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Preparation of double-doping $LaFeO_3$ thin film for ethanol sensing application

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Abstract:

Recently, perovskite-based nanomaterials are used in diverse applications. Many perovskite nanoparticle-based devices have been demonstrated with extraordinary sensing performances to various chemical species in both solid and solution states. In this context, a new ethanol gas sensor $La_{0.8}Ca_{0.1}Pb_{0.1}FeO_3$ has been developed. $La_{0.8}Ca_{0.1}Pb_{0.1}FeO_3$ film has been prepared using the drop coating method onto SiO_2/Si transducers with platinum interdigitated electrodes based on the use of nanopowders which are prepared by sol-gel method. X-ray diffraction (XRD) showed that our sample was still perovskite phase with orthorhombic structure with $Pnma$ space group. The electrical properties showed the P -type semiconductor behavior of our sensor and the ethanol sensing measurements revealed his good sensitivity towards C_2H_5OH -gas. The response ($S = R_g/R_a$) was found to increase from 2.040 to 3.250 when concentration increased from 5 ppm to 30 ppm at the operating temperature of 250°C.

Keywords : $La_{0.8}Ca_{0.1}Pb_{0.1}FeO_3$, films, sensors, ethanol, response, 5 ppm.

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I. Introduction :

Perovskite materials (ABO_3), where A is a rare earth and B is a transition metal, are very used as gas sensors materials due to their thermal stability at higher temperature [1-4]. One of these functional materials is $LaFeO_3$ which is P -type semi-conductor material in air [5, 6]. In the surface of this sensor, oxygen adsorption enhances the conductivity of these materials on account of the increase of holes concentration, which are the main charge carrier species in P -type semiconductor [7]. For this reason, the resistance of these materials increases by applying reducing gases like ethanol [8]. In literatures, many studies have reported that $LaFeO_3$ is characterized by higher resistance which present a

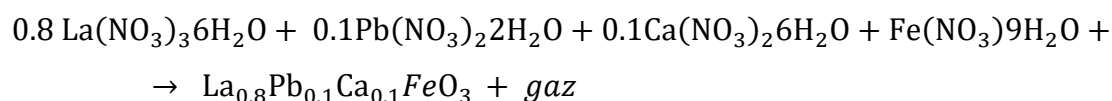
disadvantage in the gas sensing application and the partial substitution of *La* by lower valent cation elements, such as *Pb* [9] and *Ca* [10] could reduce the resistance of the mother compound. Besides, and using Kroger-Vink defect notations, its charge carriers are holes h^\cdot , which are produced by the ionization of La^{3+} ions vacancy defect $[V_{La}^x]$ and the ionization reaction is $(V_{La}^x \rightarrow V_{La}'' + 3h^\cdot)$. When La^{3+} ion is substituted by Pb^{2+} one, the ionization reaction of this element is $(Pb_{La}^x \rightarrow Pb_{La}' + h^\cdot)$ (where Pb_{La}^x is related to the point defect which is formed when La^{3+} is replaced by Pb^{2+} in the crystal and (h^\cdot) are the generated holes). As a result of $[Pb_{La}^x] \gg [V_{La}^x]$, the mobility of holes increases, and consequently the resistance of the doped compound decreases compared to the mother compound $LaFeO_3$ [11].

In this context, the aim of this work is to study the effect of introducing *Pb* and *Ca* at La-site in the ethanol-sensing properties of $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ sample.

II. Experimental

II.1. $La_{0.8}Ca_{0.1}Pb_{0.1}FeO_3$ nanopowder and film preparation:

The $La_{0.8}Ca_{0.1}Pb_{0.1}FeO_3$ sensitive layer has been prepared using the drop-coating method at room temperature on SiO_2/Si transducer with platinum interdigitated electrodes. Our preparation was started by preparing nanopowder by the sol-gel method using nitrates: $La(NO_3)_3 \cdot 6H_2O$, $Pb(NO_3)_2 \cdot 2H_2O$, $Ca(NO_3)_2 \cdot 6H_2O$ and $Fe(NO_3)_3 \cdot 9H_2O$ as precursors according to the proportion indicated in the equation below:



After that, we add the polyethylene glycol (PEG) and citric-acid in order to ensure homogenization and gel formation. Before, the obtained powders are calcined at 300 °C, 600 °C and 900 °C. Besides, a slurry of 20 mg of perovskite mixed with 20 ml of 2-propanol has been prepared and ultrasonicated for 10 min at 40 °C in order to ensure a good homogenization for the mixture. Subsequently, the deposition of thin films has been assured by coating the substrate drop by drop. Films thickness is found to be equal to 300 nm and it was measured using a DEKTAK6M mechanical profilometer. This method is completed by annealing our films at 500 °C.

II.2. Gas sensing measurements:

The electrical and gas sensing measurements were measured using a test chamber with a volume of 2L which allowing the control of the operating temperature and the gas

concentration. Dry air was used as reference gas and voltage has been fixed to 0.05 V. The resistance measurements of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor were carried out in dry air and in presence of ethanol gas at various working temperatures from 200 °C to 280 °C. Ethanol concentrations were obtained by diluted the saturated-ethanol with different amount of dry air in order to provide a final ethanol concentration between 5 and 30 ppm. The sensing response was defined as $S = R_g/R_a$, where R_a defines the resistance in air and R_g is the resistance in presence of ethanol gas.

