosters the dissemination of PROTEUS towards more than 13000 key contacts potentially interested in PROTEUS outcomes.



## 2.4 External events

### 2.4.1 IoT week Lisbonne



Figure 9 SMAS Almada Presentation in IoT Week 2015

During the IoT week 2015 which took place in Lisbon, PROTEUS made its place in the field of Smart Water management within the Industry Day:

Bruno Almeida introduced approaches to build IoT solutions tailored to specific market needs while preserving interoperability at higher levels. See IoT Week 2015 Bruno's presentation

Paulo Nico presented the challenges faced by utility companies such as SMAS Almada and how IoT can support them. See IoT Week 2015 Paulo's presentation (Figure 9).

### 2.4.2 WssTP Water Innovation Europe



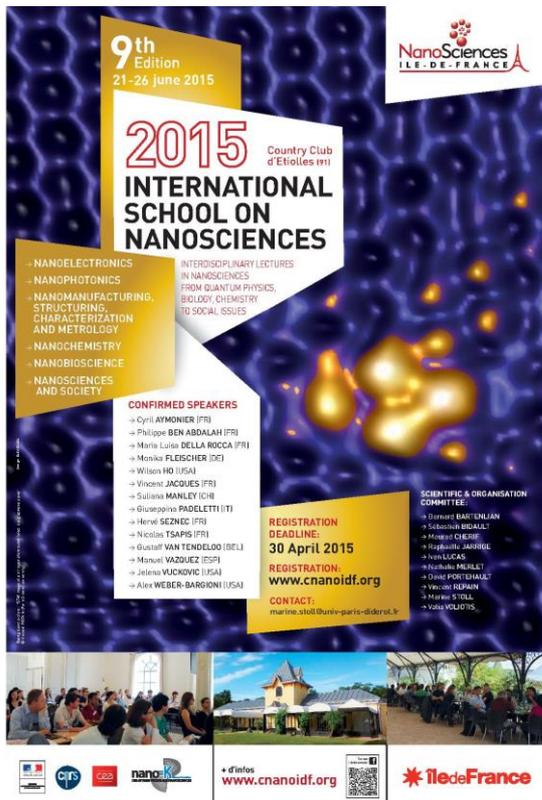
Figure 10 WssTP Water Innovation Europe

The 2015 edition of Water Innovation Europe took place from June 23th to 26th in Brussels. Focusing on "The role of water in the circular economy", (Figure 10) this summit aimed at identifying challenges and opportunities in that field and at defining how research and innovation may contribute to meet them.

Among the 250 European attendees, WINGS ICT Solutions took part in Group meetings and conferences to perform liaisons with other projects and initiatives and presenting main objectives and concept of PROTEUS for gaining visibility among water industries.



### 2.4.3 International Summer School on Nanosciences



From June 21th to 27th, C’Nano IDF organized the 9th edition of this summer school (Figure 11), welcoming about 50 researchers and PhD students. Lectures, seminars and debates built a rich scientific program mobilizing key international speakers on nanosciences and related disciplines (Electronics, photonics, biosciences...).

ESIEE Paris, represented by Massimo Pellegrino, attended this high added-value training and also presented a poster on PROTEUS outcomes. This training event was a right place to foster debates and create project awareness within the nanoscience community.

Figure 11 International Summer School on Nanosciences

### 2.4.4 MNBS Workshop 2015

On October 14th-15th, IFSTTAR, WINGS ICT Solutions and Easy Global Market represented the consortium in the 9th edition of MNBS Consultation and Concertation workshop on Micro-Nano-Bio Convergence Systems, Figure 12.




**Joint Event**

**EPoSS General Assembly & Annual Forum 2015**  
 “State of the (Sm)Art: Smart Systems Responding to Demand Side Requirements”

**MNBS 2015 9<sup>th</sup> Annual Concertation and Consultation Workshop on Micro-Nano-Bio Convergence Systems**  
 “Translate Technologies into Competitive, Validated and Manufacturable Products to Impact Quality of Life”

**12- 15 October 2015**  
**Imec**  
**Leuven, Belgium**  
[www.smart-systems-integration.org/annual\\_forum](http://www.smart-systems-integration.org/annual_forum)

Figure 12 EPoSS - MNBS

Conferences, posters demonstrations and panels animated the two-day event focusing on future stakes in the field and how to “translate technologies into competitive, validated and manufacturable products to impact quality of life”.



Paraskevas Bourgos, WINGS, participated in the poster session whereas Berengère Lebental, IFSTTAR, gave an overall presentation of PROTEUS project during the session on Smart Agriculture, Food and Environment. Spreading the word, this participation was also the opportunity to network with European projects in related fields such as agriculture, food sectors where ensuring quality, safety through monitoring are key requirements.

## 2.4.5 Aquatech / Aquamatch Amsterdam 2015



Figure 13 Aquatech Amsterdam

In the wings of the Amsterdam International Water Week, WINGS ICT Solutions participated on behalf of PROTEUS to two main events gathering international water technology professionals: Aquatech and AquaMatch (Figure 13).

The 2015 Aquatech Amsterdam trade show gathered 856 exhibitors with a visitor attendance of more than 18 000 people from 139 countries. This show being world's leading trade exhibition for process, drinking and waste water, this participation of WINGS enabled the consortium to get a wide overview of key stakeholders.

To capitalize on this first experience, WINGS participated in the AquaMatch event, a B2B pre-arranged speed dating. Tightening links with the water community, spreading the word towards companies, research institutes and universities, this event fostered the PROTEUS awareness and opened future cooperation.

## 2.4.6 Info day on Smart cities and communities



Figure 14 Info Day on Smart Cities and Communities

On November 6<sup>th</sup>, WINGS ICT Solutions participated in the Smart Cities & Communities Info Day in order to promote PROTEUS solutions for smart water management and water quality sensing in the broader context of smart cities, since water is a valuable resource and should be handled in an intelligent manner by cities of the future.

As a result, dissemination of concept and objectives of PROTEUS for further exploitation took place during the event.



## 2.4.7 10<sup>th</sup> Water conference



Figure 15 Paulo Nico Casimiro presentation



Figure 16 10th Water Conference

On November 11th and 12th, SMAS Almada participated in the 10th edition of Water Conference (Figure 15), dealing with the future of the Portuguese water sector and especially with how restructuring and innovation may act in the current context of a mutating sector.

Paulo Nico Casimiro (Figure 14) brought its testimony in the debates by presenting the PROTEUS project in the session “New innovation ideas, research and technological development applied to water market”. Central and local authorities, the whole water community but also academics showed a real enthusiasm for the project expected outcomes.

## 2.4.8 Green city Business 2015



Figure 17 Green city Business

On November 12th, IFSTTAR represented the consortium on its own stand during the Green City Business event held in Paris/Marne La Vallée, France (Figure 16). Organized on the fringe of the COP21, the United Nations conference on climate change, this event aimed at identifying innovation and reliable models for sustainable cities.



A PROTEUS roll-up presented the main expected outcomes, giving an example on how technology can help significantly for a smarter water management but also creating project awareness towards academics, public authorities and SMEs networks.

## 2.4.9 WssTP Brokerage and Working group event 2015



Figure 18 WssTP Brokerage and Working group event 2015

On November 23-24<sup>th</sup>, PROTEUS participated in the WssTP H2020 Brokerage and WG event 2015 (Figure 17). Within the different working groups, WINGS ICT solutions presented the PROTEUS project, discussing, interacting and exchanging with other European projects.

It constituted a new opportunity for project awareness and networking experiences. More specifically, the participation aimed at performing liaisons with other projects and initiatives, as well as at exploiting the distribution channels of the WssTP members in the water sector.

## 2.4.10 Optics valley seminar



Événement en partenariat avec:

Figure 19 Optics valley seminar

On November 27<sup>th</sup>, ESIEE Paris joined the debates during the Optics Valley seminar organized by the eponymous high technology cluster and hosted by IBM.

Even if drink water is one of the most controlled food products, there are some breakthrough innovations to develop before reaching a smart and efficient water management system. Health, environment, the seminar tried to deal with “Water stakes in urban areas: photonics solutions”.

