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Micro-Analyzer with Optical Detection and Wireless Communications

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***Abstract:-* There is a need to monitor chemical variables online in the environment and in industry. Novel on- line strategies are needed to optimize the measurement and control of these variables. This calls for the development of instrumentation systems capable of in situ measurements. This paper describes the development of a chemical micro-analyzer with an optical detection. The structure was manufactured using low temperature co-fired ceramics (LTCC) technology. The optical detection and the flow systems were integrated modularly to micro-analyzer and controlled by wireless communications. The contin- uous flow system was implemented with miniaturized actuators to provide autonomy and portability. The response of the optical detection was evaluated using a dilutions system by multi-commutation. The result was validated with a commercial instrument. The development described and proved in this paper would open up opportunities to new applications with wireless chemical micro-systems networks, a field with great potential.**

Keywords: Micro-analyzer LTCC,Optical detection,Miniaturized flow system, Wireless communication

1. INTRODUCTION

Environmental problems as a result of human activities like pollution of air, soil, surface water and groundwater have spurred the development of systems for the monitoring, and control of environmental parameters. These systems are also useful for risk assessment and for the evaluation of remediation measures.

Additionally, online systems have proved to be useful monitoring and controlling chemical variables in industry. Nowadays this monitoring is achieved frequently off-line. A sample is taken, transported to a central laboratory to be stored, processed, analyzed until a report is issued and a control decision is made. This takes a lot of time so, when the control action is taken, the control action lags behind the variable of interest. This drives the development of in situ instrumentation systems, with high selectivity and sensitivity, preferably autonomous, capable of being applied to more than one type of sample that allows for simple modification and adaptation of the analytical method enabling on-line measurements in real time.

Since the 20th century, analytical systems have reduced steadily the size of the sample [1]. The overall size of the system has been reduced as well thus arriving to the concept of micro-total analysis system, μTAS [2]. Nowadays, the automation of these systems includes the micro-fluidic optimization and a decrease of operational costs as reagent use is reduced [3]. Micro-systems integrate one or more analytical steps in a small scale including: sampling, sample transport, separation, reaction, measurement, transduction, signal acquisition and processing [4]. Moreover, flow injection systems generally use large actuators like peristaltic pumps and injection valves, resulting in large systems that usually need to be housed in special facilities limiting portability. Several alternatives have been reported to optimize fluid management [5–7]. A common approach to micro-systems manufacturing involves silicon, glass or polymers requiring costly facilities and procedures. LTCC manufacturing, also known as green tape is advantageous to silicon, glass and polymer fabrication in several ways [8]. LTCC (low temperature co-fired ceramics) fabrication is an economical, simple and versatile manufacturing technique. It can produce tridimensional structures for fluid management and it can also be a substrate for electronic printed circuits [9,10]. This material and its associated fabrication techniques are used increasingly in the fields of environmental and biomedical monitoring [11–14] and also in the development of several types of sensors and actuators [15]. A popular automated analytical method is flow injection analysis, or FIA, as it can produce measurements in real time [16]. To achieve miniaturization, it is necessary to evaluate the operational characteristics of selected detection technique, including: sensitivity, small sample volumes (pL, nL, μL) at low concentrations [17], reliability, short response time, low power and autonomy. Optical methods have a great acceptance in qualitative and quantitative determinations. A detection system based on a light emitting diode (LED) and a photodetector offers versatility, reliability, accuracy, small size, low cost and low power consumption that can be used in spectrophotometric methods [18]. Robust analyzers have been developed by integrating other devices

