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Development of a Highly-Miniaturised Wireless ISE/pH Sensor

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Abstract: The goal of this work is to fabricate robust, highly-miniaturised, wireless sensor modules that incorporate ion-selective electrodes (ISEs). pH is one of the main parameters in assessment of the quality of our environment (water, soil) and these ISE/pH sensors will be deployed in a miniaturised, programmable modular system. The simplicity of ISEs (low costs and low power requirements) allow for the preparation of sensors that are all very similar in construction but can at the same time be easily made for variety of different environmentally important ions (i.e. heavy metals). This is important because of the increasing focus on the impact of the quality of the environment on society, both locally, and globally. The work described will contribute to a widely distributed sensor network for monitoring the quality of our environment, focused mainly on soil and water quality.

Key words: Ion Selective Electrodes, Miniaturisation, Modularisation, Wireless Sensors Networks

1. INTRODUCTION

Under the theme of CLARITY's research programme - bringing information to life – which refers to the harvesting and harnessing of large volumes of sensed information, this work is a collaborative effort from electronic engineers in Tyndall National Institute and chemical engineers in DCU. What has taken place in DCU is work on development of miniaturised potentiometric sensors (ion selective electrodes (ISEs)) for variety of ions including pH, lead, cadmium, sodium, potassium and other environmentally and health important ions.

This paper outlines the development and characterisation of the system giving details of the integration of ISEs with Tyndall's 25mm² motes into WSN and testing/deployment of this WSN system prototype in an environmental testing chamber. This iteration of research is necessary for the essential components of the mote to be realised in the 3D integration solution. This will then provide miniature but efficient data transfer modules enhanced for low power and high RF range. The development of the sensor layer is described, and the difficulties involved in the development of a highly miniaturised modular system outlined.

2. DESIGN

2.1. ISE Sensor

Project requirements for the integration of the ISE sensors are the ability to read an analog voltage (potentiometric difference) through a high input impedance and convert it to a digital signal to be transmitted. Ion-selective electrodes (ISEs) have been prepared using screen-printing technology. The electrical contacts were made by printing a carbon ink-based layer on top of a silver ink-based layer. The actual ISE consists of two polymer layers. A conductive polymer polyoctylthiophene (POT) serves to establish well-defined potential between the solid contact and polymer-based ISE. It is drop cast using the 10⁻²M POT solution (relative to monomer) in chloroform.

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Ion selective membrane was prepared by dissolving 1 wt% of hydrogen ionophore H II, 0.5 wt% of NaTFPB, 33 wt % of PVC and 65.5 wt% of o-NPOE (all obtained from Fluka) in 3 ml of THF. The cocktail was drop-cast on top of dried layer of POT and left overnight for the solvent (THF) to evaporate. ISE prepared in this way corresponds to well-studied systems described earlier. [1]

Before the measurement, the electrodes were conditioned overnight in 10^{-3} M universal buffer (mixture of citric acid, boric acid and potassiumdihydrogen phosphate – all in the concentration of 10^{-3} M). Such ISE was connected against a commercially available reference electrode. The two electrodes are in contact with solution and show results comparable to commercially available pH detectors.

The working range of these ISEs is pH 2 to pH 9 where one pH unit corresponds to 59 mV. Prototypes have been developed in forms of a pen-like structure, screen printed (for mass production) and a pen with 7 electrodes. The integration of the screen-printed membrane ISE's are described in this paper.