To give some elements of a response, Tarik Bourouina, presented the research expected outcomes of PROTEUS project during the “Water quality” session. A real interest arose from scientifics and from the water community attending this event, as was highlighted in an article of the cluster newsletter.



## 2.4.11 ENEG Congress 2015



Figure 20 ENEG Congress 2015

On December 1st to 4th, all the Portuguese water management actors met in Porto, Portugal at the ENEG Congress 2015 (Figure 19) to analyse and debate on the challenges to overcome for a sustainable water sector. SMAS Almada was naturally there.

All along these four days, the delegation participated in conferences, exhibition and events, rising awareness on PROTEUS project. In parallel, a survey was conducted to define users' needs and requirements. Results should be available soon and will be disseminated within the consortium and beyond. It built a true progress for PROTEUS but also for SMAS, that got two more awards in this edition: "Golden tube" in "Best institutional and business information" - 1<sup>st</sup> place in "Portugal Pipe Contest 2015".

## 2.4.12 European Nanoelectronics Forum 2015

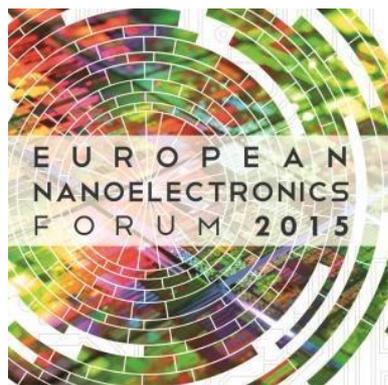


Figure 21 European Nanoelectronics Forum 2015

From December 1st – 2nd, PROTEUS project participated to the annual European Nanoelectronics Forum in Berlin, Germany (Figure 20). Welcoming public authorities, the business world and the research community around the theme of "Driving Digital Economy", this event was a real showcase for the PROTEUS project.

Among 70 innovative projects, PROTEUS took part in the exhibition with a poster in the "Project village", showing an overview of the technology and objectives being developed by the consortium. As the project recently started, João P. Oliveira from UNINOVA, on behalf of PROTEUS, met potential partners that will open, within the next weeks, new exchanges within the Nano-electronics community.

## 2.4.13 IEEE Mems Shanghai



Figure 22 IEEE Mems Shanghai

From January 25<sup>th</sup> – 28<sup>th</sup>, PROTEUS project participated to the International Conference on Micro Electro Mechanical Systems held in Shanghai, China (Figure 21, 22). This 29<sup>th</sup> edition welcomed more than 700 participants, involved or interested in the major areas of activity of MEMS research and applications.



Figure 23 IEEE Mems Shanghai

A delegation of Esiee, composed by Tarik Bourouina, Frédéric Marty and Philippe Basset, represented PROTEUS at the event, establishing contacts with other MEMS related projects and ensuring dissemination with a selective diffusion of leaflets.

During side meetings, Esiee took part of ICAN 2016 organization committee as reported in the January newsletter. This international contest of applications in nano-micro technology will be held on July in Paris, a PROTEUS demonstration is likely to be done during this large-scale high-tech innovation contest initiated by scientists from both academy and

industry in micro/nano fields.

## 2.4.14 Leeuwarden Water week



Figure 24 Leeuwarden Water week

On February 2016, the city of Leeuwarden in the Netherlands was the place to be for Water related stakeholders. Ponsel (Bertrand Vergne), WINGS ICT Solutions (Paraskevas Bourgos) and Easy Global Market (Philippe Cousin & Nicolas Giunta) represented PROTEUS project during three main events. For this occasion, a new double sided version of the project leaflet was released.

On February 9<sup>th</sup>, Nicolas Giunta from Easy Global Market presented an overview of PROTEUS to the ICT4water cluster (Figure 24). This side event, presenting all the projects members of the cluster, provided us with the opportunity to tighten the links

with other cluster members but also with the water community overall and stakeholders attending the presentations. Paraskevas Bourgos, WINGS, contributed the more technical details and expected outcomes to the debates. The evening networking reception, held in Wetsus research centre, strenghtened this first approach .

On February 10<sup>th</sup>, all represented Proteus partners attended the 3<sup>rd</sup> EIP Water Conference gathering 550 people from research, industry, public authorities and other stakeholders around the theme "How is water innovation succeeding in Europe?". Insightfull conferences gave a tempo to the day, as wells as booth visits in the exhibition space gathering research projects and industrial solutions.

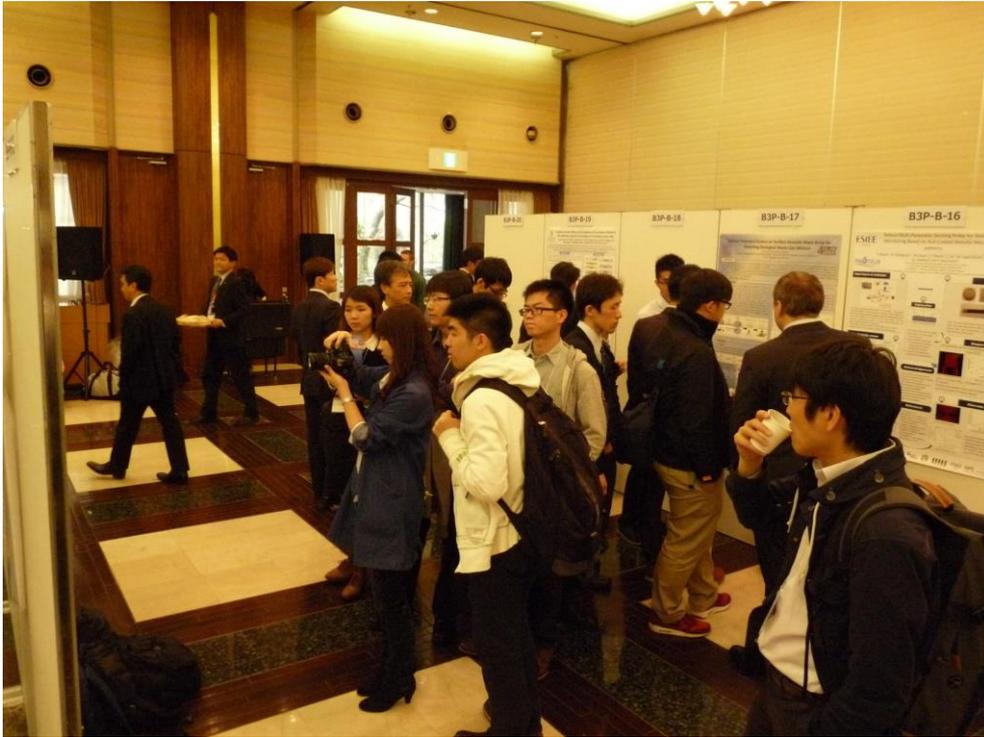


Figure 25 EGM at the ICT4Water Cluster

On February 11<sup>th</sup>, PROTEUS members attended the Mayors and water conference aiming at networking and disseminating towards 150 public structures. Conferences highlighted the main stakes for water management and how innovation can make the cities smarter.



## 2.4.15 IEEE Nems Conference



*Figure 26 IEEE Nems Conference*

PROTEUS partners participated into the 11th Annual IEEE International Conference on Nano/Micro Engineered and Molecular Systems (IEEE-NEMS 2016) held in Matsushima Bay and Sendai MEMS City in Japan (Figure 25). From 17<sup>th</sup> to April 20<sup>th</sup>, this international conference presented the latest scientific and technological research on Nems and related topics such as fabrication or integration with devices, sensors or systems.

On the basis of the joint work done, Esiee and Ifsttar submitted a paper on “Robust Multi - Parameter Sensing Probe for Water Monitoring Based on ALD - Coated Metallic Micro-Patterns” presenting some of the advances on PROTEUS work concerning long-term reliability of sensors immersed in water:

- multi-sensing probe for water network monitoring enabling simultaneous measurements of water electrical conductivity, flow-rate and temperature
- coating process using Atomic Layer Deposition (ALD) in order to reduce both electro-erosion and biofouling, while keeping the sensor’s electrical and thermal functionalities

These results were presented during the poster session on April 19th, towards an heterogeneous public gathering researchers, academics, students...