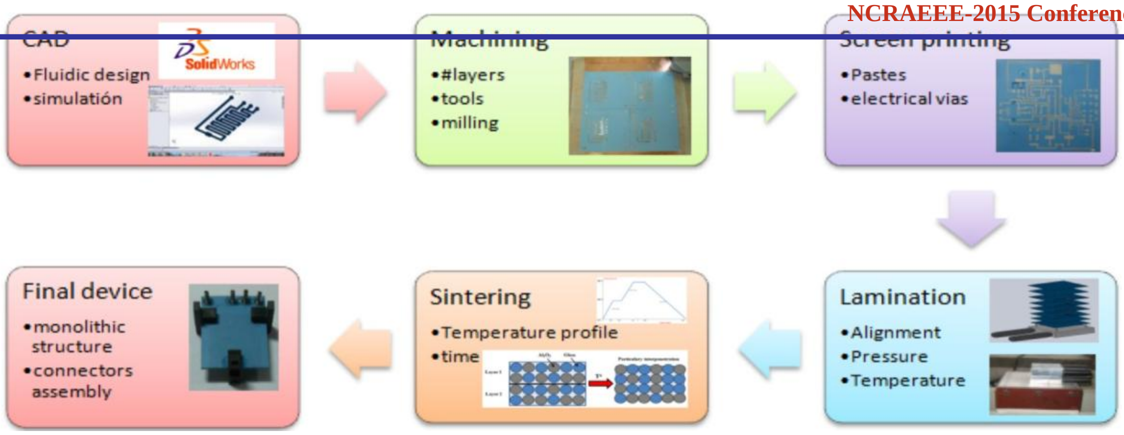

Fig. 1. LTCC manufacturing process.

such as optical fibers [19,20], photomultiplier tubes [21], phototransistors [22], photodiodes [23–25] and reversed biased LEDs [19,26–28].

The need for chemical information in the monitoring and control of chemical processes require real time and on-line response. With the development of wireless communications it is possible to measure in remote locations, far removed from a central laboratory. The integration of wireless communication with analytical instruments it is possible to produce devices for in situ measurements [29]. The development of smart sensors called "motes" has been reported recently. Motes are autonomous nodes with capability of micro-process, signal acquisition, data transmission, battery power management and the communication with others motes [30,31]. A system formed by a network of connected motes linked to a remote server by a gateway is a kind of wireless sensor network (WSN). In the present work, a micro-analyzer fabricated on LTCC (low temperature co-fired ceramics) technology featuring optical detection and integrating Bluetooth communication capabilities [32] are described. The transmitted data are acquired, processed, stored and displayed with a virtual instrument. The continuous flow system has been built with miniaturized actuators and multicommutation techniques. The flexibility provided by the electronic design allows the micro-analyzer to be operated at different wavelengths by changing the LED. Also, the sensitivity can be adjusted through the modification of the sampling and commutation times using the virtual instrument. This permits the application of the micro-analyzer to other analytes. The performance of the micro-analyzer was evaluated by determining concentration of $KMnO_4$. The configuration and control were made by wireless communications.

2. Experimental

2.1. Materials and equipment

The micro-analyzer was manufactured using LTCC technology featuring DuPont 951PX green tapes (DuPont Microcircuit Materials, NC, USA). The in/out fluid connectors on the LTCC structure were made of titanium (MAFSA, S.A de C.V., Torreon, Mexico) and glued using epoxy. The measurement cell was integrated inside the micro-fluidic structure and it was covered with a 0.1 mm thick borosilicate glass that allows light beam to pass through the cell. The borosilicate glass allows 90% transmittance whenever working at wavelength between 400 nm and 2 μm [7], and it will not affect the absorbance measurements.

For the continuous flow system piezoelectric micro-pumps (Takasago Electric, Inc., Nagoya, Japan) were used, dimensions 25 mm × 25 mm × 4.8 mm. All external connecting tubes were polypropylene with a 1.5 mm internal diameter. The solutions were injected by multi-commutation [33–35] using two-way solenoid micro-valves (161T010, NResearch, NJ, USA).

The detection system was implemented using an ultrabright LED (Kingbright Co., LA, USA) with a wavelength $\lambda = 525$ nm. The detector was a TAOS TSL257-LF, light to voltage converter (TAOS Inc., Plano, USA) with an integrated transimpedance amplifier.

The control electronic circuit was based on the micro-controller PIC18F4550 (Microchip Technology Inc., Chandler, USA). Surface-mount-device (SMD) electronic components were used to reduce the dimensions of the board. To enable the portability of the system, it was powered with a lithium-ion rechargeable battery VSO-F550H (Amstron PS, Valencia, USA) of 7.2 V–2.2 A/h. Wireless communications between the device and control electronics was achieved using Bluetooth modules LM-058 (LM Technologies, London, UK).

The micro-analyzer was evaluated using dilutions of $KMnO_4$ (Sigma–Aldrich Co., St. Louis, USA) and bi-distilled water. A $0.05753\ g\ L^{-1}$ of $KMnO_4$ was prepared as stock solution.

