Centre for Nanotechnology and Smart Materials



SmartEEs2 Training Workshop

Sensing Technologies and Applications

02-06-2021

Session 2

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OUTLOOK

- ✓ Basic Principles
- ✓ Sensing Technologies
- ✓ Sensor R&D @CENTI





Basic Principles





Sensors vs Transducer

"A sensor is a device that receives a signal or stimulus and responds with an electrical signal while a transducer is a converter of one type of energy into another"

Sensor Technology Handbook, edited by Jon Wilson, Elsevier, 2005 : Excerpted from Practical Design Techniques for Sensor Signal Conditioning, Analog Devices, Inc., www.analog.com.

"A sensor is a device that responds to a physical stimulus (as heat, light, sound, pressure, magnetism, or a particular motion) and transmits a resulting impulse (a signal relating to the quantity being measured). (...)" wikianswers.com

"Transducer: a device that changes (transduces) the signal to or from an electrical domain as a voltage or current. Sensor: also called detector, a device that senses a physical or chemical stimulus and converts it into a signalthat can be electrical, mechanical, or optical "

ISAT 253. Analytical Methods V: Instrumentation and Measurement (James Madison University)





Sensor Classification

Form of Signal	Measurands
Thermal	Temperature, heat, heat flow, entropy, heat capacity
Radiation	Gamma rays, X-rays, ultra-violet, visible, infra-red, micro-waves, radio waves
Mechanical	Displacement, velocity, acceleration, force, torque, pressure, mass, flow, acoustic wavelenght and amplitude
Magnetic	Magnetic field, flux, magnetic moment, magnetisation, magnetic permeability
Chemical	Humidity, pH level and ions, concentration of gases, vapours and odours, toxic and flammable materials, pollutants
Biological	Sugars, proteins, homones, antigens
Electrical	Charge, current, voltage, resistance, conductance, capacitance, inductance, dieletric permitivity, polarisation, frequency





Sensor Characteristics

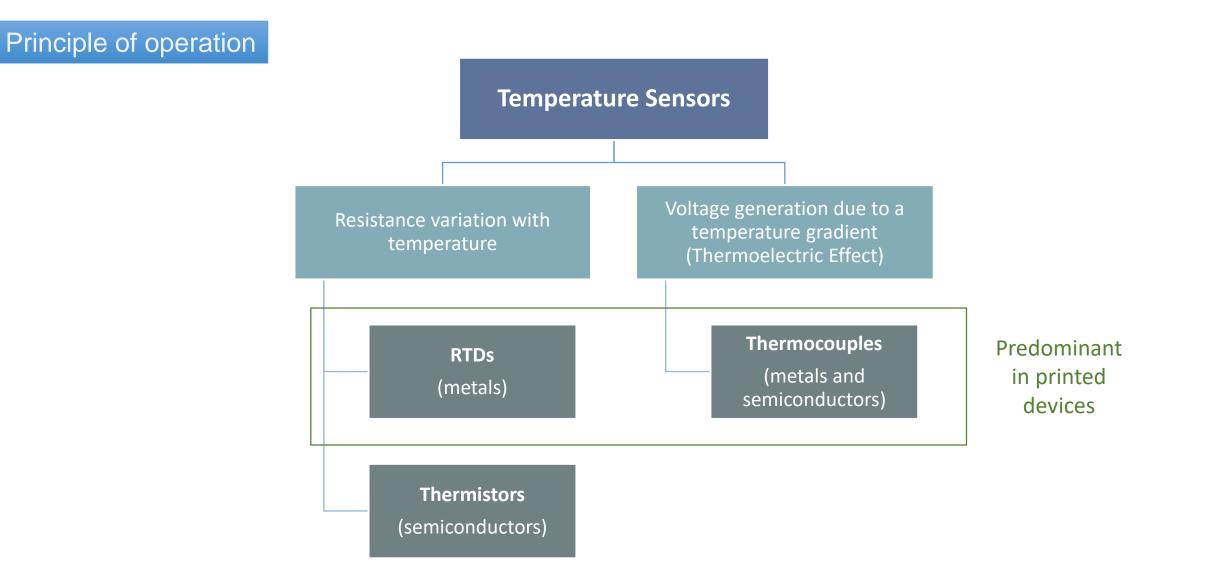
- * Transfer function Function that relates the sensor output to the measured quantity;
- Sensitivity Indicates how much the sensor's output changes when the input quantity being measured changes;
- **Resolution** Smallest increment of magnitude that the sensor can detect.
- ✤ Accuracy Sensor capacity to provide a measured value close to the real value;
- Precision Sensor capacity to provide similar values in successive measurements;
- Hysteresis Difference between two separate measurements taken at the same point, the first is taken during a series of increasing measurement values, and the other during a series of decreasing measurement values;



Sensing Technologies











Structures and materials selection

RTDs: signal sensitivity is dependent of the material resistivity, ρ , and the respective temperature coefficient (TCR), α .