## 2.4.16 IoT Week Belgrade



*Figure 27 EGM at IoT Week*

Each year, the IoT week gathers the key players of the internet of things community. Philippe Cousin and Franck Le Gall, from Easy Global Market, participated from May 31<sup>st</sup> to June 2<sup>nd</sup>, presenting relevant outcomes from our research projects. Easy Global Market gave some highlights by presenting to IoT leaders, researchers, decision makers and company representatives a part of our work on the topic.

PROTEUS project was depicted, giving an overview of concrete and innovative examples of IoT applications and especially on the use of nanotech based sensor for water monitoring developed within the project.



## 2.4.17 Water Innovation Europe



*Figure 28 WINGS receiving the WssTP SME Award*

With over 200 participants from all around Europe, Water Innovation Europe is the landmark event for the European water sector. The event brings together all the aspects of the sector: scientists and technology developers, utility representatives, large water users, European and national/ regional policy makers and finance experts. Water Innovation Europe offers an open platform for information gathering and networking among the most influential stakeholders from within and beyond the water sector. Participation goals: Shape Europe's water future. Connect and collaborate with your peers. Raise your profile and visibility. Stay ahead of the curve.

WINGS participated to the following sub-events of Water Innovation Europe:

- Participation to a panel on "Water on the interface between digital and the physical world" session. Which is the role of technological SMEs in water & environment challenges and how do you see the impact of connectivity, IoT, wearables, software networks or big data analytics?"
- SME contest and awards. Presentation of WINGS technology innovation pitch as an SME and also as part of the PROTEUS project. WssTP SME contest 2016 award reception.
- SME Award reception.

WINGS ICT Solutions received a WssTP Water Innovation SMEs Award (Figure 27) for the software technology innovations developed in the context of PROTEUS. For this third edition, held the first day of Water Innovation Europe 2016, the judging panel rewarded the advanced features of the solution offering:

- The required adaptability that enables a single device to monitor several, highly differentiated, water parameters (e.g., flow-rate, temperature, nitrates, chlorine)
- Predictive capabilities for quickly and reliably identifying problems



This prize identified the best practices in their field of application with high market potential, announcing promising results for PROTEUS outcomes. This event also gave key opportunities to network with key European stake-holders over three days, gathered around the theme of ‘Water-Smart! European Solutions for a Smart Water Society’ in Brussels.

## 2.4.18 MIXDES Conference 2016



*Figure 29 UNINOVA at MIXDES Conference 2016*

Held in Lodz, Poland, the 23<sup>th</sup> edition of the MIXDES Conference (Mixed Design of Integrated Circuits and Systems) acted as a key European forum presenting recent progresses in micro- and nano-technologies components and related items. From June 23 to 25<sup>th</sup>, this event offered a broad overview by dealing with all innovation processes such as design, modelling, simulation, testing and manufacturing.

Pedro Joao Oliveira (UNINOVA) participated to this event as invited keynote speaker (Figure 28), presenting an overview of the research developed within the PROTEUS project. The presentation, carried out during the plenary session, focused on the advanced sensing platform for water monitoring, and was entitled “Advanced Amplification Techniques for Nanoscale CMOS in the Context of IoT Node Sensors”. An homonymous paper on this topic will be soon published in IEEE database.



## 2.4.19 EUCNC Conference 2016



Figure 30 Panagiotis Vlacheas (WINGS) presenting PROTEUS Demo to Eva Kaili, Member of the European Parliament

The 25<sup>th</sup> edition of the EUCNC (European Conference on Networks and Communications) took place in Athens from June 27<sup>th</sup> to 30<sup>th</sup>.

A team from WINGS ICT Solutions presented during this event the first demonstration of the PROTEUS project (Figure 29). Nikos Monios, Panagiotis Vlacheas and Paraskevas Bourgos animated the dedicated booth showing the status of on-going research in advanced networks applied to water field.

This demonstration, depicted in Figure 30 below, entitled “Embedded and Cloud-based software for smarter (reactive, predictive, cognitive) Smart Systems for Water Monitoring” gave an overview of reactive and predictive capabilities designed by WINGS within the PROTEUS project with an introduction of the hardware platform used for the prototyping of the smart system and a presentation showing system features based on testing scenarios.

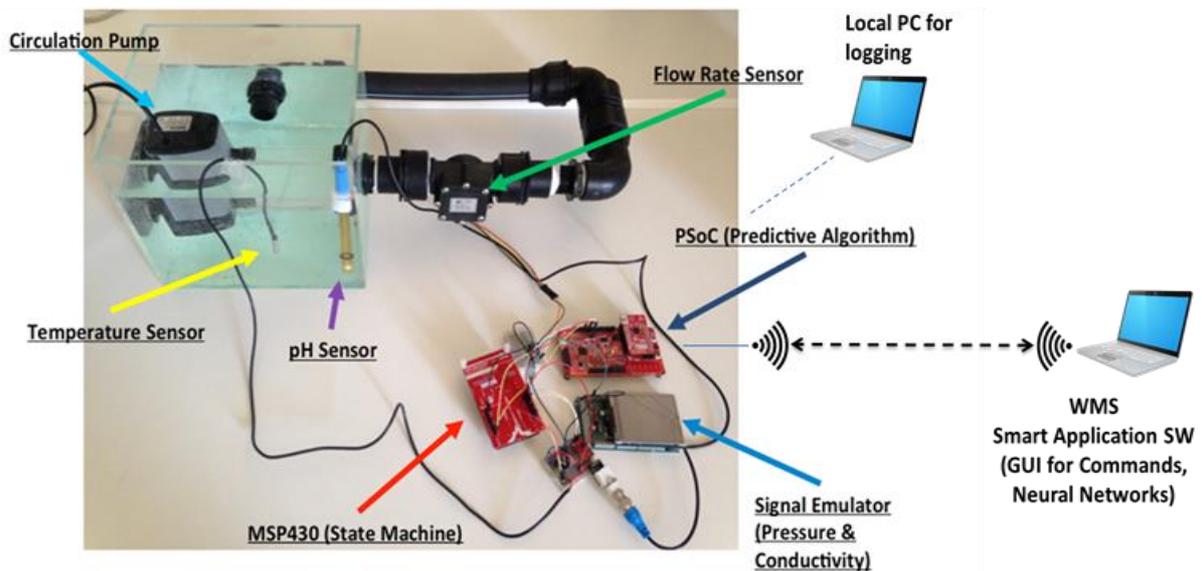


Figure 31 Demonstration Architecture



As long-term management of water resources requires widespread, low-cost monitoring system able to connect with highly differentiated requirements and adaptive capabilities, this software demonstration showed a relevant example of smart system in water network monitoring. From real environment to digitized networks, WINGS presented how a single device architecture may support several, highly differentiated applicative goals and highlighted the fallouts of cognitive capabilities in the way to manage evolving contexts and requirements during exploitation even in a predictive manner.



Figure 32 Paraskevas Bourgos (left) and Nikos Monios (Right) at the WINGS booth

The scope of the demo was to present the Software Integrated Prototype developed by WINGS. We introduced the hardware platform used for the prototyping of the smart system and we presented the demonstration and testing scenarios targeting the innovations listed below:

- To enable reactive capabilities in the smart system.
- To enable predictive capabilities in the smart system and SCADA.

## 2.4.20 ISOCS-MiNaB-ICT-MNBS Joint event



Figure 33 ISOCS-MiNaB-Ict-MNBS

From June 27<sup>th</sup>, this workshop gathered in Otranto, Italy, over three days about 40 projects representatives on the theme “Sensing for Smart Anything Everywhere: Materials, Technologies, Applications”, Figure 32.

Easy Global Market, member of the PROTEUS consortium, participated in and presented the project towards this selected audience. Focusing on results exploitation and market access, we briefly showed Proteus outcomes and current status, and highlighted the innovation process and market access strategy. Experts, researchers, project leaders mainly in e-health, food & safety showed a real interest in the water monitoring example.

