Applying odds of carbon nanotubes-based sensors for environmental monitoring

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Abstract. Simultaneously with the development of a multitude of technological applications in various activity fields of human society undoubtebly having the goal of increasing humanity quality of life, inadvertent spillover effects have especially emerged regarding environmental pollution on different levels. Animated discussions have started first on scientific level with the goal of finding best strategies for solving created complex situation to succeed maintaining good environmental quality. After finally understanding that technological advance could be connected also to undesired effects, carried out debates on a global level brought the concept of "sustainable development" as a potential solution. It was clear pointed out that progress in technical, economic, as well as environmental and social fields must be emphasized by considering several processes in these fields. In this regard technology assessment (TA) can play an important role by emphasising various potential impacts of technological applications especially on environmental field. Registered advance in nanotechnology, first as scientific efforts in physics combined with technological field, has pointed out applying possibilities also in environmental field. Going into details, applying carbon nanotubes (CNTs) for environmental monitoring by approapriate sensors is representing an innovative scientific approach bringing for sure advance in environmental area.

Key Words: carbon nanotubes, environmental monitoring, environmental protection, environmental sensors, holistic approach, nanotechnologies.

Introduction. Future developments on a global level regarding each human activity field should take per se into consideration the concept of sustainable development of humanity (Jischa 2005; Mousa & Tulbure 2020). After long debates on a global level concerning future human development odds, the concept of sustainable development has been finally defined in 1987 in the Brundtland Report (Hauff 1987). The simple the definition of sustainable development has seemed to be, the complex its concrete application has demonstrated to be for real practical situations (Tulbure 2003; Banse 2011). Manifold tryings have been in the meantime registered especially on scientific level in order to develop methodologies for operationalizing sustainable development. Nevertheless, difficulties have been emphasized in simultaneously taking into consideration inter- and multidisciplinary aspects, coming from various fields, as from technological, economic, environmental, as well as socio-political ones (Jischa 2005).

Main aspect related to assuring sustainable development of human society is represented by the fact of carrying out economic activities under consideration of keeping a good environmental quality. The necessity of environmental protection has emerged after remarking numerous effects of developing industrial activities, having as foremost goal raising humanity quality of life, but having as a side effect unthought environmental pollution (Meadows et al 1972; Grunwald 2002). Having finally recognized unwanted impacts of industrial applications on environment, debates have started to find solutions for emerged situation applicable by considering regional aspects (Tulbure 2003).

Noted advance in nanotechnology, because of scientific efforts in physics combined with technological field, has pointed out its multiple applying odds also in environmental field. In this context applying carbon nanotubes, CNTs in the field of environmental protection is representing a new scientific approach, which brings notable advance in environmental area. As it is known, concerning using environmental protection technologies in a first step existing pollution challenges should be assessed (Jischa 2005). In this regard the aim of this contribution is to emphasize applying odds of CNT-based environmental sensors for carrying out a comprehensive environmental monitoring as a first condition for environmental assessment of human economic activities using for this goal different tools of technology assessment.

Material and Method. Future development odds of various technological applications can be emphasized by considering tools of technology assessment (TA), as presented in Figure 1 (Tulbure 2013). For assessing potential impacts of industrial processes on environment, several tools of TA can be applied with good results depending on the task which has to be analysed by considering in the stage of their concrete application CNT based environmental sensors (Mousa et al 2021). Most used tools of TA are the following ones, as to be remarked also in Figure 1 (Tulbure 2013):

- environmental management system;
- life cycle assessment;
- ecoaudit;
- ecobalance.