 $R(T) = R_0 \cdot [1 + \alpha(T - T_0)]$

Thermocouples: signal sensitivity depends on the Seebeck coefficient, S, of both materials.

$$\mathbf{U}_{\mathrm{A,B}} = (\mathbf{S}_{\mathrm{B}} - \mathbf{S}_{\mathrm{A}}) \cdot \Delta \mathbf{T}$$

Most common conductors for temperature sensors applications:

Material	α (/°C) a 20°C	ρ (Ω.m) a 20°C	Seebeck (µV/°C)*
Silver	0.00380	1.59x10 ⁻⁸	1.5
Platinum	0.00392	1.06x10 ⁻⁷	5
Copper	0.00404	1.68x10 ⁻⁸	1.5
Nickel	0.00600	6.99x10 ⁻⁸	-20
Gold	0.00340	2.44x10 ⁻⁸	1.5

*For a temperature reference of 0°C

Table values extracted from:

https://www.engineeringtoolbox.com/resistivity-conductivity-d_418.html https://www.electronics-cooling.com/2006/11/the-seebeck-coefficient/





Thin film Vs Convencional Electronics

Advantages

- Fabrication of robust and compact devices without jeopardizing the accuracy of the sensors;
- Adaptability to several types of surfaces, allowing an easy integration of the sensors;
- Decrease of the time of response when compared to conventional devices.

Disadvantages

- Reading instabilities related to external factors, such as strain effects that result from the flexibility of the devices and can change the measured resistance;
- More rapid degradation (p.e. oxidation) of the film, resulting in losses of accuracy.



Conventional RTD¹



2. https://blog.wika.us/products/temperature-products/wire-wound-vs-thin-film-resistance-temperature-detector/



Thin-Film RTD²





Printed Temperature sensors

Parameters for materials, fabrication and sensing of various printed sensors:

Materials	Substrate	Process	Range (°C)	Sensitivity (%/°C)
PEDOT: PSS/graphene/EGC	PU	Inkjet	35-45	0.064/0.034
PEDOT: PSS/CNT	PI	Shadow Mask	22-50	0.61
PEDOT: PSS/DMSO	PEN	Inkjet	20-70	2.5×10^{-3}
PEDOT: PSS/CNT (3:1)	PET	Screen printing	26-45	0.89
PEDOT: PSS	SU-8	Inkjet	-20-50	0.018
PEDOT: PSS/CNT	PET	Screen printing	20-45	1.3
Ag	PI	Inkjet	20-60	2.19×10^{-3}
Ag	PET	Inkjet	0-100	1.076×10^{-3}
Ag	PET	Inkjet	30-100	0.1086 Ω/°C
Au, PTC & NTC pastes	PEN/PET	Screen printing	20-80	0.06 V/°C
Ag, Ni	PET	Inkjet, Electrode- position	-10-60	$1.82 imes 10^{-3}$
Flake graphite/CNT/PDMS	PET	Screen printing	40-80	0.028
Mn ₂ O ₃ /NiO/Co ₃ O ₄ /CuO/ZnOPVDF, PDMS, CYTOP	Ы	Screen printing	40-140	91.76% (full range change)
Ba'TiO ₃ , activated carbon, thermoset polymeric	PET	Screen printing	25-55	0.022
MoSe ₂ , Ag	Glass	Drop-cast	-0.15-99.8	~-0.51
Polylactic Acid-Carbon black	Free standing	3D Printing	25-36	-
Polyvinyl chloride/carbon black	PET	Screen printing	18-44	-0.148
Ag	Paper	Inkjet	-20-60	1.1×10^{-3}
Ag	Paper	Inkjet	20-80	1.63×10^{-3}
Ag & PEDOT: PSS	Paper	Inkjet	25-45	$\begin{array}{c} 0.938 \times 10^{-3} \ \& \\ -13.9 \times 10^{-3} \end{array}$

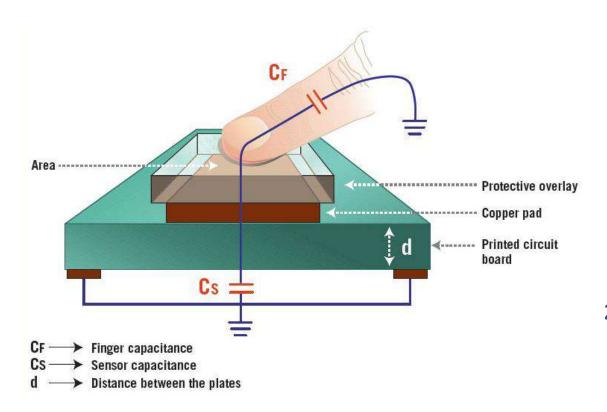
Barmpakos, D., Kaltsas, G. (2021) "A Review on Humidity, Temperature and Strain Printed Sensors—Current Trends and Future Perspectives", Sensors 2021, 21(3), 739



CAPACITIVE TOUCH SENSORS



Principle of operation



https://runtimerec.com/making-capacitive-touch-sensors-water-tolerant/

1. Principle

- Capacitance is the amount of electrical charge accumulated in a system when an electrical potential is applied;
- When any object with capacitive characteristics-such as a finger-comes close to a capacitive touch sensor, it acts as another capacitor due to its dielectric nature. This varies the effective capacitance of the system, which is used to detect the touch

2. Main applications and materials:

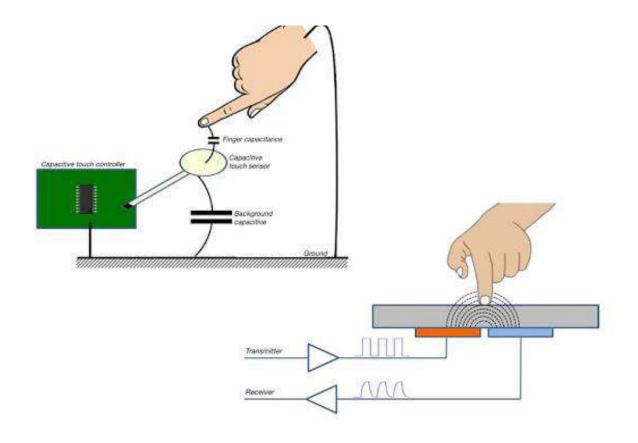
- Main uses: touchpads or switches;
- Materials: conductive material such as silver, copper or ITO are widely applied as electrodes in capacitive sensors;.