As a non water related events, this joint event was a real opportunity to network with other initiatives and get inspired by what it is done in other fields regarding to technology, methodology and strategy.



## 2.5 Scientific Publications

	Publications in conference proceedings		
Title	A Multi-cell SC DC-DC Converter Controller with Power Aware Output Ripple Reduction	Gm-C Biquad Filter for Low Signal Sensor Applications	Analysis and implementation of a power management unit with a multiratio switched capacitor DC-DC converter for a supercapacitor power supply
Authors	Ricardo Madeira, João P. Oliveira, and Nuno Paulino	Richa Arya, João P. Oliveira	Madeira, Ricardo and Paulino, Nuno
Title of the journal or equivalent	12th Conference on PhD Research in Microelectronics and Electronics	Mixed Design of Integrated Circuits & Systems (MIXDES), 2016, To be published in IEEE database	International Journal of Circuit Theory and Applications
Number date			Mar-2016
Publisher	FCT-UNL	FCT-UNL	FCT-UNL
Year	2016	2016	2016
Peer-review			Analysis and implementation of a power management unit with a multiratio switched capacitor DC-DC converter for a supercapacitor power supply



	Publications in conference proceedings		Article in journal
Title	Improving the efficiency of a 2/1 SC DC-DC converter using the parasitic capacitances	A low voltage low power temperature sensor using a 2nd order delta-sigma modulator	Effect of boundary conditions on piezoelectric buckled beams for vibrational noise harvesting
DOI	10.1109/DCIS.2015.7388607	10.1109/DCIS.2015.7388608	10.1140/epjst/e2015-02593-5
ISSN or eSSN			1951-6355
Authors	Madeira, Ricardo; Paulino, Nuno	Quendera, Filipe; Paulino, Nuno	Cottone, F.; Mattarelli, M.; Vocca, H.; Gammaitoni
Title of the journal or equivalent	Conference on Design of Circuits and Integrated Systems (DCIS)	Conference on Design of Circuits and Integrated Systems (DCIS)	The European Physical Journal Special Topics
Number date	Nov-2015	Nov-2015	Volume 224 Issue 14-15
Publisher	IEEE	IEEE	Springer
Place			
Year	2015	2015	2015
Pages	1-5	1-6	
Public & private participation			NO
Peer-review	YES	YES	YES



	PhD Dissertation	
Title	Wideband CMOS Low Noise Amplifiers	Wireless Nano Sensors for Embedded Durability Monitoring in Concrete
Authors	Ivan Bastos	Fulvio Michelis
Title of the journal or equivalent	Phd Thesis	Phd Thesis
Number date	Dec-2015	Oct. 2015
Publisher	FCT-UNL	Ecole Polytechnique
Year	2015	2015
Public & private participation	YES	



## 2.6 Networking

### 2.6.1 ICT4water cluster



The PROTEUS project officially joined the ICT4Water cluster which gathers international projects combining ICT actors and the Water management community. This new membership brings key information and contacts for the project and opened new collaborations for subsequent events such as the Water Utility Week in November 2016 in Barcelona. Synergies will be pursued in 2017 with presentations from some cluster partners in workshops, fostering networking, and giving additional dissemination such as:

- Publication in the January newsletter
- February overview presentation during the Leeuwarden water week, Netherlands
- Project status delegated presentation at the cluster meeting in June 2016, Jerez de la Frontera, Spain.



## 2.7 Strategy update

As PROTEUS outcomes tend to yield significant results, as we have seen above, via various awards obtained by the partners (SMEs Award for WINGS software part during the Water innovation Europe, PRIME2016 Bronze leaf Certificate Paper Award for the CMOS chip, 2<sup>nd</sup> prize for UNINOVA and ESIEE students joint team at the ICAN - International Student Competition on Innovation on July 2016), PROTEUS members will pursue their efforts to enhance dissemination and exploitation.

Each partner played a key role in communication by publishing, presenting, and networking. Now, as the experimentation step is starting in Proteus, our work will focus further on exploitation in the second period. Dissemination will continue, strengthening awareness around the project, capitalizing on networking contacts to build up continuously prospects and potential stakeholders. This will lead to wide broadcast of concrete materials via newsletters (1<sup>st</sup> on September 2016). This will aim at giving continuous information on the project progresses and at staying connected with people interested in PROTEUS outcomes.

This constant information feed will be completed by press releases, creating « event » for medias announcing key progress in PROTEUS, such as on September 2016, the Sense-city experimentation. This dynamics will be emulated by a summer school organized by NIPS in August 2016 and by two main workshops, giving clues of our exploitation strategy:

- First workshop in Pollutec, Lyon, December 2nd presenting PROTEUS fallouts for water related actors.
- Second workshop to be defined on “Technology translation” witnessing on Smart Water Monitoring experience and widening debates on other applications of PROTEUS main technologies in others areas, fields and industries.



## 3 Exploitation of Results

### 3.1 General Approach

Within the next three years following the project, it is expected that about 1000 units of PROTEUS pre-series will be distributed, thus providing added values to the first end-users (network operators, including SMAS, partner of PROTEUS):

- Investment cost: at constant number of sensor nodes, lower investment for improved monitoring capabilities; at same investment level, more widespread monitoring capabilities
- Installation cost: decreased due to fully wireless capabilities (quicker and simpler to install); less constraints on positioning in network (does not require power line)
- Maintenance cost: no onsite operator, reduced maintenance operations, longer lifetime

Further lowering of OPEX and CAPEX is expected due to PROTEUS capability to monitor different types of water networks based on its re-configurability by design. However, no quantitative information on pricing is available for such applications, as waste and rain water network monitoring is more marginal than drink water network monitoring (though more important for environmental purposes).

The distribution in small quantity of PROTEUS first set of lower cost, compact multifunctional water probe will increase the dissemination of PROTEUS solution to a wide range of end-users. It is expected that within three years after the end of PROTEUS, based on these dissemination actions, the market will be ready (large sales volume expected) to welcome an industrial version of PROTEUS system. Indeed, the main cost in PROTEUS pre-series is the cost of CMOS chip, which is directly related to distributed volume (about 500€). If a large enough market opens up, full CMOS run may be envisioned, dividing the cost of the CMOS chip by at least a factor 10 or more, typically reaching less than 50€. Altogether, following industrialization, the full system might actually be commercialized at less than 100€, a price range that should finally truly enable massive deployment of multifunctional probes for water monitoring.



## 3.2 Partners' Perspectives

Partners	Plan	Actions Performed
IFSTTAR	<ul style="list-style-type: none"> <li>– CNT sensor technology: technology transfer to industrial partner (established company or start up) for product manufacturing and commercialization</li> <li>– Automated water sensor calibration bench: know-how transfer to industrial partner</li> <li>– Paid access to Sense-City water loop for water network technology validation by industrial stakeholders</li> </ul>	<p><b>Technical Workshops Participation</b></p> <ul style="list-style-type: none"> <li>– TNT 2016, Freiburg, Switzerland, September 2016 – planned participation</li> </ul> <p><b>Meetings with external stakeholders</b></p> <ul style="list-style-type: none"> <li>– Horiba Jobin Yvon</li> <li>– Veolia</li> <li>– SOMEI (Marseille water company)</li> </ul> <p><b>Synergies Established</b></p> <ul style="list-style-type: none"> <li>– MBA Entrepreneurship Track (technology transfer of CNT sensors – planned participation)</li> </ul> <p><b>Communication with Other Projects</b></p> <ul style="list-style-type: none"> <li>– PLATINE program at Ecole Polytechnique (reliability of nanodevices)</li> <li>– Sense-City project</li> <li>– Poseidon project</li> </ul>