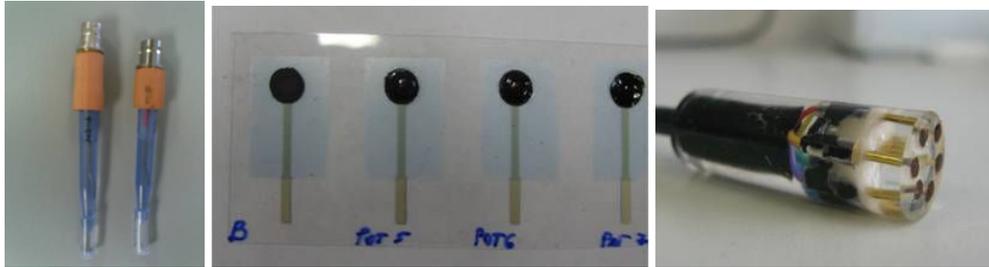


Figure 1: ISE's of pen-like structure, screen printed and a pen with 7 electrodes

2.2. ISE Reference Issues

Calibration has to be performed on pH sensors with either raw data matched with calibration files or perform calibration within the graphical user interface (gui). The system should tag the calibrated pH in the known calibration solution. The connectors of the ISE's should be such that the sensors are easily interchangeable as they are mass-produced and a variety of membranes can be chosen for test purposes.

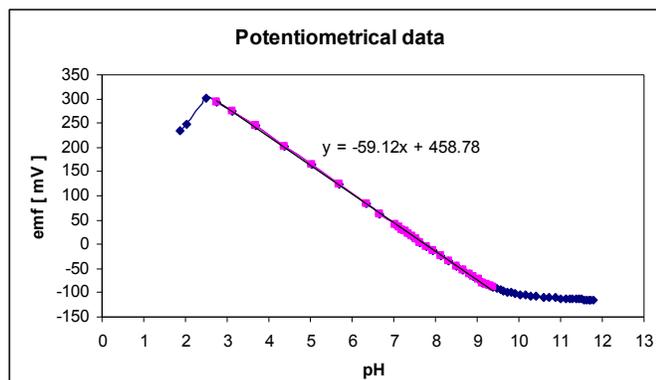


Figure 2: Potentiometrical Data of an ISE relative to a reference electrode.

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2.3. Sensor Layer Design

The sensor layer developed has a programmable gain instrumentation amplifier in series with a fixed 10x gain (non-inverting amp). For pH: prog. gain set to 1, for lead, prog. gain set to 2 etc. The dynamic (input) range can be set based on this. The input range set for the pH electrode is -0.5V to +0.5V and is connected to the positive input of the Analog Devices instrumentation amplifier AD8250, while then the reference electrode and the negative input of the instrumentation amp. are normally connected to ground. In this case, the reference is set at the mid-point of 0V and the supply voltage, therefore putting the input voltage range around this mid-point.