CAPACITIVE SENSORS



Self Vs Mutual



1. Two main types:

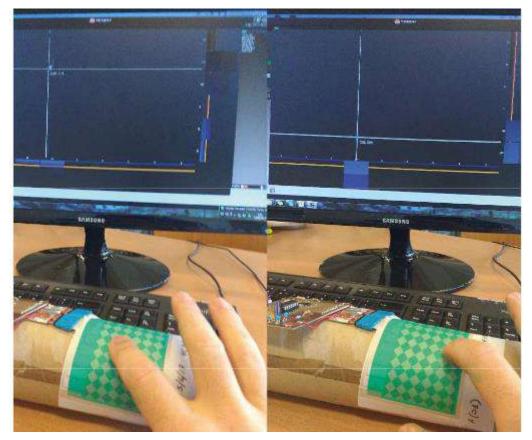
- Self-capacitive (top left image): self capacitance is the electric charge needed to raise the electric potential by one Volt. The interference of a finger changes this value;
 - o Ideal to use in systems with few sensors;
 - Simple use and less interference between close sensors.
- Mutual (bottom right): Two electrodes separated by a dielectric material have fixed capacitvie when a electrical potential is applied. The proximity of a finger (or other object) changes this value;
 - Ideal to used in systems with a lot of sensor;
 - Allows simple controlling electronics using matrix of sensor instead of individual sensors.



CAPACITIVE TOUCH SENSORS



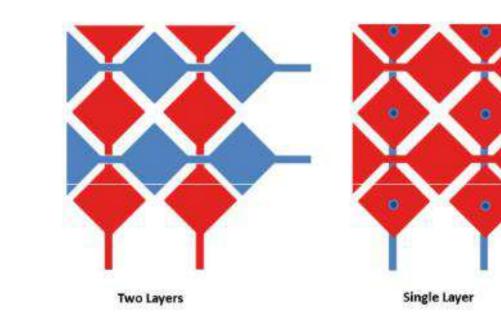
Sensors in textile substrates



Ferri et al., in SENSORDEVICES (2017)

A printed capacitive touchpad printed in textile substrates:

- 2 different 9x6 matrix design used:
 - Two layers design (silver electrodes printed with a dielectric layer in between);
 - One layer design (all electrodes in the same layer).
- Effective response of the printed sensors.

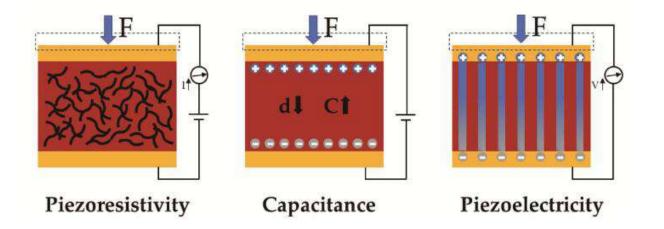




PRESSURE SENSORS



Principle of operation



Pressure sensors can be divided in three types according to their principle of operation **Piezoresistive flexible pressure** sensor is based on the change of pressure applied from the external force, which is reflected as a change in the corresponding resistance value;

Capacitive flexible pressure sensor is based on the change of the distance between electrodes when pressure is applied, which causes an alteration of the capacitance. These sensors tend to exhibit low sensitivity.

Piezoelectric flexible pressure sensor is based on the generation of electrical charges when mechanical stresses are applied. Piezoelectric sensors are good candidates for developing lowpower-consumption or even self-powered sensing devices.

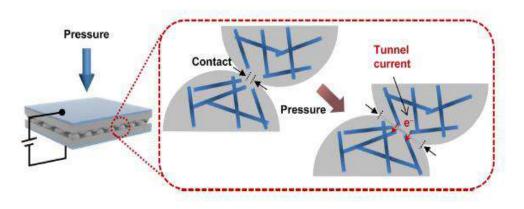


PRESSURE SENSORS



Flexible Pressure Sensors

- A typical configuration of these sensors consists in a "sandwich" structure composed of two conductive electrodes and an active layer in between them;
- Common substrates used in flexible electronic devices include PDMS, polyethylene terephthalate (PET), polyimide (PI), and polyethylene naphthalate (PEN).
- PDMS polymer is the common choice to work as the active layer and can be doped with carbon nanotubes to improve its electrical properties;
- An important characteristic to retain is the <u>presence of voids</u> within the active layer.



^{*} Park, J., Lee, Y., Hong, J., Ha, M., Jung, Y. D., Lim, H., ... & Ko, H. (2014). Giant tunneling piezoresistance of composite elastomers with interlocked microdome arrays for ultrasensitive and multimodal electronic skins. *ACS nano*, *8*(5), 4689-4697.