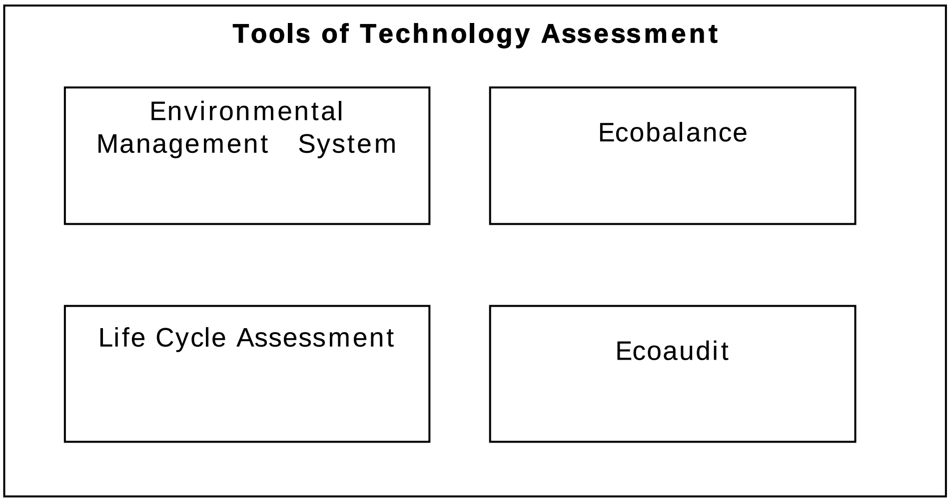

Figure 1. Tools of technology assessment.

One of the most used tools of TA is represented by life cycle assessment (LCA), as being one considering all phases of the general life cycle of different products, to which can be referred by considering applying possibilities of carbon nanotubes (Mousa et al 2021). This approach can be also used for analyzing potential environmental impacts in different life cycles of various products.

Noted tools are actually applied in the context of carrying out different studies related to environmental impact assessment when using various technological applications. Approaching environmental impacts has as a main goal to assure carring out only such activities with minimum environmental impacts (Jischa 2005). Going into details following issues have to be taken into account when applying TA:

- possible results and consequences of a project have to be searched, described, and evaluated; and

- results of analysis have to be delivered to authorities which have to decide next steps basing on existing results.

In order to carry out such an analysis the project which has to be approved must contain information about the project itself, potential environmental impacts, proposed measures to diminish potential negative effects, as well as information about other alternatives (VDI-Richtlinie 3780, 2000). The application field for such studies is especially represented by comprehensive public projects. Main requirements with respect to environmental impact assessment of a project are especially represented by the demand that carried out assessments have to be transparent, and public, as well as applied methods shall be unified, and results have to be comparable.

Life cycle assessment (LCA). The LCA is representing an analysis which registers environmental impacts of a product during its life, "from the cradle to the grave", from the phase of production to consumption and recycling (ISO 14000, 2015). The general life cycle of a product is presented in Figure 2. It is to be remarked that besides production and consumption processes also transport processes are taken into consideration. With “T” are indicated transport processes within the life cycle of a product.

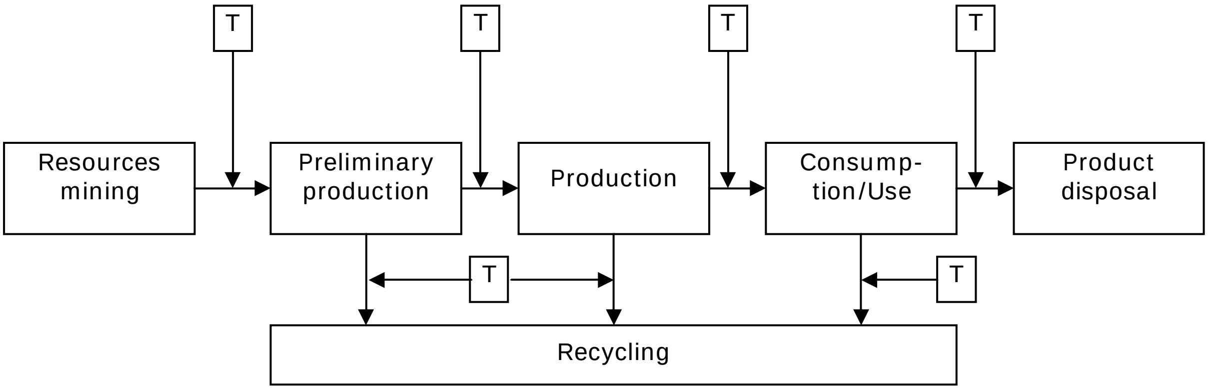

Figure 2. General life-cycle of products.