<p>ESIEE</p>	<ul style="list-style-type: none"> <li>– Monolithic MEMS chip platform for multi-parameter physical and chemical sensor integration: technology transfer of IP and know-how to industrial (established company or start up) for product manufacturing and commercialization.</li> <li>– ALD – coating for improved reliability of water sensors: Technology transfer of IP know-how.</li> <li>– Manufacturing services of MEMS chip multiparameter sensor platforms from prototype to small-scale production and until technology transfer to third party for large scale production.</li> </ul>	<p><b>Meetings with external stakeholders</b></p> <ul style="list-style-type: none"> <li>– ICAN 2016 international student competition on innovation, July 7, 2016, Paris (joint project UNINOVA-ESIEE won the second prize)</li> <li>– VEOLIA innovation contest (planned participation to this contest)</li> </ul>
<p>UNINOVA</p>	<ul style="list-style-type: none"> <li>– Mixed-mode design, technology transfer through IP and consulting services to industry (established company or start up) for product development, with special emphasis on low-voltage and low power design developed for PROTEUS, using the latest CMOS technology nodes.</li> <li>– Mixed-mode design consulting and testing services, with special emphasis on low-voltage and low power design developed for PROTEUS, using the latest CMOS technology nodes.</li> <li>– Mixed-mode design training, with special emphasis on low-voltage and low power design (consolidated in PROTEUS) using the latest CMOS technology nodes.</li> </ul>	<p><b>Meetings with external stakeholders</b></p> <ul style="list-style-type: none"> <li>– ICAN 2016 international student competition on innovation, July 7, 2016, Paris (joint project UNINOVA-ESIEE won the second prize)</li> <li>– VEOLIA innovation contest (planned participation to this contest)</li> </ul> <p><b>Communication with Other Projects</b></p> <ul style="list-style-type: none"> <li>– Project DISRUPTIVE</li> </ul>
<p>SMAS</p>	<ul style="list-style-type: none"> <li>– Promotion activities among national water stakeholders to present PROTEUS achievable results</li> <li>– Participating in educative training activities, demonstrating how the results achieved by</li> <li>– PROTEUS can contribute to reach a more sustainable and efficient operation.</li> </ul>	



<p>PONSEL</p>	<ul style="list-style-type: none"> <li>- Aqualabo Controle forecast to use the sensors alone (without radio communication but with RS485).</li> <li>- Aqualabo Controle will integrate the radio communication system developed in the project for its range of sensors.</li> <li>- Aqualabo Controle will integrate in its automatic factory calibration the sensor to be able to provide a calibration service to end users.</li> <li>- Aqualabo Controle expect to rent the sensors and to sale it to the company operating on the rental market.</li> </ul>	<p><b>Technical workshops participation</b></p> <ul style="list-style-type: none"> <li>- One presentation by a flyer distribution to the public to the African Water Association in relation with the subject “potable Water Security in Africa” during the AFWA Exhibition in Nairobi/Kenya.</li> <li>- Presentation in Pollutec Lyon in December 2016.</li> </ul> <p><b>Meetings with external stakeholders</b></p> <ul style="list-style-type: none"> <li>- Aqualabo Controle is in relation with strategic applications of the big Water Operator (Veolia/Suez/Eau de Paris/Saur).</li> </ul> <p><b>Communication with Other Projects</b></p> <ul style="list-style-type: none"> <li>- Aqualabo Control is a partner to a French National Project (FUI): “CAPENC” working on a fouling sensor based on conductivity and thermic measurement. The sensor will be a MEMS. The PROTEUS partners help us for that project.</li> </ul>
<p>EGM</p>	<ul style="list-style-type: none"> <li>- EGM planned to act as a PROTEUS system integrator, acting as a relay for diffusion of the project solution. Our experience in interoperability, IoT platform and testing will be used in order to set up Smart Water solutions within drink and waste water industry. Others target industries may be reached in a second time with technology adaptation such as Maritime, Bio environment monitoring, smart city and individuals markets.</li> </ul>	<p><b>Meetings with external stakeholders</b></p> <ul style="list-style-type: none"> <li>- EGM established links with marine research centre to evaluate innovation potential for the PROTEUS solution. Some PROTEUS outcomes are already required by these actors, some aspects needs technology translation to meet with special needs of environmental monitoring. EGM is also working with Africa for fish farming where such low-cost solutions are in demand.</li> </ul> <p><b>Synergies Established</b></p> <ul style="list-style-type: none"> <li>- A partnership has been established with PONSEL-AQUALABO to investigate the exploitation of PROTEUS results, starting with</li> </ul>



		<p>integration of existing sensors from PONSEL and EGM IoT platform. This aims at being experimented in real conditions in Sophia-Antipolis region with PROTEUS sensor proposed as a midterm evolution for wider scale deployment.</p> <p><b>Communication with Other Projects</b></p> <ul style="list-style-type: none"> <li>– EGM maintains links with other EU projects related to IoT topics and especially WAZIUP project that fosters use of IoT in sub-Saharan Africa. As the PROTEUS solution is designed for a large-scale implementation and with reduced industrial costs, we identified strong synergies.</li> </ul>
<p>WINGS</p>	<ul style="list-style-type: none"> <li>– Enhance WINGS’s Cloud-IoT platform by developing, integrating and validating smart components, for the predictive and cognitive management of water resources; these components will be optimally distributed from the chip level to the cloud level.</li> <li>– Interaction with water industry experts for deriving requirements, accessing relevant data and validating the new solutions.</li> <li>– Existence of multifunctional and reconfigurable sensing hardware to apply the software solutions.</li> <li>– At first stage, both conditions are satisfied through the collaborations inside PROTEUS. At second stage, WINGS aims at developing a complete solution towards full commercialization through point to point collaborations in water management domain, and by seeking funds from venture capitals.</li> </ul>	<p><b>Technical workshops participation</b></p> <ul style="list-style-type: none"> <li>– Participation in the WssTP "Strategic Innovation Research Agenda (SIRA) 2030 Workshop" and in the "Water &amp; ICT" work group at 21 June, as chapter leader in the "Water &amp; ICT" roadmap.</li> </ul> <p><b>Meetings with external stakeholders</b></p> <ul style="list-style-type: none"> <li>– Participation in the Water and Innovation Event in Brussels at 21-23 June 2016, organised by WssTP. <ul style="list-style-type: none"> <li>– Participation as a panelist in the "Water on the interface between digital and the physical world" session.</li> <li>– Presentation of WINGS technology innovation pitch as an SME and also as part of the PROTEUS project.</li> </ul> </li> <li>– Participation in Aquatech, the international water technology exhibition in Amsterdam 3-6 November 2015 at the RAI conference center, in the context of PROTEUS Project. Participation in the Aquamatch bilateral meeting at the exhibition area on Thursday 5 November 2015.</li> <li>– Participation in Water Event in Leeuwarden 9-11 February 2016, the</li> </ul>



		<p>Netherlands. Specifically, participation in EIP Water Conference at 10 February, side work group meeting at 9 February and site visits at wastewater treatment facilities at 11 February.</p> <p><b>Synergies</b></p> <p>WINGS is a member of WssTP, which is the Water Supply and Sanitation Technology Platform. With the values of innovation, expertise, professionalism and team spirit at cornerstones of its operation, WssTP strives to:</p> <ul style="list-style-type: none"> <li>- Foster collaborative, innovative and integrated European Research and Technologies Development</li> <li>- Ensure the European Growth and Competitiveness of the Water Sector</li> <li>- Provide Global answers to Global Challenges for the next generations</li> <li>- Address the challenges of an integrated and sustainable management of water resources</li> </ul>
<p>UI</p>	<p>UNPARALLEL is targeting to different exploitation assets:</p> <ul style="list-style-type: none"> <li>- The Long range network infrastructure in Almada which will be deployed during the project</li> <li>- The Tool to model reconfigurable systems, which is being used in PROTEUS to model the Analog Front End.</li> </ul>	<p><b>Meetings with external stakeholders</b></p> <ul style="list-style-type: none"> <li>- UNPARALLEL had several meetings with Almada municipality regarding the Long Range network infrastructure deployment.</li> </ul> <p><b>Synergies Established</b></p> <ul style="list-style-type: none"> <li>- Due to the recurrent meetings with Almada municipality a good synergy has been established</li> </ul> <p><b>Communication with Other Projects</b></p> <ul style="list-style-type: none"> <li>- The device being developed in PROTEUS is being promoted in WAZIUP-H2020 project.</li> </ul>
<p>NiPS</p>	<ul style="list-style-type: none"> <li>- IP foreground and innovative outcome regarding of systems and materials for energy harvesting that</li> </ul>	<p><b>Technical workshops participation</b></p>