2.2. Manufacturing of the micro-fluidic structure

The design of a fluidic structure in LTTC follows a layered approach. Every single layer is designed and then machined individually using a CNC mill (LPKF Laser & Electronic, Garbsen Germany). When all layers are stacked and aligned, the desired structure is achieved. The stacked layers are laminated using an automated hydraulic press (MAFSA, Torreon, Mex.) with hot plates (100 °C) at 750 psi prior to sintering. The laminated LTCC is sintered in a programmable furnace (Cole Palmer, Vernon Hills, USA). The general fabrication process for LTCC devices has been described in detail elsewhere [8,14,36]. A simplified diagram of the process is shown in Fig. 1.

A micro-analyzer may integrate micro-fluidic, control and data acquisition electronics or it may follow a modular approach to facilitate maintenance. The micro-fluidic structure is a passive laminar micro-mixer [37] with an integrated detection cell, composed of eight layers: the two top layers where four-liquid inlet/outlet ports are located, four layers to define the geometry of the channels and two bottom layers. Each layer is 0.254 mm (±13 μm). After sintering, the channel dimensions were 0.873 mm (±0.02 mm) wide and 0.85 mm (±0.013 mm) depth. The dimensions of the detection cell were 3.5 mm × 1.7 mm × 1.72 mm for a 10.5 μl to total volume. The micro-analyzer had a total area of 45 mm × 30 mm and is shown in Fig. 2.

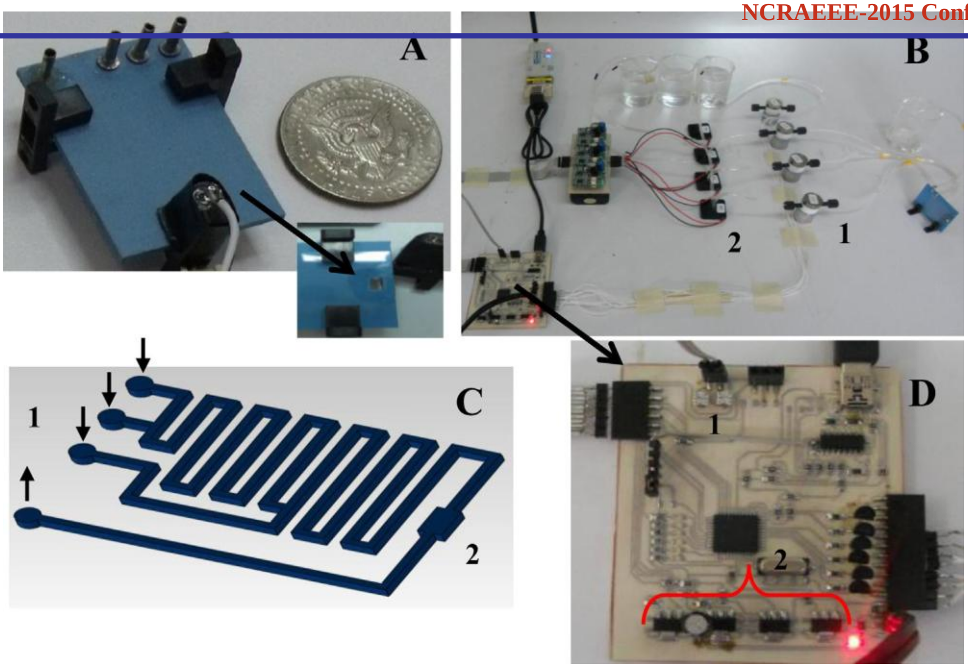

Fig. 2. Micro-analyzer with optical detection and wireless communications. (A) Micro-analyzer (45 mm × 30 mm). (B) Miniaturized flow management; 1: Micro-valves; 2: micro-pumps. (C) Internal geometry; 1: inlet/outlet; 2: flow and detection cell (3.5 mm × 1.7 mm × 1.72 mm). (D) Electronic control and wireless modules; 1: intensity control; 2: regulators.