The LCA is a pretty much applied tool in order to try improving production lines of diverse products, to compare different products and to optimize life cycles of products by considering energetic as well as environmental aspects (Tulbure 2013). The LCA is understood to be a kind of an ecobalance which can be performed depending on the followed goal as a singular study or as a comparative study (Grunwald 2002). In this regard the ecobalance registers material and energetic flows in the fabrication process of products or within a process or a company, even with a region. Such an analysis neads several steps (Tulbure 2003):

a) definition of goal and scope;
b) inventory analysis;
c) impact assessment;
d) interpretation of results.

a) Definition of goal and scope – followed goal shall unambiguously state intended application, reasons for carrying out the study as well as intended audience, this means to whom the results of the study are intended to be communicated. In defining the scope of a LCA following items shall be considered and clearly described: functions of analysed product, functional unit, system boundaries, methodology of impact assessment, data requirements, assumptions, as well as limitations;

b) Inventory analysis - it involves data collection and calculation procedures to quantify relevant inputs and outputs of a product system. These inputs and outputs may include resources usage and emissions into air, water and soil associated with considered system;

c) Impact assessment - it is aimed at assessing significance of potential environmental impacts using results of inventory analysis. The impact assessment may include elements as: assigning of inventory data to impact categories, inventory data modelling within impact categories and possibly aggregating results in specific cases. It is to be mentioned that the methodological and scientific framework for impact assessment is yet not being well developed and clarified. Often in the assessment step aggregated indicators are used in order to allow carrying out transparent assessment processes;

d) Interpretation of results - in this phase findings from inventory analysis and impact assessment are combined together. Interpretations often take the form of conclusions and recommendations to decision-makers, consistent with declared goal of carried out study.

With respect to LCA a difficult step is usually represented by getting on needed data and information about various products and connected production processes. To compare different life cycle stations of a product from the point of view of environmental impacts by considering pollutants emissions a method has been developed at Clausthal University of Technology and applied for different products (Tulbure 2003). The obtained results emphasize that in most of the cases in the usage phase of a product most pollutants are emitted compared to its production phase. At this stage it is to be mentioned that transport processes should be taken into account, anyway, often is not the case because of registered lack of data.

Ecoaudit. The ecoaudit is representing a management tool applied for systematical, documented, periodic, objective assessment of carried out environmental management in a company or institution. The environmental management in a company as stated in the norms DIN-ISO 14000 represents the whole measures directed to organize and perform activities in a company related to environmental protection including specific plants for environmental protection and for environmental monitoring (ISO 14000, 2015). The ecoaudit is representing an instrument which has a function of prevention with respect to environmental protection, as in a company actually the available current situation is emphasized. Results state the degree of respecting legislative measures in the field of environmental protection as well as company goals in this regard. Taking results into account it shall by this improve the environmental protection program of the company (Tulbure 2013). It is to be mentioned that the aim is that companies take voluntarily part in with the conviction of gaining in the end certain economic advantages. It is to be mentioned that databases are very important in this regard, this means collecting, processing, and evaluating data and certain information related to the considered company.

Ecobalance. The ecobalance, so-called environmental performance evaluation represents an instrument for systematically analysis of products, and processes or even of companies, and regions regarding environmental impacts (Grunwald 2002). The ecobalance can be performed as a singular study or as a comparative study. The ecobalance usually registers material and energetic flows in production processes or within a company or a region. An ecobalance is in most of the cases to be carried out in 4 main steps: definition of goal and scope; inventory analysis; impact assessment; and interpretation of results (Tulbure 2013).