Advantages:

Flexibility, low cost, and compatibility with large-area processing techniques;

• Great and stable electricity

Disadvantages:

- Instability in conductivity;
- High energy consumption

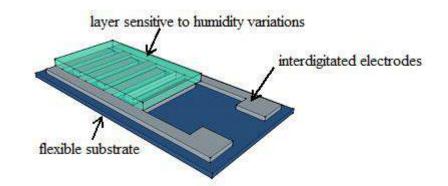


HUMIDITY SENSORS



Principle of operation

- Humidity sensors can be based on a capacitive or resistive principle.
- In the first case, the change in relative humidity is measured from the change in capacity of the resulting sensor, while in the second case the change in impedance with the humidity of the medium is the reading parameter.
- For both types, the sensors have a conductive layer of interdigitated electrodes (IDE), on which a layer of a material sensitive to humidity variations can be deposited, in order to promote the sensitivity of the sensor.



Material	Principle	Deposition
PDMS (Dimetil polissiloxano)	Capacitive	Drop-coating
CAB (<i>Cellulose acetate butyrate</i>)	Capacitive	Inkjet printing
pHEMA (Polyhydroxyethylmethacrylate)	Capacitive	Gravure printing
Nafion	Capacitive	Inkjet printing
PI (polyimide)	Capacitive	Película
PANI (polyaniline)	Resistive	Drop-casting
PMMA	Resistive	Dip-coating
PEDOT:PSS	Resistive	Spin-coating





Printed pH sensor

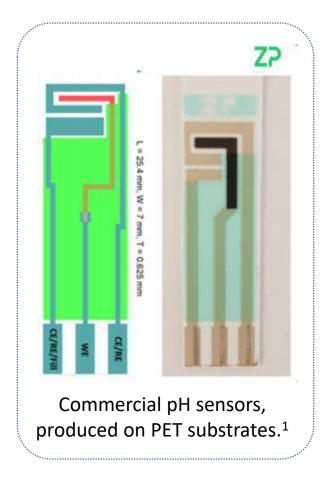
- Printed pH sensors can be potenciometric, capacitive, chemiresistive and luminescence.
- Potentiometric pH sensors are the most common, and they operate by measuring the electromotive force between a work and reference electrode They allow the creation of sensors with a reduced size, with a low fabrication cost is low.
- The reference electrode is used to maintain a constant electrical potential, regardless the sample composition and temperature, with a mix of silver/silver chloride being normally used, due to its potential stability and environmental friendly;
- The work electrode is responsible for creating a electrical potential, proportional to the activity logarithm of an ionic species. PANI is widely used as an ion selective membrane due to its high conductivity, durability, environmental stability, and low cost. It can be drop casted or electrodeposited.
- An insultation layer must be applied in order to protect the sensor.

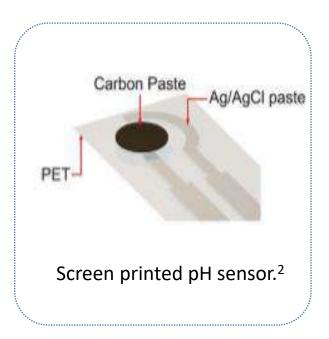






pH sensor - examples





¹pH Sensors - zimmerandpeacock (zimmerpeacocktech.com)

²Park H., Yoon J. et al "Potentiometric performance of flexible pH sensor based on polyaniline nanofiber arrays", Nano Convergence (2019) 6:9



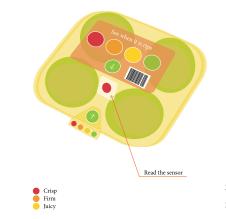


Colorimetric pH sensor

- The colorimetric method is based on the property of acid-base indicator dyes, which show a specific color according to sample pH;
- These type of sensors can be used in intelligent food packaging. Although its simplicity and low cost, they are instable, and the printing reproducibility is challenging;
- The development of colorimetric pH sensors implies the combination of two main components: polymers, and sensitive dyes. For example, the use of chitosan, as a polymer, and anthocyanins, as a dye, allows the production of a biodegradable and biocompatible pH sensors.



Yoshida, C., Maciel V. et al, "Chitosan biobased and intelligent fi Ims : Monitoring pH variations. LWT", Food Science and Technology (2014), 55(1), 83–89.



Fuertes G., Soto I. et al "Intelligent packaging systems: sensors and monitor food quality and safety", Journal of Sensors (2016)





Principle of operation

Conductivity sensors indirectly measure conductivity by generating an electric field in the test solution and reading the circuit it creates;

- Resistivity sensors measure the electrical resistance of the solution, considering the system a resistor;
- Capacitive sensors measure the capacitance of the solution between the electrodes, that acts like a capacitor;
- Biosensors measure the scale of a specific enzymatic reaction and correlate it with the conductivity of the solution;



Conductivity sensors from Metrohm <u>https://www.metrohm.com/en/products/electrochemistry/electrochemistry-electrochemistry/electrochemistry-</u> <u>electrodes/#</u>



IONIC CONDUCTIVITY SENSOR



Ionic conductivity sensor

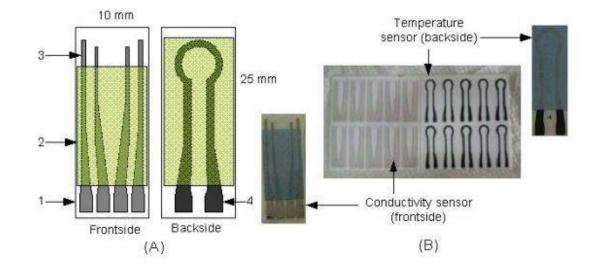


Figure 2. (A). The design of thick film conductivity and temperature sensors on a 10 mm x 25 mm alumina substrate: [1]. Alumina substrate (Al₂O₃), [2]. Dielectric layer, [3]. Elecrodes (AgPd), [4]. Temperature sensor (RuO₂), (B). The resulting conductivity and temperature sensor device after manual cutting.

