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COST-EFFECTIVE SENSORS AND SENSOR NODES FOR MONITORING ENVIRONMENTAL PARAMETERS

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Abstract. *This paper reviews the design and characterization of humidity and pH sensors manufactured in the printed circuit board (PCB), ink-jet, and screen printing technologies. The first one (PCB technology) provides robust sensors with PET film which can be exposed to harsh environment. The second (ink-jet technology) can manufacture sensors on flexible substrates (foils and papers). The third (screen printing technology) has been used to implement a thick-film sensor. In addition to this, a multi-sensor cloud-based electronic system with autonomous power supply (solar panels) for air and water quality monitoring has been described. Finally, a flexible and modular hardware platform for remote and reliable sensing of environmental parameters has been presented.*

Key words: *humidity sensor, pH sensor, sensitivity, stability, sensor node*

1. INTRODUCTION

Advanced applications require different types of sensors which can be manufactured in various technologies. The manufacturing method determines the performance and price of the sensors. This paper deals with the two types of sensors: humidity sensors and pH sensors.

Humidity sensors play an important role in many measurement and control applications in meteorology, agriculture, environmental protection, industry, and medicine. In the past years, a lot of effort has been made to develop high-performance humidity sensors exhibiting the large sensitivity, fast response and recovery, and small hysteresis. Various transduction techniques, such as capacitive, resistive, acoustic, optical, and mechanical, have been adopted for the design of humidity sensors. Their cost depends on the accuracy requirements, response time, hysteresis, sensitivity, mechanical and chemical characteristics, power

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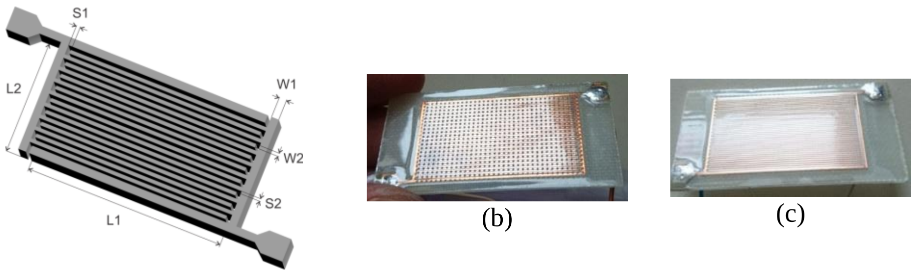
consumption, etc. PET film as one of the most common substrates in industry is used as a sensitive layer for humidity measurements. PET film, compared to different thermoplastics, has equal or better water vapour transmission rate, dimensional stability, service temperature range, etc. Novel sensitive materials, such as graphene-oxide (GO), have recently been introduced in manufacturing process of humidity sensors [1-8]. For instance, the sensitivity of interdigitated capacitive (IDC) humidity sensors has been significantly improved by using the GO as a sensitive material [9].

The monitoring of water quality is an essential task having global impact. This requires determining the parameters such as pH, dissolved oxygen, content of ammonia, conductivity, turbidity, temperature, and dissolved metal ions [10]. Among these the pH is one of the most important as it measures the acidity or basicity of water and directly affects the health of individuals [11]. The pH measurement has wide range of applications including environmental monitoring, chemical processing [12], medical [13], food and beverage [14], biomedical applications such as blood analysis [15], and monitoring of pH fluctuations in the human brain [16]. These applications require highly reliable and accurate pH sensors with the reduced level of maintenance and long lifetime. A range of electrochemical and non-electrochemical methods have been explored for pH measurements [17-19]. Among these the glass electrode based pH sensor has been the most attractive and reliable [17-20]. However, the lack of applicability of the existing solutions in environments that are corrosion prone, or have high temperature and high pressure conditions is a limitation, which provides a strong motivation to develop new pH sensors. In this regard, the metal oxide based pH sensors are attractive as they offer a number of potential advantages over glass electrode pH sensors, including low-cost, smaller dimensions, and ease of manufacturing. Due to high chemical stability, the TiO_2 based films are considered good for pH sensitive layers and a few studies concerning TiO_2 as a pH sensitive layer have been reported as well [21, 22].

