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A Calibrated Intelligent Sensor for Monitoring of Particulate Matter in Smart Cities

^{1,*} Mariana Rodrigues Villarim, Douglas de Farias Medeiros,
Larissa Cavalcanti de Sousa Medeiros, Cleonilson Protasio de Souza,
Márcia Helena Pontieri, Nataly Albuquerque dos Santos
and Orlando Baiocchi

¹ Federal University of Paraíba, João Pessoa, 58051-900, Brazil

² University of Washington Tacoma, Tacoma, WA 98402, USA

¹ Tel.: +558330450215

E-mail: mariana.villarim@cear.ufpb.br

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Abstract: Air pollution is directly related to both the cause and aggravation of various diseases and the cause of ecological damage to the environment. According to the World Health Organization (WHO), 4.2 million deaths are attributed to air pollution each year and 91 % of the World's population lives in places that exceed the indicated air quality limits. In this work, an intelligent and compact wireless sensor for air pollution monitoring is presented having as main target to alert the population and to allow certain control of pollutants emissions by governing authorities. The proposed wireless intelligent sensor monitors Particulate Matter (PM) using the cheap and compact PPD42NS sensor, which uses light scattering to measure particles concentration. To consider the PPD42NS sensor as the PM sensor to the proposed wireless sensor, a calibration process was conducted using as the reference equipment the HiVol HVS 3000, fabricated by the Ecotech and approved by the EPA as a Federal Reference Method. After the calibration process, the PPD42NS shows to be very effective to measure PM concentration in real time allowing the proposer wireless sensor to compute the Air Quality Index (AQI) in the area it is deployed. Another result takes the PPD42NS power consumption in consideration and points out a possible approach to reduce it, namely, eliminating its electronic circuit. The conclusion is that just 5% of power consumption is achieved with this solution since the most responsible for the consumption is the heater resistor.

Keywords: Air quality, Particulate matter, PPD42NS sensor, HiVol HVS 3000.

1. Introduction

Air pollution is a major environmental issue associated with several diseases in humans and being the cause of millions of deaths worldwide annually [1]. In addition, air pollution also is responsible for ecological damage to the environment [2]. The World Health Organization (WHO) has estimated that 4.2 million deaths are attributed to air pollution each year

and 91 % of the World's population lives in places that exceed the indicated air quality limits [3].

Outdoor air pollutants have complex chemical and their physical features are direct dependent on their sources, which can be from human activities, such as industrial and vehicles emissions, agricultural activities or natural hazards [4].

The pollutants presented as inorganic forms that cause the greatest health concerns are Particulate

Matter (PM), Ozone (O_3), Nitrogen Dioxide (NO_2), Sulfur Dioxide (SO_2), Lead (Pb), and Carbon Monoxide (CO) [3]. In developing countries, due to their huge industrialization, the main sources of atmosphere pollutants including the means of transport, such as cars and trucks, and industries [5].

Several studies have demonstrated the negative health effects related to PM emissions [6, 7]. PM can cause numerous health damage as it can be deposited in various sites of the respiratory system and in the bloodstream, and, as a result, it is associated with serious respiratory and cardiovascular problems [8]. In addition, emissions of PM into the atmosphere have been contributing to global warming, climate change and air pollution increase [8].

PM is formed by solid and liquid micro-sized particles from different sources, such as carbon-centered and secondary inorganic combustion. According to the U.S. Environmental Protection Agency (EPA), PM can be classified in three different categories according to its diameter:

- TSP (Total Suspended Particulate Matter): particles with aerodynamic diameter less than or equal to 50 μm ;
- PM_{10} : Inhalable Particles with aerodynamic diameter particles less than or equal to 10 μm . When inhaled, they can pass through the body upper airways;
- $PM_{2.5}$: Fine Inhalable Particles with aerodynamic diameter less than or equal to 2.5 μm . When inhaled, they can pass through the body lower airways, settle in the lungs and cause great health risks.

For average concentration of $PM_{2.5}$, PM_{10} , and TSP, EPA defines the 24-hour reference period as the basis for assessing the risk for PM.

In general, PM measuring devices are based on the following methods: gravimetric, microbalance, and optical [9] as shown in Fig. 1. The gravimetric method consists of a quantitative analytical method based on the difference between the final and initial value of the mass of the particles collected by a filter in a pre-defined sampling period [10]. The gravimetric method is defined as the reference method for measuring PM concentration by EPA.

In the microbalance method, the particles are deposited on the surface of an oscillating device and the MP concentration value can be obtained based on the modified oscillation period [11].

Considering the optical method, there are three types for measuring concentration of MP according to the interaction of the light behavior through the particles, namely: light scattering, light absorption, and light extinction, as pictorially shown in Fig. 2. In the light scattering method, when the incident light beam hits the particles, they are dispersed in several directions. In light absorption, a part of the light on the particles is absorbed and can be transformed into another form of energy, such as heat, and the PM concentration can be measured by particles heating and light attenuating [11]. Finally, light-extinction based PM concentration measurement is obtained from the difference between the incident and the transmitted light through the particles and the PM

concentration is obtained from the fraction of the incident light that hits the receiver (opacity meter) [9].

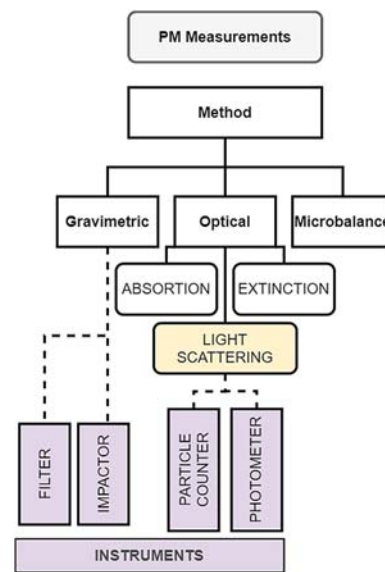


Fig. 1. Methods and instruments for measuring PM concentration [9].

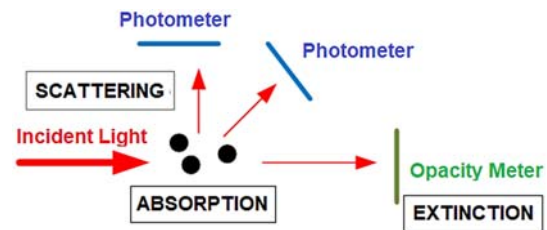


Fig. 2. Optical methods for PM measurement [11].

In a practical point of view, acquiring environmental data by monitoring techniques are needed to assess air pollution in the environment and the results of such actions are very important to government for environmental policy making. However, particularly in developing countries, air pollution monitoring stations are rare since they are very expensive to deploy and to maintain. Low-cost sensor-based approach can be of helping in this context. In addition, despite their precision, a large amount of them deployed around a given area, for instance, a city or a portion of it, can form a spatially distributed air pollution monitoring making possible to identify specific places and times with higher levels of emissions and, then, adopt local strategies to reduce the levels of pollutants. In this scenarios, Internet of Things (IoT) may be a very interesting approach.

IoT is a paradigm that allows several kinds of interaction actions between real-life objects (called “things”), embedded with sensors, microcontrollers and radio transceivers, share data wirelessly to the internet [12] making them able to communicate autonomously with each other and also with the users [13] for getting data and, optionally, make decisions.

For distributed environmental monitoring, IoT is a solution for sharing data of environmental variables in real time [14] such as PM concentration, temperature, atmospheric pressure, humidity, harmful gases concentration, etc. and also computing the Air Quality Index (AQI).

A limitation of an IoT solution for real-time air quality monitoring systems is the calibration of the used sensors [15], mainly, when low-cost sensors are taken in consideration to guarantee some level of data quality.

Although, low-cost sensors have low accuracy for certain applications, they can be considered suitable to provide information on levels of environmental pollution [16]. In this way, low-cost sensors must be evaluated according to the applications and the application needs for high or low precision. On the other hand, air quality can be assessed in several sites covering a large area by using an adequate number of low-cost sensors [17].

There are some alternatives for low-cost environmental monitoring and several commercial sensors for measuring PM. One of them is the PM sensor model PPD42NS, which is based on light scattering method. However, the use of such a sensor has to be validated through calibration against a Reference Method approved by an environmental agency, like the EPA/ EUA, to verify the quality of the data.

The main objective of this work is to evaluate the performance of the PPD42NS sensor as the PM sensor to a proposed wireless sensor for distributed PM monitoring where a calibration process was conducted using as the reference equipment the HiVol HVS 3000, fabricated by the Ecotech and approved by the EPA as a Federal Reference Method. In addition, some considerations about the PPD42NS power consumption are taken and possible solution to reduce it is addressed.

2. PPD42NS PM Sensor

A low-cost PM sensor unit fabricated by SHINYEI Technology is the PPD42NS that is based on the light scattering method [18]. The sensor is capable to detect particles continuously and consists of three main parts: an Infrared LED (IRED) and a phototransistor (PT), as seen in Fig. 3, and a signal condition electronic circuit, shown in the Fig. 12. The air flow is self-aspirated by means of a built-in heater resistor that drive external airborne particles into the sensor enclosure from the bottom inlet towards the top outlet, as seen in Fig. 3, and when the IRED emits infrared light beam that hits the particles, they scatter the incident light and part of them are sensed by the PT.

The signal condition electronic circuit of the PPD42NS converts PT output signal into a pulse output that corresponds to the concentration per unit volume of particles measured. Specifically, to get the PM concentration from the sensor, it is needed firstly to compute the ratio between the LPO (Low Pulse

Occupancy) time, which corresponds to the cumulative time at low level, and the a given time period, normally, 30 seconds, as shown in Fig. 4. After this and using the computed ratio, it is possible to find the corresponding PM concentration value using the characteristic curve provided by the manufacturer.

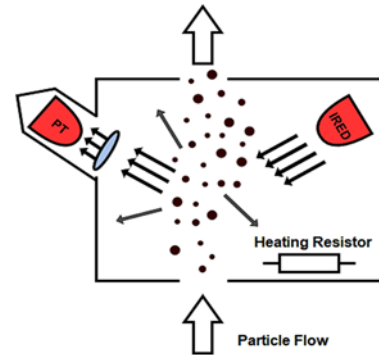


Fig. 3. Light scattering method used in the PPD42NS sensor [19].

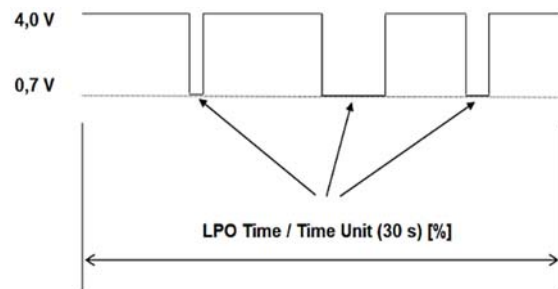


Fig. 4. Low Pulse Occupancy (LPO) [20].

The smallest particle diameter that can be detected by the PPD42NS is $0.5 \mu\text{m}$ and it is an adequate alternative to estimate PM concentration levels in environments exposure to air pollution when calibrated against a reference equipment [21]. As humidity can influence measurements from PPD42NS sensor since at humidity levels above 90 %, its measurement is affected [19] as the water particles absorb part of the infrared radiation, reducing the light intensity received by the phototransistor and interfering with the operation of the sensors circuit. For this reason, the das sensor BME680 from Bosch Sensortec was used as it measures relative humidity and other variable: barometric pressure, ambient temperature and some Volatile Organic Compounds (VOC) gases.

3. HiVol 3000 Reference Equipment for PM Measurement

The HiVol HVS 3000 equipment is fabricated by Ecotech Pty Ltd and is a particulate sampler and performs remote unattended sampling of $\text{MP}_{2.5}$, MP_{10} or PTS and others basic meteorological parameters. It

presents the following dimensions: 380 mm (W) x 380mm (D) x 1200mm (H), and is shown in Fig. 5. The HiVol 3000 allows to sample $MP_{2.5}$, MP_{10} or PTS depending on the particle separator mechanism [22] and holds approval of the U.S. EPA as a Federal Reference Method.

The first stage of the HiVol 3000 is the air sampling where an electronic volumetric flow control system, using acceleration nozzles, maintains a constant air flow to collect a representative sample of particulate matter. In the acceleration process, the heavier particles gain greater momentum and are trapped on an impact plate with a greased surface to stick them. After this first stage, only the smallest particles are left and dragged to the second stage through a porous-material filter that collect the remainder particles [22].



Fig. 5. A picture of the HiVol HVS3000 particulate sampler [22].

After a pre-determined sampling period, generally, 24 hours, the filter, with the accumulated particles deposited on it, is weighed to measure the final mass of the filter after the sampling period, M_f , and the PM concentration, PM , can be computed by the following equation:

$$PM = \frac{(M_f - M_i) \cdot 10^6}{vol}, \quad (1)$$

where PM is the PM concentration in $\mu\text{g}/\text{m}^3$, and M_i , the initial mass of the filter before the sampling period, 10^6 means the conversion to μg and vol is the total volume of air sampled in m^3 .

It is important to highlight that the weighting process and the computation of the PM concentration by using the HiVol HVS 3000 is manually accomplished.

4. The Intelligent Sensor for PM Monitoring

The developed compact and low-cost intelligent sensor scheme for PM monitoring and measurement of others basic environmental parameters is presented in Fig. 6. The intelligent sensor is composed of PM sensor PPD42NS and the BME680 sensor for environment variables, such as ambient temperature, humidity, barometric pressure, and VOC gases. An ARM Cortex MO+ is used to process the sensor signals and a LoRa-communication transceiver (TX) is used to transmit sensor data to a receptor module (RX), which can be interconnected to a PC computer by a USB connection.

Both the processor and the transceiver are part of a development board from Adafruit, called Feather M0 RFM95. The intelligent sensor is powered by a battery charged by a 6 V / 2 W solar panel. LoRa-communication solution has been chosen because it allows the sensor to be deployed in a very large distance ($\approx 3 \text{ km}$) from the receptor module.

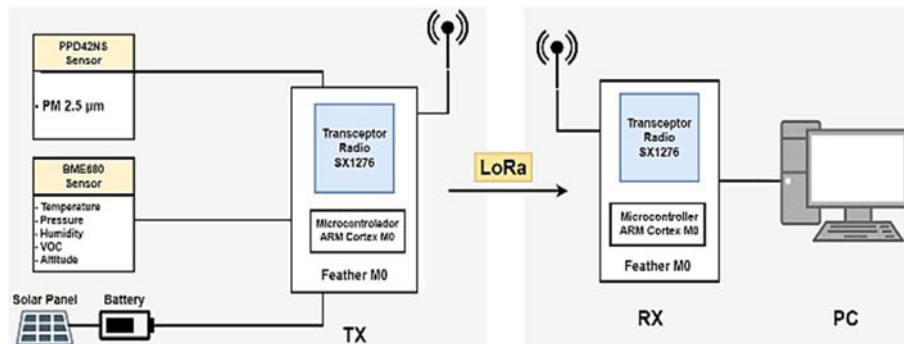


Fig. 6. Transmitter and receiver connections.

The sensors (PPD42NS and BME680) are enclosed into a climate shelter formed by a pile of plates to let air flows through them and protect them

from rain. The electronic parts of the intelligent sensor are enclosed IP66-protection-level case, as shown in Fig. 7.

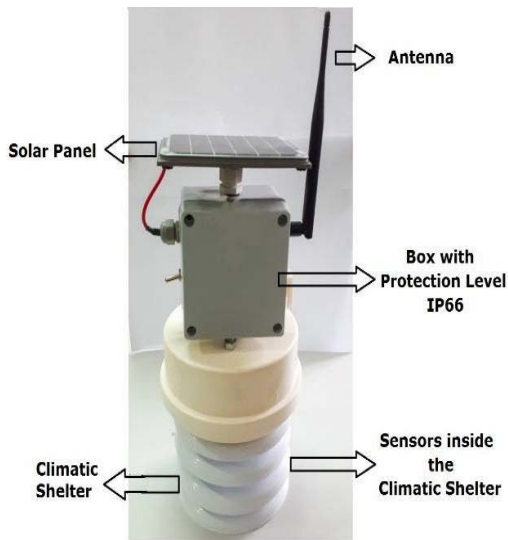


Fig. 7. Portable sensor for monitoring particulate matter and environmental variables.

In the Table 1, it is described the basic algorithm running in the sensor processor to acquire a set of measuring data along 3 minutes with steps of 30 seconds.

This approach has been used to reduce the number of transmissions and, consequently, reduce power consumption decreasing the use of the LoRa radio. In this way, after each 3 minutes, the set of data is transmitted: PM_{30} , PM_{60} , PM_{90} , PM_{120} , PM_{150} , PM_{180} , where PM_t is the PM_{2.5} concentration at the time $t = \{30, 60, 90, 120, 150, 180\}$ and the BME outputs (ambient temperature, humidity, barometric pressure, and VOC gases) in a single LoRa

package. However, despite this transmission rate, the sampling rate is 30 seconds.

Table 1. Algorithm for Sensors Reading and Transmission.

Algorithm: Sensors Reading and transmission.	
1:	{Step 1} Reset the Timer
2:	Wait 30 seconds
3:	{Step 2} Read the PPD42NS and save as PM_{30}
4:	Wait 30 seconds
5:	{Step 3} Read the PPD42NS and save as PM_{60}
6:	Wait 30 seconds
7:	{Step 4} Read the PPD42NS and save as PM_{90}
8:	Wait 30 seconds
9:	{Step 5} Read the PPD42NS and save as PM_{120}
10:	Wait 30 seconds
11:	{Step 6} Read the PPD42NS and save as PM_{150}
12:	Wait 30 seconds
13:	{Step 7} Read the PPD42NS and save as PM_{180}
14:	Read the BME680
15:	{Step 8} Transmit PM_{30} , PM_{60} , PM_{90} , PM_{120} , PM_{150} , PM_{180} and BME outputs
16:	{Step 9} Go back to Step 1

Fig. 9 shows the obtained PM_{2.5} data obtained during 4 days. As can be observed the higher PM_{2.5} concentration values occur from 6 am to 3 pm, approximately, and the highest near 12 noon when the local temperature is the highest and humidity is the lowest. The field experiment took place in the city of João Pessoa in northeastern Brazil. On the weekend, it is possible to observe that the PM_{2.5} concentration decreases compared with the other days and even more notable on Sunday when the movement of people and vehicles is reduced.

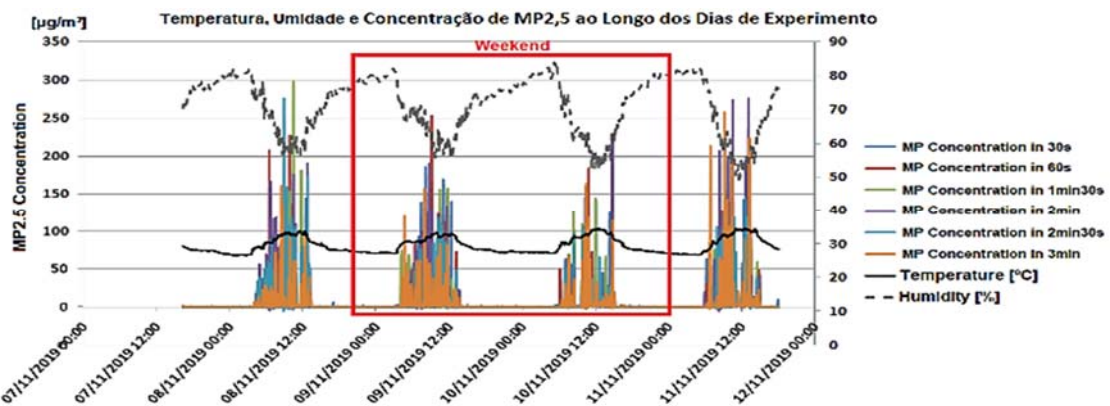


Fig. 9. Concentration data of PM_{2.5} during the days of the field experiment.

5. Results and Discussion

The HiVol 3000 provides data on a daily base, that is, a PM_{2.5} measurement is obtained every 24 hours as preconized by EPA that defines 24-hour reference time period for PM risk. In a practical point of view, the filter of the HiVol3000 is weighted and changed at 5 pm every day of the experiment and the considering

24-hour time period if from 5 pm of the day to 5 pm of the other coming day. Therefore, the same time period was considered to compute the 24-hour-basis PM_{2.5} concentration using the PPD42NS.

To convert the 30-seconds measurement values during 24 hours to a 24-hour-basis PM_{2.5} concentration, PM_{24} , the Eq. (2) was used:

$$PM_{24} = \frac{\sum_{k=1}^n (PM_{30}^k + PM_{60}^k + PM_{90}^k + PM_{120}^k + PM_{150}^k + PM_{180}^k)}{6n}, \quad (2)$$

where n represents the number of receiver packets in 24 hours and PM_t^k , the PPD42NS reading as describes in Table 1, at a k sample.

The experimental results for the average daily PM_{2.5} concentration of the HiVol3000 and the PPD42NS sensor are in the Table 2.

Table 2. Experimental obtained measurement values.