Wiranto, Goib & Hermida, I Dewa Putu & Hiskia, & Rama, Beni & Rusdiana, Dadi. (2017). Liquid Conductivity Sensor Based on AgPd Paste Fabricated on an Al2O3 Substrate Using Screen Printing Technique. Materials Science Forum. 887. 108-115. 10.4028/www.scientific.net/MSF.887.108.

- This resistive sensor measures the resistance of the solution as part of an electric circuit, by applying an electric field;
- The electrodes are made of a AgPd paste printed on an Al₂O₃ substrate for better behavior of the circuit;

While the circuit resistance is good, it can only be used on low concentration solutions in order to minimize possible electrochemical reactions;

 4 electrodes are used for the measurement. The outer ones generate the electric current, while the potential difference is read using the inner ones, minimizing the effect of ionic polarization near the sensing electrodes;

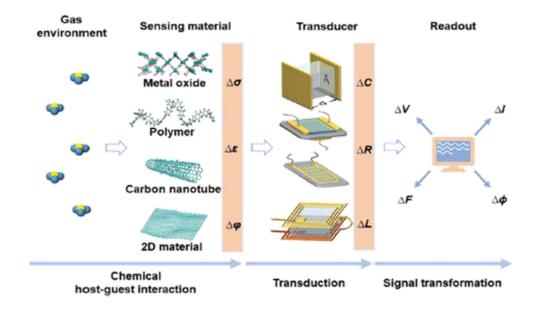
• The backside is a temperature sensor, necessary to correctly calibrate the conductivity sensor;





Principle of operation

- Gas sensors are typically classified according to their operating principle of signal:
 - •Optical gas Sensor.
 - •Electrochemical gas Sensor.
 - •Acoustic based gas Sensor.
 - •Thermometric gas Sensor.
 - •Gravimetric gas sensor
- Electrochemical gas sensors are the most investigated, and are composed of two main components: the sensing material and the transducer;

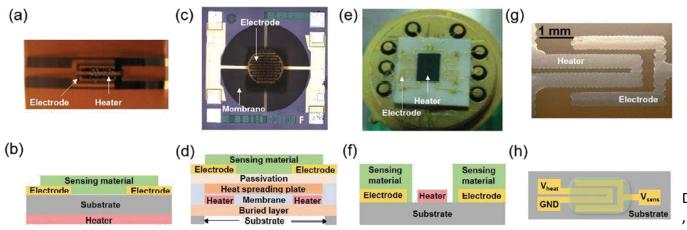






Printed gas sensors

- The printed devices are predominantly chemiresistive sensors, due to their relatively simple configuration and working principle these sensors.
- Consists of one to several pairs of electrodes with a layer of sensing material deposited on top of.
- A constant current or potential is applied across the device, and upon adsorption or desorption of a gaseous analyte, the electrical resistance or conductance of the sensing material changes, facilitated as the output.
- Despite the simple setup, chemiresistors are limited by their single type of output, that is, the resistance, which is easily influenced by environmental perturbations.

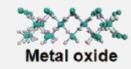






Sensing materials

- Gas sensing materials are generally conducting or semiconducting in nature and undergo changes of electrical properties upon exposure to gas.
- Examples: metal-oxide semiconductors, conducting polymers, carbon nanotubes and 2D materials.



Detection: Metal oxides (example: NiO, SnO₂, Fe₂O₃, ZnO); Sensing mechanism: the gas induced charge transfer and doping, sometimes with surface reactions; Sensitivity: polar or reducing/oxidizing gases.



Detection: organic vapors; Sensing mechanism: Their tailorable molecular backbones, modifiable end groups and side chains make their electrical properties and surface chemistry highly tuneable toward specific gas response.



Detection: strong electron receptor or donator gases (NH_3 and NO_2) and less reactive gases at room temperature, (H_2 and H_2S);

CNTs possess good chemical and mechanical stability, excellent electronic properties, and ultrahigh surface to volume ratios.





Electroluminescent gas sensor

- Electroluminescence (EL) is an optical phenomenon of non-thermal light emission under a strong electric field. The EL devices operating with alternating current (AC) have attracted much attention because of high lighting efficiency, uniform light emission over a large area, high flexibility, simple device structure and low production cost;
- The use of sensitive materials with AC-EL causes a variation in light depending on the amount of gas

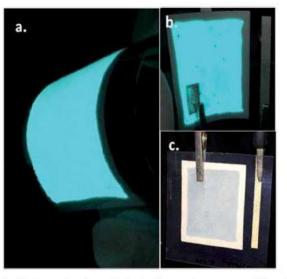


Fig. 2 Photographs of an electroluminescent gas sensor: (a) under bending on ITO side and without bending on PEDOT:PSS side with (b) power-on and (c) power-off.