2.3. Continuous flow system

Autonomous and portable analytical systems need to realize measurements in real time at the location where the analyte is. The flow system was designed with micro-actuators to improve mixing conditions and minimize reagent use and waste generation. This implementation achieves a smaller, lighter and less power demanding system. Two piezoelectric micro-pumps were configured at 5 Hz of frequency (from 0 to 60 Hz) in order to provide a flow rate of 1.5 mL min^{-1} at discharge pressure 5.6 kPa. The micro-pumps suck and discharge at the reverse timing. Two micro-valves were configured at 200 ms multi-commutation speed. The injections parameters were set and controlled by a virtual instrument program. In the present work micro-actuators replace bulky pumps and injection valves normally used in flow injection analysis (FIA). This brings our developed system closer to the autonomy and portability needed for in situ analysis.

2.4. Implementation of the electronic control

Control data acquisition and signal processing electronics were based on a PIC micro-controller. The system presented here also includes wireless communications between a personal computer (PC) and the micro-controller. The wireless modules use a serial communication and it is converting to Bluetooth for transmitting signals. The serial micro-controller port was used to connect one module and the other module was connected using the PC USB port. Voltage supplies were 5 V for the micro-actuators and 3.3 V for the micro-controller. A start button and a reset button were included in the circuit. The start button runs an activation program prior to normal functioning. The reset button can be pressed at any time to terminate the program and to start over. In each of the inputs of the micro-pumps there were drive circuits that translate the frequency provided by the micro-controller to a signal that drives the micro-pumps to the desired flow rate. The micro-controller also controls the optical detection system including the acquisition of the detector response. The program for the micro-controller was developed in PIC-CCS. The micro-controller generates the control frequencies to drive the piezoelectric micro-pumps and sets the times for the opening and closing of the micro-valves in multi-commutation.

2.5. Optical detection system

In spectrophotometry, the radiation emitted or absorbed by a sample occurs in a very short time. The radiation source is a continuous beam of light with the relevant wavelength. Any fluctuations in the beam intensity can affect measurements. Fluctuations in light intensity in an optical detection system such as the one used in the present work may be due to voltage supply variations. The power supply used in the present work is a high-capacity (2200 mA h) lithium-ion battery followed by a voltage regulator. This ensures a steady voltage that will produce a steady light intensity. In addition, after each measurement, the detector was calibrated using bi-distilled water as reference. As a radiation source an ultrabright LED with a h = 525 nm, a diameter of 5 mm with an illumination angle of 20° and transparent plastic bodies was employed. The power emitted as light is directly proportional to the current through the LED. The LED emission power is 25 mW and the current required to develop its light intensity is 20 mA. The maximum supply voltage is 5 V, so the resulting limiting resistance is ≥32 K. A variable resistor was placed to control the intensity of radiation emitted by LED. The photodetector TAOSL-257-LF is a light-to-voltage converter. Its output voltage is directly proportional to light intensity (irradiance) on the photodiode. The active area has a round shape with a diameter of 0.75 mm and is located at the center of the lens [38]. When light strikes the photodiode, this current is converted to voltage by a transimpedance amplifier integrated in the detector. With this configuration the intensity of incident light is proportional to the output voltage of the detector. The detector and the LED were fixed in a coupler with two cavities, one where the LED was fixed and the other for the photodiode. The distance between these components was 5 mm. This module was placed externally and perpendicularly to the micro-analyzer cell to achieve maximum light measurement. Using this setup the components may