Day	Reference Equipment HiVol 3000 ($\mu\text{g}/\text{m}^3$)	PPD42NS Sensor ($\mu\text{g}/\text{m}^3$)
1	10.345	7.558 ± 0.725
2	10.681	7.347 ± 0.715
3	7.838	5.057 ± 0.763
4	10.969	8.112 ± 0.881
Average	9.958	6.483 ± 0.771

5.1. Calibration Curve

Taken in consideration the data from the Table 2, the calibration curve was obtained by using the *polyfit* function of Octave tool, in which returns the coefficients of the polynomial that best fits the data. The obtained calibration curve was the first-order polynomial $y = 1.278x + 0.983$ and the curve is shown in the Fig. 10.

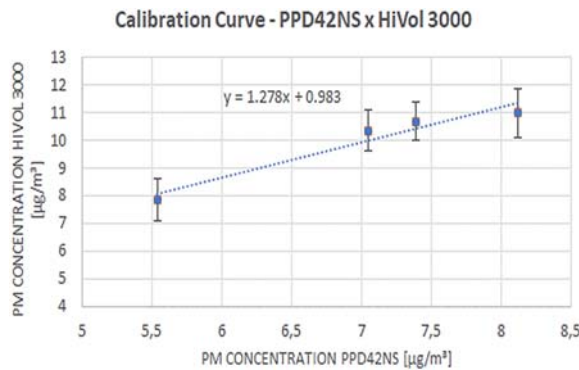


Fig. 10. Obtained calibration curve: PPD42NS sensor versus HiVol3000.

5.2. Air Quality Index (AQI)

The obtained PM_{2.5} concentration measurement values obtained from the calibrated PPD42NS sensor can be used to compute the Air Quality Index (AQI) to provide the people a simplified air quality information at a given site, and varies from 0 to >200, which is categorized in 5 Quality Level: N1, N2, N3, N4, and

N5. The higher the AQI value, the worse the air quality, for example, N5 indicates poor air quality, and N1 means good air quality, that is, no impact on health. The categories of AQI and its different impacts on human health are summarized in Table 3.

Table 3. Air Quality Index categories and their different health impacts [23].

Quality Level	AQI	Health Effects
N1 Good	0-40	It has no effect on health.
N2 Moderate	41-80	People from sensitive groups (children, elderly and people with respiratory and heart disease) may experience symptoms such as dry cough and tiredness. The population, in general, is not affected.
N3 Bad	81-120	The entire population may experience symptoms such as dry cough, tiredness, burning eyes, nose and throat. People in sensitive groups can have more serious health effects.
N4 Very Bad	121-200	The entire population may experience worsening symptoms such as dry cough, tiredness, burning eyes, nose and throat and shortness of breath and wheezing. Even more serious effects on the health of sensitive groups.
N5 Hazardous	>200	The entire population may present serious risks of manifestations of respiratory and cardiovascular diseases. Increase in premature deaths in people from sensitive groups.

The AQI is a dimensionless value that represents the concentration of pollutants in the atmosphere and is computed using the Eq. (3) given as follows:

$$AQI = I_{ini} + \frac{I_{fin} - I_{ini}}{C_{fin} - C_{ini}}(C - C_{ini}), \quad (3)$$

where

- I_{ini} : Index value that corresponds to the initial concentration of the range;
- I_{fin} : Index value that corresponds to the final concentration of the range;
- C_{ini} : Initial concentration of the range where the measured concentration is located;
- C_{fin} : Final concentration of the range where the measured concentration is located;
- C : Measured pollutant concentration.

The AQI was also computed considering the PM_{2.5} data of the PPD42NS sensor obtained during the 4 days of experiment shown in the Fig. 9.

Fig. 11 shows the computed AQI calculated during the days of the experiment and the colored horizontal lines indicates the AQI quality levels. It can be observed that the great majority of the time the air is good or moderate, and sometimes the air is very unhealthy (above the purple line).

6. PPD42NS Power Reduction

The circuit diagram of the PPD42NS is shown in the Fig. 12 where can be seen the Infrared LED

(LED1), the phototransistor (PIC1), and the heating resistor (RH1=100 Ω). The PM sense signal is at the test point, TP16, that is input to the signal conditioning circuit based on operational amplifiers forming filtering stage (a and b) and 2 comparators (c and d). The outputs of these comparators are P1 and P2 that digitally indicate the PM concentration of particles whose size is around 1 μm or larger and 2.5 μm or larger, respectively, as shown in Fig. 4.

The attempt to reduce the PPD42NS power consumption came from the premise that the OpAmp-based signal conditioning circuit can be bypassed or eliminated since normally the PPD42NS is interconnect to an embedded ADC of the used microcontroller-based SoC (System-on-Chip).

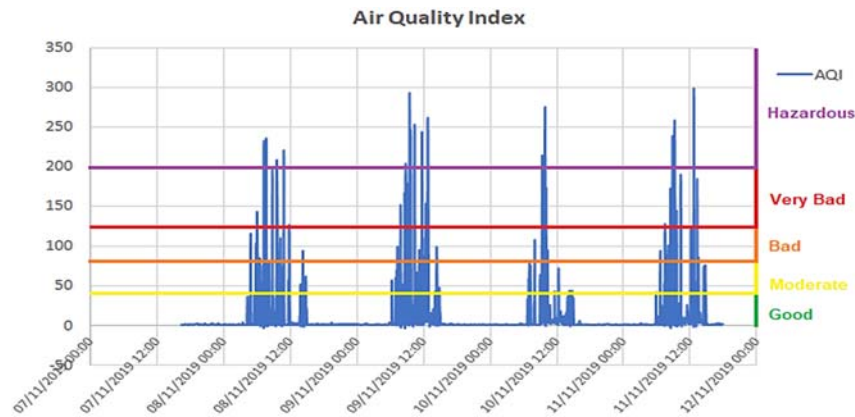


Fig. 11. Air Quality Index (AQI) during the days of the experiment.

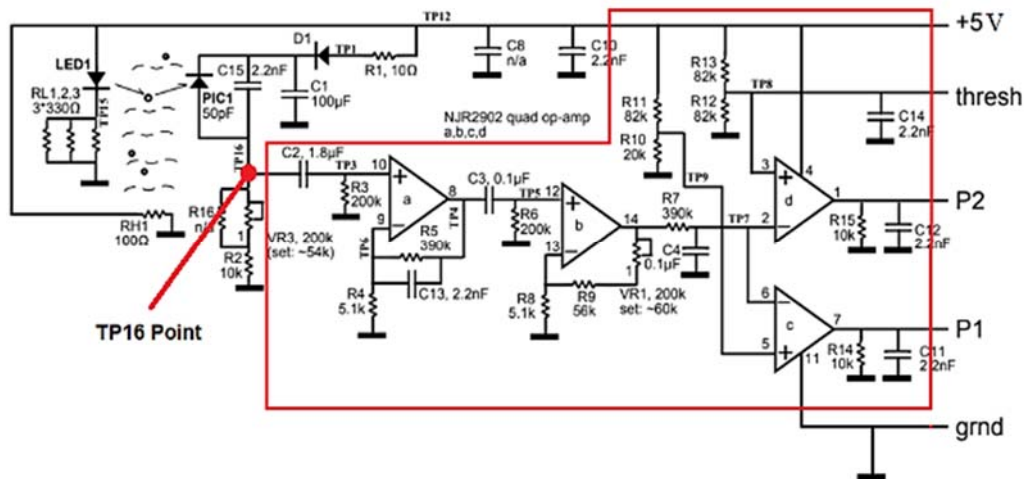


Fig. 12. Schematic diagram of the PPD42NS [24].

In this way, the TP16 signal is directly connect to a 12-bit ADC input and the filtering stage and comparators can be digitally processed by the microcontroller using a first-order type IIR digital filter and comparator code.

To evaluate the PM_{2.5} concentration measurement based on the TP16 signal with digitally processing and compare it with the PPD42NS Sensor Response.

In laboratory, in order to create different levels of PM_{2.5}, an amount of some paper sheets was burned to generate the particulate. This laboratory experiment began with a certain amount of paper sheets burned at a determined distance from the PPD42NS sensor. After this, the same experiment is conducted but with the double amount of paper sheets. This last step was carried out more three times. Table 4 describes the

amount of paper sheets in the four stages of PM generation. Between the end of a stage to the following one, 10 minutes was waited to clean the air.

Fig. 13 shows the TP16 signal with digitally processing (blue line) and the PPD42NS Sensor Response (red line). As can be observed, the signals are very similar and, as a result, to eliminate the PPD42NS signal conditioning circuit is possible option to reduce power consumption or even its cost.

The issue after proving that it is possible to eliminate PPD42NS signal conditioning circuit and still be possible to accomplish PM_{2.5} measurement is to evaluate the power consumption reduction. For this, the PPD42NS sensor was simulated using the software Proteus, as seen in Fig. 14. The simulation result showed that the current of the PPD42NS sensor is

90 mA and it without the signal conditioning circuit is 84.8 mA, result in power reduction of 5.8 %. It can be concluded that the most power consuming part is the heating resistor and any solution of power reduction needs to consider it.

Table 4. Number of burnt paper sheets *per stage*.

Stage	Number of Papers
1.	3 paper sheets
2.	6 paper sheets
3.	12 paper sheets
4.	24 paper sheets

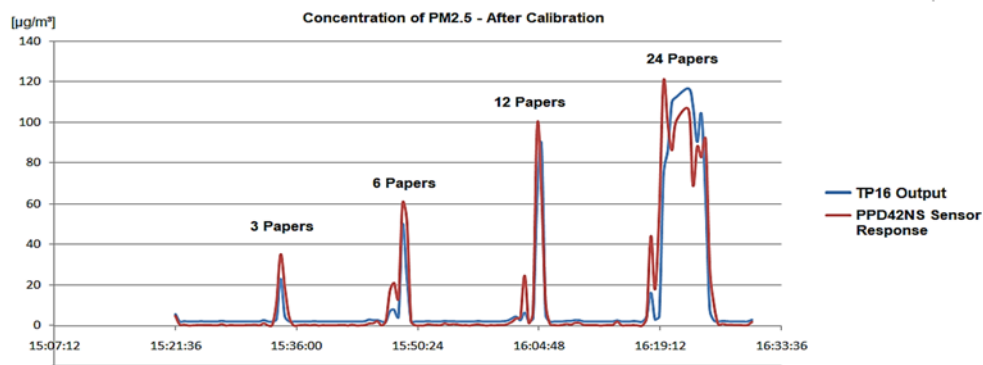


Fig. 13. Concentration curve of PM_{2.5} obtained by the TP16 signal with digitally processing (blue line) and the PPD42NS Sensor Response (red line).

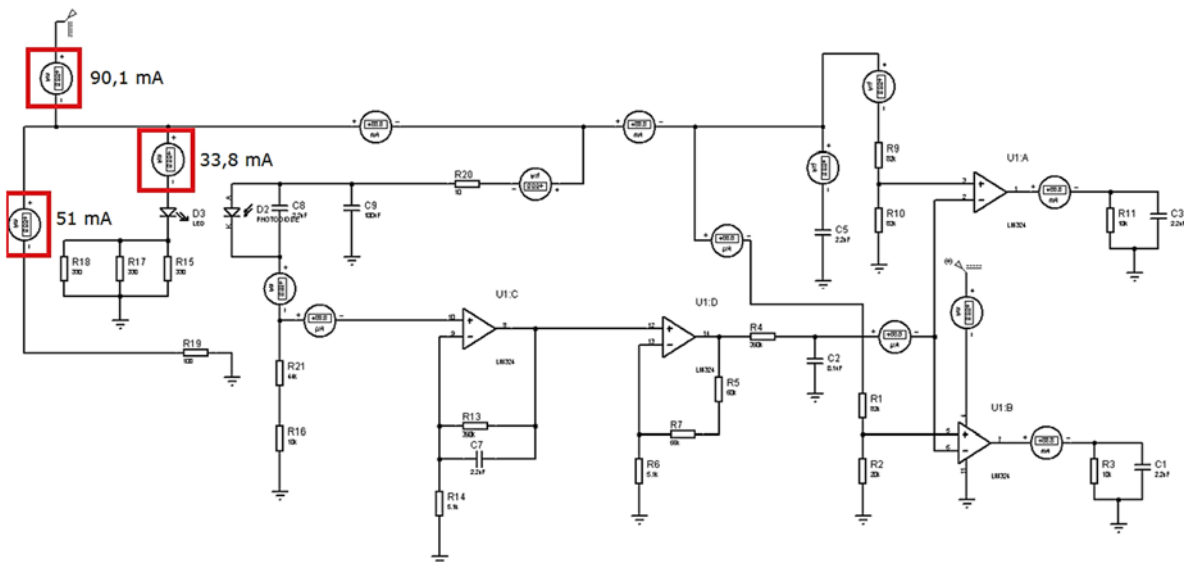


Fig. 14. Schematic diagram of the PPD42NS in Proteus.

7. Conclusions

A portable and wireless intelligent sensor was introduced, which is composed by the environmental sensor BME680 informing data of temperature, humidity, among others, and the PPD42NS sensor,

providing concentration data of PM_{2.5} from light scattering method. In order to analyze the data quality of the PPD42NS, the PPD42NS sensor was calibrated using the reference equipment Ecotech HiVol 3000. The low cost, the linear correlation above 0.9 observed in the calibration process, and the dispensability of

daily filter changes, as need when HiVol 3000 is used, suggests that the proposed PPD42NS-based intelligent sensor can assist in sampling PM_{2.5} for monitoring the quality of the air. In addition, aiming to reduce the current consumption, the electronic part of the PPD42NS was replaced by digital processing, but the observed power reduction was not significant.

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Pressure Transducer Optimization by Multiphysics Simulation

¹ Ricardo PEREIRA and ^{2,*} Carlos CIMA

¹ UNISINOS, Avenida Unisinos, 950 – São Leopoldo, 93022-750, Brazil

² UNISINOS, Avenida Unisinos, 950 – São Leopoldo, 93022-750, Brazil

² Tel.: +55.51.3590-8483, fax: +55.51.3590-8305

E-mail: carloscima@unisinos.br

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Abstract: Pressure sensors are used in a wide range of applications with a significant market share in the industrial sector. The piezoresistive pressure sensor is quite popular due to its low production cost. It is normally made up of sensing elements attached to a deformation membrane. The analysis of the mechanical deformation in the elastic element of the sensors plays a critical role in its design. In this paper, the design of a pressure sensor with semiconductor piezoresistive elements was tested and validated via simulation with the COMSOL software. The emphasis was put on the evaluation of the membrane thermal expansion effect on the overall pressure sensor operation. This observation allowed the conduction of a study to minimize the observed thermal effect. Through the simulation it was also possible to evaluate different arrangements and positioning of the semiconductor sensors in order to obtain the best sensitivity and less non-linearity. The best adhesive for the semiconductor resistors attachment was determined through experimental sensitivity and creeping tests. The complete pressure sensor was then prototyped and fully characterized. The final experimental results confirmed the simulated data and the proposed method for reducing the sensor temperature sensitivity.

Keywords: Pressure sensor, Piezoresistor, Displacement analysis.

1. Introduction

In industrial applications, pressure is one of the most popular variables measured in process control systems. Its widespread usage is mainly due to the relative ease of measurement and versatility. It is also possible to infer other process variables, such as level, volume, flow, and density from the pressure values. Considering their wide range of applications, pressure sensors are routinely submitted to the most diverse working conditions and environments while keeping in compliance with the industry performance and reliability requirements.

The piezoresistance effect in silicon was discovered by C. S. Smith in 1954 [1]. The first piezoresistive semiconductor sensors used in strain

measurement were reported in the early 1970s in applications for the automotive industry. Pressure sensors based on silicon piezoresistive devices have been the subject of intense research and used in commercial applications ever since [2]. Its main advantages are the high sensitivity and the high resistance value, as well as the small size.

Currently, pressure sensors based on the phenomenon of piezoresistivity in silicon are widely used not only due to their good performance figures, but also for its low production cost. However, the process of encapsulating these pressure sensors is still delicate [3].

The pressure sensor presented in this work was designed to operate in the industrial environment and its principle of transduction is the piezoresistivity

effect in the semiconductor material. The sensor design seeks to create and optimize a low-cost solution. The device uses silicon sensors, but the semiconductor substrate is not directly exposed to the material on which the pressure is exerted. Instead, the sensors are fixed on a stainless steel membrane, which guarantees contact compatibility with a wide range of materials.

Usually, the sensor output signal needs correction and compensation. Some non-linearities and/or influences of other physical quantities, such as temperature, must be corrected for achieving its best performance. Hence, the characterization of the sensor is essential to describe its behavior and evaluate the main factors affecting its operation.

2. Piezoresistive Pressure Sensors

Most piezoresistive pressure sensors nowadays are intended for the automotive and healthcare sectors, however, the industrial area also has a relevant market share [4].

The piezoresistive effect is a phenomenon observed in metals and semiconductors, which relates the variation in the electrical resistance of the material with the applied mechanical strain. A useful variable used to quantify the piezoresistive effect is the Gauge Factor (GF), defined as the ratio of the relative change in resistance (R) to the relative change in material length (l). The GF value in semiconductor materials can be up to two orders of magnitude higher than that of metals. This high sensitivity allows the use of a lower excitation voltage. It also permits the mechanical structure deformation to remain in a region further away from the point of plastic deformation [5].

In many applications, the piezoresistive pressure sensor consists of two basic elements: a deformation membrane and the piezoresistive deformation sensor [6]. There are many techniques to carry out the sensor construction and manufacture. The most common ones involve fixing the piezoresistors on a membrane which undergoes a tension (stress) proportional to the pressure applied to it. The piezoresistive deformation sensor will change its electrical resistance as a result of the mechanical deformation generated by the pressure.

The piezoresistive deformation sensors must be positioned on the membrane surface taking into account its deformation condition. In addition, the piezoresistors are commonly connected in an electrical configuration based on the Wheatstone bridge. The Wheatstone bridge can be fed either by a current source or by a voltage power supply. There are three basic configurations for the construction of the Wheatstone bridge: quarter bridge, half bridge, and full bridge. Among these possibilities, the full bridge is usually the most widely used, because it provides good pressure response and low sensitivity to temperature variations. Fig. 1 shows the configuration of a full Wheatstone bridge.

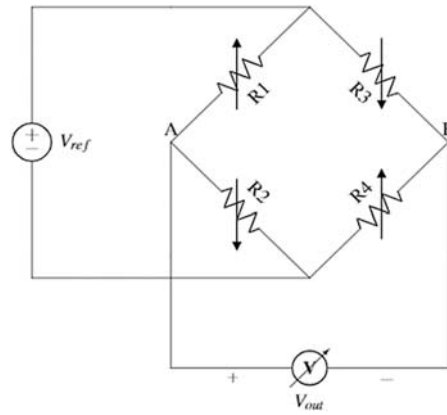


Fig. 1. Full Wheatstone bridge.

3. Pressure Sensor Design and Fabrication

The piezoresistive deformation sensors were manufactured using 6-inch Czochralski-grown silicon (Si) wafer with a thickness of 625 μm and $\langle 100 \rangle$ crystalline orientation. Originally, the wafer had a thin native layer of silicon dioxide (SiO_2) on the surface, which was removed during a cleaning step before any processing. The wafer was subsequently oxidized in a diffusion furnace, resulting in a thick layer of high quality silicon dioxide (~ 850 nm). A polycrystalline silicon (poly-Si) film was then deposited by LPCVD (Low Pressure Chemical Vapor Deposition) on the wafer using phosphorus (P) as the doping element. The resulting poly-Si layer has a uniform n -type doping concentration of $1.05 \times 10^{20} \text{ cm}^{-3}$ and a thickness of 450 nm. This layer is the piezoresistive element in the semiconductor structure. After the LPCVD step, the wafer was submitted to a photolithography process, in which the shape of the sensing element is defined by an appropriate mask and the subsequent etching. The aluminum (Al) pads for the poly-Si resistor were deposited by PVD (Physical Vapor Deposition) and defined by another photolithography step and chemical etching. After annealing, the entire wafer was grinded to a desired thickness (70 μm). The final step is the wafer dicing, producing the individual resistors. The strain device has a length of 1 mm and a width of 0.6 mm, as shown in Fig. 2.

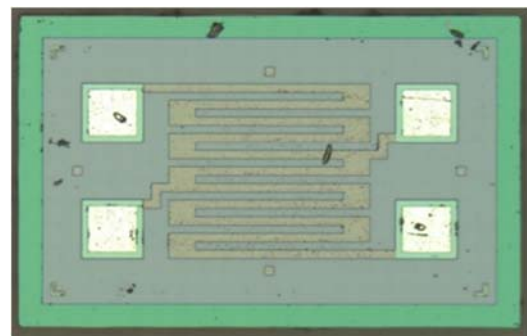


Fig. 2. Piezoresistive strain sensor.

At room temperature, the piezoresistor average resistance is 3.7 k Ω (without being submitted to any deformation) which corresponds to an average sheet resistance (R_s) of 17 $\Omega/\square$. According to the working principle of the piezoresistance, the change in resistance, caused by an induced change in the resistivity ($\Delta\rho$), is related to the stress (σ) by the constitutive relationship presented in Eq. (1), where Π is the piezoresistance tensor (SI units: Pa⁻¹· Ω ·m), a material property.

$$\Delta\rho = \Pi \sigma \quad (1)$$

The piezoresistive tensor coefficients depend on carrier concentration and operating temperature [7]. In general, the relationship between them is inverse, i.e., the higher the temperature and/or the carrier concentration, the lower is the value of the tensor coefficients, thus decreasing the sensor sensitivity.