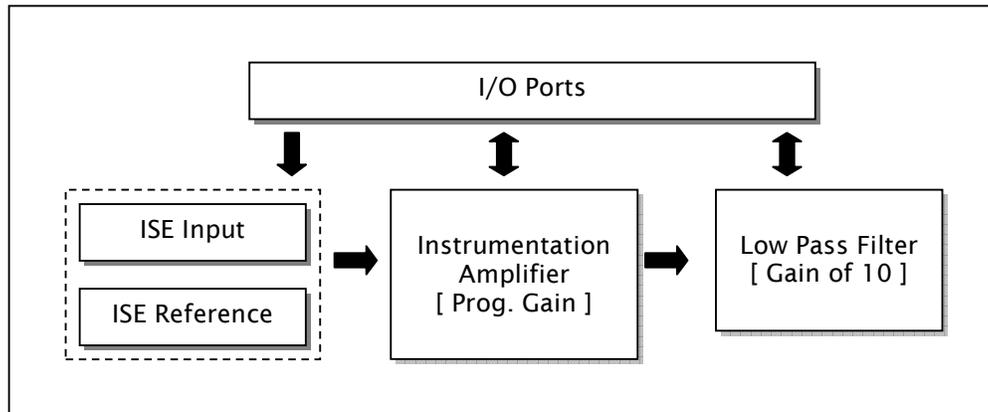


Figure 3: Block Diagram of Sensor Layer module

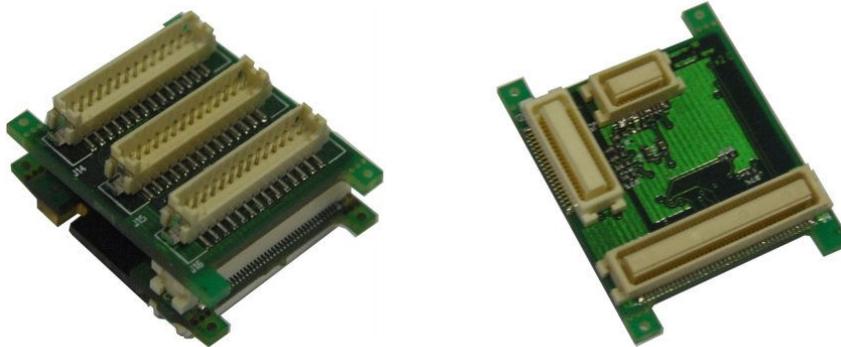


Figure 4: Sensor Layer module on Microcontroller/Transceiver Layer | Bottom side of Sensor Layer

The Tyndall Mote modules use a stackable connector system to make the electrical and mechanical interconnections between layers [2]. Layers developed already include a microcontroller/RF transceiver layer, and a power supply layer. The stackable configuration enables ease of connectivity between the layers depending on the system level requirements. The specific sensor interface /communications layer has been designed in-house and built for this project.

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2.4. Functionality

Using the 'C' programming language to define the functions within the microcontroller, the ISE voltage output can be read by the on-board ADC via the sensor layer board and set up to periodically send this data along with its unique node id. The mote's wireless capability has a Zigbee ready (802.15.4 compliant) transceiver implementation that has been developed at Tyndall to provide a standards based communications protocol for wireless sensor networks. This development of the modular system focused on the Atmel Atmega128 device so as to provide for compatibility across the family of devices.

The development of a graphical user interface for this wireless sensor network is of grave importance, for a chemical engineer does not require programming capability. Presently, the sensor data of each node is tagged, recorded and set up to be viewed on a PC or laptop using LabVIEW via a base transceiver module.

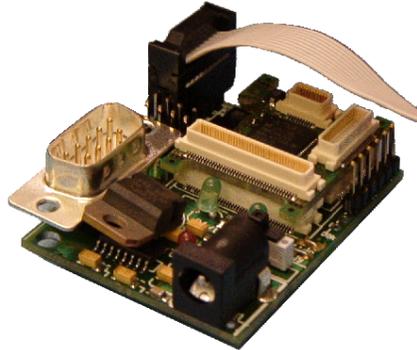


Figure 5: Tyndall Mote programming board with serial interface

3. TEST RESULTS

Results of the deployment of the sensors are currently taking place at time of writing. The system prototype is being tested in a specially constructed environmental chamber located in DCU's chemical science-adaptive sensors laboratory.

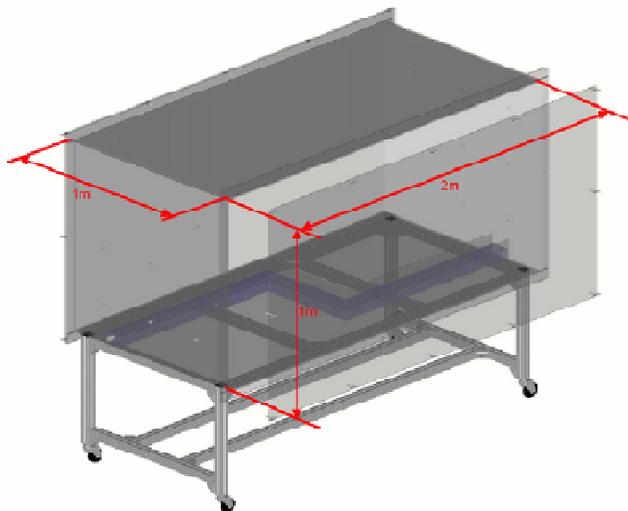


Figure 6: Environmental Sensing Chamber (ESC), dimensions 2m x 1m x 1m, developed for testing wireless sensing networks.