Environmental monitoring. As already mentioned, in order to succeed applying most appropriate environmental protection technologies there is a need in a first step to carry out a comprehensive environmental monitoring (Mousa at al 2021). On a global level many attempts have been registered having the goal of developing and establishing flexible adaptive systems for environmental monitoring on different levels. In the corresponding literature it has been much debated the fact that an important part of environmental monitoring systems is represented by environmental indicators, which can be simple or more complex ones, by aggregating various single environmental variables and parameters (Tulbure 1997; Jischa 2005). For developing corresponding environmental indicators depending on established available targets, as well as depending on considered levels, there is a need to consider most relevant environmental variables, for which measurements can also be carried out in certain time intervals (Jischa 2005).

An accomplished model regarding environmental indicators is the pretty well-known *pressure-state-response* model (Eurostat 2001). This mentioned model containes about 60 indicators, some of them for the pressure part, others for the state part, and others for the response part. Actually, environmental indicators are used for emphasizing air, water, or soil pollution, and are currently applied in many countries (Jischa 2005). The interest in indicators describing air, water or soil quality is actually based on the desire to succeed carrying out environmental monitoring, as a first step for assessing environmental quality in various regions (Tulbure 2013). In order to concretely carry out a propor environmental monitoring in a first step a lot of measurements of singular environmental variables and parameters have to be performed by using in this regard proper measuring devices, as emphasized in Figure 3. Such measuring devices in environmental field are mostly based on various environmental sensors, which have the capacity of detecting certain pollutants, for which they have been designed (Tulbure 1997; Jischa 2005). Environmental sensors are represented by linked objects capable of providing various types of information, depending on their types and properties, such as location, position, velocity, and data collected for the certain environmental pollutant element for which they have been designed. Currently several categories of sensors are available, as especially electrochemical sensors, and optical sensors.