The other essential component for the construction of the pressure sensor is the deformation membrane. Several different deformation membrane shapes can be used. The most common shapes are the circular, the rectangular and the square ones. The circular membrane shows the highest deformation under the same applied pressure condition. Additionally, this deformation happens with the lowest level of stress, when compared to the other shapes [8]. However, the square membrane has the highest sensitivity and the best linearity. Considering membranes with the same area, circular and square shapes produce the best performance [9]. Although the square membrane is the one with the highest sensitivity, a circular membrane was chosen to develop the present work, because this shape has a simple construction process with metallic materials. This is aligned with the purpose of developing a low-cost sensor.

The membrane thickness and radius settings have been defined based on the sensor working pressure range. These dimensions were determined in order to obtain a pressure sensor with a working pressure of up to 1 MPa. The working pressure of the sensor is extremely important to design the mechanical system of the pressure sensor. A displacement and stress analysis must be done to check the behavior of the mechanical deformation. The presence of plastic deformation would compromise the proper work of the sensor, since the piezoresistor element would remain submitted to a deformation that is no longer related to the applied pressure. Therefore, the maximum operating pressure to which they could be subject must not exceed the flow limit of the membrane material. In order to determine the maximum pressure the membrane can withstand without exceeding its flow limit, a few factors must be taken into account, such as the shape of the membrane, its material properties, thickness, and surface roughness. In the case of the circular membrane, Eq. (2) defines the maximum pressure (P_b) that can be applied to it, where F_{Max} is the flow limit of the membrane material, A is the area of the diaphragm, h is the thickness of the diaphragm

and ν is the Poisson's ratio of the material (supposing the material membrane is ductile) [10].

$$P_b = \frac{3.4F_{Max}h^2}{A(1-\nu^2)} \quad (2)$$

Defining the membrane material as 316 stainless steel, the diameter as 8 mm, and the maximum pressure as 1 MPa, Eq. (2) was used to determine the diaphragm thickness as 220 μ m. Fig. 3 shows a deformation membrane with the four piezoresistors placed in initial positions, since their precise location has not yet been defined (the outer diameter is 18 mm).

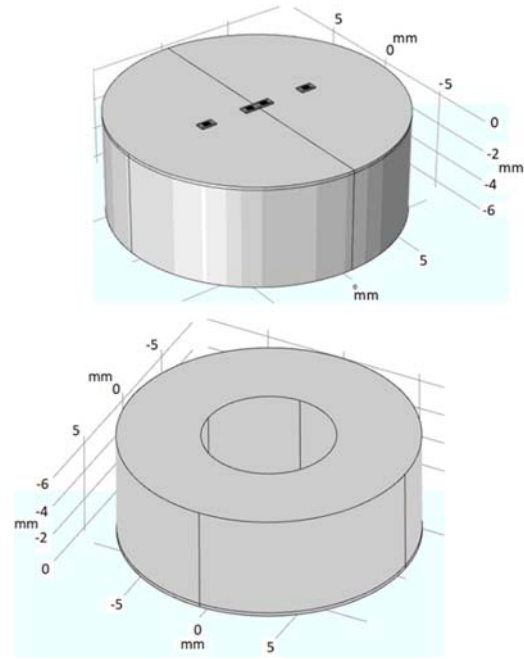


Fig. 3. Deformation membrane (upside and downside views).

The silicon devices must be attached to the membrane with an adhesive that can transmit the deformation of the membrane to the piezoresistor sensors in a reliable way. Therefore, different types of adhesives must be tested to check which one has the best performance. In this context, four adhesives were chosen to be submitted to a test: two epoxy, one cyanoacrylate and one phenolic-based material. For each type of adhesive, 6 piezoresistors were fixed on a 200 μ m thick stainless steel cantilever beam. The electrical contact to the sensor terminals was provided by 17 mm-thick gold wires soldered using a wire bonding tool. This structure allowed the measurement of the resistance behavior for each piezoresistor under different deformation conditions. Fig. 4 shows one of the beams with the piezoresistor sensors bonded on its surface.

One of the ends of the beam was fixed, whereas the other end received the application of a mechanical force causing a deformation in the metal sheet. This mechanical strain was created by calibrated masses

suspended at the beams' edge. Fig. 5 illustrates the experimental arrangement used in the deformation analysis.

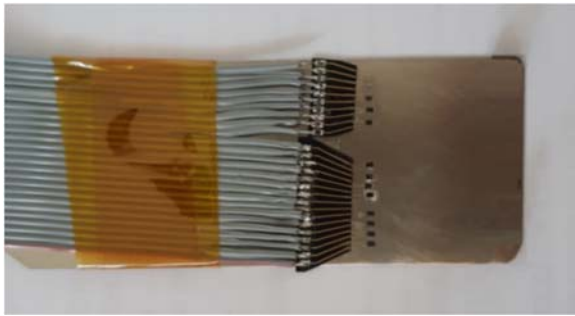


Fig. 4. Piezoresistor elements attached to a cantilever beam using different adhesives.

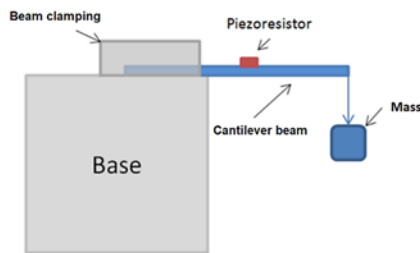


Fig. 5. Experimental arrangement used for the adhesives' testing.

The resistance measurement was done using the Keysight 34401A digital multimeter. For each weight value applied to the beam, a series of 6 measurements was performed. The experiment was carried out applying 7 different weight values: 0, 20, 50, 70, 100, 120, 150 g. Hence, for each adhesive under test, a total of 252 measurements were taken. In the graph shown in Fig. 6, one can view the resistance behavior with the different tested adhesives. Each point plotted in the graph of the figure denotes the average of all measurements taken in the 6 piezoresistors used for the respective combination of adhesive and weight. The phenolic and the cyanoacrylate-based adhesives presented a very similar sensitivity performance (-0.236 and $-0.259 \Omega/\text{g}$, respectively), however this is not enough to warrant the choice of the best material for sensor construction.

Another adhesive test was then executed to further evaluate the adhesion performance of the piezoresistor elements on the deformation membrane using the same experimental setup. This additional experiment is called the creeping test, a procedure based on the observation of the piezoresistor resistance variation over a long period of time while keeping the cantilever beam under a constant deformation condition. The numerical value of the creeping parameter is calculated taking the total resistance variation value ($\Delta R = R_{\text{initial}} - R_{\text{final}}$) and dividing it by the initial resistance value ($\text{Creeping} = \Delta R / R_{\text{initial}}$). This provides a good tool for evaluating the entire adhesive

set performance comprising the adhesive itself, the deformation membrane, and the piezoresistor element. In the creeping experiment, the cantilever beams with the attached piezoresistors were subject to a constant deformation for a full week (168 hours). During this time, the temperature of the samples was kept constant and resistance measurements were periodically taken. For each tested adhesive, a total of six piezoresistors were again attached to the membrane, thus minimizing the effects of possible construction variations on the final results. The graph in Fig. 7 shows the average resistance of the piezoresistors throughout the creeping test for different adhesives. The best calculated creeping values were shown by the phenolic and the cyanoacrylate-based adhesives (72 and 130 ppm, respectively). In view of these results, the phenolic adhesive was chosen to build the final sensor prototypes.

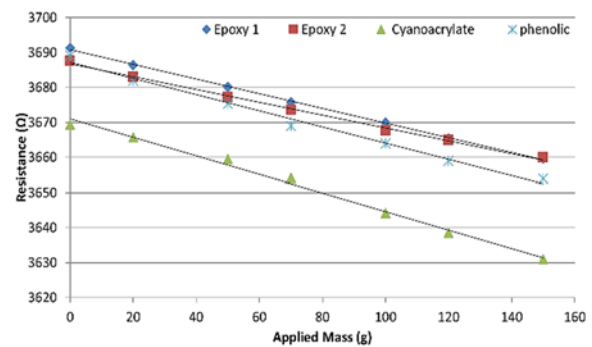


Fig. 6. Experimental resistance variation using different adhesives.

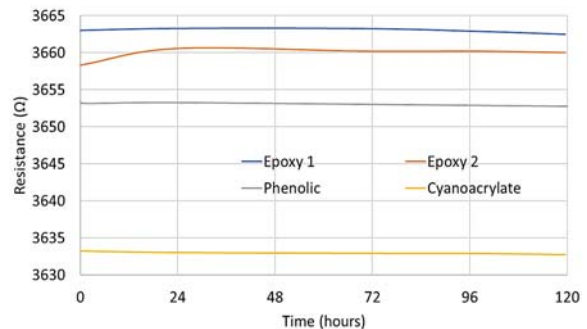


Fig. 7. Piezoresistors resistance during the creeping test.

Before building the transducer, the positioning of the semiconductor sensors on the membrane must be defined through a deformation study of the metallic membrane. The purpose of the analysis is to position the sensor elements at the points where the highest tensile and compression deformation intensities occur. The study was carried out using the physical simulation software COMSOL. For a better performance of the Wheatstone bridge, two of the piezoresistors must be positioned in places where the compression effort occurs and the other two where there is a traction effort. In the circular membrane, the

radial and axial stresses on its surface have the same intensity when equidistant from its central point. The points of the same radius, therefore, measured from the center of the membrane, are submitted to the same strain. During the simulations with the COMSOL software, it was possible to verify the behavior of the stress generated by the deformation along the membrane. This allowed the optimization of the piezoresistor positioning on its surface. The graph in Fig. 8 shows the distribution of the mechanical stress alongside the radius of the deformation membrane for a wide range of pressure values. The regions with the highest stress values are indicated in the figure (center and edges).

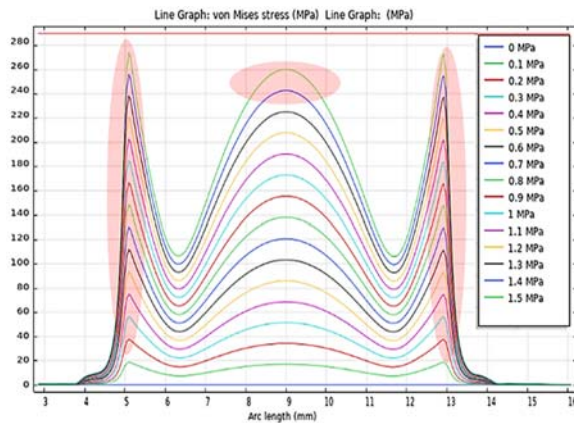


Fig. 8. Distribution of the mechanical stress along the radius of the deformation membrane (simulation).

In addition to the sensitivity, the positioning of the semiconductor sensors on the membrane can also affect its linearity. Another study was then carried out to evaluate the response of the pressure sensor as a function of the different positions where the strain sensors are fixed on the membrane. Distinct arrangements and positions of the strain sensors were simulated, and, in turn, their responses were evaluated. During the simulation, the pressure was varied from 0 MPa to 1 MPa. Fig. 9 shows the proposed positioning arrangements.

For each arrangement shown in Fig. 9, a simulation was done in which the sensor's output signal was checked for different pressure values. During the simulation, the Wheatstone bridge was excited with a current of 2 mA and the measurement of the differential voltage occurred between the intermediate terminals of the Wheatstone bridge. The graph in Fig. 10 shows the output voltage for each position arrangement as a function of the applied pressure.

As is clearly shown in Fig. 10 the arrangement that produces the best sensitivity to the applied pressure is the one identified as Position 1 in Fig. 9. In addition, this arrangement has the smallest non-linearity in its response, as indicated by the graph shown in Fig. 11. Therefore, this arrangement model was chosen for the construction of the pressure transducer.

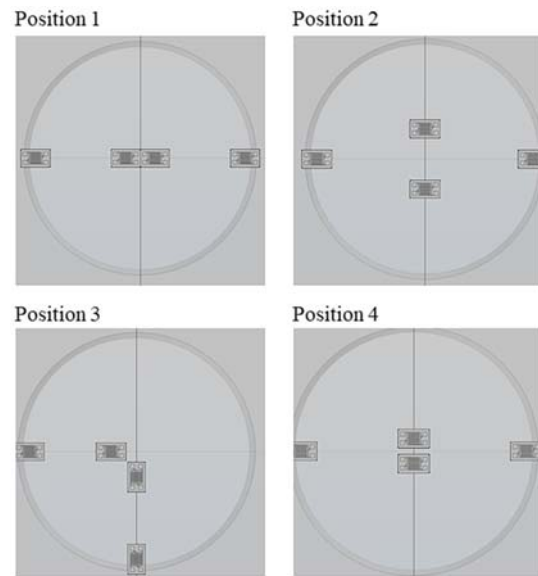


Fig. 9. Proposed arrangements and positions of the strain sensors on the membrane.

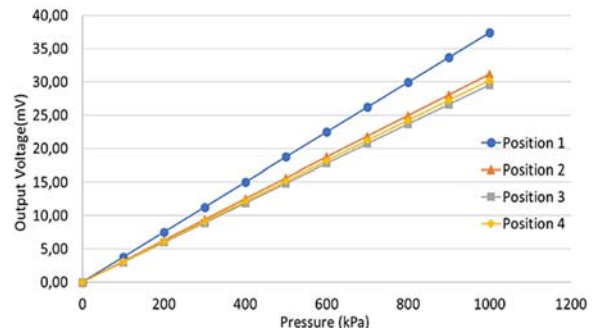


Fig. 10. Sensor output signal for each tested position arrangement (simulation).

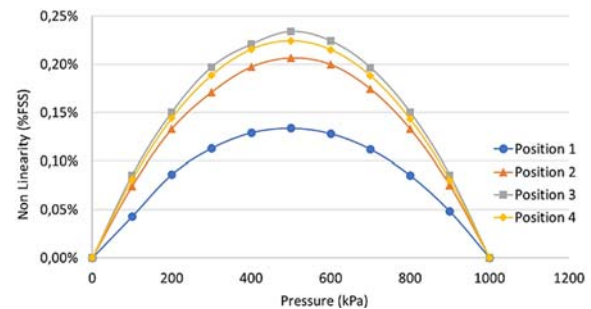


Fig. 11. Non-Linearity of the sensor output signal for each position (simulation).

The simulation of the pressure transducer was also done for different environmental conditions. Keeping the piezoresistors attached to the membrane interconnected in a Wheatstone bridge excited with a constant current of 2 mA, the simulation checked the temperature effect on the overall sensor performance. In this case, the temperature was varied from 0 °C to 100 °C, with the same pressure range of the previous simulations. A combination of simulations was then

carried out under these new conditions in order to validate the sensor's operation in a comprehensive way. Fig. 12 shows the output signal generated by bridge for the simulated temperatures and pressures (using the sensors in Position 1).

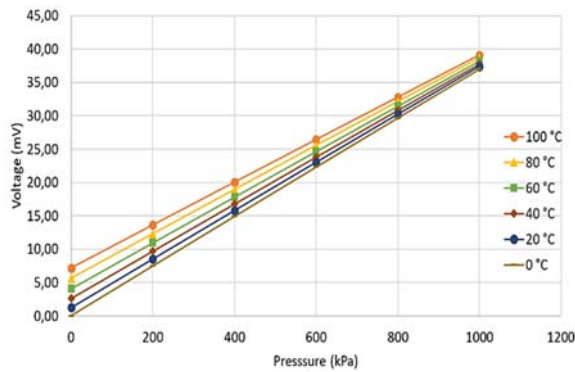


Fig. 12. Simulated output signal from the transducer for different temperatures and relative pressures.

Observing the transducer response curve (Fig. 12), it is evident that the pressure sensor sensitivity is greatly influenced by the temperature. This happens because the temperature affects both the physical properties of the deformation membrane and the semiconductor piezoresistance (which decreases with the temperature variation). It is worth noting that the highest variation in the transducer output voltage (under the same pressure) occurs when there is no pressure applied to the sensor. This represents a significant variation in the sensor offset. In order to determine the cause of this effect, the thermal expansion of the metallic membrane due to the temperature variation was also simulated. The graph in Fig. 13 shows the deformation caused by the membrane thermal expansion alongside the cross section passing through its center under two different temperatures (0 and 100 °C). The value for the partial derivative of the deformation caused by the thermal expansion in relation to the position of the cutting axis under analysis is plotted too.

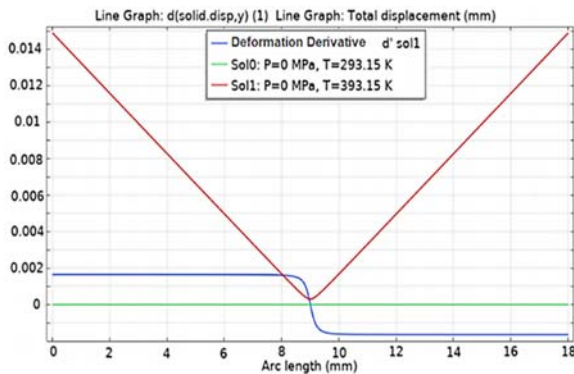


Fig. 13. Deformation caused by the membrane thermal expansion alongside a cross section through its center (simulation).

It is possible to observe that the change rate modulus for thermal expansion is constant in almost all the membrane surface. However, there is a central region in which the thermal expansion does not have a linear behavior. This region coincides with the location where two of the bridge's piezoresistors are positioned. It was expected that these thermal expansion effect would be minimized, since the sensors would be experiencing an expansion of the same intensity, being cancelled by the Wheatstone bridge. The simulation revealed this is not observed, since the central piezoresistors do not experience the same deformation due to the thermal expansion as compared to the piezoresistors at the edge of the membrane, causing a bridge imbalance generated by the temperature variation. In order to minimize this effect, the central region piezoresistors were repositioned so that they remain entirely in the area where the thermal expansion change rate is constant and of the same intensity as in the other piezoresistors. This was accomplished by moving each central piezoresistor 200 μm to the edge of the membrane. Fig. 14 shows the sensor response to different pressures and temperatures after adjusting the position of the central piezoresistors. One notes that there was a significant reduction in the offset of the transducer output signal. In the original arrangement, when the working temperature was changed by 100 °C, the offset voltage changed by 7.2 mV. After changing the position of the piezoresistors, the offset variation was reduced to 0.45 mV.

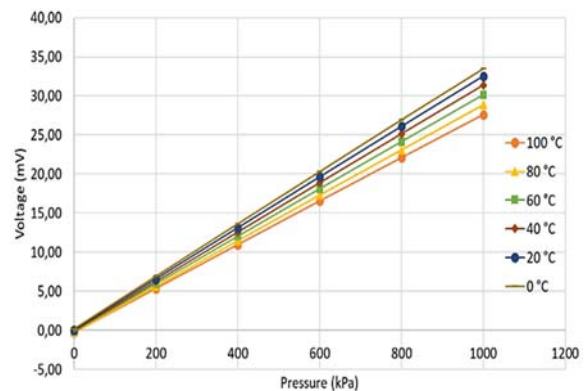


Fig. 14. Transducer output signal for different temperatures and relative pressures after the sensor repositioning (simulation).

After the optimization of the sensor design by simulation, the devices were built and submitted to a set of tests for the characterization. The sensors were subjected to different pressures using a laboratory infrastructure composed of a pressure controller in an environment with controlled climatic conditions. Then the sensors' response was measured under different pressure values. Intermediate pressure values always correspond to a multiple of 20 % of the sensors' full scale. Hence, the pressure was varied from 0 to 1 MPa with an interval of 0.2 MPa between each

measurement. Notice that the pressure values used to describe the experiments and the results refer to a relative pressure (with the atmospheric pressure as zero reference). The measurements were carried out by increasing and subsequently decreasing the pressure values applied to the sensor. In addition to checking the pressure applied to the sensor, the sensors' working temperature was controlled and monitored during the tests. Therefore, the sensors' response tests were performed at several temperatures, ranging from 0 to 100 °C. Fig. 15 shows the diagram of the laboratory infrastructure built to perform the tests.

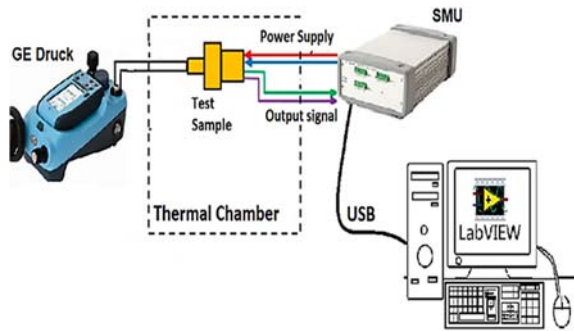


Fig. 15. Laboratory infrastructure diagram.

The measurement data was acquired using an Agilent U2733A SMU (Source Measurement Unit), which makes it possible to automate the experiments using LabView. Therefore, a program was developed using the LabView software, which communicates with the SMU, acquiring the measured data and showing it in the measurement environment. The SMU is responsible for supplying the Wheatstone bridge with the desired current, as well as for measuring the differential voltage of the bridge. The data is acquired for each pressure and temperature condition. A quantity of eight measurements is taken at five-second intervals for each point. Fig. 16 presents the actual laboratory infrastructure used to perform the tests.



Fig. 16. Laboratory infrastructure used to perform the tests.