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4. APPLICATIONS

Potentiometric sensors (ion-selective electrodes or ISEs) are well known and have been used for many years for important applications in clinical measurements (e.g. blood electrolyte analysis) and environmental monitoring (e.g. measurement of phosphates, nitrates and chlorides in rivers and lakes). Perhaps the best-known sensor of this type is the glass electrode, which is normally used for pH measurements.

The best commercial success ISEs have found in clinical analysis for analysis of blood electrolytes (i.e. Na^+ , K^+ , Cl^- , Ca^{2+} , Mg^{2+} etc) and ISE-based instrumentation are today in routine use in hospitals around the world. The new advances in the area have allowed that the ISEs find application in environmental analysis for measurement of heavy metal pollution [3], and even for the detection of DNA [4] and proteins [5].

5. CONCLUSIONS | FUTURE WORK

Additional work underway is to implement a way of improving device performance by replacing the conventional wire bond chip-to-board interconnect with a solder interconnection to eliminate or minimise RLC parasitics, etc. The technology to be utilised in module construction relates to a build up process for the batch manufacture of electronic devices each of which comprises one or more die or passive chips. This wafer level packaging process embeds the die in polymer and eliminates wirebond connections by using plated copper interconnect. It is an objective to develop an efficient process that overcomes at least some of the problems with the known devices.

Down the line the mote would also possibly require more sensor channels because of the complexity of environmental analysis and requirements to measure multiple parameters in the same time.

A further objective is to provide a batch fabricated low parasitic packaging technology that does not require specially thinned die and can integrate in a 3D topology, the essential mote components which are of various thicknesses. Both the ISE and complete electronic system will ultimately be incorporated together as one fully functional miniature wireless sensor and be integrated into deployable autonomous sensing network.

REFERENCES

- [1] C.M. McGraw, T. Radu, A. Radu, et al. "Evaluation of liquid- and solid-contact, Pb^{2+} -selective polymer-membrane electrodes for soil analysis", - *Electroanalysis*, Volume: 20, Issue: 3, Pages: 340-346, Feb 2008
- [2] S. Bellis, K. Delaney, B. O'Flynn, J. Barton, K. Razeeb, C. O'Mathuna, "Development of Field Programmable Modular Wireless Sensor Network Nodes for Ambient Systems" , - *Computer Communications - Special Issue on Wireless Sensor Networks and Applications* Volume 28, Issue 13, Pages 1531-1544
- [3] A. Radu, S. Peper, C. Gonczy, et al. "Trace-level determination of Cs^+ using membrane-based ion-selective electrodes" - *International Conference on Electrochemical Sensors*, NOV 13-18, 2005 Matraufured, Hungary; - *Electroanalysis*, Volume: 18, Issue: 13-14, Pages: 1379-1388, Jul 2006
- [4] A. Numnuam, K.Y. Chumbimuni-Torres, Y. Xiang et al. "Potentiometric Detection of DNA Hybridization", - *Journal Of The American Chemical Society*, Volume: 130, Issue: 2, Pages: 410+, Jan 2008
- [5] K.Y. Chumbimuni-Torres, Z. Dai, N. Rubinova, et al. "Potentiometric biosensing of proteins with ultrasensitive ion-selective microelectrodes and nanoparticle labels", *Journal Of The American Chemical Society*, Volume: 128, Issue: 42, Pages: 13676-13677, Oct 2006
- [6] E. Pretsch, "The new wave of ion-selective electrodes" – *Trends in Analytical Chemistry*, Volume 26, No. 1, 2007, Pages 46 – 51
- [7] <http://www.analog.com/en/amplifiers-and-comparators/instrumentation-amplifiers/ad8250/products/product.html>
- [8] <http://www.dcu.ie/~best/ise.htm>

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