2. COST-EFFECTIVE SENSORS MANUFACTURED IN DIFFERENT TECHNOLOGIES

Humidity sensor with PET lamination film

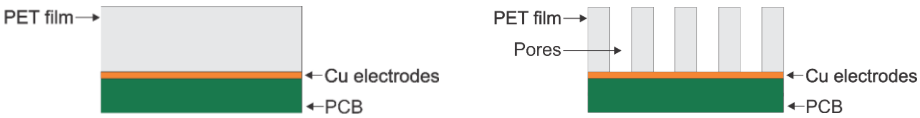
PET film as a sensitive layer has been chosen since, compared to thermoplastics, it has equal or better water vapour transmission rate, dimensional stability, and service temperature range. This film (with and without 400 µm pores) has been laminated on copper electrodes. Three types of IDC structures have been designed and manufactured on the standard FR4 dielectric substrate with a conductive copper layer. Geometrical parameters of the IDC structures have been optimized in order to obtain the targeted capacitance values (from 25 pF to 45 pF). The layout of the IDC humidity sensor is shown in Figure 1a, while the representative samples of the manufactured sensors (with and without macro-porous cover) are presented in Figure 1b and Figure 1c, respectively.



(a) (b) (c)

Fig. 1 a) Layout of IDC sensor, b) Porous PET sensor, c) Standard PET sensor

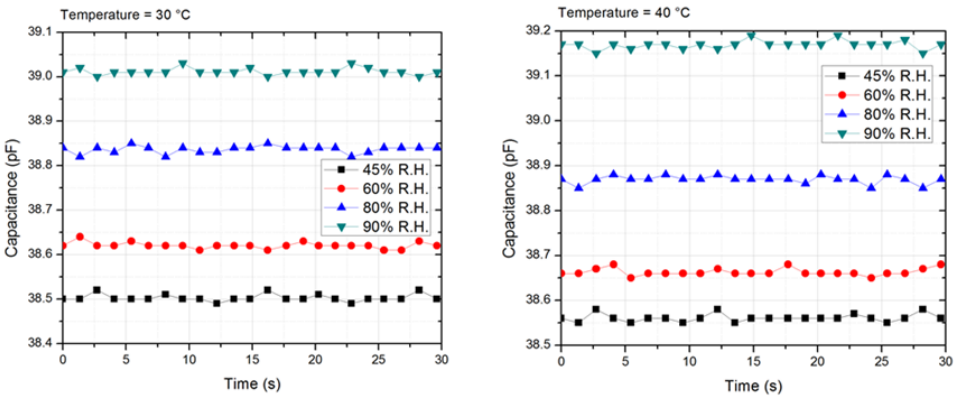
The IDC structures have been measured with LCZ meter (HP 4277A). They have been milled with LPKF ProtoMat S100 machine. A porous PET film with 400-µm pore diameter has been used (Figure 2b).



(a) (b)

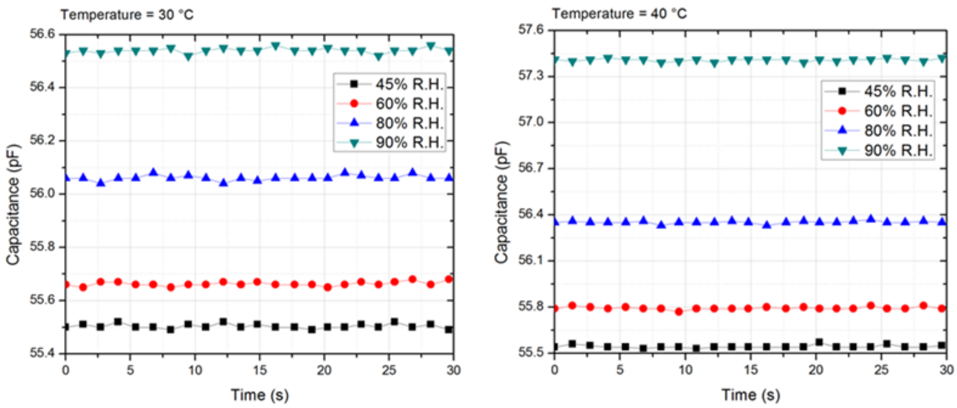
Fig. 2 Schematic structures of proposed humidity sensors: a) PET film laminated on the copper electrodes, b) Porous PET film laminated on the copper electrodes