In order to check the reduction of the offset variation in a practical way, an experimental test was carried out using three samples for each of the two different positions. In this case, no pressure was applied to the sensor, only the working temperature was changed. During the test, the differential voltage of the Wheatstone bridge was monitored, generating a graph that shows the variation of the sensors' offset voltage for different working temperatures. As can be seen in Fig. 17, the experiment proved the result obtained in the simulation environment. The change in the position of the central sensors resulted in the reduction of the offset variation caused by the temperature.

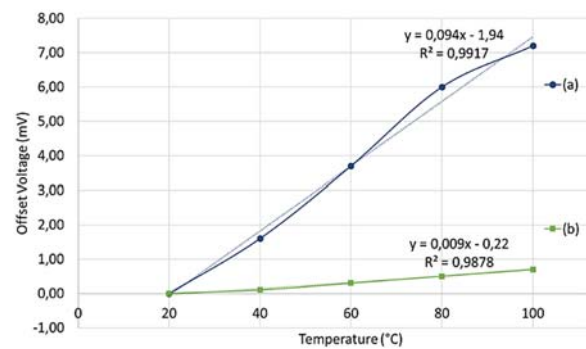


Fig. 17. Experimental variation of the sensors' offset voltage for different working temperatures (no pressure applied): (a) Original position; (b) After repositioning.

After the tests performed to verify how the temperature affects the pressure sensor, experimental measurements with real pressure applied to the sensors were also realized. These tests were performed using pressures ranging from 0 to 1 MPa, and then observing the differential voltage of the Wheatstone bridge. The temperature was varied from 0 to 100 °C for each pressure experiment. Fig. 18 shows the experimental measurements obtained (with the position of the central piezoresistors already corrected). The vertical axis shows the signal of the output voltage with the discount of the offset value at 0 MPa, i. e., only the variation of the output voltage is shown in order to facilitate the visualization of the graphs.

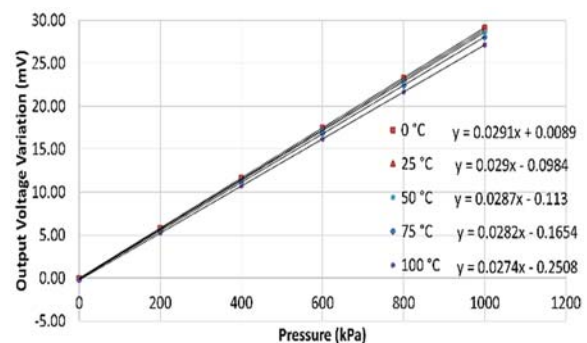


Fig. 18. Experimental output voltage variation under different pressures and temperatures applied to the sensor.

As can be seen in the results obtained during the experimental tests, the sensors show a linear behavior regarding applied pressure. The average variation of the sensors' output voltage, considering the full pressure range, is 29.107 mV. The variation of the offset voltage remained low (0.26 mV) as expected after the sensor repositioning. Conversely, the sensor output signal temperature dependence is still visible, but now it is mostly related to the semiconductor material properties.

When one compares the simulation and the practical results, it is clear that both show the same behavior under the influence of the temperature variation. As the temperature is raised, the sensitivity of the sensor deteriorates, i. e., higher temperatures make the sensor less sensitive to pressure variation. This happens because the temperature reduces the values of the piezoresistance tensor coefficients, resulting in a smaller resistance variation caused by a specific pressure difference.

4. Conclusions

The simulations carried out in the COMSOL software, covering different pressure and temperature conditions, characterized the behavior of a pressure sensor. The analysis of the simulation results was important to validate the sensor operation principle, as well as to evaluate the influence of different physical phenomena on its performance. In particular, the simulation revealed the critical importance of the metal membrane thermal expansion and the sensor positioning for the transducer offset value at zero pressure.

The adhesive used to attach the piezoresistors to the membrane was chosen by an experimental procedure. After the complete simulation of the transducer, prototypes were built in order to check its real behavior. The transducers showed good linearity and low thermal offset variation in the experimental tests, confirming the effectiveness of the piezoresistors repositioning optimization. The interaction between experimental and simulated data proved to be a fast and reliable strategy to optimize the sensor design and fabrication.

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Highly Sensitive Formaldehyde Based on Hierarchical ZnO Nanostructure to Defeat Sick Building Syndrome

¹ Paramita Chowdhury, ^{2,*} Sunipa Roy, ² Nabaneeta Banerjee,
³ Swapan Das and ⁴ Utpal Biswas

¹ Department of Electronics & Communication Engineering, Netaji Subhash Engineering College,
Kolkata-700152, India

² Department of Electronics & Communication Engineering, Guru Nanak Institute of Technology,
Kolkata-700114, India

³ IC Design and Fabrication Center, Department of Electronics and Telecommunication Engineering,
Jadavpur University, Kolkata 700032, India

⁴ Department of Computer Science & Engineering, University of Kalyani, Nadia-741235, India
Tel.: 9830751850

E-mail: sunipa_4@yahoo.co.in

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Abstract: Zinc oxide (ZnO) hierarchical Nanofoam structure on ZnO nanofibers grown SiO₂ coated p-Si substrate were successfully constructed under chemical deposition technique by forming a homogeneous solution of Sodium Zincate (Na₂ZnO₂) in Teflon-lined Autoclave at 120 °C. The seed layer of hierarchical ZnO nanostructure was formed by Sol-gel technique followed by CBD (Chemical bath deposition) technique to grow ZnO nanofibers. The surface morphology and crystallinity of the thin film was characterized by scanning electron microscope (SEM) and X-ray diffraction (XRD) and controlled by adjusting the pH of the reaction solution. The hierarchical ZnO Nanostructure based Formaldehyde (HCHO) Sensor was tested in the resistive mode at dynamic range of 190 ppm to 2020 ppm formaldehyde vapour to defeat sick building syndrome. It was found to offer high response magnitude 94.8 % at 2020 ppm at very low optimum operating temperature 100 °C with fast response time (20 s). Finally, the possible mechanism of decomposition of formaldehyde sensor has been discussed with detailed explanation of higher response magnitude on exposure of formaldehyde vapour with energy band diagram.

Keywords: ZnO hierarchical nanostructure, ZnO nanofibers, Teflon-lined Autoclave, Formaldehyde sensor, Sensing mechanism, Energy band diagram.

1. Introduction

Hierarchical nanostructure has ushered with immense possibilities as there are much scope to explore the opportunities with the use of it [1-3]. By controlling their physical, chemical properties with modifying their shapes and morphology the

effectiveness of this hierarchical nanostructure can be varied in a broad way. There are many reports till date with 3-D nanostructures [2-4]. But lower surface area with limited gas interaction facets, these types of nanostructures cannot compete as a next generation gas sensor. Researchers are looking for some new developments as next generation gas sensors are

already in the market. Synthesis of these hierarchical nanostructures with prescribed dimension is quite tricky as it requires some improved technology to perk up their physical properties which will positively enlarge their area of application. There are several methods to available to fabricate hierarchical nanostructures. Many of them require sophisticated hi-end equipment. Moreover, control of morphology, thickness is getting restricted several times. To circumvent the above mentioned problem, hydrothermal synthesis is one of the best possible methods of producing hierarchical nanostructures owing to its ease of production, lower cost and scalability [2-4]. Moreover the structure offers high purity with narrow particle size distribution. Metal oxide semiconductor based gas sensors are there in the market since last two decades as the band gap can be manipulated judiciously to obtain the optimum output. It is heavily used in optoelectronics, sensors, nanopiezotronics and catalysis [1-8].

Among different metal oxides Zinc oxides, (ZnO) is one of the proven material in gas sensor family. As it has got unique properties like higher band gap, direct band gap, porosity, smaller grazing size etc., the morphology and structuring of ZnO is multidimensional.

A fast detection sensor is required to continuously monitor the ambience around us and alert us about the presence of toxic gases in extremely low concentrations even in the indoor places where common people are unaware about it.

Among different silent diseases, sick building syndrome is one of the silent killer that cannot be detected at an early stage sometime. This disease is caused by harmful volatile organic compounds (VOCs) formaldehyde. Formaldehyde is found frequently in acrylic paint, adhesive resins that bind powdered wood products like particle board etc. Formaldehyde is also used in the textile manufacturing industry. Prolonged exposure of it may cause headaches, fatigue, nausea, and many more. As it is toxic, it may cause leukemia after prolonged exposure even where the concentration is low, *i.e.*, ppb level [3-5].

Not only that this hazardous gas is also formed during ozonation which one of the part of water treatment processes, and water animals are highly affected. As per the standard, formaldehyde levels of greater than 1 ppm grounds cause problem to different organs and above it may affect the human lives precariously.

As per the existing safety standards, a human can withstand maximum exposure of 2 ppm for 8-hour, while inside room levels should not go beyond 0.08 ppm (80 ppb) over 30 minutes. The Chinese Environmental Protection Agency limits exposure to 0.06 ppm over 30 minutes [7, 8]. So, formaldehyde detection is very much needed to save the life of common people across the globe. Functional material synthesis is quite a challenging task that can be eliminated by the use of hierarchical nanostructures. The way the formaldehyde is detected should be competent as very low level detection is required.

Several metal oxide based formaldehyde sensor have been reported by the researchers [1-3, 7, 8] and compared with the present work. Li, Cheng and Wang, *et al.*, have been successfully synthesized low temperature (120 °C) hierarchical Sn/SnO₂ based formaldehyde gas sensor using hetero-junction at 1 ppb concentration [3]. A hierarchical Co₃O₄ nanostructure has been made-up by calcinating Co₅-based metal organic structure by Zhou, Wu and Zhao, *et al.* at 170°C to detect formaldehyde maintaining long term stability [1]. Y-doped SnO₂ hierarchical flower-like nanostructures was made via one-step hydrothermal method for improving the formaldehyde sensing at 180°C has been successfully prepared by Zhu, Ma, Tie, Zhang, *et al.* [2]. Deng, Li and Wang, *et al.* [7] reported a hierarchical flower-like CuO nanostructure based formaldehyde gas sensor with a high response of 1.378 to 50 ppb formaldehyde at 250 °C (11.9 sec response time and 8.4 sec recovery time). Li, Guoans Su, *et al.* [8] have reported Hierarchical morphology-dependent gas-sensing performances of 3D SnO₂ nanostructures to detect formaldehyde with comparison of other VOCs at 200 ppm at optimum temperature of 325 °C.

Table 1. Comparison of different electrochemical exfoliation technique for synthesis of graphene with the present work.

Material	Testing temp.	Testing conc.	Response magnitude*	Response time **	Recovery time **	Range of gas concentration (ppm)	Ref.
ZnO	100 °C	1000 ppm	90 %	29 s	76 s	190 - 2020	Present work
Co ₃ O ₄	170 °C	200 ppm	93 %	8 s	11 s	10 - 500	[1]
SnO ₂	180 °C	50 ppm	94.4 %	16 s	20 s	1 - 100	[2]
Sn/SnO ₂	120 °C	50 ppm	93 %	60 s	75 s	1 - 50	[3]
CuO	250 °C	1 ppm	57.9 %	10 s	17 s	0.05 - 1	[7]
SnO ₂	325 °C	100 ppm	95.6 %	15 s	20 s	5 - 200	[8]

* All sensitivity/response magnitude of all the references were calculated as $S = ((R_a - R_g) / R_a) \times 100$ and compared with the present work.

** Response & recovery time were calculated as time taken to reach 90 % of the saturation value and to reach back to the original value respective.

So, developing a fabrication-friendly method to synthesize ZnO hierarchical nanostructure with distinct, definite morphology is still a major concern. This paper, presents a simple hydrothermal method for the fabrication of foam like ZnO hierarchical nanostructure using twin method of CBD and chemical deposition technique at very low temperature (100°C). The gas sensing capability of this structure was checked by the incorporation of formaldehyde in the test chamber.

2. Experimental

2.1. Materials

All the chemicals were of analytical reagent grade and used as-received, without further purification. Zinc Acetate Dihydrate (Merck, 99.9 %), 2 Propanol (25 ml), Diethanolmine, Hexamethylenetetramine [(CH₂)₆N₄] Deionized Water (Resistivity ~18.2 MΩ-cm).

2.2. Sample Preparation and Gas Measurement Set-up

The schematic of the hierarchical ZnO nanostructured Formaldehyde sensor is shown in Fig. 1. The base (seed layer) of ZnO hierarchical Nanofoam structure on ZnO nanofibers is formed by Sol-gel technique by reacting Zinc Acetate Dihydrate ((Merck, 99.9 %), 3 gm) and 2 Propanol (25 ml) on SiO₂ coated the p type silicon (Si<100>) substrate.

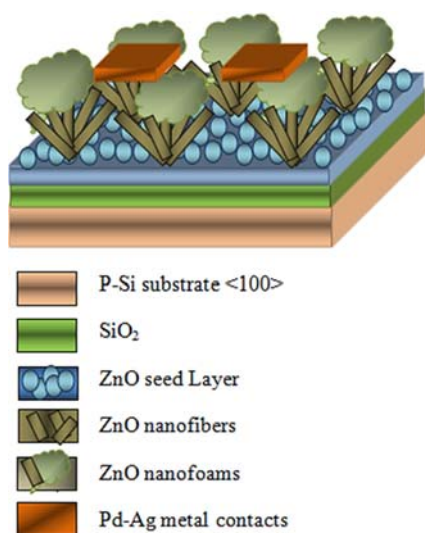
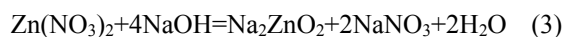
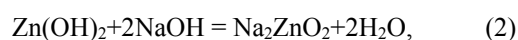
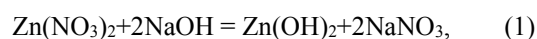


Fig. 1. Schematic diagram of formaldehyde sensor.

3 ml Diethanolmine was also then added drop by drop to get a crystal clear solution followed by 24 hours aging. The samples were spin coated (1000 r.p.m.) for 25 s and put in an annealing furnace at 450 °C for 1 hours and subsequently ZnO seed layer was formed

by thermal decomposition of Zinc Acetate Dihydrates. To prepare ZnO nanofibers, a 50 ml solution was prepared by mixing Zinc Acetate Dihydrate and HMT [Hexamethylenetetramine [(CH₂)₆N₄]] in deionized water (resistivity ~18.2 MΩ-cm) solution in the mass ratio of 1:3. The substrates were then immersed into the solution and after removal kept in a baking oven for 1 hr at 100 °C. Samples were allowed to cool down gradually.

For getting ZnO hierarchical nanostructure, ZnO nanofoam layer was formed by a chemical deposition technique by preparing a homogeneous solution of Sodium Zincate (Na₂ZnO₂) by mixing Zinc Nitrate (Zn(NO₃)₂·6H₂O) and Sodium Hydroxide (NaOH) in the ratio (wt.%) of (1:3) and was magnetically stirred at room temperature for 20 minutes.



After stirring, the mixture was then transferred to a 50 mL Teflon-lined autoclave, sealed, and kept at 120 °C for 12 h and finally cooled to room temperature. Two metal electrodes (Pd-Ag) were then deposited by e-beam vacuum evaporation technique on top of the sensing layer using Al shadow mask. The device dimension was [5 mm × 5 mm] having an electrode area of [2 mm × 2 mm] (each electrode area having 1 mm × 1 mm).

The details of vapor measurement set-up have already been reported earlier [5, 6, 10]. The formaldehyde vapors were bring in the compartment by passing IOLAR grade N₂ (carrier gas) through the formaldehyde kept in a conical bottle (Fig. 2).

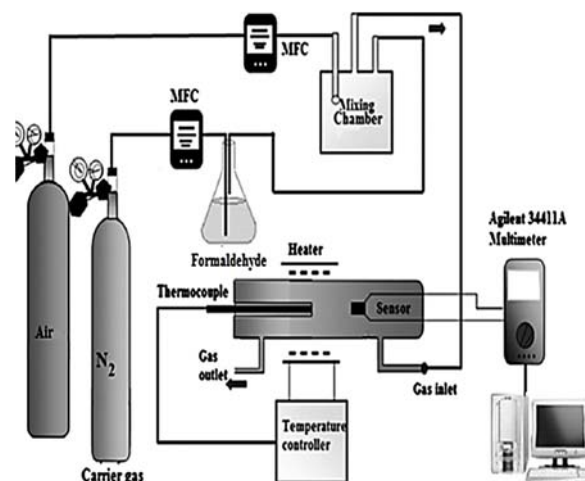


Fig. 2. Gas sensor measurement set-up.

Assuming that carrier got satisfied with the formaldehyde, the desired concentrations of the vapor were obtained in the sensing chamber by controlling the flow rate of N₂ through formaldehyde and adding

IOLAR grade N₂ (diluent) through a separate gas flow line. The mixture that is consistent in nature carrying the sufficient target vapor was fed into the chamber with a bendable PVC pipe. To measure the sensor resistance, electrodes are connected to an Agilent U34411A Multimeter with Agilent GUI Data logging software (v2.0). The final concentration of formaldehyde vapors was calculated using this formula [9, 10].

$$C = \frac{\frac{P \times L}{760 - L}}{\frac{P \times L}{760 - L} + L + L'} \times 10^6 \quad (4)$$

2.3. Characterization of the Sensor

A comprehensive study of shapes and structural characterization of the ZnO hierarchical layer has been reported here. XRD (Fig. 3) (using ULTIMA-III, CuK α target, X-ray Diffractometer, $\lambda=1.54\text{\AA}$) confirmed the strongest (002) preferential c-axis growth (at 34.44°) with $5.12\text{ \AA}$ interplanar spacing. The other diffraction peaks positioned at 2θ values of 31.76° and 36.21° can be indexed to the hexagonal wurtzite phase of ZnO (JCPDS card no: 79-2205) with crystallinity of (100) and (101) planes respectively.

The surface morphology of the hierarchical ZnO was characterized by atomic force microscopy. Fig. 4 show a typical height profile of ZnO nanostructure on SiO₂ substrate. AFM micrograph of nearly $50 \times 50\text{ }\mu\text{m}$ area of Si/SiO₂ substrate supporting hierarchical ZnO nanostructure is presented here. The thickness was calculated from the height difference between the surface of ZnO and that of the substrate and has been recorded as $\sim 300\text{ nm}$.

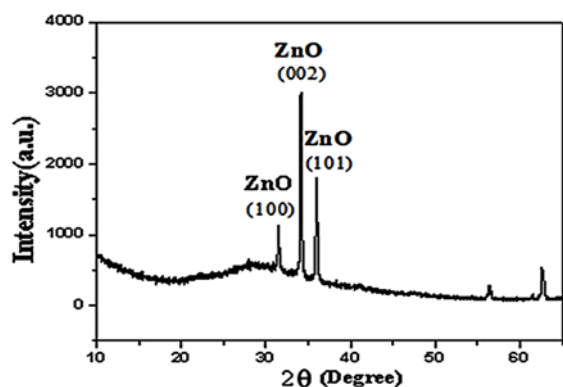


Fig. 3. XRD pattern of ZnO hierarchical formaldehyde sensor.

The average lateral size of these electrochemically hierarchical ZnO nanostructure ranges from 300 to 350 nm.

FESEM (Fig. 5) result depicts the uniformly distributed ZnO nanofoam having average dimension

of $\sim 300\text{ nm}$ with average fiber diameter of $\sim 20\text{ nm}$ and length 400 nm .

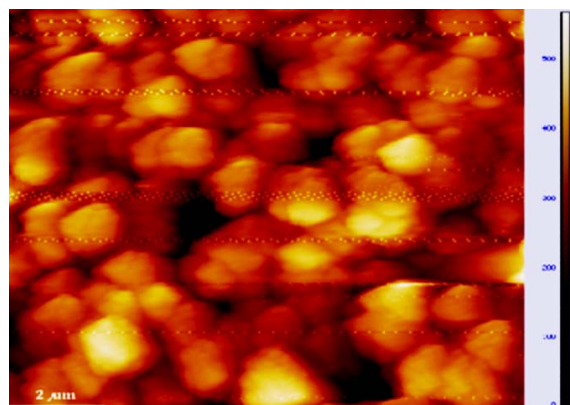


Fig. 4. AFM image of ZnO hierarchical formaldehyde sensor.

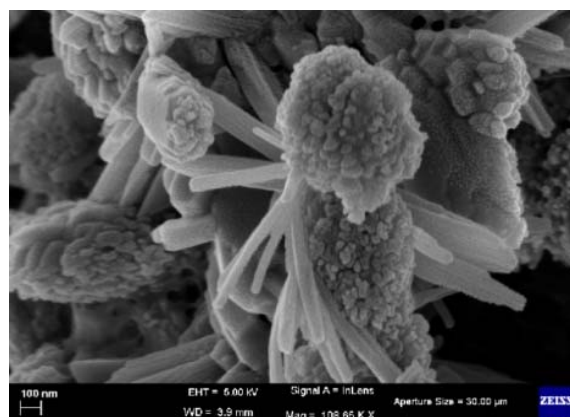


Fig. 5. FESEM image of ZnO hierarchical formaldehyde sensor.

3. Results and Discussions

3.1. Sensor Study

To report the optimum operating temperature of sensing, sensor resistance was calculated in presence of air and formaldehyde vapor and the equivalent response magnitude was calculated as a function of temperature. Response Magnitude (RM) of the sensor was considered with this formula:

$$RM = \frac{R_a - R_g}{R_a} \times 100\%$$

where R_g is the sensor resistance in the test vapor (formaldehyde vapor diluted with air) and R_a is the sensor resistance in air. In this study, response time is defined as the time required for the sensor to reach 90 % of the saturation value and the recovery time is defined as 90 % of the time required to come back to the original value from saturation value [11].