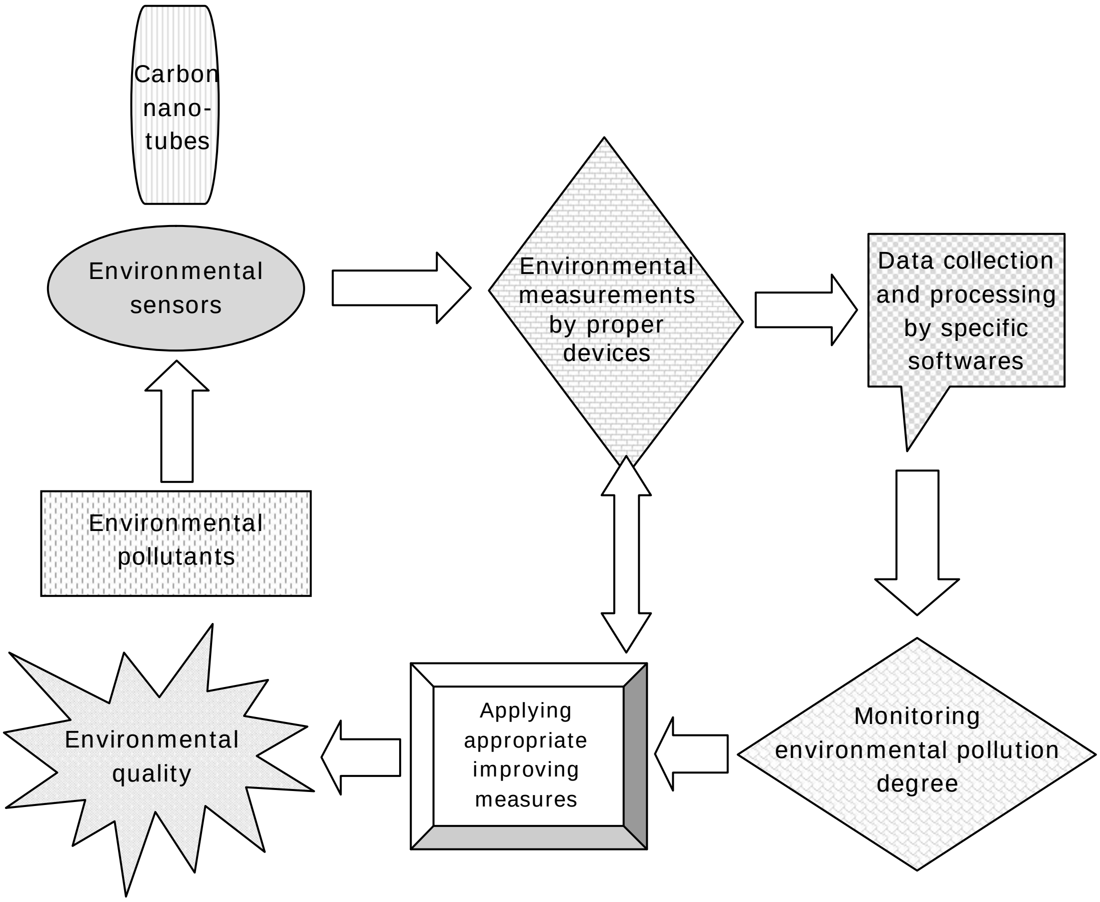

Figure 3. Environmental monitoring by environmental sensors based on carbon nanotubes.

Electrochemical sensors are based on a chemical reaction between gases in the air and the electrode in a liquid inside a sensor. Designing such sensors is a pretty difficult job, as signals from sensors not only depend on the air pollutant of interest, but they can be influenced by several effects, such as other interfering compounds, temperature, humidity, pressure, and signal drift. Nevertheless, at high pollutant concentrations the signal from the pollutant is strong enough, but mixed in the environment the signal is weaker, and interfering effects can influence it pretty strong (Goldoni et al 2003). It follows that the quality of measurements, made by sensors, therefore strongly depends on applied technology as well as on concrete implementation, meaning by this the application, site, conditions, as well as set-up (Mousa et al 2021). This is the reason why often reproducing sensor outputs at different measurement sites made by considered sensors is pretty difficult (Gupta et al 2020). Anyway, due to the influence of meteorological parameters on the sensor signal, correction is in more of the situations not possible. Nevertheless, in certain well-defined cases, measurement uncertainties of these devices may be neglectible compared to made advance by detecting real pollution rates in considered situation (Gupta et al 2020).

Environmental sensors by carbon nanotubes. In the last time, because of rapidly developing industrial activities on a global level, big number of toxic chemicals have been released into the environment in terms of airborne, water, and solid pollutants. Chemical pollutants, such as heavy metals, aromatic compounds, pesticides, and microorganisms can cumulatively cause severe damage to human health as well as to environment. Thus, it is important to have a sensitive, selective, portable, reliable, and rapid response to this issue, and cost-effective sensors for monitoring environmental pollution (Gupta et al 2020; Mousa et al 2021). Carbon nanotubes (CNTs) are representing a multiphase solid material in which at least one phase has one, two, or three dimensions less than 100 nm (Goldoni et al 2003). CNTs with exceptional electrochemical properties, high surface area, high adsorption, and effective catalytic activities are mostly used for fabricating CNT-based sensors for environmental applications (Li et al 2011; Llobet 2013). Mentioned properties that CNTs possess offer several odds for fabricating portable nano-sensors for successful detection of environmental pollutants present in the air, and water (Mousa 2018). At this point it is to be mentioned that sensors based on pristine CNTs have certain limitations, such as sometimes low sensitivity to analytes for which they have low adsorption energy or low affinity, lack of selectivity, or irreversibility or long recovery time (Rigoni et al 2013). To overcome these limitations, several research groups are currently working on the rational functionalization of CNTs with different methods (covalent and non-covalent) and with different nanocomposite materials (polymers, metals oxide, and catalytic metal nanoparticles) to alter their chemical nature and enhance their sensing performance (sensibility, selectivity, and response time). During the past two decades, considerable progress has been made on CNT-based sensors for environmental monitoring, owing to the development of nanotechnology. CNTs-based sensors can be successfully applied for environmentally relevant analytes in both gas and liquid media (Goldoni et al 2003).