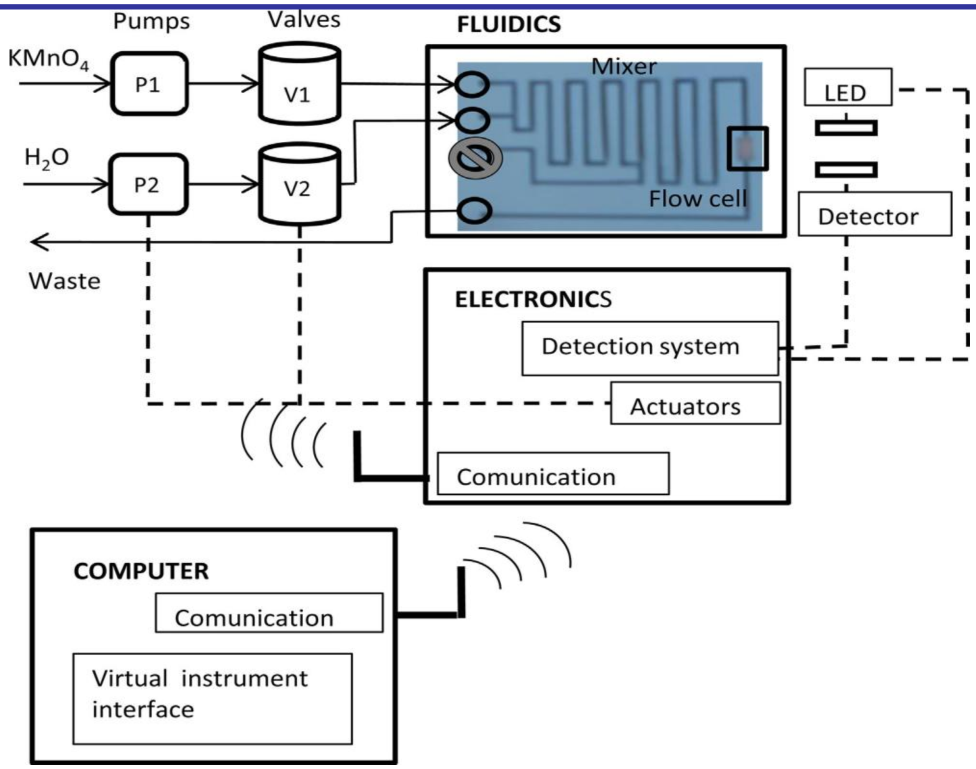

Fig. 3. Block diagram of experimental set-up.

be replaced if necessary, without altering the electronic assembly.

2.6. Procedure

The micro-analyzer was covered with a black box to isolate the detection system from ambient light avoiding interference from spurious signals. The micro-analyzer was configured from a virtual instrument running in the PC. The process begins by activating the wireless communication system to transmit and receive signals via Bluetooth. The communication port was selected on virtual instrument. Once communication is established micro-actuators are activated first and then the detection system. Hydrodynamic variables such as flow rate and injection volume of the solutions are adjusted and controlled through a virtual instrument interface by wireless. The dilutions are generated by multi-commutation and transported to the detection cell where the analysis is done obtaining an analytical signal that corresponds to the light transmitted and thus to the concentration of the solution. This analytical signal is acquired by an A/D converter in the micro-controller from the sensor and transmitted to the PC via Bluetooth. The virtual instrumentation program displays the results by a time/voltage graph and stored them in a text file and then processed. The absorbance value was calculated by the Eq. (1) [39]:

$$A = \log \frac{S_a - S_d}{S_r - S_d} \qquad (1)$$

where S_a is the analytical signal, S_r is the reference signal measuring bi-distilled water and S_d is the dark signal (signal intensity obtained with the LED turned off).

The micro-analyzer was evaluated using an experimental set-up shown as a block diagram in Fig. 3.

The experiment was made to determine the absorbance of the dilutions obtained by the micro-analyzer. Solutions at different concentrations were prepared automatically in micro-analyzer from the stock solution of $KMnO_4$. Measurements were made using a Perkin Elmer Junior III Spectrophotometer 6/8, (Perkin Elmer, Waltham, USA) for comparison purposes.

3. Results and discussion

3.1. Micro-analyzer evaluation

In the development of miniaturized analytical autonomous systems using flow injection methods, the mixing of solutions may entail the occurrence of a chemical reaction [40,41]. In this work multi-commutation was configured by wireless to prepare dilutions from a stock solution and no reaction took place. The solenoid micro-valves switching between potassium permanganate (valve V1) and bi-distilled water (valve V2). Once the dilutions process was validated in the micro-analyzer, the optical system was tested. The intensity of radiation emitted by LED was 4.24 V. It was measured by the photometer TAOSL-257-LF during the course of 5 days. A RSD = 0.002% (n = 20; 98% confidence) was obtained. The relationship between output voltage and output noise voltage for the voltage regulator used is 10 $\mu V V^{-1}$ or 10 ppm [42].