En-on, J. *et al* (2017) "Flexible alternating current electroluminescent ammonia gas sensor" RSC Adv. 7, 16885

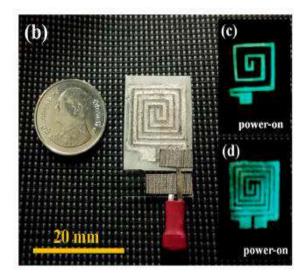


Fig. 2. (b) power-off and power-on (c) before and (d) after coating of graphene sensing layer.

Seekaew, Y. and Wongchoosuk, C. (2019) "A novel graphene-based electroluminescent gas sensor for carbon dioxide Detection" Applied Surface Science 479, 525–531.





Wearables



Penn State and Northeastern University

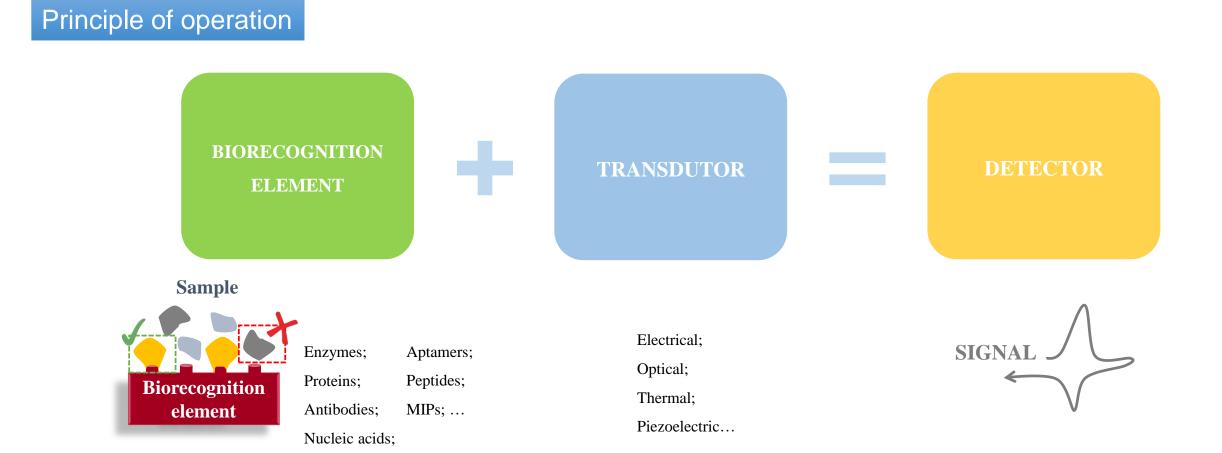
A wearable gas sensor can monitor environmental and medical conditions.

The nanomaterials used in this work are reduced graphene oxide and molybdenum disulfide, or a combination of the two; or a metal oxide composite consisting of a core of zinc oxide and a shell of copper oxide





- Biosensors are compact analytical devices made with biorecognition and transducing elements.
- These devices are especially suitable for local applications when build with an appropriate size, and on a portable fashion, producing quick responses, with low cost.







- Biosensors allow quick diagnosis and application over wide disease screening programs, being especially suitable for biomedical monitoring.
- Biosensors have been emerged as a promising alternative to the conventional methods, once they allow detecting and quantifying specific analytes, eventually in point-of-care.

Conventional methods



ELISA assay

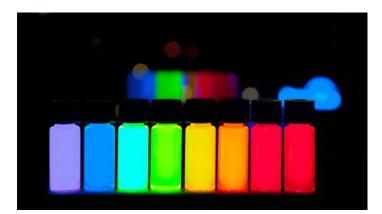
(Enzyme-linked immunosorbent assay)

www.jove.com/v/5061/the-elisa-method



Chromatographic methods (HPLC, GC, GC-MS, among others)

www.ajvs.com/new/about_us.php



Fluorescence spectroscopy

www.nersc.gov/news-publications/nerscnews/science-news/2015/quantum-dot-blinking/

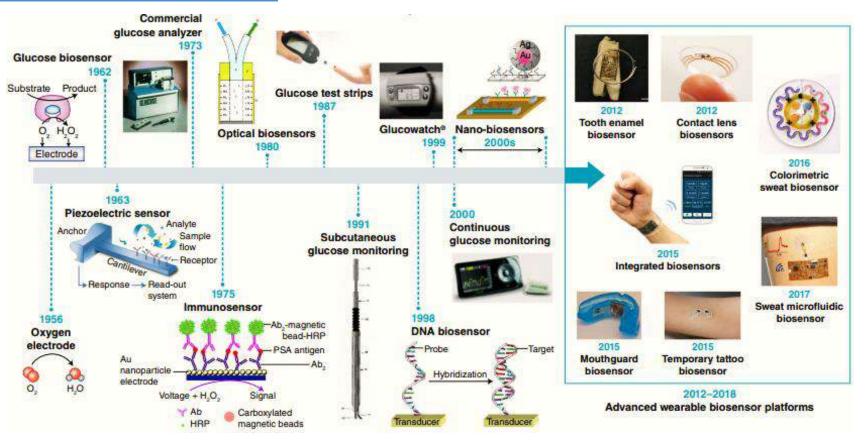
Among other conventional methods....





Wearable biosensors have been emerged as a potential sensing devices that incorporate a biological recognition elements into the sensor operation, allowing a continuous, real-time and non-invasively monitor of physiological information.