The change in the capacitance of presented humidity sensors is related to the three different processes [23]. The first is adsorption on the polymer surface (given rise of a new thin layer on the top of the polymer), second is absorption into the polymer phase (changing the dielectric constant of the polymer) and the third is swelling of the polymer layer. Sensor's sensitivity can be increased by adding pores into PET laminated layer which significantly increase the water molecules adsorption inside this porous dielectric film. In order to investigate the humidity response, the sensors have been installed in a chamber with humidity and temperature control (Heraeus Vötsch VLK 08/450). The adjustable humidity range has been between 45% and 90%, while the temperatures were fixed at 30°C and 40°C. The measurements have been carried out with a LCZ meter (HP 4277A), which was connected via Agilent 82357A USB/GPIB interface converter with a laptop. An in-house developed program (created using LabVIEW) has been used for data acquisition. Characteristics of the tested sensors have been determined by observing capacitance variations at 50 kHz. Capacitance values of the two sensor types (with standard PET film and porous PET film) have been measured. Capacitance responses of the sensors for a fixed environment temperature (30°C and 40°C) and relative humidity (45% RH – 90% RH) are presented in Figure 3 and Figure 4. The capacitance stability of the sensor has been observed for time of 30 seconds. Results show a very high stability of the sensor capacitance in time.



(a) (b)

Fig. 3 Standard PET sensor capacitance stability as a function of relative humidity (RH): a) For temperature of 30°C, b) For temperature of 40°C



(a) (b)

Fig. 4 Porous PET sensor capacitance stability as a function of relative humidity (RH): a) For temperature of 30°C, b) For temperature of 40°C

Comparison of the humidity sensor sensitivities is presented in Table I. Above 80% RH at 30°C, the porous sensor has a sensitivity of 48 fF/%RH, while the sensitivity of the standard sensor is 17 fF/%RH. Also, for humidity above 80% RH and temperature of 40°C, the sensitivity of the porous sensor is 106 fF/%RH, while the sensitivity of the standard sensor is 30 fF/%RH.

Measurement results show that the sensitivity of sensors laminated with PET film (standard and porous) increases with increase of the temperature and RH. Similar results have been reported in [24]. Since the adhesive layer is placed between PET film and copper electrodes, the sensor sensitivity is reduced. This is because the adhesive layer, in some way, prevents the transfer of PET film dielectric constant change (due to the water adsorption) to IDC structure and its capacitance.

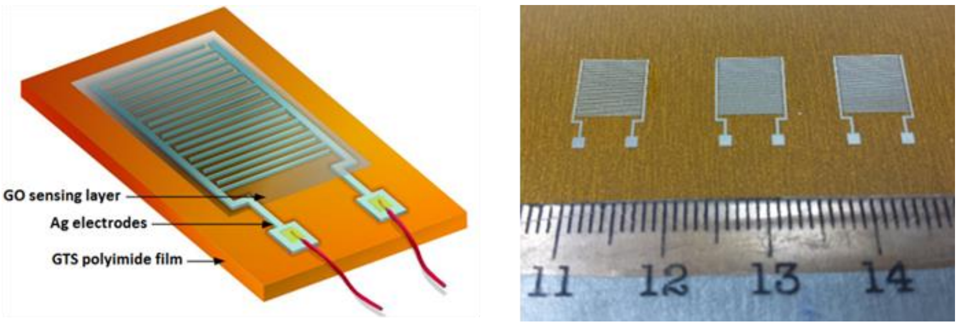
Table 1 Sensor sensitivity in a humidity range from 45% to 90%

Temperature	Sensor	45-60%	60-80%	80-90%
30 °C	PET sensor	8 fF/%RH	11 fF/%RH	17 fF/%RH
	Porous PET sensor	11 fF/%RH	20 fF/%RH	48 fF/%RH
40 °C	PET sensor	8 fF/%RH	12 fF/%RH	30 fF/%RH
	Porous PET sensor	17 fF/%RH	28 fF/%RH	106 fF/%RH

Response time of the sensors is measured to 90% point of the final steady-state capacitance during the relative humidity change from 45% RH to 90% RH at 24°C. Also, the recovery time is measured as time in which the sensor capacitance changes with 90%RH of its maximum value to the initial value while humidity content is reduced from 90%RH to 45%RH. The response and recovery times of the standard PET sensor have been found to be 35 s and 57 s, respectively. Likewise, the response time of the porous PET sensor is 42 s, while the recovery time is 47 s.