	<p>will be exploitable for industrialization and market will be evaluated for patenting and licensing to SME and commercial partners.</p> <ul style="list-style-type: none"> <li>- The Proteus energy harvesting technology (EHT) will be shared within the academic community. In addition, the EHT will be transferred to other research areas, such as: sensing, ICT, biomedical and smart materials.</li> </ul>	<p>The research activity about the PROTEUS energy harvesting system was presented and discussed by NIPS group during meetings and international workshops and conferences:</p> <ul style="list-style-type: none"> <li>- NiPS Summer School 2015</li> <li>- BIT 2nd World Congress on Smart Materials 2016</li> <li>- EMRS – European Material Research Society – 2016 spring conference</li> <li>- ERASMUS+ projects workshops</li> </ul> <p><b>Meetings with external stakeholders</b></p> <ul style="list-style-type: none"> <li>- NiPS met ETO MAGNETIC GmbH, a company of magnetic shape memory alloys, which has shown his interest on supporting the research on novel design of energy harvesting systems suitable for PROTEUS.</li> </ul> <p><b>Synergies Established</b></p> <ul style="list-style-type: none"> <li>- NiPS established collaborations and synergies with the research group of Hydraulic Infrastructure at the Department of Engineering at University of Perugia who will share their laboratory for future testing.</li> </ul> <p><b>Communication with Other Projects</b></p> <p>The research outcome of the PROTEUS project is being exploited by other projects carried on by NIPS:</p> <ul style="list-style-type: none"> <li>- ICT Energy (FP7 EU)</li> <li>- ERASMUS+ (H2020)</li> <li>- Fondazione CRPG 2016</li> </ul>
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## 4 Health and Environmental Risks of using CNT

The previous stage of this study (see Deliverable 2.1 First version of Smart System Co-design) was dedicated to assessing the health and environmental risks of using CNTs in sensors for water monitoring. We stated the following conclusions:

- There are consistent reports on toxicity of CNTs in the literature, with effects ranging from mild to intense. The actual health risk however depends both on the actual morphology and chemistry of the CNTs under study, and on the specificity of the exposure (duration and concentration of particles). It can be drastically reduced by minimizing exposure, and by selecting types of CNTs with low toxicity.
- Exposure scenarios for the workers manipulating the CNTs and or manipulating the final PROTEUS nodes are described. Mitigation and prevention actions were listed. They are easily applicable in research labs.
- Exposure of the general public to CNT via future PROTEUS sensors may occur via release of the CNT from the surface and contamination of the drink water, either directly from drinkwater sensornodes, or indirectly after environmental storage of the CNTs (when the sensors are used in rain of waste water, or during waste storage or recycling of the sensors). Note that the quantity of CNTs involved in PROTEUS process are extremely low compared to most nanomaterial-based products.

Based on this first analysis, we proposed to study further the following aspects:

- Detailed analysis of toxicity (including threshold) for non-functionalized MWNTs with 10nm diameter and less than 1.5 $\mu$ m length.
- Analysis of release mechanisms of CNTs from silica and glass substrates, in order to assess the potential volume of CNTs released in the drinkwater and in the environment from PROTEUS nodes.
- SOTA of current practices in terms of occupational handling of nanomaterials.

These three topics constitute the core of the current report.



## 4.1 Toxicity of Multi-walled carbon nanotubes

The present paragraph aims at identifying the potential health impact of the specific type of CNTs that are being used in PROTEUS, in view of determining if they should be replaced in post-project developments with different types of CNTs. Namely, we are using Nanocyl MWNT, non-functionalized, with diameter 9.5nm, length less than 1.5µm, purified at 95%.

A wide number of recent in-vitro and in-vivo studies as well as various meta-analysis of the scientific literature confirm that CNTs cause both genotoxicity and inflammatory responses,<sup>2,3,4,5,6</sup>. To summarize the most recent of those (typically from 2013), namely refs 7, 10 and 11,

- Small CNTs tend to foster genotoxicity, probably because they have usually a more reactive surface (due to their curvature) and thus react more easily with the physiological medium. Bigger CNTs cause inflammatory responses (akin to the effect of asbestos), because they cannot be easily disposed of by the immune systems (mostly because of size-hindrance to transport in the biological medium and subsequent accumulation). The limit between the two regimes seems to be around 20µm in length and 10 to 20 nm in diameter. The MWNTs we are using thus falls clearly in the small CNT range. Overall, the difference between single- or multi-walled CNTs does not appear critical per se, but mostly important because it leads to CNTs with different geometry and surface reactivity.
- More reactive CNTs surfaces (particularly with surface defects, usually carboxylic functions), or CNT with more reactive functionalizing molecules, cause more genotoxicity and inflammation than less reactive CNTs surfaces, because of oxidative reactions. However, more reactive CNTs have a shorter persistence inside the body than high crystallinity CNTs because they can be more easily processed by the body, a parameter whose impact is unclear in the current CNT toxicity framework. In our case, our MWNTs are definitely crystalline with a significant number of defective carbon rings (relatively sharp G and D peaks under Raman spectroscopy). With respect to the literature, it would suggest an intermediate reactivity but still opening up the possibility of CNT degradation inside the body. It could thus be seen as a favorable case. **To go beyond these simple elements However, detailed results on specifically Nanocyl nanotubes are needed. A few of studies are already available and will be analyzed in the next literature study (M27).** Regarding to the functionalization, literature tends to indicate that each type of functionalizing molecules needs to be studied on a case by case basis for its toxicity. **As our molecules are specifically made for the goal of PROTEUS, there is no**

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<sup>2</sup> Determinants of carbon nanotube toxicity, Sophie Lanone, Pascal Andujar, Ali Kermanizadeh, Jorge Boczkowski, *Advanced Drug Delivery Reviews* 65 (2013) 2063–2069, <http://dx.doi.org/10.1016/j.addr.2013.07.019>

<sup>3</sup> Toxicité et écotoxicité des nanotubes de carbone, Etat de l'art 2011-2012, ANSES.

<sup>4</sup> Chiu-wing Lam, John T. James, Richard McCluskey, Sivaram Arepalli & Robert L. Hunter (2006) A Review of Carbon Nanotube Toxicity and Assessment of Potential Occupational and Environmental Health Risks, *Critical Reviews in Toxicology*, 36:3, 189-217. <http://dx.doi.org/10.1080/10408440600570233>

<sup>5</sup> Genotoxicity and carcinogenicity risk of carbon nanotubes. Shinya Toyokuni. *Advanced Drug Delivery Reviews* 65 (2013) 2098–2110. <http://dx.doi.org/10.1016/j.addr.2013.05.011>

<sup>6</sup> Occupational Exposure to Carbon Nanotubes and Nanofibers, *Current Intelligence Bulletin* 65, April 2013, National Institute for Occupational Safety and Health



**preexisting result on those, but we will look for toxicity results on similar molecules (M36).**

- The presence of metal catalysts mixed in with the CNTs strongly increases toxicity (actually caused by the metal nanoparticles and not the CNTs). It is highly recommended to work with purified batches of CNTs, which is our case (5% remaining impurities as indicated in the datasheet). A further purification step could be considered to remove still more impurities. However, besides reducing the process yield, the purification process is widely known to create defects in the surface of nanotubes, thus possibly increasing surface reactivity (with its associated toxicity impact). A balance between both aspects should be found.
- Despite confusing results, there are strong hints that toxicity depends on dose and duration of exposure. The contradictions in the literature are expected to be caused by agglomeration effects of the CNTs.
- A strong impact of rigidity of CNTs is expected, but no clear result on this is available at the time.
- Work is starting to understand the impact of the “protein corona” forming around CNTs in biological conditions. There are expectations that its feature would be more determining for toxicity than the CNTs considered by themselves.