Before testing with gas I-V characteristics of the Pd-Ag contact on aligned ZnO nanofoams were studied at different temperatures. The I-V characteristics (Fig. 6) indicated a good ohmic nature of the Pd-Ag contacts for all three temperatures.

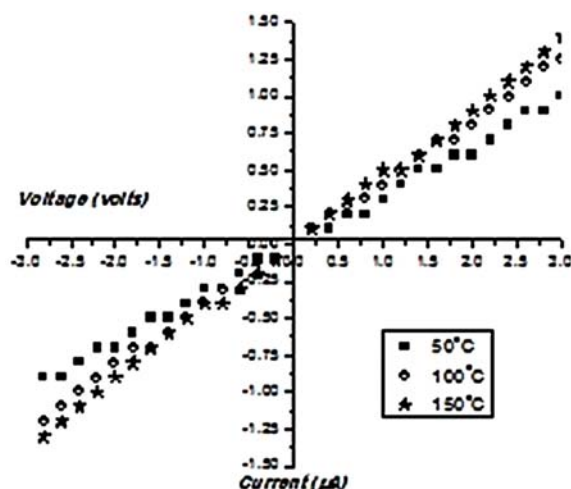


Fig. 6. I-V characteristic of Pd-Ag contact on ZnO sample.

The change in resistance of the resistive sensor was measured in order to get the optimum operating temperature using 1000 ppm formaldehyde vapor (in air) as shown in Fig. 7. Transient response characteristics of ZnO hierarchical Nanofoam structure on ZnO nanofibers resistive sensor at its respective optimum operating temperature (100 °C) for different formaldehyde vapor concentrations (190-2020 ppm) are shown in the Fig. 8.

It can be envisaged from the graph (Fig. 8) that the sensor resistance was initially high due to absence of free electrons on the ZnO sensing surface. After introducing the formaldehyde vapor, the sensor resistance decreased due to release of free electrons; on cutting off the vapor supply, the sensor resistance increased due to recapturing of the adsorption site by oxygen species (from air) causing a reduction in the available surface electron concentrations. At 2020 ppm concentration of the formaldehyde sensor shows high response magnitude (94.8 %) with 20 s response time and 88s recovery time but at lower concentration (190 ppm) the sensor offers 70.4 % response magnitude with high response (45 s) time. As a result response time decreases to the higher concentration but the recovery time increases due to slow desorption rates of the formaldehyde molecules from ZnO surface. At higher concentration, formaldehyde sensor acquires a tendency towards saturation due to occupancy of all the adsorbent sites of the surface. It is also noticed that there are minimal baseline shift due to the fact of some target vapor species remained adsorbed even after the vapor pulse is cut off and the amount of such adsorbed species increased as the vapor concentration increased. The variation of response magnitudes as a function of formaldehyde concentrations (190, 380, 570, 760,

950, 1260, 1530 and 2020 ppm) at particular optimum operating temperature (100 °C) shows in Fig. 9 by the bar chart representation.

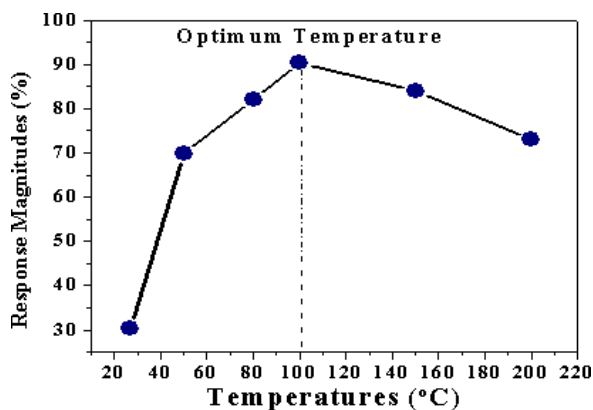


Fig. 7. Response magnitude (function of temperature) for 1000 ppm formaldehyde concentration.

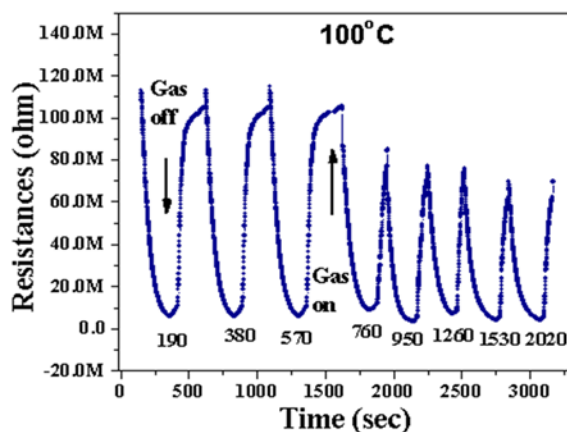


Fig. 8. Transient response characteristics of the Sensor at 100 °C.

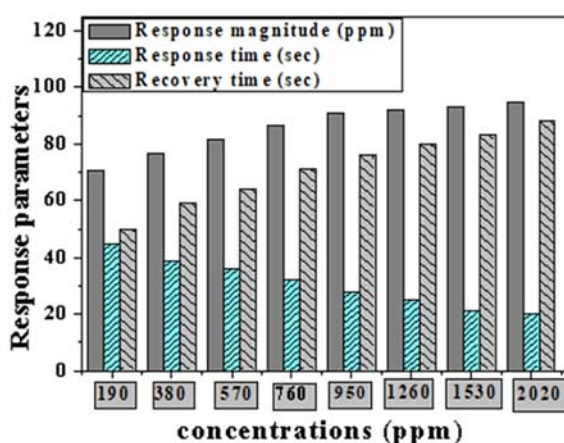


Fig. 9. Comparison charts for response magnitude, response and recovery time of the sensor for different concentrations of gas.

Overall results (different sensing parameters in a concentration range of 190-2020 ppm at 100 °C) for

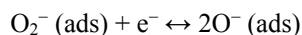
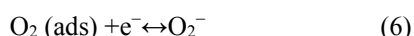
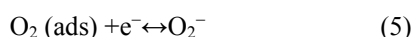
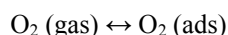
the formaldehyde sensor with Pd-Ag contact is represented in a tabular form below (Table 2).

Table 2. Response parameters in the range of (190-2020) ppm of Formaldehyde sensor.

ZnO hierarchical nanofoam based formaldehyde sensor (100 °C)			
Concentrations (ppm)	Response magnitude (%)	Response time (s)	Recovery time (s)
190	70.4	45	50
380	76.6	39	59
570	81.8	36	64
760	86.4	32	71
950	90.8	28	76
1260	91.9	25	80
1530	93.2	21	83
2020	94.8	20	88

3.2. Formaldehyde Sensing Mechanism

Like other gas/vapor sensing mechanism, for formaldehyde gas sensing also, initial oxygen adsorption plays a pivotal role in the sensing mechanism of Zinc oxide (ZnO) hierarchical Nanofoam sensor. Oxygen ion adsorption process (on ZnO surface) reduces conduction electron concentrations resulting in an increase of ZnO film resistance. Reactive oxygen species such as O_2^- , O_2^{2-} and O^- are adsorbed on the ZnO surface. Therefore during the chemisorption, atmospheric oxygen forms ionic species such as O_2^- and O^- by taking electrons from the conduction band of ZnO surface. The reaction kinetics by chemisorption of oxygen on ZnO surface is shown below [15-17]:



The overall formaldehyde sensing mechanism of Zinc oxide (ZnO) hierarchical nanofoam structure is shown in Fig. 10 (a and b). When the ZnO sensor is exposed to formaldehyde vapor, after thermal decomposition of formaldehyde, the activated fragments of formaldehyde (which are mainly H) spill over onto the ZnO surface, then water vapour (H_2O) and carbon-di-oxide (CO_2) are formed by reacting the products (H_2 and CO) of formaldehyde with chemisorbed oxygen of ZnO surface and release electrons into the conduction band of ZnO resulting in a decrease in the ZnO film resistance. The combined vapours exposure (water vapour and CO_2 vapour) after formaldehyde dissociation offers higher response magnitude of the sensor.

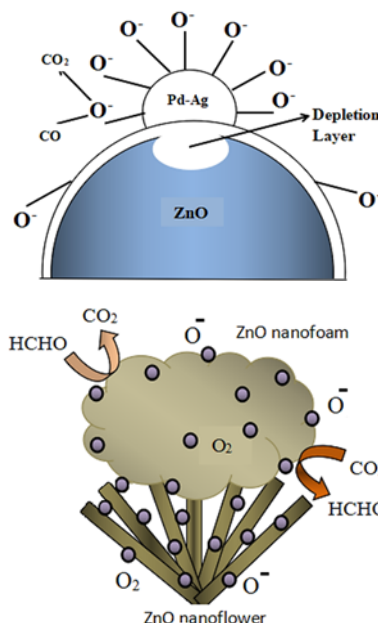


Fig. 10. Formaldehyde sensing mechanism of Zinc oxide (ZnO) hierarchical nanofoam structure.

3.3. Energy Band Diagram

Formaldehyde sensing mechanism of ZnO hierarchical nanostructure can also be reinforced by the corresponding energy band diagram of inter-granular junction and Pd-Ag with ZnO (Metal-Semiconductor junction) in presence and in absence of formaldehyde vapour shown in Fig. 11.

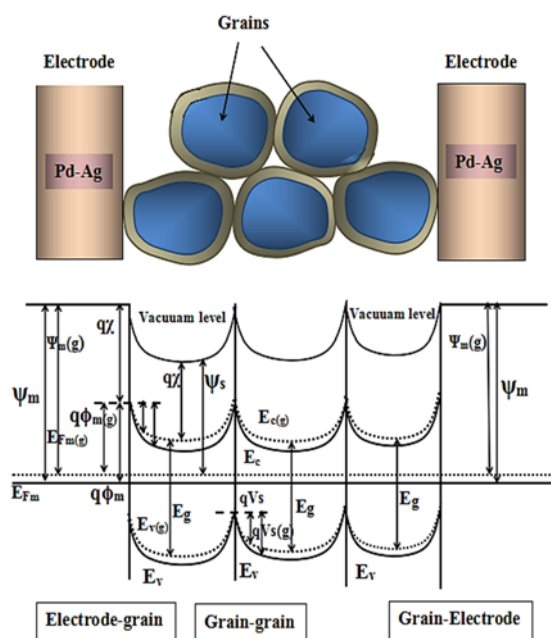


Fig. 11. Geometry of the hierarchical nanostructured ZnO thin film with Pd-Ag electrodes and its corresponding energy band diagram.

In the present case, the work function (ψ_m) of metal [Pd: 5.12] is greater than the work function of ZnO

[ψ_s : 4.45]. Initially, Pd attracts atmospheric oxygen to form weakly bonded Pd:O which eventually increases the work function of Pd. The increase of Pd work function decreases the barrier height of the Pd-ZnO contact and accordingly increasing the resistance of the sensor. But when some formaldehyde vapor is introduced, it (vapor) reacts with adsorbed oxygen (O^-) of ZnO surface and decreases the work function of metal due to reduction of O^- from surface of metals thereby increasing the barrier height. Therefore the total majority carrier (electrons) increases in the conduction band of ZnO (with respect to that in air) which reduces the resistance of formaldehyde sensor.

3.4. Stability Study

Stability study of the sensor was carried out with IOLAR grade N_2 and 1 % HCHO in N_2 for 24 h with a duration of 6 h per day in 4 days, at the optimum temperature ($\sim 100^\circ C$) (Fig. 12). It was observed that the sensor exhibits quite linear response for both N_2 and 1 % HCHO in N_2 with nominal variation of sensor resistance from its reference baseline value (3.35 M Ω in N_2 and 2.2 M Ω for 1 % formaldehyde in N_2).

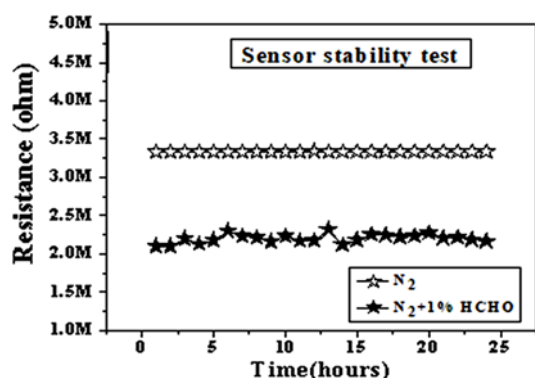


Fig. 12. Stability study of sensor with N_2 and with 1 % HCHO in N_2 at $100^\circ C$.

4. Conclusion

This paper focuses on the formaldehyde sensing characteristics, corresponding sensing mechanism with energy band diagram, electrical characteristics and stability study of ZnO hierarchical nanofoam based resistive sensors. Autoclave played a crucial role for the formation of this hierarchical ZnO nanostructure. After detailed structural characterizations (XRD, AFM etc.), the sensor was employed to detect formaldehyde. Catalytic effects, to improve the sensing performance, were incorporated by Pd-Ag catalytic contact on ZnO hierarchical nanostructure. The sensor was tested in the range of 190 to 2020 ppm by offering much higher response magnitude (94.8 %) at very low operating temperature ($100^\circ C$). The mechanism of decomposition of formaldehyde sensor has also been explained with energy band diagram.

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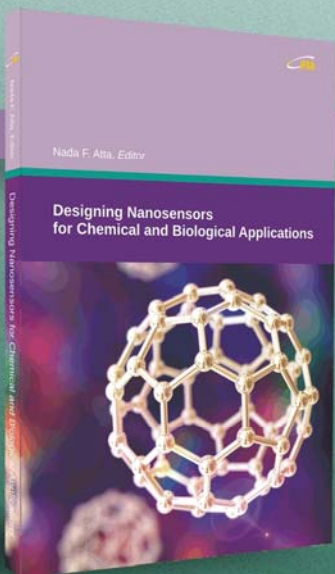


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Nada F. Atta, *Editor*

Designing Nanosensors for Chemical and Biological Applications



The present book aims at providing the readers with some of the most recent development of new and advanced materials and their applications as nanosensors. Examples of such materials are ferrocene and cyclodextrines as mediators, ionic liquid crystals, self-assembled monolayers on macro/nano-structures, perovskite nanomaterials and functionalized carbon materials. The emphasis of the book will be devoted to the difference in properties and its relation to the mechanism of detection and specificity. Miniaturization on the other hand, is of unique importance for sensors applications. The chapters of this book present the usage of robust, small, sensitive and reliable sensors that take advantage of the growing interest in nano-structures. Different chemical species are taken as good example of the determination of different chemical substances industrially, medically and environmentally.

The book will be useful for scientists and researchers, doctors and students working in medical research, engineers and students working in environmental research, professionals working in industrial field.

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Polyvinylidene Fluoride-Praseodymium Oxide Nanocomposite Force Sensors for Monitoring Human Physiological and Muscle Motion Signs

^{1,*} Ashok BATRA, ¹ James SAMPSON, ² Kuzhivelil ARUN and ³ Aschalew KASSU

¹ Materials Science Group, Department of Physics, Chemistry, and Mathematics
Alabama A&M University, Normal (Huntsville), AL 35762, USA

² Department of Physics, Sree Kerala Varma College, Thrissur, Kerala 680011, India

³ Department of Mechanical, Civil Engineering and Construction Management
Alabama A&M University, Normal (Huntsville), AL 35762, USA

¹ Tel.: 2563728109, fax: 2563725622

E-mail: ashok.batra@aamu.edu

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Abstract: In the present investigation, praseodymium oxide (Pr₂O₃) nanoparticles embedded polyvinylidene fluoride (PVDF) thick films were fabricated via solution casting/intercalation technique. The Pr₂O₃ nanoparticles were synthesized by the hydrothermal technique. The Fourier Transform Infrared Spectroscopy (FTIR) has been recorded in the range 400 to 4000 cm⁻¹, and functional groups were identified in the nanocomposite films. Infrared vibrational spectroscopy (FTIR and Raman) curves revealed ferroelectric β-phase in the annealed nanocomposite films intrinsically. The electrode films so produced were sandwiched between Kapton film to form a sensing device. Based on reasonable linear piezo-capacitive behaviors obtained, PVDF-Pr₂O₃ nanocomposites films can be used to create highly flexible pressure sensors.

Keywords: PVDF, Praseodymium oxide, Nanocomposites, Solution casting, Pressure sensors.

1. Introduction

Electroactive polymers (EAPs) are gaining immense interest as smart materials [1]. There are many EAPs, namely, Nylon-11, polylactide and aniline pentamer copolymer, poly(lactic-co-glycolic acid) (PLGA), and poly(vinylidene fluoride) (PVDF) and its copolymers with trifluoroethylene (TrFE) [2-6]. However, PVDF and PDVF-TrFE exhibit the best electroactive properties, such as piezo-, pyro- and ferroelectricity, and optoelectronic. As a result, PVDF and its copolymers are of choice for the increasing number of possible organic microelectronics

applications, such as electro-optic transducers, waveguides, sensors, actuators, energy harvesting, electro-optic memory, biomimetic robotics. PVDF is polar. With respect to the carbon atom in the unit, the hydrogen atoms are positively charged, and the fluorine atoms are negatively charged. The repeat unit of (–CH₂–CF₂–) or CH₂CF₂)_n, in which the carbon-hydrogen bonds with the electrically polar carbon-fluorine, can take up several stable configurations determined by the treatment of the polymer. PVDF can be crystallized into at least four different polymorphous phases, depending on the film preparation conditions. Two forms β (or I) and α

(or II) - are of particular importance. In form α , PVDF is non-piezoelectric, the bonds are in a trans-gauche-trans-gauche configuration, and the molecules are stacked, giving a nonpolar unit cell. The α phase can be converted into form β by stretching and electrically poling with a right field. In form β , the molecular groups are in an all-trans configuration, and the molecules are assembled to give a polar unit cell. Form β is orthorhombic with point group Amm^2 . Several research investigations have highlighted the benefits of using nanoparticles, including carbon, dispersed in the polymer matrix to design and fabricate multifunctional materials with higher strength, including with above-cited properties in hybrid sensors or devices for medical and industrial applications. Tactile sensors, most commonly are referred to as strain, and pressure sensors, can obtain mechanical property data of the human body and local environment. It is recognized where contact, pressure, or traction is exerted on the skin and some internal organs. The tactile sense is one of the five traditional senses of the body. The tactile sense is received through millions of nerve ending on the skin, detecting even the slightest touch sensations and reporting it to the brain [1].

For brevity, we define four crucial attributes of smart materials [2-3]. *Piezoresistivity*: Tactile sensors based on piezoresistivity mechanisms change electrical resistance when the mechanical stimulus is applied. Piezoresistivity is the widely utilized technology to implement a tactile sensor due to its simple structure and cost-effective fabrication techniques. Based on the simple principle of operation of the sensor, few prototypes have been fully designed and developed. *Piezoelectricity*: It is the development of voltage, which is caused by electric dipole moments observed in some anisotropic crystalline materials, in response to mechanical stimuli and vice versa; and *Pyroelectricity*: It refers to electric polarization of certain smart materials to generate a transient voltage in response to applied temperature change. Thus, by converting thermal energy to electricity, the pyroelectric effect can be applied to build a self-powered temperature sensor that automatically detects temperature change without the need of an extra power supply or battery. *Piezocapacitive*: Pressure sensors based on piezo-capacitance mechanisms change electrical capacitance when the mechanical stimulus is applied.

A great deal of research has been devoted to improving the outputs and piezoelectric properties of crystals and ceramic materials. In recent years, the ease of production and processing of PVDF films compared with piezoelectric crystals and ceramics, the relatively lower cost of polymer materials, the flexibility, and the sensitivity of PVDF nanocomposite polymer films are becoming an active area of research for developing sensors and functional devices. The piezo- and pyroelectric properties of PVDF polymer makes it an attractive polymer for various applications including energy harvesting [7-9], nanogenerators [7, 10], tactile sensors [11-14], humidity sensors [14],

force sensors [15], capacitors [14], strain sensing [12, 16, 17], for developing wearable functional electromechanical devices [16, 18-22], for tissue engineering including regeneration and repair [10, 23].

In the biomedical domain, tactile piezoelectric sensors detect movements, including human body motion, and convert the deformations into electrical signals [13, 14]. Yu, *et al.* (2019) demonstrated the application of PVDF nanocomposite polymer films as nanogenerators producing usable energy from mechanical energy generated during running and walking [8]. Based on the study, the fabricated PVDF-nanofiber-based 'shoepad' nanogenerator produced about 6.45 μ W output power with a 5.5 M Ω load resistance. The application of PVDF-based nanocomposite used for harvesting usable energy from regular human body motion and human biological activities, including respiration, for developing small size biocompatible nanogenerators to power wearable and implantable biomedical devices have also been demonstrated [9]. To enhance piezo-responses of PVDF-based nanocomposite, several design approaches, fabrication methods, and nanocomposite materials have been explored [16, 19].

PVDF has all the desired features to fabricate flexible pressure sensors such as the ultrahigh stretchability, bending, wearability, which can be easily integrated into artificial and electronic skins. Thus, it was thought exciting and worthy of exploring PVDF and its composite films embedded with Pr_2O_3 nanoparticles, in particular their suitability in pressure sensors for biomedical sensing. With this train of thought, we investigated piezo-capacitance relevant sensing characteristics under both Transverse and shear stress.