Humidity sensor based on graphene-oxide

The sensors have been manufactured by an ink-jet printing process using the Dimatix deposition material printer (DMP-3000) and spin-coating. It has been widely acknowledged that the ink-jet manufacturing technology is cost-effective in the case of humidity sensors [25]. An interdigitated capacitor with 20 pairs of electrodes has been designed, as shown in Figure 5. It consists of a polyimide substrate, interdigitated Ag electrodes, and sensing GO material.



(a) (b)

Fig. 5 Capacitive humidity sensor based on GO: a) Schematic of the sensor, b) Sensor’s electrodes before deposition of GO

The second sensor layer has been manufactured by spin-coating 3 layers of the Graphenea GO ink on top of the electrodes. Measurements have been performed using an in-house measurement setup shown in Figure 6. It consists of a chamber (plastic box) and humidity source (aerosol). Capacitances and resistances of the manufactured sensors have been measured using the Agilent 4284A LCR meter. The Lascar EL-USB-2 humidity and temperature data logger has been used to measure the humidity level inside the chamber.

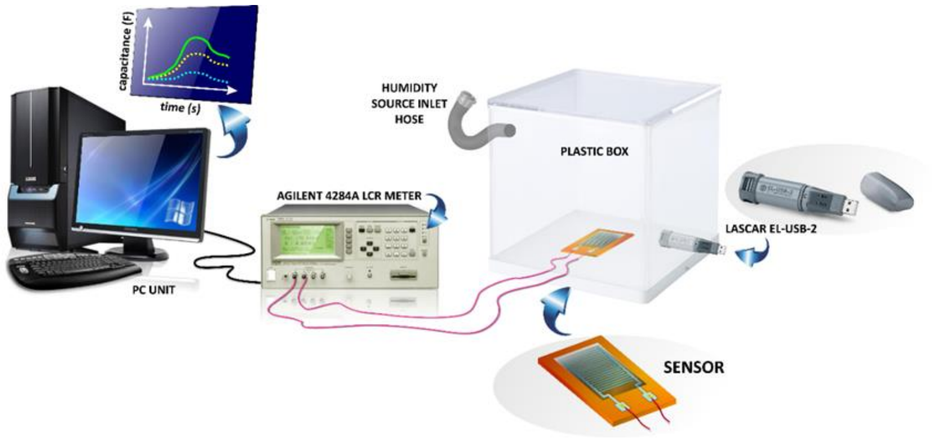


Fig. 6 Humidity sensing and measurement setup

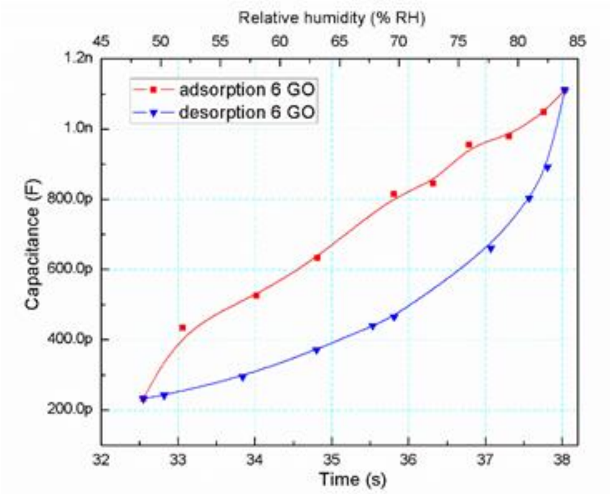


Fig. 7 Capacitance hysteresis curves of the GO-based sensor

The capacitance hysteresis characteristic of GO sensor has been observed by increasing the relative humidity from 45% to 85% for water molecules absorption and then decreasing back to 45% for water molecules desorption. The measurement results are shown in Figure 7. The capacitance values range from 200 pF to 1100 pF, for the relative humidity in the range from 45% to 85%. This indicates that the proposed GO sensor has much higher sensitivity comparing to the others described in open literature. In order to compare the response speed of the analysed GO sensors, the behaviour in absorption and desorption phases has been observed. Figure 7 shows a significant hysteresis (lagging) of the sensor capacitance behind RH variations. This could be explained by different speeds at which the humidity within the chamber (plastic box in Figure 6) has been changed.

Namely, the humidity has been raised by introducing an aerosol device and, after some time, reduced back by self-drying in laboratory conditions.