**As a summary, though the literature does provide an analysis on the impact of morphological and chemical factors in CNT toxicity, the complexity of the topic does not enable to conclude univocally on the actual toxicity of Nanocyt CNTs used in PROTEUS. In short, they are expected to behave more like short CNTs, causing more genotoxicity than inflammation. But only detailed studies on these specific CNTs can provide reliable information. It will be the focus of the next PROTEUS study on the topic.**

Let us remark here that most of the literature focuses on the respiratory route for exposure to CNTs, as it is the most acute societal issue today. However, in PROTEUS, it is only a marginal part of the problem, as CNTs are processed in powder form only very briefly and in small volumes (no production of CNTs is envisioned, and even in the large scale manufacturing phase, the volumes of processed particles should remain fairly low compared to volumes considered in production facilities), and in a containable manner (in glovebox to avoid any air release of CNTs). In PROTEUS, the main issue for occupational exposure as well as secondary exposure for manufacturers or end users is by dermal contacts or ingestion, and not at all by inhalation.

As such, we have looked into the literature on dermal exposure and gastrointestinal and have found it very limited<sup>7</sup> compared to the more general literature on inhalation/lung-related toxicity. **For the second next study (M36), we will look into more details on this literature to determine whether this scarcity of literature is a fact or simply a bias in the search method. If more literature is found, we will report on the toxicity of CNTs via dermal exposure or gastro-intestinal ingestion.**

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<sup>7</sup> Human health effects of residual carbon nanotubes and traditional water treatment chemicals in drinking water. Geoffrey S. Simate, Sunny E. Iyuke, Sehliselo Ndlovu, Mike Heydenrych, Lubinda F. Walubita, Environment International 39 (2012) 38–49



## 4.2 Occupational handling of nanoparticles: best practices

Based on the toxicity results, several countries have emitted recommendations for workplace handling of CNTs. At the time, there is very few legal framework, mostly sets of recommendations. While some of them are specific to CNTs (for instance the NIOHS report<sup>8</sup>, the Australian Information sheet on CNTs handling<sup>9</sup>), others are more generic and address all types of nanoparticles (like the EU<sup>10</sup> or the French INRS institute<sup>11</sup> propose). Recommendations for CNTs are usually more severe than recommendations for generic nanoparticles, though the risk management strategies are consistent from one document to the other.

Due to the general toxicity of CNTs identified in the literature, decision has been taken to minimize exposure by applying a hierarchy of actions. Citing here the NIOSH report from 2013 (on CNT only), a bit more detailed than its counterparts throughout the world, it is recommended to, by order of priority:

- Use available information to continually assess current hazard potential related to CNT and CNF exposures in the workplace
- Identify and characterize processes and job tasks where workers encounter bulk (“free-form”) CNT or CNF and materials that contain CNT/CNF (e.g., composites).
- Substitute, when possible, a nonhazardous or less hazardous material for CNT and CNF. When substitution is not possible, use engineering controls as the primary method for minimizing worker exposure to CNT and CNF.
- Establish criteria and procedures for selecting, installing, and evaluating the performance of engineering controls to ensure proper operating conditions. Make sure workers are trained in how to check and use exposure controls (e.g., exhaust ventilation systems).
- Routinely evaluate airborne exposures to ensure that control measures are working properly and that worker exposures are being maintained below the NIOSH REL of 1 µg/m<sup>3</sup> using NIOSH Method 5040
- Follow exposure and hazard assessment procedures for determining the need for and selection of proper personal protective equipment, such as clothing, gloves, and respirators.
- Educate workers on the sources and job tasks that may expose them to CNT and CNF, and train them about how to use appropriate controls, work practices, and personal protective equipment to minimize exposure.
- Provide facilities for hand washing and encourage workers to make use of these facilities before eating, smoking, or leaving the worksite.

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<sup>8</sup> Occupational Exposure to Carbon Nanotubes and Nanofibers, Current Intelligence Bulletin 65, April 2013, National Institute for Occupational Safety and Health

<sup>9</sup> Safe handling and use of carbon nanotubes in the workplace INFORMATION SHEET, Safe work Australia

<sup>10</sup> Guidance on the protection of the health and safety of workers from the potential risks related to nanomaterials at work, Guidance for employers and health and safety practitioners, EC Employment, Social Affairs & Inclusion, JUNE 2013

<sup>11</sup> Nanomatériaux, Prévention des risques dans les laboratoires, INRS, Janvier 2012, ED 6115



- Provide facilities for showering and changing clothes, with separate facilities for storage of nonwork clothing, to prevent the inadvertent cross-contamination of nonwork areas (including take-home contamination).
- Use light-colored gloves, lab coats, and workbench surfaces to make contamination by dark CNT and CNF easier to see.
- Develop and implement procedures to deal with cleanup of CNT and CNF spills and decontamination of surfaces.
- When respirators are provided for worker protection, the OSHA respiratory protection standard [29 CFR 1910.134] requires that a respiratory protection program be established (...) The voluntary use of respirators are permitted, but must comply with the provisions set forth in CFR 1910.134(c)(2)(i) and CFR 1910.134(c)(2)(ii).
- Information on the potential health risks and recommended risk management practices contained in this CIB should, at a minimum, be used when developing labels and Safety Data Sheets (SDS), as required [<http://www.osha.gov/dsg/hazcom>].

The EU recommendations are very similar (on all nanoparticles), describing a hierarchy of controls:

- Eliminate / Substitute: The risks posed by a MNM can be eliminated by either avoiding its use or replacing it by a less hazardous agent, taking account of its conditions of use. MNMs (or its bulk form) which are classified as carcinogens or mutagens should be considered as a priority for substitution.
- Modify process: Change the process to reduce the extent of concern by, for example:
  - Handling MNMs in liquid media or binding MNMs in solid media
  - Reducing the amount of MNMs handled, on any given occasion or
  - Changing work procedures so as to minimise the exposure
- Isolate or Enclose: All operations which involve the likely release of MNMs into the air are performed in contained installations or in facilities that can be operated remotely from a protected area
- Engineering Control All processes where there is the potential for creating dusts or aerosols of MNMs are carried out in areas with efficient local exhaust ventilation. Wet cutting is recommended for cutting solid articles containing MNMs
- Administrative Control Working procedures are developed for the safe handling of MNMs and job rotation programmes to minimise individual exposure are implemented. Workers potentially exposed to MNMs are consulted and informed about the results of the Risk Assessment and training courses about the control measures implemented are provided. An Emergency Management Plan should be established.
- Personal Protective Equipment (PPE) PPE is a last resort control measure or a supplemental option to help support higher levels of exposure control. PPE may include respiratory protection devices, dermal protection and eye protection.

In the French INRS report (on all nanoparticles), the scheme is as follows:

- Risk identification
- Avoid risks by removing nanomaterials from process
- Evaluate unavoidable health and safety risks depending on process and work modes
- Implement risk prevention and mitigation (including individual and collective protection apparatus)
- Verify efficacy of policies
- Ensure training and information of workers.



A critical parameter in all these recommendations is the Recommended Exposure Limit, namely the concentration in air that a worker can be exposed to over the course of 45 years, 8 hours a weekday, with only minimal lung effects. Presently, for CNTs, the REL is derived from the toxicology literature, based on an estimated retention volume in the lungs of airborne particles (using work on the PM2.5 airborne particles) and on an extrapolation, from the short-term studies on mice, of the maximum total dose acceptable over a lifetime by a human being. So it is clearly an approximate method devised to apply at best the precautionary principle. The NIOSH report provides a detailed description of the method.

Presently, the REL for CNT is  $1\mu\text{m}/\text{m}^3$  in the USA and  $7\mu\text{m}/\text{m}^3$  in France. Based on all the uncertainties, the discrepancy is not surprising at all. These values are actualized every few years to account for new literature findings.

These values for REL raise two secondary problems: **Are such low REL achievable? How to monitor them? We will carry out a more detailed study on this in the next stage of this study.** In a preliminary manner, it is our understanding that concentration of CNTs in large volume production facilities have been measured to be around 30 to  $50\mu\text{g}/\text{m}^3$ , so above the REL, the riskier phase being the phase of powder manipulation<sup>12</sup>. A more extensive literature search is needed to understand if these values are generic or not. In any case, PROTEUS does not concern manufacturing of CNTs, so these results do not apply.