Results and Discussion. CNTs are innovative nanostructured materials for new sensing technologies for low-cost air and water quality control and environmental monitoring. This is of strategic importance for green economy and sustainable development of human society, including protection of public health. Here, a survey of CNT-based sensors for air and water applications used in environmental monitoring is shortly discussed, being a main part when assessing potential environmental impacts of applied technologies.

Air quality control. In the past, noticeable scientific efforts have been made to develop functional materials and devices for air quality control (AQC), mainly cost-effective nanosensors (Wang & Yeow 2009). The current research for materials tackles both the specifications of continuous and ubiquitous environmental monitoring and the industrial demands for device integration. Moreover, strong inputs are pushing the development of new low-dimensional nanostructured materials for gas detection of ppb level at low power consumption and low cost. Carbon nanomaterials such as nanotubes, fiber and graphene are rapidly developing and offer the potential for increased surface area and composition control to address gas detection as low as ppb level in real-world applications by considering the interfering effects of temperature and humidity (Hu & Hu 2009). As a rolled-up layer of graphene, the hollow structure and high surface area to volume ratio enable carbon nanomaterials to be good absorber of gas molecules through both physical and chemical absorption. Theoretical studies and simulations of gas molecules absorption on carbon nanomaterials have been successfully reviewed (Bajic et al 1989; Mousa 1994, 1998). It is believed that the change in electrical property of carbon nanomaterials under gas exposure is first related to the electron transfer among gas molecules and carbon nanomaterials. Second, it is related to the carrier lifetime. Penza et al (2008) demonstrated a gas chemisensor fabricated directly onto alumina substrates using plasma-enhanced chemical vapour deposition (PECVD)-grown multiwalled CNTs (MWCNTs) for gas detection of air pollutants, at a working temperature of 200^oC. Functionalisations of