TiO_2 thick-film pH sensor

Interdigitated electrode (IDE) TiO_2 thick-film based pH sensor has been designed, manufactured, and characterized [26]. A pH measurement system based on the integrated circuit AD5933 [27-29] (which can be used for sensor impedance characterization, as well as sensor readout electronics) has also been implemented. The manufacturing process of the conductimetric pH sensor is similar to that reported in [11, 30]. We have chosen alumina as a substrate to investigate the performance of pure metal-oxide and to avoid any reaction at the metal/metal-oxide interface. Initially, a planar IDE has been deposited on an alumina substrate by screen printing of Ag paste (Ag/Pd ESL 9695). The screen printing of metal paste is a faster way of manufacturing devices at low cost [31]. Illustration of the conductimetric pH sensor is shown in Figure 8a. The major advantages of the IDE pH sensor, compared to the other reported approaches, are: faster and low-cost manufacturing, lack of reference electrode, large surface area, and low energy consumption during measurements. In addition, the screen printing technology could open avenues for integrating the pH sensors with electronics on flexible substrates [31].

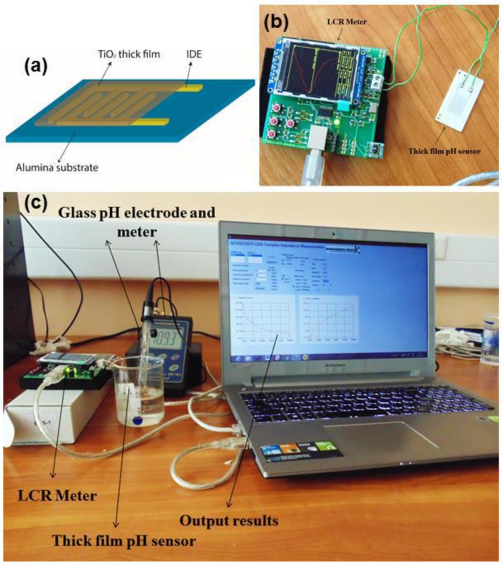


Fig. 8 a) Illustration of TiO_2 pH sensor, b) Impedance measurement device connected to pH sensor, c) Experimental setup for pH sensor characterization

The AD5933-based impedance measurement system reported earlier [27-29] has been used for sensor characterization. Figure 8c shows the experimental setup for spectroscopic analysis of the sensor impedance. The sample under test has been connected to the measurement device and placed into a beaker with a solution. The sensor can be employed in water pollution monitoring, with an expected operating pH range from 6 to 9, thus test solutions with pH ranging from 4 to 10 have been prepared by adding 1 mol% of HCl or KOH to distilled water. A standard glass-electrode pH and conductivity meter (ELMEIRON, CPC-411) with temperature probe has been used to control the pH level of test solutions and measure the conductivity of each test solution. The sensor has been washed with deionized water and dried with a paper towel after each measurement to reduce the contamination of the electrode surface by solutions with different pH. All measurements have been done at room temperature with the liquid temperature close to 23°C. To measure the electrical parameters of TiO_2 films at different pH values, the sensor has been dipped in the solution for 10 min prior to operating to ensure the steady-state. The impedance measurement has been done by performing frequency sweep in range of 5-20 kHz with AC voltage of 200 mV. Fig. 9 illustrates the variation of impedance magnitude and phase angle as a function of frequency in the range 5-20 kHz for different pH values of test solution. The magnitude and phase of the sensor impedance decrease with increase of the pH value of solution. For a constant pH, there is a decrease in magnitude and an increase in phase as the frequency increases.

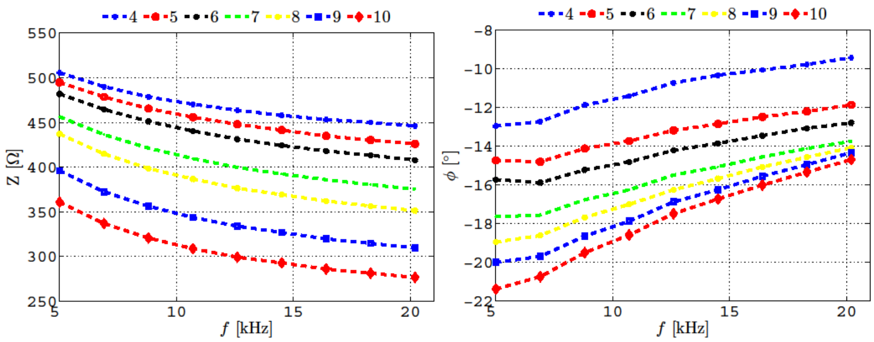


Fig. 9 Impedance magnitude and phase angle plots for TiO_2 thick-film pH sensor for different pH values of solutions over a frequency range from 5 kHz to 20 kHz