In a more practical manner, we have started work in 2015 and 2016 in our lab to implement best practices in terms of safe handling:

- Improvement of confinement while handling dry CNTs powders, by investing into a glove box system with nanoparticle filtering
- Improvement of confinement while handling CNTs dispersion (liquid phase), by carrying out
  - CNT sonication with a dedicated system, in an enclosed box
  - Enforcing 15 min resting time after CNT centrifugation to ensure re-deposition in the (dedicated) equipment of possible CNT aerosols.
  - CNT printing work in a fumehood
- A large lab meeting to inform all workers of best practices, as well as additional training actions for teams working on CNTs
- Intervention of national (CNRS) experts to evaluate current practices and suggest improvements. Experts found our practices to be at a very good level for French CNRS labs.

**In the future, we will investigate the two following directions:**

- **Carry out a monitoring campaign for air-borne CNTs in the lab**
- **Keep improving compliance with first French, then EU recommendation on nanoparticle handling.**

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<sup>12</sup> Occupational Exposure Assessment in Carbon Nanotube and Nanofiber Primary and Secondary Manufacturers: Mobile Direct-Reading Sampling Matthew M. Dahm, Douglas E. Evans, Mary K. Schubauer-Berigan, M. EILEEN Birch, and James A. Deddens. Ann. Occup. Hyg., Vol. 57, No. 3, pp. 328–344, 2013 doi:10.1093/annhyg/mes079



## 4.3 CNT release mechanisms from surfaces

Another concern in PROTEUS is the life cycle of the CNT-based sensors immersed in water, namely, whether CNTs might be released from the SiO<sub>2</sub> surface at different stage of the life cycle:

- 1) During service time in drink water, CNT could migrate in the water network and be ingested by the end users
- 2) During service time in rain and waste water, CNT could end up in the “general” environment.
- 3) During recycling and waste management operations, depending on their modalities, CNT could be released in air or in water.

**The last topic of release during recycling and waste management of CNT devices is far-reaching and complex, and will be addressed in another phase of this study (M36).**

Here we focus only on the CNT release during service time in water. **The impact of CNTs after release (either regarding to ecotoxicity or toxicity via ingestion) will be addressed in later stage of this study also.**

From the literature<sup>13</sup>, it appears that CNT can be released partially or even fully from flat SiO<sub>2</sub> surfaces in aqueous solutions with salt dispersion. However, the release rate strongly depends on CNT surface chemistry (here, the studies concern oxidized, rather hydrophilic CNTs, contrary to ours, which are pristine and rather hydrophobic) as well as the detailed composition and pH of the solution<sup>14,15</sup>. More generic results on particle adhesion on substrates also suggest that the release rate also depends on the flow rate, on the size of the CNTs and on the deposition and drying process<sup>16</sup>.

As such, it appears difficult to draw an unambiguous conclusion on the release rate of CNTs. **We will proceed to carry out experiments on the topic, in priority in static water (EDI, drink water, sea water, grey water), then in water flows (in Sense-City loop for instance).** The challenge will be to provide an indicator of the quantity of CNTs on the surface before and after exposure to water. An evolution of the resistance of the devices could indicate some change in the CNT layers, but the resistances might be impacted by other factors, such as oxidation of the electrodes. As such we propose instead to extensively use profilometry and imaging techniques, following the protocol described below.

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- Study to be carried out on 3 identical devices on the same chip for comparison.

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<sup>13</sup> Release Kinetics of Multiwalled Carbon Nanotubes Deposited on Silica Surfaces: Quartz Crystal Microbalance with Dissipation (QCMD), Measurements and Modeling, Peng Yi and Kai Loon Chen, [dx.doi.org/10.1021/es405471u](https://doi.org/10.1021/es405471u) | Environ. Sci. Technol. 2014, 48, 4406–4413

<sup>14</sup> Influence of Solution Chemistry on the Release of Multiwalled Carbon Nanotubes from Silica Surfaces, Peng Yi and Kai Loon Chen, [dx.doi.org/10.1021/es403133r](https://doi.org/10.1021/es403133r) | Environ. Sci. Technol. 2013, 47, 12211–12218

<sup>15</sup> Influence of Surface Oxidation on the Aggregation and Deposition Kinetics of Multiwalled Carbon Nanotubes in Monovalent and Divalent Electrolytes, Peng Yi and Kai Loon Chen, [dx.doi.org/10.1021/la104682b](https://doi.org/10.1021/la104682b) | Langmuir 2011, 27, 3588–3599

<sup>16</sup> Hydrodynamic particle removal from surfaces, G.M. Burdick, N.S. Berman, S.P. Beaudoin, Thin Solid Films 488 (2005) 116 – 123



- Pre-exposure characterization:
  - Measurement of resistances
  - Measurement of vertical profiles of the layer along 3 different lines of each devices perpendicular to the electrodes, and along the gap between electrodes (4 vertical profiles per devices). The profile along the gap will be used to derive the thickness of the layer.
  - SEM images at high magnification at 4 different well-identified (with respect to the electrodes sides) spots in each devices, to be analyzed to extract at least surface coverage of CNTs (by grey level thresholding) and if possible number of CNTs by square micrometer (technically very complex in high density CNT deposition)
- Exposure for one month at room temperature (in an air-conditioned lab) to drink water (with no electrical characterization in-between to avoid possible parasitic effect)
- Post exposure characterization
  - Measurement of resistances
  - Measurement of the same 4 vertical profiles as during pre-characterization and comparison by simple averaging and by more complex data statistics.
  - SEM images at same magnification of the same spots, to compare surface coverage and CNT density. If possible, image correlation will be carried out between images before and after.

Depending on the outcome of this experiment, the characterization process might be amended; in addition, it will be decided whether to extend the experiment in time or to duplicate it in other aqueous solutions. **The status of this experiment will be reported on in the next stage of this study.**



## 4.4 Summary and next stage of the study

As a summary, let us emphasize the following conclusions:

- All types of CNTs feature a form of toxicity, whose actual specificity depends on the geometry and chemistry of the CNTs under study. As such, each CNT type should be analyzed on a case by case basis. In PROTEUS, the CNTs used (produced by Nanocyl) are expected to behave rather like short CNTs, causing genotoxicity but few inflammation.
- Accounting for this health risk, recommendations have been proposed throughout the world to avoid worker exposure to CNTs, which are being applied in our lab. Recommended exposition limit in France is  $7\mu\text{g}/\text{m}^3$ .
- Most literature and recommendation focus on inhalation risks and not ingestion risk or risk by dermal contact, because the health impact is expected to be lower. It is a problem for PROTEUS, as it is the two types of exposure that are the most relevant to the project.
- Strong release of CNTs from  $\text{SiO}_2$  surface has been observed in aqueous solution, but it depends very strongly on CNT surface chemistry and the composition of the solution, so it is unclear how this result applied to PROTEUS case. As such, an experimental campaign is described, to be carried out later in the project.

In the next chapter of this study (M27), we will report on:

- SOTA on Nanocyl CNTs toxicity
- Study on potential toxicity of CNTs functionalized by PROTEUS new molecules
- SOTA on air-borne CNT monitoring, and application to actual lab measurements
- Status on improvement of lab practices in terms of CNTs handling.
- Status of the CNT release experiments

In the final chapter of this study (M36), we will report on:

- SOTA on CNTs toxicity via ingestion and dermal contact.
- SOTA on exotoxicity of CNTs
- SOTA on recycling and waste management issues regarding to devices containing CNTs
- Status on improvement of lab practices in terms of CNTs handling.
- CNT Status of the CNT release experiments



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## 5 Conclusion

This deliverable has reported on the major dissemination activities carried out for the PROTEUS project during the first 18 months of the project. Dissemination activities include publications in journals and conferences, demonstrations and presentations at water related events and fora. A significant number of dissemination activities have already been carried out during the first 18 months of the project including publications and presentations in various international forums. An important goal in terms of dissemination for the upcoming months is to keep up awareness of the projects results and to produce high quality scientific results that are published in the most prestigious international conferences and journals. Moreover, exploitation plan and exploitation activities have been addressed in this deliverable. Finally, health and environmental risk arising from the use of CNT have also been presented.