2. Experimental

2.1. Fabrication of the Nanoparticle and Films

Praseodymium oxide nanorods were prepared by the hydrothermal synthesis method. In this study, 0.1 molar solution of Praseodymium chloride is mixed with 0.1 molar solution of Sodium hydroxide. The water used in this reaction was doubly distilled and deionized. By this method pH value of the solution was adjusted to 9. After stirring the colloidal reaction solution, it was transferred into an autoclave made up of Teflon lined stainless steel. Then the solution is subjected to heat at 300°C for 6 hours in an oven and subsequently cooled down to room temperature. The settled particles were collected and washed repeatedly with deionized water under vacuum. Praseodymium hydroxide nanoparticles were heated at 80°C. Hydroxide nanorods were turned into oxides after heating to a temperature of 300°C. Annealing of the sample to a temperature of 700°C was also conducted. It was found that annealing results in a change in color, which is an indication of the change in particle.

500 mg of PVDF was dissolved in 20 ml of DMF under vigorous stirring at 60°C for the complete dissolution of PVDF in DMF. Then, a certain weight percentage of the Praseodymium oxide nanoparticles were added to the PVDF solution and vigorously stirred for one hour to obtain a homogeneous mixture. The nanocomposite films were prepared by casting the mixture in a properly cleaned and dried petri dish, and the solvent was evaporated at 90°C in the oven. 1 wt%, with 2 wt%, 3 wt%, 4 wt% and 5wt% Praseodymium oxide nanoparticles loaded PVDF films were prepared. They were coded as PrO50; PrO100; PrO150; PrO200 and PrO250 respectively, including pristine PrO0.

2.2. Optical Characterization

Raman spectral analysis in the range from 250-2250 cm^{-1} was carried out by using a Raman spectroscopy system (Enwave Optronics, Inc.) operating at 785 nm laser wavelength. The Fourier Transform Infrared Spectroscopy (FTIR) has been recorded in the range 400 to 4000 cm^{-1} . The surrounding medium was air, and all the measurements were performed at room temperature.

2.3. Electrical Characterization

LabVIEW interfaced Quadtech LCR Bridge network can be used to conduct the dielectric study, and FTIR study provides the normal vibrational modes of PVDF and nanocomposites. The real and imaginary parts of the dielectric permittivity (ϵ' and ϵ'') can be obtained by measuring terminal parallel capacitance C_p and loss tangent at a fixed measurement frequency of 1 kHz using LCR meter, and measurements were taken during heating run with temperature controller. A thermocouple installed near the sample, and the temperature can be measured with the help of a digital multi-meter. The details of the setup are described elsewhere [5]. The relevant parameters required or infrared sensing and energy harvesting, which include the dielectric constant (ϵ') and pyroelectric coefficient (p) were calculated. A real part (ϵ'), imaginary part (ϵ'') of the dielectric constant were determined as:

$$\epsilon' = \frac{C_p d}{\epsilon_0 A}, \quad (1)$$

$$\epsilon'' = \tan \delta \cdot \epsilon', \quad (2)$$

where C_p is the parallel capacitance of the sample at a signal frequency of 1 kHz, $\tan \delta$ is the dielectric loss, A is the electrode area of the silver electrode, d is the thickness of the sample, $\epsilon_0 = 8.854 \times 10^{-12}$ F/m is the permittivity of vacuum.

Piezocapacitance response is calculated as $\Delta C / C_0$; where ΔC is the change in capacitance on applied force.

2.4. Force Measurement Custom-Made Apparatus

Custom-made setups shown in Fig. 1 were designed and fabricated for testing these nanocomposite pressure/strain film sensors for use in human motions and robotics. Set I consist of a Starrett Press and Force indicator. With set up I, one can apply force on the device under test (DUT) placed between the two holders and note down corresponding capacitance. We apply a transverse force from 0 to 100 N. Thus, DUT can be used for measuring transverse pressure. Set II consists of a custom-made feature to apply sheer force on a curved device under test. This type of sensor can be used to mimic finger motion. The device is held with the help of two jaws, and sheer pressure is applied with the help of a cylinder placed under the device. By moving the cylinder upward, pressure can be applied on the device under test, and accordingly, the capacitance can be measured.

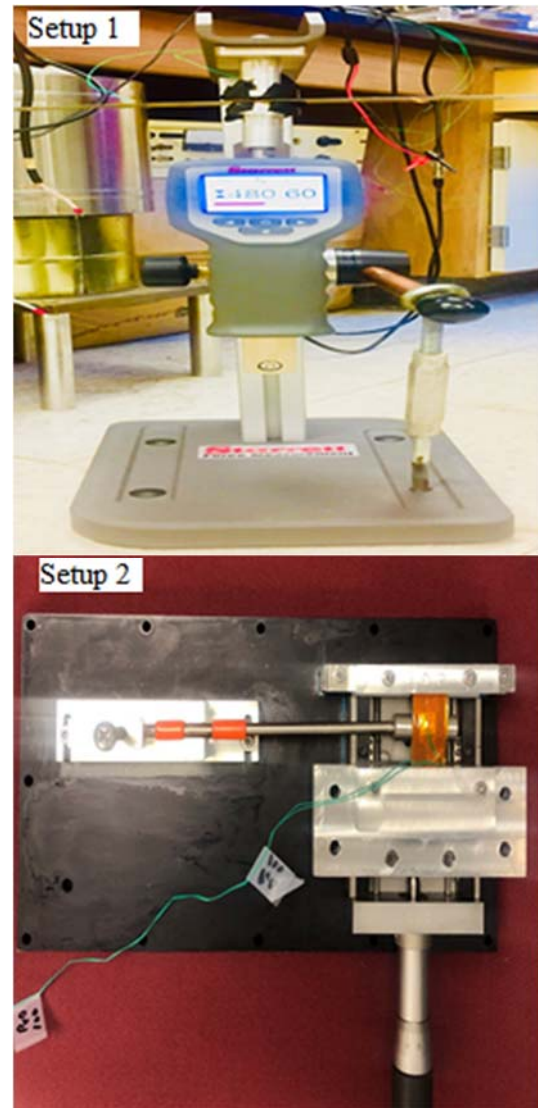


Fig. 1. Custom-made sensor performance determination setups: Setup 1(transverse pressure), Setup 2 (shear).

Custom-made setups shown in Fig. 1 were designed and fabricated for testing these nanocomposite pressure/strain film sensors.

3. Results and Discussions

From the recorded Fourier Transform Infrared (FTIR) and Raman spectra in the range of 400 to 4000 cm^{-1} , functional groups were identified in the nanocomposite films (Fig. 2). Infrared vibrational spectroscopy (FTIR) revealed ferroelectric β -phase in the annealed nanocomposite films intrinsically around 840 cm^{-1} [24, 25].

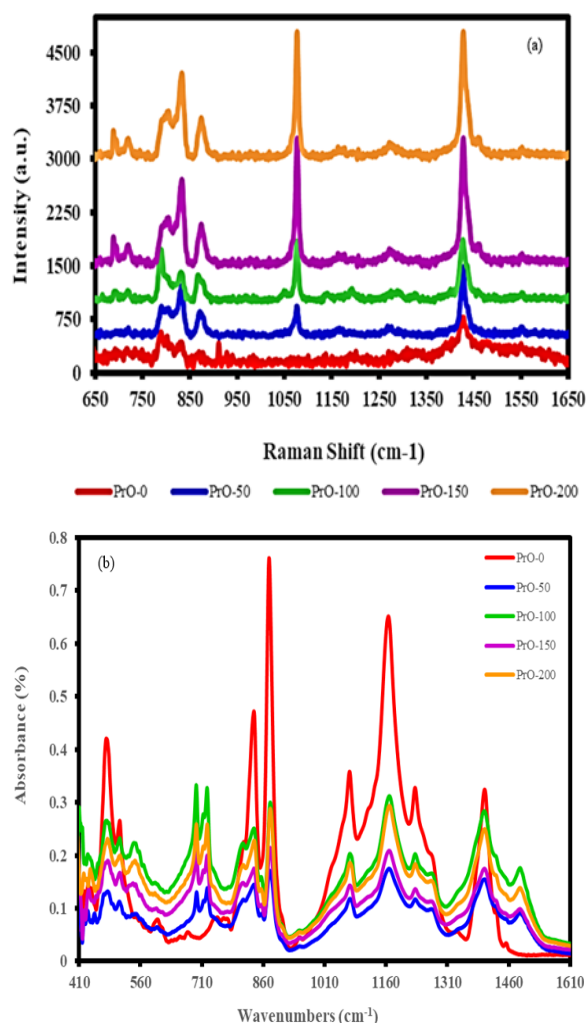


Fig. 2. Raman spectra (a) of PVDF thin film measured with a Raman system equipped with 785 nm wavelength laser and FTIR spectra (b).

For piezo-capacitive results, pressure was applied from below the electroactive nanocomposite samples via the cantilever in the earlier mentioned experimental setup initially displayed in Fig. 1. As the cantilever changes position, and consequently mechanical force, a measure of the change in dielectric capacitance is measured for the two nanocomposite

thin-films named PrO-100 and PrO-200. The relative DC electrical capacitance/sensitivity change versus position is first recorded in this manner. Secondly, electrical capacitance/sensitivity change versus force applied in Newtons is recorded utilizing a Starrett force materials testing system and graphically presented as well. Polymer composite material consisting of an insulating polymeric matrix and conductive reinforcing nanoparticles have a total resistance that is due to both components present. Assuming the resistance of the matrix be constant, the total resistance of the nanocomposite smart material will depend on number of particles as well as conducting paths present. That being said, the characteristics of the implemented conductive network will be the main factors which may ultimately determine the total resistance of the composite materials. The cantilever/position method exhibits the properties of the material under flexion, while the Starrett method utilizes applied mechanical force to exhibit the behavior of the nanocomposite-film under compression. Fig. 3 exhibit the experimental results in relation to the PrO-100 nanocomposite. Fig. 4 displays result of nanocomposite PrO-200.

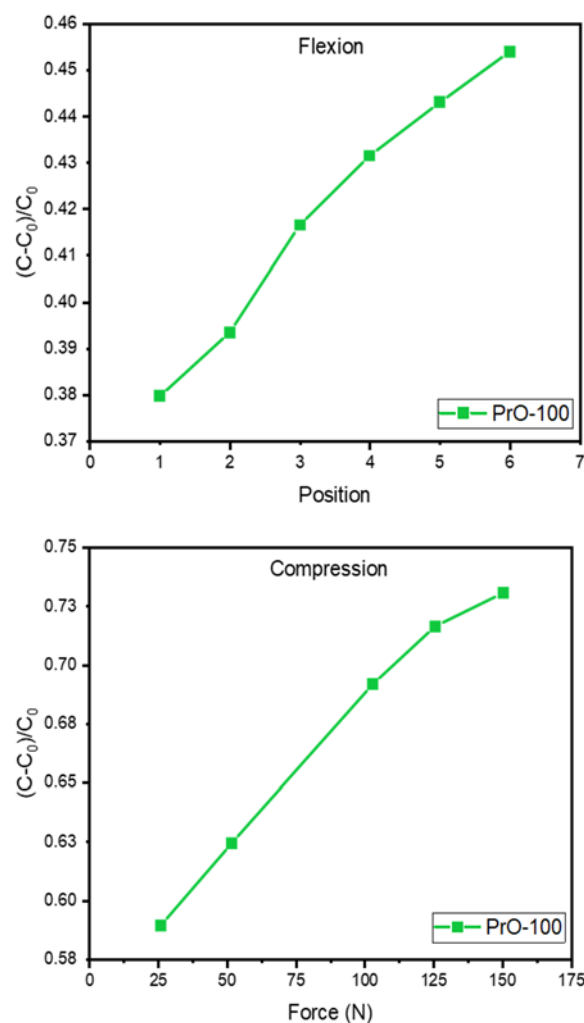


Fig. 3. Capacitance-change under flexion vs. position (arb. Units) and compression for PrO-100 versus force (N).

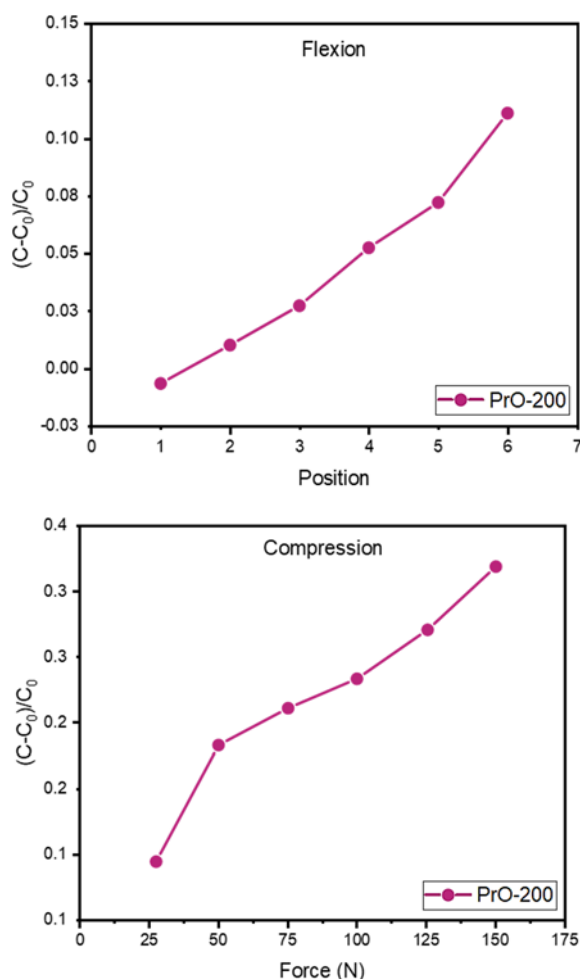


Fig. 4. Capacitance-change under flexion (shear stress) vs. Position (arb Units) and compression for PrO-200.

Stress and strain are the main driving forces and concepts in question when investigating piezo-capacitive and piezo-resistive characteristics of smart materials. The strain has three general categories, depending upon the change produced in a body and the type of stress applied. In terms of strain, there is longitudinal strain; volume strain; and shearing strain. As far as stress is concerned, there are five types of stress: tensile, compressive and shear, torsional, and bending. The results obtained using our custom-made experimental setups are indirectly related to these kinds of stress/strain parameters as well. By simulating stress/strain upon the nanocomposite film samples, information regarding their optimal placement and integration into systems as small biomedical energy harvesters or sensors can be determined. In terms of piezocapacity, common medical devices such as pacemakers could be attuned with small piezoelectric energy harvesters where the force from the flexion and compression of the contractions of the heart and pacemaker could be utilized to create an endless battery and functioning life. Piezoresistive/piezo-capacitive based smart materials would prove useful in the example of a medical patient recovering from recent knee replacement surgery. These smart devices integrated

into the artificial knee could help a physical therapist track patient's the knee strength and progression of a patient during their road to recovery.

4. Conclusions

In this study, good quality and homogeneous nanocomposite PVDF films embedded with Pr_2O_3 nanoparticles have been fabricated. We have demonstrated that technology based on Pr_2O_3 nanoparticles embedded in flexible PVDF matrix has proven successful in developing flexible pressure sensors. From the results of the FTIR analysis of the fabricated PVDF-praseodymium nanocomposite polymers films, we can clearly see the band at 840 cm^{-1} , confirming the formation of the β -phase in the films [23-28]. It is worth mentioning that compared to resistive sensors, capacitive sensors generally have higher sensitivity and lower detection limits [29, 30]. However, their inadequate linearity response and the presence of parasitic capacitance can be a significant challenge in practical applications.

Acknowledgments

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Visual Intelligence-Based Contactless Bio Signals Measurement and Diagnosis Sensor to Overcome COVID-19

¹ Chan-Il Kim, ² Sangwoo Cho, ^{1,*} and Jong-Ha Lee

¹ Dept. of the Biomedical Engineering, Keimyung University, Daegu, South Korea

² The Center for Advanced Technical Usability and Technologies, Keimyung University, Daegu, South Korea

* E-mail: Segeberg@gmail.com

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Abstract: COVID-19 is mainly transmitted either directly by droplets from infected people or indirectly through contact with vehicles in contaminated environments. Coronavirus, which is highly contagious, is highly likely to result in a secondary infection between the patient and the medical staff during the first diagnosis, and if it is left alone, the likelihood of a tertiary infection is increased. In order to prevent this, a biometric means capable of diagnosing body temperature and heart rate in a non-contact manner is needed in consideration of the characteristics of corona symptoms accompanying high fever and various complications.

In this paper, the body temperature image and pulse image are acquired simultaneously using a general camera to measure the health status of the subject. Using this, it is possible to monitor in real time and to react immediately to the patient's acute disease. This study aims to develop a system capable of simultaneously estimating temperature by measuring heart rate using image sequences and measuring changes in peripheral blood flow. First, the system recognizes the user's face from a sequence of images collected through the camera, then tracks it to continuously extract the color of the same area and measures the user's heart rate using the color change of the corresponding pixel. Second, the system measures changes in peripheral blood flow due to external temperature. The body maintains normal body temperature by regulating peripheral blood flow. In this study, the user's body temperature was estimated by measuring the difference in this delay with the user's body temperature.

As a result of the experiment, we confirmed that there is a close relationship between the external temperature and the stabilization time of blood flow, and based on this, a body temperature inference equation was derived.

In conclusion, this study, a user's face was recognized using a webcam, and a system was developed to infer heart rate and body temperature based on the color data of the recognized user's face.

Keywords: Mobile health, Non-contact, Heart rate, Temperature measurement, Artificial intelligence, Covid-19.

1. Introduction

Coronavirus disease 2019 (COVID-19) was first discovered in Wuhan, China in December 2019 due to the outbreak of pneumonia of unknown origin [1-3]. It rapidly spread from China to other countries around

the world, prompting the World Health Organization (WHO) to declare a pandemic in March 2020. In South Korea, a traveller from Wuhan, China was first diagnosed with COVID-19 on January 19, 2020. Since then, the government raised the COVID-19 alert level to "caution" due to the sporadic occurrence of patients

caused by foreign inflows, and then to “serious” in February as new cases gradually spread across the country [4-6].

Many transmission epidemiology researches conducted so far has identified acute respiratory infectious diseases and pneumonia are transmitted from person to person, and via droplets. COVID-19 is supposedly transmitted through the droplets originating from infected people or indirectly transmitted through the contact with media in a contaminated environment, as with most respiratory viruses [7]. COVID-19 patients exhibit very mild symptoms at the beginning, but they can release the virus in the asymptomatic phase before transitioning to the symptomatic phase. Therefore, failing to diagnose and impose a quarantine early leads to the widespread community transmission of COVID-19. Airborne transmission is possible as well, but it occurs only in special environments, such as medical institutions where aerosols can be generated [8, 9].

Therefore, the highly infectious coronavirus is likely to cause the secondary infection between the patient and the medical staff during the primary diagnosis, and the likelihood of the tertiary infection increases if it is not properly handled [10, 11]. To prevent the secondary infection, it is deemed necessary to develop bio signal measurement systems capable of detecting the body temperature and heart rate in a contactless manner, considering the characteristics of COVID-19 symptoms involving high fever and various complications. This study introduces a new means to remotely measure bio signals and the body temperature, which can effectively reduce a chance of additional infection during primary diagnosis process of a probable COVID-19 patient.

1.1. Clinical Features and History

Among the many complications from the coronavirus, the most prominent is high fever. Many medical institutions first measure the body temperature of a patient to diagnose COVID-19. Since secondary infection may occur during this process, hospitals introduced a contactless temperature measurement method that uses an infrared camera. It is a concept developed before the COVID-19 pandemic, but since the outbreak, this topic has started to draw extensive attention from both academia and industry, making it the most fundamental element for diagnosis and treatment. The most common methods uses thermal imaging cameras, a kind of infrared cameras. They measure the amount of the infrared rays emitted by an object and represent it as a relatively temperature to a reference point. However, if the reference point is inaccurate or is lost, the measured infrared rays cannot be used to determine the absolute temperature. In addition, infrared rays cannot penetrate transparent glass or acrylic materials. The information that infrared cameras receive in presence of a transparent barrier between the object and the

camera, such as glass window, is the thermal image of the barrier, not the object. In contrast, conventional cameras can retrieve light information through the barrier. Additionally, it is difficult to widely use thermal imaging cameras due to their high prices compared to conventional thermometers even though the prices have been reduced by the active distribution of the cameras.

To overcome these problems, this study proposed a method to measure bio signals without any physical contact, which reduces the chance of additional infection and also provides convenience to both medical personnel and patients. The health status of the participants was measured by collecting body temperature and pulse images concurrently using image processing. Then the results were used to develop a model that can enable real-time monitoring which provide sufficient time to medical personnel to take immediate action to the acute diseases of the patient.