From Figure 9, it can be noted that the impedance is lower when the sensor is in a solution of a higher pH. Variations of the solution resistance with different pH values contribute to changes of the sensor impedance. The observed dependence is mainly caused by a lower resistance of the applied alkaline solutions as compared with the acidic solutions. The variation in impedance with frequency can be attributed to the effect of intercrystalline capacitance [32]. In the kHz-range, this value is sufficient for short-circuiting the spaces between the grains, which reduces the resistance of sensor [32].

Obtained impedance data has been used for more detailed sensor characterization regarding sensor sensitivity. It is very important to determine impedance changes of the sensor compared to changes of the pH value of the analysed solutions. The developed impedance measurement device (used for the sensor characterization) can be used as readout electronics as well, if the measurement error is lower than the sensor sensitivity. Sensor sensitivity regarding the relative change of impedance magnitude (Z) with pH value change can be defined as

$$S_Z(\mathrm{pH}) = \frac{|Z_{\mathrm{pH}} - Z_{\mathrm{pH\text{-}1}}|}{Z_{\mathrm{pH\text{-}1}}} 100\%,$$

for pH values between 4 and 10. Sensor sensitivity regarding relative change of impedance phase angle (ϕ) with pH value change can be defined as

$$S_\phi(\mathrm{pH}) = \frac{|\phi_{\mathrm{pH}} - \phi_{\mathrm{pH\text{-}1}}|}{\phi_{\mathrm{pH\text{-}1}}} 100\%.$$

In Figure 10, relative changes of the impedance magnitude and phase angle of the manufactured pH sensor are shown. The five frequencies (5 kHz, 8.8 kHz, 12.6 kHz, 16.4 kHz, and 20.2 kHz) in the analysed frequency range are chosen to establish a linear frequency distribution.

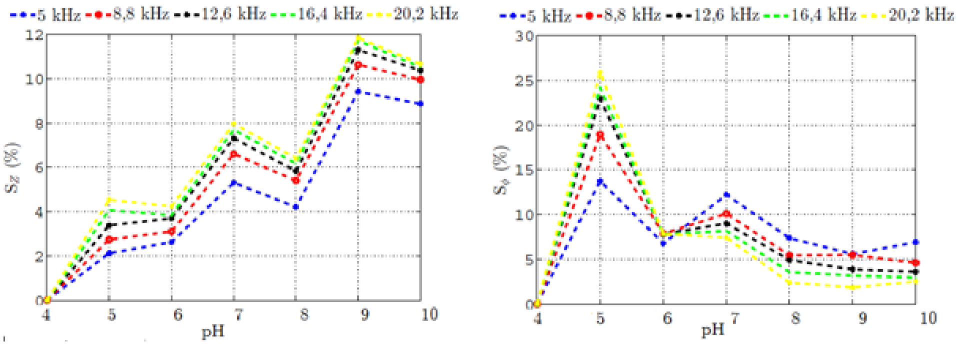


Fig. 10 Sensitivity of sensor impedance magnitude and phase angle

As it can be seen from Figure 10, the relative change of the impedance magnitude is higher than 2% and it increases with frequency increase. Additionally, there is decrease in relative change of the impedance phase angle with increase of the pH value. Therefore, for pH values lower than 7, it is more convenient to measure the phase angle, while for pH values higher than 7, it is better to measure the impedance magnitude. Moreover, it can be concluded that the reported measurement error of 2% of developed AD5933-based device [27-29] is acceptable in typical applications.

3. Wireless Sensor Nodes for Environmental Parameter Measurements

A TiO_2-based sensor has been used with commercial sensors in realization of a wireless sensor node for environmental parameters monitoring (pH, temperature, relative humidity, volatile organic compounds, etc.) [33]. It is a low-cost, portable, and low-power system powered by a solar-panel charger unit, thus providing automated in-situ measurements and data storage operations. Compared to the systems presented in literature, the design shown in Figure 11 offers the advantage of remote multi-parameter measurements in real-time [33].