Advances of biosensors



Modern WEARABLE BIOSENSORS

 $\hat{\Gamma}$

real-time and non-invasive biomonitoring applications



Blood monitoring biomedical devices used for a wide range of healthcare applications.

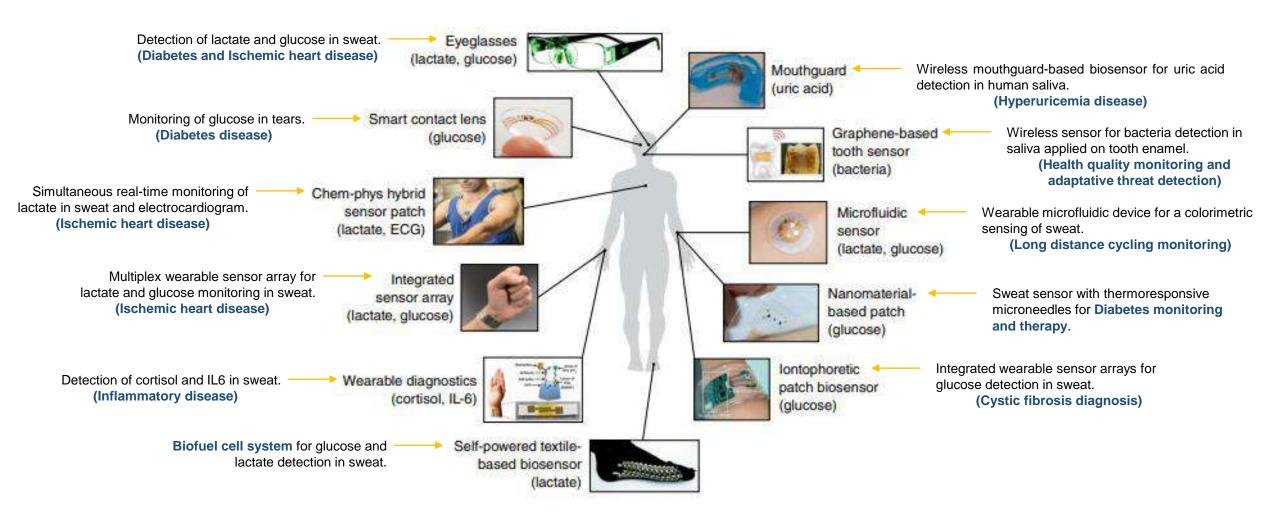
Historical path of biosensing technology advances for wearable biosensors.







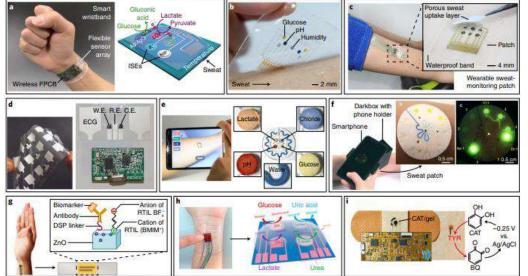
Wearable biosensors



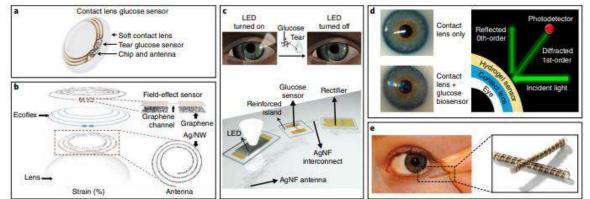




Wearable biosensors



Tear-based biosensors



Epidermal biosensors for real-time monitoring of

biomarkers present in sweat

Iontophoresis electrode WE R./C.E. Anodic Cathodic compartment compartment Glucos . 0 0.0 Gluconi hine intophoresis electrodes PVB re **Dual** iontophoresis 1 cm

Epidermal iontophoretic biosensors



Sensor R&D @CENTI

iPVest INTELLIGENT PROTECTIVE VEST



Topics:



Main Objectives 1.

- \geq Development of multifunctional textiles and intelligent hybridization for technical and functional clothing of multi-risk protection, as well as sensing system, control hardware and firmware, interface system and software and transmission of the data.
- 2. **CeNTI R&D** specific contributions to the main objectives
 - Development of the sensor system, hardware and control \succ firmware;
 - Development of interface system and data transmission \geq software;





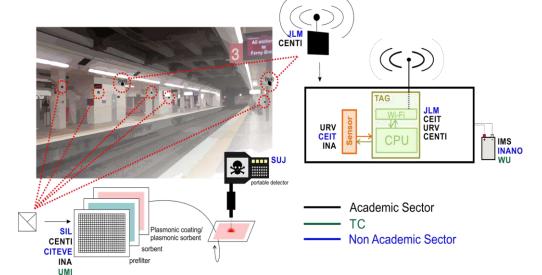






Sensoft





Topics:

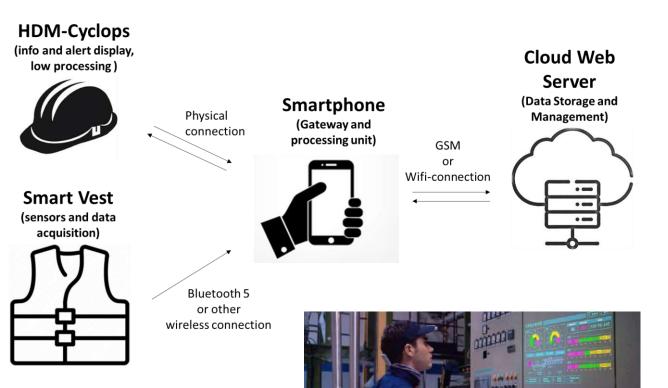


Main goals:

- Develop rapid field screening systems capable of monitoring the presence of gaseous species in the air in closed environments, to carrying out an identification of the chemical risk (HCN, Sarin and BTEX as selected molecules);
- Generate a real-time rapid alert alarm on the spot, to improve the perception of the situation (a few minutes after the attack);
- Knowledge transfer between the scientific community and SME partners, to bring scientific knowledge to industry and the market.