MWCNTs tangled bundle films with nominally 5-nm-thick Pt and Pd nanoclusters, prepared by sputtering, provided higher sensitivity for significantly enhanced gas detection of NO_2, H_2S, NH_3 and CO up to a low limit of sub-ppm level. The electrical resistance at room temperature of unmodified and Pt- and Pd-functionalised MWCNTs was measured as 32.5, 14.1 and 13.4 kΩ, respectively. Thus, the metal modification of CNTs was found to decrease electrical resistance of CNT-networked films. The measured electrical conductance of the metal functionalised MWCNTs upon gas exposure was modulated by charge transfer with p-type semiconducting characteristics. Pt and Pd-cluster-functionalised MWCNT sensors exhibited better performance compared to unmodified MWCNTs, making them promising candidates for air pollutants monitoring. The calculated limit of detection of the most performing Pt-modified CNT sensor was as low as 3 ppb NO_2, 4 ppb H_2S, 200 ppb NH_3 and 4 ppm CO, working at 200^{o}C. Also, a voltage output of sensor signal shows the detection at sub-ppm level ranging from 100 to 600 ppb NO_2 in the 30-min steppulse format using the three CNT sensors, at the operating temperature of 200^{o}C. Both metal-functionalized CNT sensors are more sensitive than unmodified CNT sensor due to the catalytic effect of the spillover with highest sensitivity for Pt-modified CNT sensor able to detect 100 ppb NO_2. This limit represents the actual NO_2 attention level for environmental monitoring in some national regulations. The resistance change induced by 100 ppb NO_2 in the Pt-modified CNT sensor is extremely high as 5.1 kΩ and is higher than that of about 4 Ω reported in the literature for unmodified CNT-based NO_2 gas sensors (Valentini et al 2003). These results able the use of metal-functionalised CNT gas sensors for environmental air monitoring applications. Additionally, Penza et al (2010) demonstrated vertically aligned MWCNT layers Radio frequency (RF)-PECVD synthesised on Fe-coated alumina substrates. A miniaturised CNT-based gas sensor array was developed for monitoring landfill gas (LFG) at a temperature of 150^{o}C. The sensor array was composed of four sensing elements with unmodified CNT and CNT loaded with 5-nm nominally thick sputtered clusters of Pt, Ru and Ag. Chemical analysis of the multicomponent gas mixtures constituted of CO_2, CH_4, H_2, NH_3, CO and NO_2 was performed by array sensor responses and pattern recognition based on principal component analysis (PCA). PCA results demonstrated that the metaldecorated and vertically aligned CNT sensor array can discriminate the NO_2 presence in the multicomponent mixture LFG. The size of metal clusters decorating CNT top surface varied in the range of 5-50 nm. Functional characterisation based on electrical charge transfer sensing mechanisms in the metal-modified CNT chemiresistor array demonstrated high sensitivity providing minimal sub-ppmlevel detection down up to 100 ppb NO_2, at 150^{o}C. Cantalini et al (2003a) demonstrated that PECVD-grown CNT sensors were fabricated onto Si_3N_4/Si substrates equipped with Pt electrodes for gas-sensing applications. The CNT diameter ranged 30-40 nm and length 100-200 nm. CNTs showed cross-sensitivities towards NH_3, H_2O and C_2H_5OH and exhibited high sensitivity towards NO_2 gas in the gas concentrations range of 10-100 ppb. The highest NO_2 gas sensitivity was measured at 165^{o}C working temperature. No response was found towards CO and CH_4 gases in the operating temperature range of 25-250^{o}C. Additionally, NO_2 gas sensitivity resulted to be improved by annealing the as-grown films at temperatures higher than 330^{o}C. This was attributed to structural changes induced by temperature exceeding 250^{o}C providing a support for a phase transition causing an electrical change from a metallic to a semiconducting response of the CNT film. Kuzmych et al (2007) demonstrated chemically functionalised CNT-field-effect transistors (FET) for nitric oxide (NO) gas detection. The NO gas to be detected was passed through acidic gas scrubbing, oxidation, and conductivity measurements by CNT-FET. Gas mixtures containing NO were passed through an Ascarite scrubber and then an oxidising material (CrO_3) which converted NO to NO_2. This gas was delivered to the CNT-FET modified with polyethyleneimine (PEI) polymer. Interaction of NO gas with PEI-modified CNT-FET resulted in a conductivity change depending proportionally on NO gas concentration. A wide range of NO gas concentrations were measured from 2 ppb to 5 ppm. A detection limit was measured as 5 ppb NO at a relative humidity of 30%. Cross-sensitivity to CO_2 and O_2 was measured as well, modelling human breath conditions. Compared to chemiluminescence methods for monitoring NO, this sensor offers the advantages of low cost, compact size, and simplicity for selfdiagnostics and home care (Cantalini et al 2003a, b).