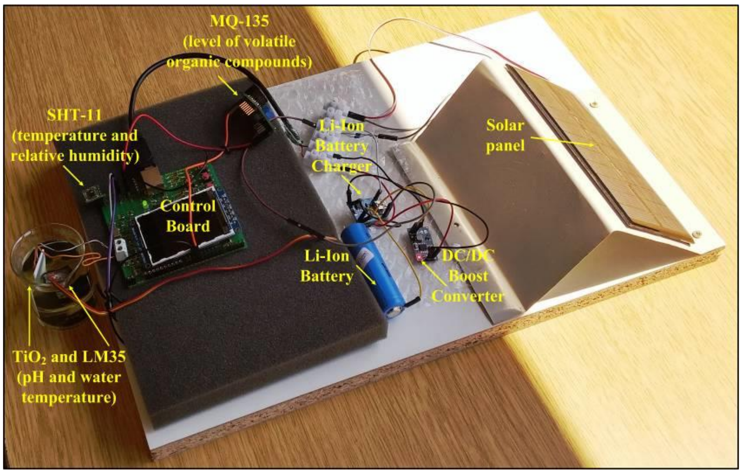


Fig. 11 Wireless sensor node with a pH sensor and additional commercial sensors

Moreover, the developed system for remote measurement and acquisition of environmental parameters has been integrated in a more complex cloud-based system which ensures remote access to the measurement data in real-time. IBM IoT platform has been used for data presentation and Internet access of measurement results, as it is shown in Figure 12.

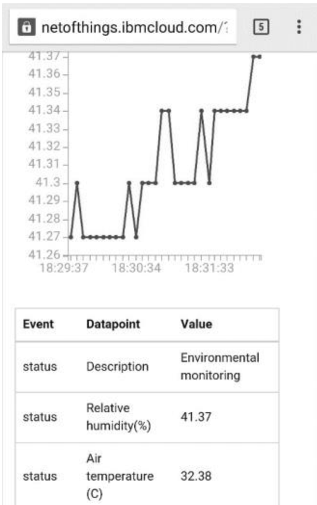


Fig. 12 Browser view of the web IBM Watson IoT platform with sensor data

IHPNode has been developed as a flexible and modular hardware platform for remote sensing in environmental and agricultural applications [34]. The node (Figure 13) is based on the Texas Instruments MSP430x low-power microcontroller and three RF transceivers, one working in the 868 MHz and two working in the 2.4 GHz frequency band. The two of these (CC1101 and CC2500) support flexible proprietary network protocols, while the third (CC2520) provides a network coprocessor for ZigBee protocol integration.

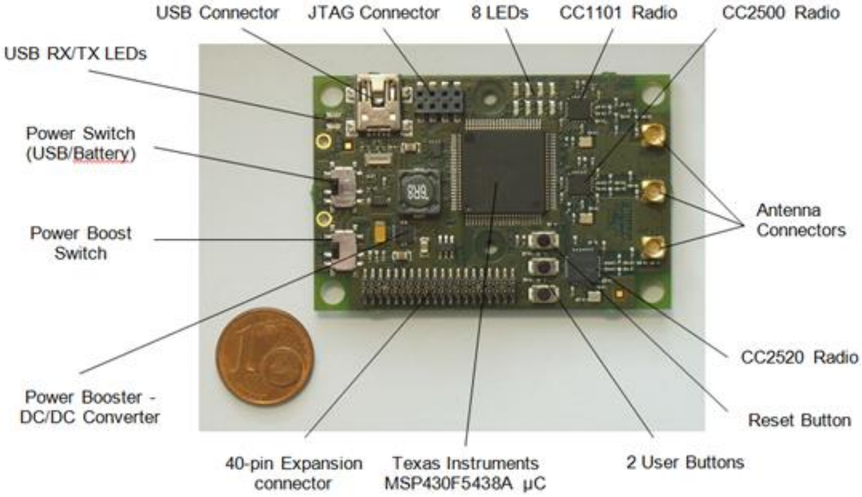


Fig. 13 IHPNode based on TI MSP430x microcontroller

4. Conclusion

Cost-effective humidity and pH sensors have been designed, manufactured, and characterised. Two wireless sensor nodes for remote monitoring of environmental parameters have been developed using the aforementioned humidity and pH sensors. The future work should complete and integrate these nodes into a smart multi-sensor cloud-based hardware/software platform for environmental and agricultural applications.

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