2. Previous Studies

2.1. Beer-Lambert Law

The Beer-Lambert law represents the correlation between the intensity of the projected light, and that of the light that penetrated through a homogeneous medium when the light is projected at a given wavelength to the medium. This law uses the following equations to evaluate the correlation for the transmitted light from the vasoconstrictive circulation state at the photoplethysmography (PPG) signal measurement point.

$$\frac{I}{I_0} = \exp(-\varepsilon_a C_a V_a - \varepsilon_v C_v V_v - \varepsilon_t C_t V_t), \quad (1)$$

$$\frac{I_t}{I_0} = \exp(-\varepsilon_t C_t V_t) \quad (2)$$

In the above Eq. (1) and Eq. (2), I_0 is the intensity of the light projected from the light source, I is the intensity of the light transmitted through the blood and tissue, and I_t is the intensity of the light transmitted only through the tissue. Eq. (1) represents the relationship between the light source and the light transmitted through the blood and tissue, and Eq. (2) the relationship between the light source and the light transmitted only through the tissue. The ε_a , ε_v , and ε_t are absorption in the artery, vein, and tissue, respectively. Constants C_a , C_v , and C_t are their concentrations, and V_a , V_v , and V_t are their volumes. Eq. (1) is divided by Eq. (2) to obtain Eq. (3).

$$\frac{I}{I_t} = \exp(-\varepsilon CV) \quad (3)$$

In the above equation, V is the total blood volume, and ε is the average absorption coefficient of the total blood. Therefore, $V = V_a + V_v$ and $\varepsilon = (\varepsilon_a V_a + \varepsilon_v V_v) / V$

hold in Eq. (3). C_a and C_v in Eq. (1) represent hematocrits. The value of C_v increases during mental stress, but it varies by only a few percent (%). Consequently, C_v and C_a can be interpreted to be the same as the average concentration of the total blood (C) in the normal circulation state. Therefore, $C_a=C_v=C$ holds. It is known that the arteries and veins in the hands are very sensitive to vasoconstrictor stimuli. In addition, reflex venoconstriction and vasoconstriction do not occur independently. This indicates that changes in alpha-adrenergic sympathetic nerve activation are suitable for detecting changes in the blood vessel state between the arteries and veins at the fingertip. Therefore, assuming $\lambda = V_v/V_a$ (ratio of the venous blood volume to the arterial blood volume), ε can be expressed as follows.

$$\varepsilon = \left[\frac{1}{(\varepsilon + 1)} \right] \varepsilon_a + \left[\frac{1}{(\varepsilon + 1)} \right] \varepsilon_v \quad (4)$$

In particular, ε is not affected by changes related to the blood volume. Therefore, the differential coefficient of I/I_t , which corresponds to V in Eq. (3), is expressed as following.

$$\frac{\delta(\frac{I}{I_t})}{\delta V} = -\varepsilon C \left(\frac{1}{I_t} \right) \quad V = \frac{\pi r^4}{8 \eta l} \Delta P \quad (5)$$

Summarizing Eq. (5) yields Eq. (6).

$$\Delta V_a = (\varepsilon C)^{-1} \Delta \frac{I_a}{I} \quad (6)$$

The total blood volume consists of the averages and pulsating elements of the arterial and venous blood volumes. When pulsation is observed only in the arterial part, ΔV and corresponding ΔI are again converted into ΔV_a and ΔI_a in Eq. (6). When the attenuation of the transmitted light seems to lower the voltage in the actual PPG signal measurement, the values of ΔI_a and I have (-) and (+) signs, respectively. Therefore, $-\Delta I_a$ is replaced with ΔI_a to have (+) for ΔI_a in Eq. (6). Based on this, PPG signals can measure the blood volume change by projecting light toward vein.

2.2. Heart Rate Measurement Using PPG

Previous studies show that the heart rate and respiration rate can be measured using a conventional camera, appropriate lighting, and PPG.

PPG is one of the most commonly measured bio signals along with the electrocardiogram signal. The blood flow velocity, blood oxygen saturation, and pulse rate can be measured using PPG signals. The PPG measurement uses a photosensor that converts light into electric charges. This sensor act as a photoelectric transformation element and photodiodes are mainly used. A photoelectric transformation element converts the incoming incident light into electric charges. These electric charges deliver the intensity of light at each pixel position to the central

processing unit through a charge-coupled device and a digital signal processor (DSP) to generate an image. The information of the object is collected from the light reflecting off the object. PPG measurement acquires the signal by irradiating light in a specific wavelength band to the human body and detecting the reflected or transmitted light. The resultant signal represents a pulse wave generated by the heartbeat.

2.3. Utilization of PPG Signals

PPG has been clinically used for diverse physiological diagnoses and the evaluation of the functions and characteristics of the cardiovascular system. The cardiovascular system is a major organ system responsible for circulation, and mainly consists of blood, heart, and vessels. Physiological changes in cardiovascular system may occur due to multiple factors, such as the characteristics of myocardial cells, active state of the heart, mechanical characteristics of vessels and blood, and autonomous nerve system (ANS). Interaction between these multiple components achieve blood circulation in the body. Therefore, PPG, which can be non-invasively measured, has been utilized in various fields even though it indirectly reflects the status of the cardiovascular system.

2.4. Contactless Heart Rate Measurement Using Image Sequences

In previous studies, face was selected as the body part to extract PPG signals from. This is because it is easy to capture the images of a face using a camera and face clearly shows the difference in blood flow depending on the heartbeat. When PPG measurement is performed using an RGB camera, the green channel is more suitable for measuring the heart rate and respiration rate than the red or blue channel. This is because the green channel covers the wavelength that shows a clear difference in light absorption between hemoglobin and oxidized hemoglobin.

In addition, another study demonstrated that it is possible to measure the heart rate and respiration rate in the presence of many people after defining the face area using a face recognition system with a webcam. In the study, three independent factors were extracted from the RGB channels of the camera using blind source separation (BSS), and an independent component analysis (ICA) algorithm was used in this process. BSS is used to extract an accurate signal source under the assumption that an accurate signal cannot be identified because the measured signal is mixed with noise and various signals.

In this study, BSS was used to measure the precise amount of light reflected by oxidized hemoglobin and non-oxidized hemoglobin. While Hemoglobin is under influence of light of all channel and wavelengths, the data residing in the range from

520 to 580 nm is dominantly used in determining the blood vessel status. Based on this, PPG was measured by extracting independent factors that affect the accurate amount of light reflected by hemoglobin. In addition, we attempted to add the process of estimating the temperature of the user through the change in peripheral blood flow due to the external temperature difference.

3. Research Method

This study aims to develop a system that can measure the heart rate using image sequences and estimate the temperature simultaneously by measuring the change in peripheral blood flow. The system can be represented as Fig. 1.

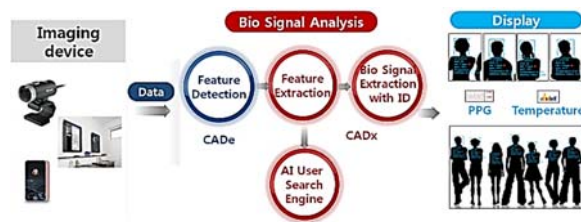


Fig. 1. Schematic of the developed system.

First, the system recognizes the user's face from the image sequences collected through the camera and then extracts feature points from the face. Then, it acquires coordinates for measuring the biometric data of the user based on the extracted feature points. The system continuously extracts the color of the same area by tracking the user's face and measures the heart rate of the user using the color change of the corresponding pixel.

Second, the system measures the change in peripheral blood flow caused by the external temperature. The human body controls the peripheral blood flow to maintain the normal body temperature. In this study, the user's temperature was estimated by measuring the difference in this delay depending on the user's body temperature.

3.1. Heart Rate Measurement

The method of measuring the heart rate using a camera in the detected face area is basically the same as the PPG measurement principle. Briefly, it is shown in Fig. 2 below.

PPG measurement is a method of estimating the heart rate by measuring the blood flow in a vessel using the optical characteristics of tissue. When blood flows to peripheral blood vessels, the flow becomes slow between the diastolic and systolic phases of the heart. In this instance, PPG detects changes in the transparency of the blood vessels.

This can be measured from any peripheral tissue of the body, the face, the fingers, or the earlobe. For conventional PPG measurement, the fingers are used. When the measurement is taken with the face, we expected that the heart rate can be recorded by detecting the variation in speed of blood flow in the vessels of the facial skin.

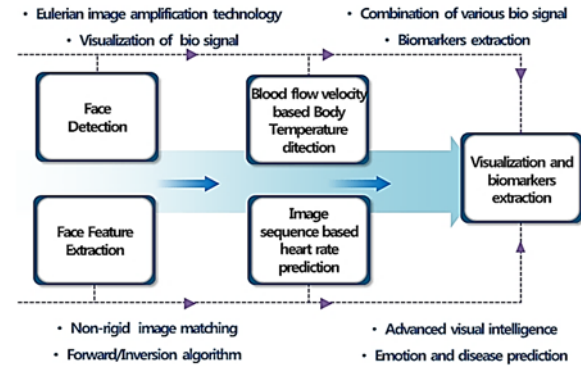


Fig. 2. Process of measuring the heart rate using a camera.

3.2. Temperature Measurement

PPG signals are generated by changes in peripheral blood flow and thus reflect the vessel state in the measurement area. As the cardiovascular system is a closed loop, it is possible to evaluate the state of various vascular systems by analyzing the components of PPG signals. The blood flow can be represented by the Hagen-Poiseuille's law (HPL) as shown in Eq. (7).

$$V = \frac{\pi r^4}{8\eta l} \Delta P, \quad (7)$$

$$R = \frac{8\eta l}{\pi r^4} \quad (8)$$

In Eq. (7), ΔP is the pressure, η is the viscosity of blood, and l is the length of the tube. Eq. (8) for the resistance in a tube can be obtained using Eq. (7). It indicates that the blood flow and the resistance against it are proportional and inversely proportional to the radius to the fourth power, respectively. Therefore, these two parameters are more affected by the change in the diameter of the tube than by the change in the length, pressure difference, and viscosity of the tube. This principle was applied to derive the change in peripheral blood vessels depending on the temperature. The surface temperature of the measurement area for PPG signal measurement is influenced by the body temperature and the surrounding environment. Local temperature changes will cause the contraction of tissue and peripheral blood vessels, thereby impacting the flow of blood. In a previous study, the correlation between the PPG signal and skin temperature under varying external temperature was revealed through an experiment after

attaching a reflective sensor to the forearm. In another study, the correlation between the blood flow and the finger temperature was revealed as Fig. 3. With these findings applied to the capillaries of the face, it is possible to estimate the body temperature using the change in the blood flow of the facial capillaries caused by the temperature change.

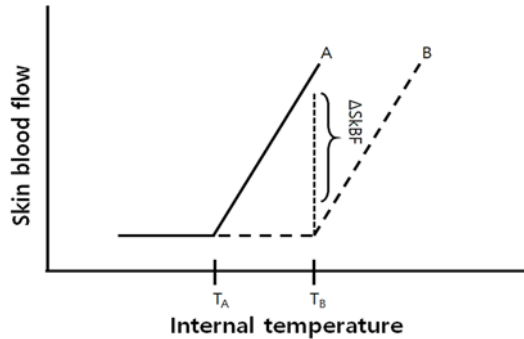


Fig. 3. Skin blood flow according to the external temperature.

The sympathetic nerve control of the skin blood flow involves the noradrenergic vasoconstrictor system and the sympathetic active vasoconstrictor system, and the latter is responsible for 80-90 % of the percutaneous vasoconstrictor caused by the thermal stress of the whole body. In short, the skin blood flow may increase to 6-8 L/min when the body temperature rises.

Thus, this study attempted to measure the body temperature based on the difference in the expansion of the skin capillaries when the body enters a stable environment from an external environment with a large temperature difference. We measured the time required for the temperature of the skin, which was previously disrupted by the environment, to stabilize (stabilization time: ST). The body temperature was calculated through the correlation between the difference of the subject's body temperature between the external temperature and ST (Fig. 4).

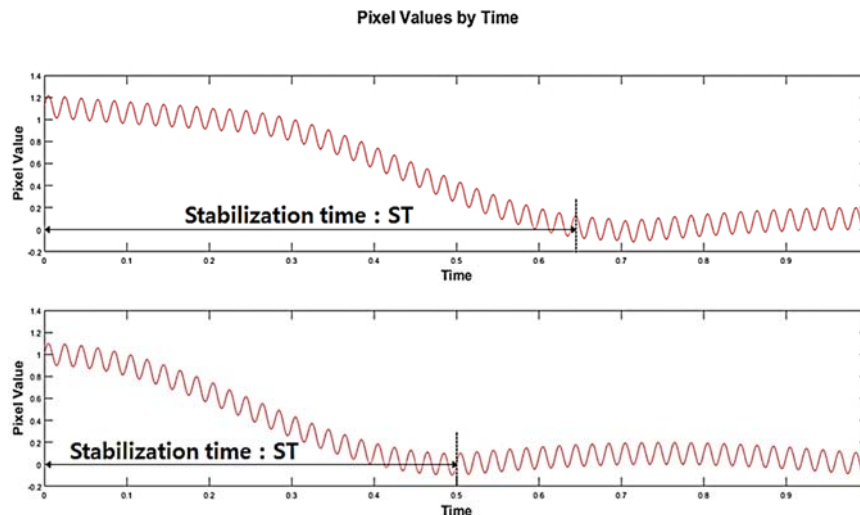


Fig. 4. ST according to the external temperature (top: external temperature of 37°, bottom: external temperature of 33°).

4. Research Results

The validity of the data collected from a camera was evaluated by comparing the variance of blood flow, which was measured through the proposed method, to the value measured through PPG measurement that used Biopac MP150 (Biopac, Goleta, CA, USA).

Biopac MP150, a device for oxygen saturation measurement from Biopac, can measure biosignals using both digital and analog methods.



Fig. 5. Experimental setup of Contactless biosignal measurement system.

4.1. Heart Rate Measurement Results

The experiment was performed ten times with seven males and three females aged between 23 and 31. In each experiment, the heart rate was measured ten times for a minute, and the average heart rate measured using the image sequences obtained by alternating the lighting conditions was compared with the average heart rate measuring using Biopac. The results are shown in the figure below. The accuracy of average heart rate measurement was found to be 99.1 %.

4.2. Body Temperature Measurement Results

In this study, an experiment was performed with test subjects aged between 23 and 33 while the temperature of the room was fixed at 25°C. ST was measured after each test subject was exposed

to an environment with an external temperature between 28°C and 38°C for ten minutes and then introduced to an indoor environment with a fixed temperature of 25°C.

The thickness of the blood vessel wall, the elasticity of the blood vessel wall, and the viscosity of blood were excluded due to their negligible influence over the peripheral blood flow. Based on the temperature difference with the largest influence, a body temperature measurement equation was derived. The results showed that T_{ST} increased as the temperature difference between the control and test environment, i.e. the indoor and outdoor respectively. As shown in the Fig. 6, the correlation between the temperature difference and T_{ST} was analyzed through the linear regression method to derive a relationship. The derived equation is as follows.

$$T_{ST} = 1.337(T_{out} - T_{in}) + 9.710 \quad (9)$$

In Eq. (9), T_{ST} denotes ST. A linear regression model was fitted by including the y-intercept of the graph. As a result, the values of the dotted line in Fig. 6 were obtained. The coefficient of determination was calculated to find relatively suitable fitting. The calculated coefficient of determination was $R^2=0.798$.

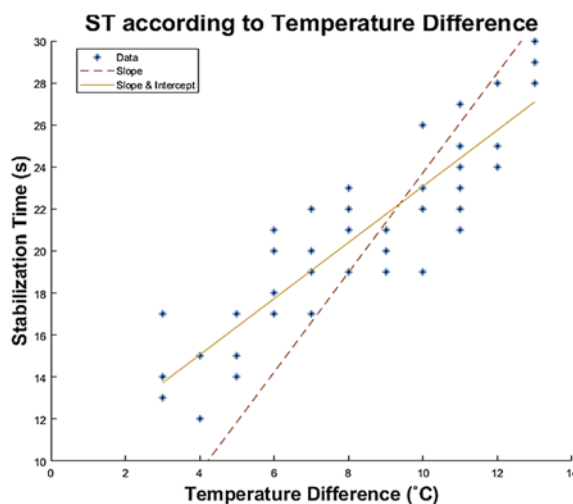


Fig. 6. ST according to the external temperature.

5. Conclusion

As the product of this study, we designed a system to measure the complex biosignals and data of the user using image sequences. The system recognizes the user's face using the image sequences collected through a camera and acquires coordinates for measuring the biometric data of the patient based on the extracted feature points. It extracts the color of the same face area by continuously tracking the user's face and measures the heart rate of the user, using the color change of the corresponding pixel. In addition to simple heart rate measurement, the blood flow in the skin capillaries was measured using the phenomenon

that the light absorption wavelengths of oxidized hemoglobin and hemoglobin are different. Based on this, an algorithm and a system for estimating the body temperature were also developed.

When the heart rate measured using image sequences was compared to photoplethysmography (PPG), which is the conventional contact-type measurement method, an accuracy of 99 % was achieved. And the calculated body temperature also exhibited high accuracy compared to the actual temperature measured with a thermometer. The results showed that the biosignals processed using image sequences have comparable reliability to those measured using the conventional biosignal measurement method.

However, the proposed method has a drawback. It estimates the body temperature by measuring the time required for the cooled body to be stabilized. And it can be used only when there is a temperature difference between the external space and the measurement space. As the method does not have control over the environmental temperature, it is necessary to develop and verify additional algorithms that can overcome this limitation.

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Advances in Artificial Intelligence: Reviews

Sergey Y. Yurish, Editor



Artificial intelligence has been one of the fastest-growing technologies in recent years. The market growth is mainly driven by factors such as the increasing adoption of cloud-based applications and services, growing big data, and increasing demand for intelligent virtual assistants. Various end-use industries have also employed artificial intelligence such as retail and business analysis that has also boosted the demand in this market. The major restraint for the market is the limited number of artificial intelligence technology experts. The Book Series on 'Advances in Artificial Intelligence: Reviews' has been launched with the aim to fill-in this gap.

The first book volume from the 'Advances in Artificial Intelligence: Reviews' Book Series contains 11 chapters written by 21 contributors from academia and industry from 10 countries: Algeria, Germany, India, Iran, Israel, Russia, Slovenia, South Africa, Tunisia and USA.

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First Results of the BeE-Nose on Mid-Term Duration Hive Air Monitoring for Varroa Infestation Level Estimation

Andreas KÖNIG

Institute of Integrated Sensor Systems, TU Kaiserslautern,
Erwin-Schrödinger-Str. 12, Kaiserslautern, 67663, Germany
Tel.: +496312053696, fax: +496312053889
E-mail: koenig@eit.uni-kl.de

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Abstract: Bees are recognized as an indispensable link in the human food chain and general ecological system. Numerous threats, from pesticides to parasites, endanger bees and frequently lead to hive collapse. The varroa destructor mite is a key threat to bee keeping and the monitoring of hive infestation level is of major concern for effective treatment. Sensors and automation, e.g., as in condition-monitoring and Industry 4.0, with machine learning offer help. In numerous activities a rich variety of sensors have been applied to apiary/hive instrumentation and bee monitoring. Quite recent activities try to extract estimates of varroa infestation level by hive air analysis based on gas sensing and gas sensor systems. In our work in the IndusBee4.0 project [8, 11], an hive-integrated, compact autonomous gas sensing system for varroa infestation level estimation based on low-cost highly integrated gas sensors was conceived and applied. This paper adds to [11] with the first results of a mid-term duration investigation from July to September 2020 until formic acid treatment. For the regarded hive more than 79 % of detection probability based on the SGP30 gas sensor readings have been achieved.

Keywords: Multi-modal bee health monitoring, Varroa infestation level estimation, Gas sensing, Machine learning, Apiary intelligence.

1. Introduction

Major issues from environmental pollution to invasive species are threatening our ecological system and the human food supply. Insects, and honey bees in particular, play a decisive role, e.g., for pollination. The varroa mite parasite is a major threat to bee keeping and the cause of many bee colony losses. The monitoring of the varroa infestation level is one important task of conventionally operating bee keepers. Though there is a community practicing treatment free bee keeping [1], the majority of bee keepers follows standard treatment practice, e.g., by formic acid, which needs to know the right time to start treatment based on the hive infestation level.

Sensors and automation approaches, e.g., condition-monitoring, and Industry 4.0, can both alleviate hive keeping and also make it much more effective (see, e.g., [8, 11]). In addition to common monitoring modalities, like temperature, moisture, visual/IR images, or sound patterns, the hive air attracted attention, both due to therapeutic interest for patients with respiratory ailments [3], and for extended hive condition monitoring based on simple and cost effective gas sensors, in particular MOX sensors [2], e.g., for CO₂ or VOC concentration measurement. The aspect of estimating the varroa infestation level by means of such gas sensors combined with pattern recognition and machine learning techniques has attracted numerous researchers. The approach shows

potential to be generalized to other parasites or illness, e.g., the emerging pest of SHB (Small Hive Beetle), or foulbrood.

In [4-7] interesting activity based on a multi-gas sensor system of Figaro gas sensors can be found. A principal and useful relation between the gas sensor readings and varroa infestation level was reported. The ground truth in [4] was obtained by employing the flotation method to the same hive. The external measurement setup, however, reportedly implied challenges, e.g., with regard to dew point issues and context dependence due to day time of measurement [6], and monitoring times were rather limited, i.e., continuous monitoring for a whole bee season or development cycle of a hive was not reported yet.