HARPSENS Head Mounted AR Platform and Smart Vest System



Topics:



1. Main Objectives

Developed a fully integrated headset with high performing mobile computer, advanced computer vision software, novel plug & play sensing capabilities, and battery management;

2. CeNTI R&D specific contributions to the main objectives

CENTI

- Develop a universal communication protocol a diverse set of sensors for plug & play functionality;
- Develop multiple sensor kits and drivers utilizing universal communication protocol;
- Develop a small communication module able to be seamlessly integrated into the textile structure of the vest;

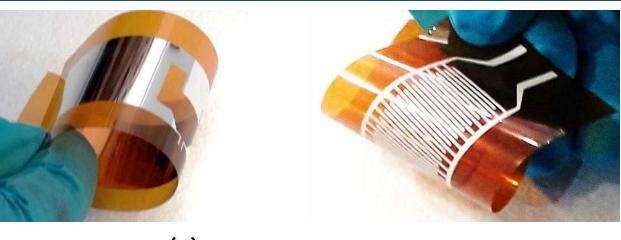


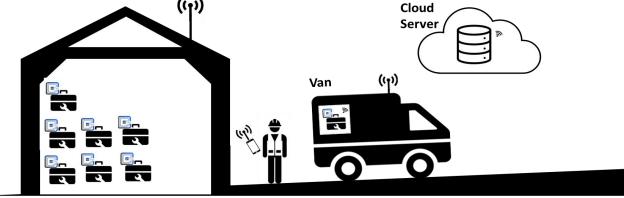
SCOOL





Vi-TAG







Topics:



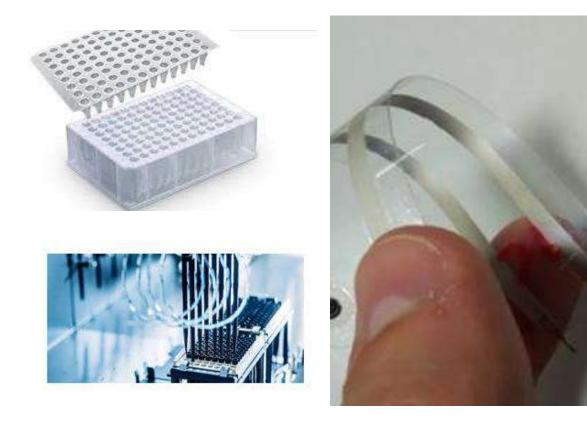
1. Main Objectives

- Creation of a new and unique inventory monitoring and control system between warehouses, factories and mobile transport units.
- 2. CeNTI R&D specific contributions to the main objectives
 - Implementation of a UHRF architecture for communication and low energy harvesting systems;
 - Printed sensors for measuring parameters such as humidity, temperature and position, among others, to monitor transported box items

COMP



SR4SB Smart Robotics for a Smarter Biotechnology



Topics:

Main Objectives

Develop a cross-cutting system based on smart electronics to apply to biotechnological downstream processes. It will consist of a multi-functional robotic device equipped with the sensors to actuate a wide range of biotechnological process.

CeNTI R&D specific contributions to the main objectives

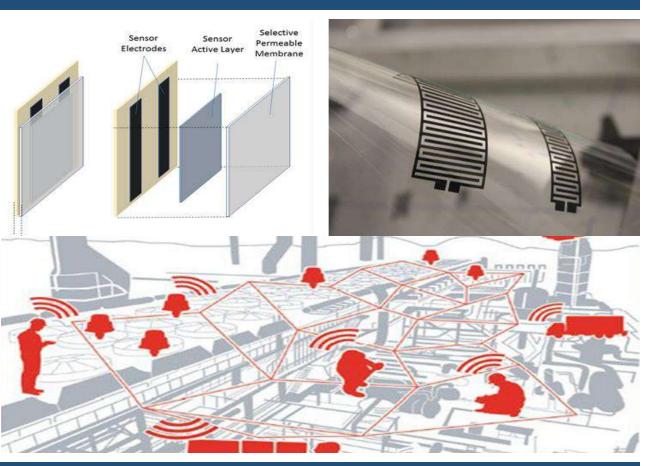
- Development of printed pH sensors for falcon tubes used in chromatography techniques and high throughput downstream processes;
- Development of printed flexible ionic conductivity sensors for falcon tubes used in chromatography techniques and high throughput downstream processes;
- Development of volume sensors for 96-well plates used in the downstream of antibodies development;
- Sensors integration in the robotic system, including the development of data acquisition firmware.







Wisen





Topics:

Main Objectives

Create an integrated solution of sensing and communication systems in industrial environments, namely for a waste management plant via.

CeNTI R&D specific contributions to the main objectives

- > Optimization of a wireless data transmission architecture;
- Development of new sensors and respective integration solutions;