Water quality control. Nanomaterials for environmental protection are emerging for new practical applications. In particular, carbon nanomaterials are good candidates for water quality control and liquid applications. CNTs have some important advantages for fabricating carbon material-based electrochemical sensors including good electrical conductivity, biocompatibility, large specific area, well-defined nanotube structure, modifiable surface, functional groups on the surface with pretreatment and capability of being solubilised or dispersed in solutions. Based on these unique properties, a large variety of research works, and reviews have been dealt with CNT-based electrochemical sensors. An extensive review on electrochemical CNT-based sensors for applications has been proposed by Hu & Hu (2009). Here, pristine and modified CNTs integrated for water and liquid applications are shortly reviewed. Barisci et al (2000a, b, c) demonstrated the possibility to use CNTs to modify electrodes in electrochemical sensors immersed in aqueous and nonaqueous solutions. In particular, an electrochemical quartz crystal microbalance (QCM) was modified with singlewalled CNTs (SWCNTs) for studying the voltammetric properties of various electrolytic solutions showing that the QCM electrode mass increased with increasing negative potentials and was associated with double-layer charging in the CNT electrode. Liu et al (2002) demonstrated the usage of carbon nanotube powder in electrochemical microelectrodes for nitrite detection in acidic solutions with a low detection limit as 10^{-7} M (where M is Molarity (parts per million)) at signal-to-noise ratio of 4. Valentini et al (2007) demonstrated functionalised SWCNTs modified microsensors for the selective response of epinephrine in the presence of ascorbic acid. Modified stainless steel microelectrodes (microwire diameter, 300 mm) were assembled using functionalised SWCNTs deposited by the electrophoretical deposition process (EPD) method. The functionalised SWCNTs, which covered the microelectrode surfaces, showed an improved sensitivity and selectivity towards the electrochemical detection of epinephrine. These chemical sensors hampered the voltammetric responses of ascorbic acid (AA) and uric acid (UA), while the electrochemical oxidation of epinephrine was significantly enhanced. Using the differential pulse voltammetry technique, epinephrine showed a very well-resolved peak centred around 240 mV, while 1 mM of AA (present in the same solution) was not detected. This optimisation resulted in microsensors with a good linear range (2-100 μM) epinephrine, good sensitivity (28.1 A M^{-1} cm^{-2}) and interelectrode reproducibility (RSD% = 7.0, n = 6), detection of limit (LOD = 3σ) of 2 μM, response time of 6 s, significant operational stability (13 h in continuous working conditions) and long-term stability (1 month). Consales et al (2007) developed an optical fibre coated by SWCNT-Langmuir-Blodgett (LB) technique at the distal end for detection of toluene (20-80 ppm) in water environment. The good stability, high sensitivity (1.2 x 10^{-4} ppm), detection limit as low as 5 ppm and good dynamics in the response and recovery (few minutes) confirmed the potentiality of this optical device for detecting chemicals in aqueous solutions.

Conclusions. The multitude of human activities has had as a result beside direct desired impacts on humanity quality of life also undesired effects on ecosystems around the world. Environmental pollution represents a side result of developing and applying lot of technological applications. From made presentation became obvious that for assessing industry environmental impacts several tools of TA can be used by considering CNT based environmental sensors in the step of monitoring. Toxic chemicals can be found in the most pristine rainforests, oceans, and in the blood of animals. To succeed in protecting the environment, early pollutants monitoring is important. With the development of nanotechnology, carbon nanotubes offer efficient possibilities for applications in the field of environmental monitoring. Most of the traditional analytical methods, including Ultraviolet (UV) Vis spectroscopy, LC (Liquid Chromatography), GC (Gas Chromatography), ICP-AES (Inductively Coupled Plasma Atomic Emission Spectroscopy), F-AAS (Flame-Atomic Absorption Spectroscopy), and XRF (X-ray Fluorescence Spectroscopy have disadvantages such as high cost, and requiring lot of time, off-site determination, and technical training. Taking these facts into consideration it follows that CNT-based sensors are of great interest due to their small size, simplicity, low cost, on-site determination, and fast response time. The modularity by which CNTs can be functionalized is allowing rapid development of sensor methods. It is to be expected by this that commercial CNTs–based environmental sensors will start to be successfully used in various environmental monitoring systems to emphasize pollution problems and to support by this designing environmental protection technologies. Finally, the future of CNT-based sensors looks very bright in combination to the various transducers used, but continued progress needs to overcome the current challenges, move towards the development of response mechanisms, and further improve performance. CNTs have large surface-to-volume ratios and unique electrical properties. To increase the response of CNTs in detection, researchers continually develop methods for functionalising the CNT surface with conducting polymers and nanoparticles, the optimisation of numerical methods of signal processing to analyse sensor output for pattern recognition applications and the development of suitable manufacturing techniques enabling mass production of CNT-based sensors. Furthermore, the current study of the CNT-based sensors should move from the fundamental sensing performance in laboratories towards real-world applications in actual working scenario. To be more specific, in this contribution has been demonstrated that CNT-based sensors have lot of potential to be applied in the field of environmental monitoring, especially air and water quality control, currently emerging as an innovative approach in environmental field bringing for sure in the future scientific advance.

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