The $KMnO_4$ solution has a maximum absorption wavelength of 530 nm, very close to that emitted by the LED (525 nm) that was used. For this evaluation, flow rate for the stock solution and bi-distilled water was set at 1.5 mL min^{-1}. Sampling time was set at 8 s to develop a volume of 200 μL according to a flow rate. To produce the in-line dilutions automatically, the commutation time was configured between V1 and V2 in a ratio proportional to the dilution required during the sampling time (8 s). The dilutions were made in

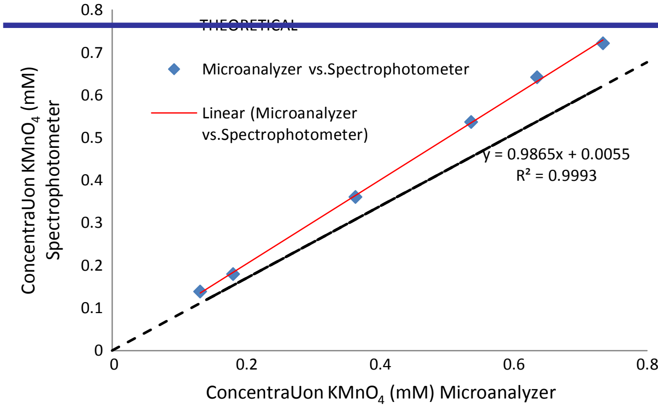

Fig. 4. Comparative response between micro-analyzer and spectrophotometer at h = 525 nm.

a range from 0.238 to 19.6 mg mL^{-1} of $KMnO_4$. After each measurement, the detector was calibrated using bi-distilled water as blank or reference. A linear response was observed. The micro-analyzer equation that describes the linear relationship between concentration ($KMnO_4$) and absorbance (A) is $A = 0.033[KMnO_4] + 0.0211$; $r^2 = 0.9967$. For a concentration of 0.734 mM, the RSD obtained was 1.2% (n = 4; 95% confidence). The detection limit was estimated as 0.139 mM (RSD (%) = 2.16, (n = 4; 95% confidence)). The result was compared with measurement made with a spectrophotometer at wavelength 525 nm.

The absorbance measured by the micro-analyzer was contrasted against measurements made using a Perkin Elmer Junior III Spectrophotometer 6/8, (Perkin Elmer, Waltham, USA). When both results are plotted against each other, the resulting line has a slope close to unity (0.9993) showing that both methods are equivalent. This is shown in Fig. 4.

According to the result described above, the micro-analyzer in this work measured $KMnO_4$ concentrations very close to the measurement of the spectrophotometer.

The LTCC micro-analyzer using wireless communications for control and measurements produced results that are equivalent to those produced by a spectrophotometer. The implementation of micro-analyzer is portable and may be used for in situ measurements.

3.2. Electrical and reagent consumption evaluation

The flow system was designed with miniaturized actuators to improve mixing conditions and to minimize reagent consumption as well as waste generation. The automation flow system reduced the reagent consumption, sample consumption and waste generation 25-fold compared with the batch procedure using a spectrophotometer with a 5 mL cuvette. This is an environmentally friendly alternative and could be considered as a step toward green chemistry.

The power consumption has been 120 mA in standby system with micro-pumps, electronic control, wireless communication modules and optical system. Each micro-pump uses 18 mW, its driver circuit uses 240 mW while driving two micro-pumps, for micro-valves the power required is 1 W. In this application it was necessary to operate two micro-pumps and the switching of two micro-valves, with a total of 1.2 W. The power consumption with the operating system was 1.8 W. The dilution procedure was accomplished in 8 s. Therefore, the battery-operated micro-analyzer can achieve 2640 sampling cycles. With a sampling rate of 2 measurements per hour the autonomous flow system of micro-analyzer can work 55 days. This result suggests the potential of micro-analyzer as a portable system.

4. Conclusions

A novel micro-analyzer using optical sensing and wireless transmission was developed using low temperature co-fired ceramics (LTCC). The resulting system is a step toward the implementation of autonomous analytical systems for in situ determination of environmental variables and for the control of industrial processes. This micro-analyzer was used to measure $KMnO_4$ using a wavelength of 525 nm as an application example.

The main feature of the developed micro-analyzer is the integration of a miniaturized flow system, including micro-pumps and micro-valves for managing fluids and a Bluetooth wireless transmission system. It includes a Li-ion power source. The miniaturization of actuators (valves, pumps) and other components (sensor, electronics, power supply) in this micro-analyzer together with reduced power consumption and small reagent volumes, result in an autonomous portable system.

The result of this application could lead to advances in the development of wireless chemical micro-systems networks.

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