In our IndusBee4.0 project, started end 2017, in-hive cost-effective integrated sensor systems and machine learning based data analysis, continuous state or condition monitoring, and automated decision making is pursued [8, 11] for longer period, preferably covering the decisive part of the bee season from the onset of breeding activity to the first formic acid treatment [11] and finding effective cues in the monitoring data to estimate the desired infestation level. In Section 2, the ground truth obtaining will be outlined, Section 3 describes the details of the data acquisition, and Section 4 gives the first results obtained from July to September.

2. Varroa Monitoring Options

There are several standard methods available for conventional varroa infestation level assessment for the required ground truth. They all have in common, that they imply substantial effort for the bee keeper and deliver results only at larger time steps. The analysis of hive debris including mites, dropping from the hive bottom and collected on a slider or tray, is most common. Usually, three days are expended until a manual, or more recently (semi) automated vision-based analysis, of the debris for the number of varroa mites can be conducted. The hive infestation level can be estimated from this count [8]. Another common approach, also denoted as flotation method, used in [4], extracts a bee sample from the hive and effectively drowns them to separate bees and varroa to count the mites. The powder sugar and the CO₂-based sedation method are two alternative, more bee-friendly, variants. Sample adequateness will probably depend on the location of extraction in the hive. A more recent principle approach tries to scrutinize in and out going bees at the flight hole for varroa mites clinging to them, e.g., [8, 9].

Here, the conventional established tray analysis was employed and more frequently repeated for the instrumented hives.

3. Data Acquisition by BeE-Nose

For the continued and unobtrusive measurements in individual bee colonies and a complete apiary, a

modular monitoring system has been conceived, that networks the instrumented hives and combines them with hive keeper assistance systems, as outlined in [8, 11]. The block diagram of the IndusBee4.0 Apiary Monitoring system is shown in [11, Fig. 1]. The basic building block is the SmartComb in-hive autonomous measurement system [11, Fig. 4]. In the monitored hive, the SmartComb is located in the middle of the second super of a three super hive, i.e., in the very center. The monitoring can be controlled and read-out via WLAN. The regarded hive has been instrumented also with hive and honey room scales in addition to temperature, moisture, sound patterns, and gas sensors readings. The focus of this report here is on the SGP30 gas sensor, with fortunate properties of baseline determination [12], and its monitoring results. At a later point, context from the other sensors will be included in the analysis.

In the following experiments, the SGP30 gas sensor readings of time intervals from July to September 2020, due to issues in employed libraries and several required restarts, have been merged.

4. Experiments and First Results

For the first hive monitoring investigations by BeE-Nose, the data was acquired in the interval from 9th July to 11th September with about 6 readings per minute.

The development of the varroa population was monitored more frequently than usual by established tray analysis as outlined in Section 2. The obtained varroa count development confirmed the validity of the chosen observation time. The regarded instrumented hive had released a strong swarm end of April.

Fig. 1 shows the SGP30 gas sensor recordings over the July to September campaign. The two raw data outputs for ethanol and hydrogen as well as the computed e-CO₂ and TVOC outputs are displayed. This is complemented by the scaled varroa ground truth, which was obtained concurrently to gas measurements by following the standard procedure of counting the daily average drop of varroa on the tray, here 0, 2.5, 8.6, 14, and scaling this to an estimate of the actual population in the hive by the standard factor 150. For the sake of visibility, in the Fig. 1 the floored resulting values were additionally scaled by a factor of 10. As can be seen, in this hive the varroa population ramps up during the campaign until the critical level for required treatment, commonly more than 10 per day, is reached in September.

Fig. 2 shows a scatter plot from the immediate SGP30 sensor data, i.e., ethanol and hydrogen outputs, picking in a hold-out approach every thousandth sample from the available 1003919 of the baseline corrected readings as training set of a size of 1003 samples, in this first step without further preprocessing or feature extraction. A corresponding test set of the same size has been picked from the same sensor readings with a temporal displacement of

500 samples to the training data. The four varroa level estimations obtained from counting and related varroa population size estimate as displayed in Fig. 1 have been translated to four classes from '1, No Varroa', '2, Low Varroa', '3, Mid Varroa', to '4, Treatment !'. However, the boundaries between classes 1, 2 and 3 are rather arbitrary, but make classification definitely much harder and error prone. Thus, with regard to the actual binary issue *ok* or *treatment needed* alert, this was simplified to just two classes '1, Ok', here summarizing former classes 1 to 3 and former class 4 as '2, Treatment !' in the remaining conducted classification experiments.

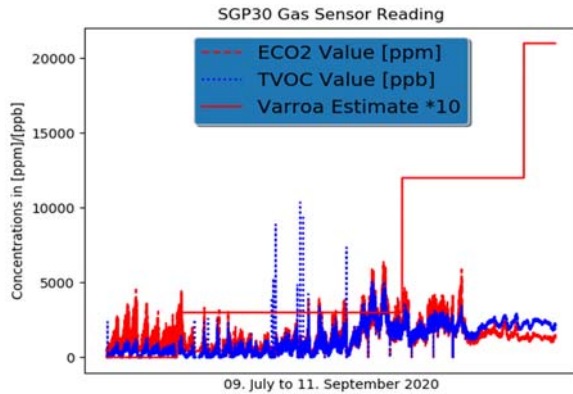


Fig. 1. SGP30 gas sensor recordings over the July to September campaign for the computed e-CO₂ and TVOC values complemented by the scaled varroa ground truth obtained from tray analysis.

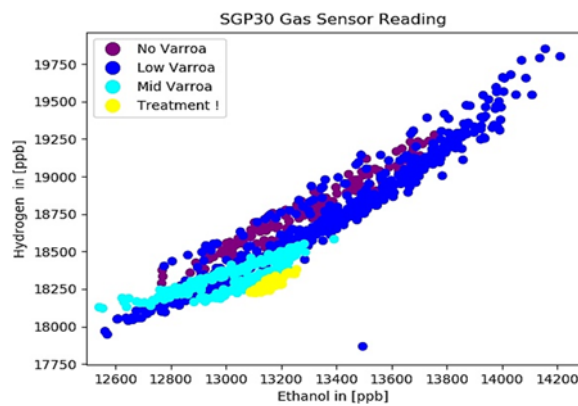


Fig. 2. Scatter plot of the training subset of SGP30 ethanol and hydrogen gas sensor readings over the July to September campaign.

The scatter plot in Fig. 2 shows for four assumed classes a mediocre support of the hypo-thesis, that the SGP30 readings give a cue on varroa infestation level. Unfortunately, even reducing to a two class problem or alert detector for treatment, inter class distance is small and a substantial overlap in the currently acquired sensor data can be observed. This advocates both the use of additional sensors as well as efficient feature computation [13-15].

Fig. 3 correspondingly shows a scatter plot for the e-CO₂ and TVOC sensor readings, training set, with a comparable outcome as observed in Fig. 2.

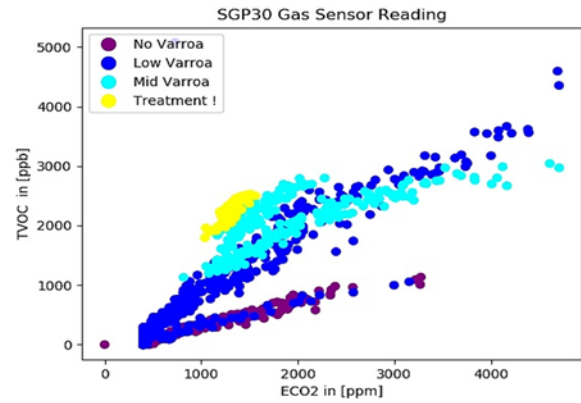


Fig. 3. Scatter plot of the training subset of SGP30 e-CO₂ and TVOC gas sensor readings over the July to September campaign.

Classification experiments were conducted for both SGP30 ethanol/hydrogen and e-CO₂/TVOC data sets for several common and relevant classifiers and the results are summarized in Table 1.

Table 1. First classification results for the SGP30 data and a voting kNN-classifier (k = 3), RNN, PNN, and SVMs.

Classifier	No. of Classes	Resubstitution	Generalization
SGP30 ethanol and hydrogen output			
kNN	4	89.0 %	79.8 %
kNN	2	98.5 %	97.4 %
RNN	2	100 %	96.91 %
PNN	2	100 %	97.41 %
SVM	2	93.15 %	93.15 %
NLSVM	2	98.00 %	97.91 %
SGP30 e-CO₂ and TVOC output			
kNN	4	88.33 %	82.85 %
kNN	2	99.1 %	98.6 %
RNN	2	100 %	97.81 %
PNN	2	100 %	97.61 %
SVM	2	98.4 %	98.0 %
NLSVM	2	98.9 %	98.6 %
SGP30 ethanol, hydrogen, e-CO₂ & TVOC output			
kNN	2	99.1 %	98.7 %

The classifications were predominantly carried out based on the sklearn package of Python, RNN and PNN were employed in the proprietary QuickCog tool. RNN does not require parameters, PNN kernel width was set to 2 in resubstitution and 4 and 7 in generalization for the reported results. For the kNN, k=3 was determined and employed. The linear SVM was employed with C = 1.0, the nonlinear SVM with rbf-kernel, C = 1.01, and gamma = $2.6 \cdot 10^{-5}$.

The kNN runs for the four class case give the worst results of 79.8 % and 82.85 % in generalization, which is not unexpected and due to the granularity of currently determined varroa counts and resulting class thresholds, and the overlap at class boundaries.

Reducing to two classes, i.e., an alert detector for *treatment needed*, however, returns more than 97 % in all cases, which is encouraging. Table 2 and Table 3 show the confusion matrices of the kNN classifications of SGP30 ethanol and hydrogen output for four and two classes of Table 1, respectively. The confusion between classes 3 and 4, and 1 and 2, respectively, gives only 82.6 % recognition (True Positives, TP) of the class 2 *treatment needed*.

Table 2. Confusion matrix for the SGP30 data and a voting kNN-classifier with $k = 3$ and four classes.

SGP30 ethanol and hydrogen output				
	1	2	3	4
1	118	38	1	0
2	45	403	56	0
3	0	36	223	14
4	0	0	12	57

Table 3. Confusion matrix for the SGP30 data and a voting kNN-classifier with $k = 3$ and two classes.

SGP30 ethanol and hydrogen output		
	1	2
1	920	14
2	12	57

This is repeated in Table 1 for e-CO₂ and TVOC output and the corresponding confusion matrices are given in Table 4 and Table 5.

Table 4. Confusion matrix for the SGP30 data and a voting kNN-classifier with $k = 3$ and four classes.

SGP30 e-CO ₂ and TVOC output				
	1	2	3	4
1	141	16	0	0
2	62	394	47	1
3	0	33	232	8
4	0	0	5	64

Table 5. Confusion matrix for the SGP30 data and a voting kNN-classifier with $k = 3$ and two classes.

SGP30 e-CO ₂ and TVOC output		
	1	2
1	925	9
2	5	64

The confusion between class 3 and 4, and 1 and 2, respectively, now gives 92.75 % recognition (TP) of the class 2 *treatment needed* for e-CO₂ and TVOC output. Finally, in the last row of Table 1, one more experiment just for kNN with $k=3$ can be observed,

where the four sensor outputs have been merged, which left resubstitution unchanged and gave a minor increase in generalization from 98.6 % to 98.7 %.

5. Discussion

The employed SGP30 gas sensor and the related measurements indicate a moderate correlation with the manually determined onset of varroa infestation in the bee hive. However, the SGP30 sensor readings cannot realistically be expected to give a direct indication of the present varroa population, the observed correlation or dependency can be accredited to indirect indication of changes in bee hive climate due to the imposed influence and stress of the growing mite population on the bee colony. This clearly advocates to add additional sensors, e.g., as inspired by [4-7], but in hive-integrated embodiment. The popular Bosch SENSORTec BME680, which even allows temperature modulation, has already been included [11] and will be incorporated in the future analysis. This will in general also demand for effective domain-specific feature computation as, e.g., presented in [13-15], which will also give credit to the temporal nature of the data. For instance, analyzing the temporal sequence of occurrence of current misclassifications, it became obvious, that these were predominantly singular or temporarily isolated events. As the treatment decision allows to accumulate decisions over a longer interval, e.g., several hours, singularities could be masked by a voting approach for a more robust alert.

Further, the number of manual inspections and related varroa counts are still sparse or coarse and would ask for a more fine grained, i.e., more frequent, determination in future measurement campaigns. Also, in the current investigation, samples have been adopted from any time of the 24 h observation cycle in the regarded campaign of three months. In the light of [6], the context of time of day and colony activity should be definitely included in the choice of classification data for improved detection and alert generation capability.

6. Conclusions

IoT, Machine Learning, and Artificial Intelligence massively move in the agro-tech domain, striving for fully automated farming solutions [10]. The importance of bees in agriculture and the overall ecosystem as well as the stringent need for technological support and alleviation of bee keeping is undisputed [4-7, 8, 9, 11]. In this work, a step towards continuous varroa infestation level determination by a low-cost, small, and unobtrusive in-hive monitoring system with gas sensor extension [11] has been achieved. First results obtained by this BeE-Nose from a mid-term observation time in a hold-out approach harmonize with those reported in [4-7], which were based on comparably macroscopic hive-external

equipment, shorter observation time, but richer sensor palette.

Though *ad hoc* use of this first SGP30 data already delivered a moderate leverage for the infestation level estimation and treatment alert generation, the role of context, robustness, and stability of the results, varroa threshold settings, as well as generalization issues to different hives have to be thoroughly investigated in the next steps.

In future work, more sophisticated feature computation [13-15] and multiple gas sensor employment, starting with adding BME680 information, will be regarded. Exploitation of further cues from acoustical data [8, 11] and merging those with the gas sensor information for increase of robust varroa infestation level estimation and treatment alert generation will be investigated (see [11], Fig. 10).

Potential generalization to other pests and illnesses as the SHB or variations of foulbrood open further opportunities.

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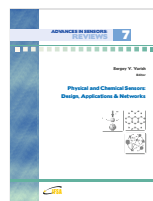
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In 1961 professor P. V. Novitskiy wrote: "... In the future we can expect, that a class of frequency output sensors will get such development, that the number of now known frequency output sensors will exceed the number of now known amplitude sensors...". It was also predicted in a technology forecast in 1971: "Many basic measurements will be related to or measured by time/frequency techniques. This situation will occur because of the increasing facility and accuracy provided through time frequency measurements... Frequency and computing counters can be expected to become smaller, lighter and cheaper, provide direct frequency readout and will be more universally used... Progress will continue in the field of time and frequency during the next five years and will contribute materially to the state of the art in electronics".

Despite the fact that this forecast was not fully justified till now, it is really the situation when "the future glimmers long before it comes to be". Today, the next big challenge has come from microelectronics. So, in 2010 professor Staphan Henzler did write: "Below the 100 nm technology node the design of analog and mixed-signal circuits becomes perceptibly more difficult. This is particularly true for low supply voltage near 1 V or below. The result is not only an increased design effort but also a growing power consumption. ... However, digital circuits become faster, smaller, and less power hungry".

Frequency/time domain signal processing and so time-to-digital converters (TDC) and frequency-to-digital (FDC) converters are well suitable for high volume microelectronics in order to circumvent analog impairments in nanometer-scale CMOS technologies. Hence, the TDC and FDC concepts are promising that have the ability to exploit fully the advantage of digital circuits. The FDC will be a trend-setting technology for ultimately scaled CMOS technologies in the nearest future, and can be used for many applications such as smart digital sensors, IoT and Industry 4.0. No any doubt that now we are living in time, when the old prediction becomes true !

On the basis of the mentioned above and Industry 4.0 and IoT 2.0 challenges it has been decided to start a new annual international conference, which will cover both: methods of measurements of frequency-time parameters of signals, and devices including appropriate sensors, transducers and converters to frequency, and frequency to digital. Sure, the conference can not ignore the other related areas connected with frequency and time, namely: generation, transfer, various oscillators and resonators, standards and references, etc.

The 1st IFSA Frequency and Time Conference (IFTC' 2019) has taken place in Barcelona, Spain (2019), and the 2nd IFSA Frequency and Time Conference (IFTC' 2020) - in Porto (Portugal). The 3rd IFTC' 2021 will take place in Palma de Mallorca (Balearic Islands), Spain in conjunction with the 7th Annual International Conference on Sensors and Electronic Instrumentation Advances (SEIA' 2021) under the same conference umbrella.



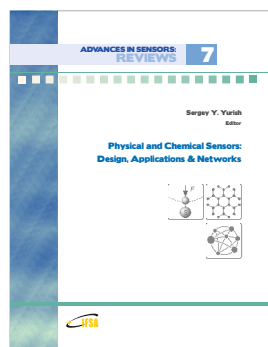
3rd IFSA Frequency & Time Conference (IFTC' 2021)

The topics of interest include (but not limited):

- Methods for Frequency, Time and Phase Measurements
- Measurements of Frequency-Time Parameters of Signals
- Frequency-to-Digital Converters (FDC)
- Time-to-Digital Converters (TDC)
- Voltage-to-Frequency Converters (VFC)
- Current-to-Frequency Converters
- ADC based on Voltage-to-Frequency Converters
- Capacitance-to-Frequency Converters
- Resistance-to-Frequency Converters
- Resistive Bridge-to-Frequency Converters
- Inductance-to-Frequency Converters
- Impedance-to-Frequency Converters
- Counters, Phasemeters and Tachometers
- Frequency Generators and Synthesizers
- Frequency, period, duty-cycle, time interval, pulse width modulation and pulse number output sensors and transducers
- Surface Acoustic Wave (SAW) sensors
- Bulk Acoustic Wave (BAW) sensors
- Quartz Crystal Microbalance (QCM) sensors
- Ultrasonic Sensors
- Oscillators, Resonators, Timers, Resonator Circuits and Filters
- Voltage-controlled Oscillators
- Atomic and Optical Frequency Standards
- Frequency References
- Frequency Control, Timing and Time Distribution
- Time and Frequency Transfer
- Ferroelectric, Piezoelectric, and Piezomagnetic Materials
- Applications, trends and markets

One Event - Three Publications!

1. All registered abstracts will be published in the conference proceedings (Flash drive edition of published Proceedings with the ISBN).
2. Authors will be invited to submit full-page extended papers to the special issue of Sensors & Transducers journal (ISSN 2306-8515, e-ISSN 1726-5479) by IFSA Publishing, which is published in both formats: print and electronic.
3. The limited number of full-page papers published in the journal will be selected by the Editorial Board to extend for the open access book chapters for the Book Series on 'Advances in Sensors: Reviews', Vol. 8, which will be published in 2020/2021, or 'Advances in Measurements and Instrumentation: Reviews', Vol. 3. These Book Series are indexed in the Book Citation Index (Web of Science) by Clarivate Analytics.



Contribution Types:

- Keynote presentations
- Invited talks
- Industrial presentations
- Regular presentations
- Special Session presentations
- Posters
- Panel discussion & round tables
- Exhibition
- Tutorials

Deadlines:

Submission (2-page abstract): **1 June 2021**
Notification of acceptance: **20 June 2021**
Registration: **15 July 2021**
Camera ready (4-6 pages paper): **30 July 2021**

Special Sessions and Tutorials

Authors are welcome to propose and manage special sessions or give a half-day pre-conference tutorial during the IFTC' 2021. Each special session will contain 4-6 papers in a related field as specified above.

Session organizers will get:

- Certificate of Appreciation
- Free Registration
- Special Publishing Theme within Conference Proceedings

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Information for Sponsors

The conference organizers are looking for sponsors from industry and academia to support various activities, such as invited, keynote speakers, gala dinner, coffee breaks, post- and pre-conferences tours and other social events. The organizers offer different sponsorship and associated benefits. For more information on sponsoring, please contact Dr. Sergey Y. Yurish by e-mail: SYurish@sensorsportal.com

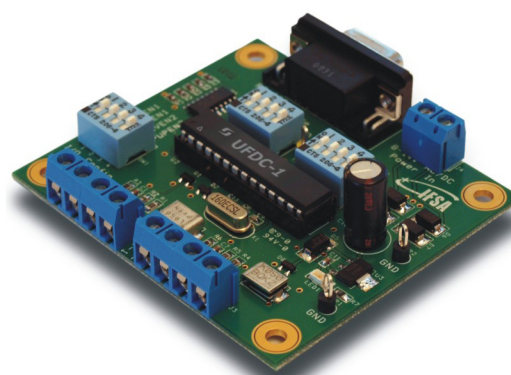
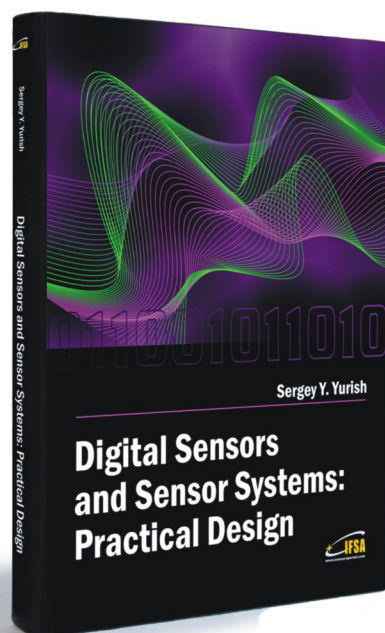
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Applications:

- Digital sensors and sensor systems
- Smart sensors systems
- Data Acquisition for frequency-time parameters of electric signals
- Frequency counters
- Tachometers and tachometric systems
- Virtual instruments
- Educational process in sensors and measurements
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