III. Results and Discussion :

III.1. XRD and Morphological studies:

The phase of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ nanopowder was performed by means of X-ray diffraction with $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) using the FULLPROF program. **Figure. 1** demonstrates that our sample crystallizes in the orthorhombic structure with the *Pnma* space group and we notice the existence of the Fe_3O_4 secondary phase. The lattice refined parameters a, b, c and the volume V are gathered in **Table. 1**. Based on the use of the Williamson-Hall's equation ($W - H$) [12] (Eq. 1), we estimated the average crystallite size D_{W-H} which is found to be equal to 83.023 nm (**Figure. 2.**)

$$\beta \cos(\theta) = k\lambda/D_{W-H} + 4\varepsilon \sin(\theta) \quad (\text{Eq. 1})$$

Where: θ defines the Bragg diffraction angle, β demonstrates the effective strain, k is the shape factor and the D_{W-H} presents the average crystallizes size.

Habitually, sensor materials with a smaller crystallite size are characterized by higher activity, which facilitates the chemisorption of oxygen species leading to an increase of the response of materials used for gas sensing application [2, 13]. For this reason, D_{W-H} value of our compound confirms its use for ethanol sensing application.

Figure. 3 and **Figure. 4** depict the SEM micrograph and EDS-mapping of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sample, respectively. The EDS study of our compound revealed that our prepared sample is composed of La, Ca, Pb, Fe and O elements.

III.2. Gas sensing properties:

In the following part, we investigate the response of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor in presence of ethanol gas with different concentrations 5, 10, 20 and 30 ppm.

In order to ensure the adsorption of our $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ nanopowders over the SiO_2/Si substrate, a view using the optical microscope, will takes place with a magnification of 1.25 to see the surface [14] (**Figure . 5**).

Operating temperature is one of crucial parameter to consider for the potential practicality application of gas sensors and sensing process, such as gas adsorption and desorption phenomena on sensors surface are subject to the sensors operating temperature [15]. General diagram of measurements setup used to characterize ethanol microsensors is presented in **figure 6**. $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor response $S = R_g/R_a$ as a function of time ($T = 220^\circ\text{C}$, $T = 240^\circ\text{C}$, $T = 250^\circ\text{C}$ and $T = 270^\circ\text{C}$), in presence of 30 ppm of ethanol reducing gas for an exposure time equal to 1 min, are presented in **Figure. 7**.

We can see that for temperatures equal to $T = 220^\circ\text{C}$ and $T = 270^\circ\text{C}$ the $R(t)$ curves doesn't return to the baseline. This variation depends on the amount of oxygen adsorbed on the surface because the electron trapped by the oxygen at the surface from the semiconductor increases the resistance by decreasing the major charge carrier (hole) concentration [16]. However, for a temperature equal to $T = 240^\circ\text{C}$ or $T = 250^\circ\text{C}$, we observe the presence of a reversibility of $R(t)$ curves.

Our captor has been exposed at different temperatures in order to identify the optimal operating one. From **Figure . 8** some observations can be selected:

- ❖ The $\text{C}_2\text{H}_5\text{OH}$ -gas response increases and reaches a maximum value at an optimum operating temperature equal to 250°C .
- ❖ The $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor demonstrates a *p*-type semiconducting properties because the resistance increases when reducing gas is introduced [17].

A typical $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor response to small ethanol concentrations is shown in **Figure. 9**. The variation of $R(t)$ demonstrates a good reversibility in presence of different amount of $\text{C}_2\text{H}_5\text{OH}$ -gas at the operating temperature of our sensor.

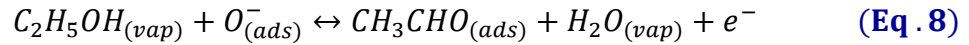
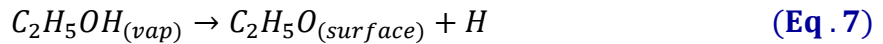
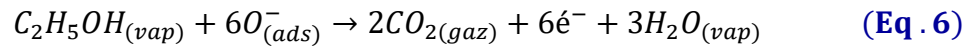
In addition, the sensitivity indicates the ability of a material to respond to low ethanol concentrations of 5 ppm compared to others perovskite materials [18, 19]. The response of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor at the operating temperature vs. $\text{C}_2\text{H}_5\text{OH}$ concentrations is depicted in **Figure. 10**. From this figure we can conclude that the intensity of the response peak increases monotonically with increasing gas concentration and consequently increasing the response (R_g/R_a).

As is well-accepted, the gas sensing mechanism of others materials such as metal oxide semiconductors involves an adsorption –desorption phenomena in which chemisorbed oxygen plays a crucial role [20]. In our case - case of perovskite materials- these two phenomena are ensured because $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor shows a good return to the

base line at the operating temperature which is equal to 250°C in presence of reducing gas. Reactions between adsorbed ionized oxygen species on the surface: O_2 , O_2^- , O^- and O^{2-} are expressed as follows **Eq(2-5)**:



When ethanol gas was introduced, recombination phenomena between electrons and holes are produced, inducing consequently a decrease in the density of holes-number in the $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ sensor. This phenomena leads to an increase in the electrical resistance of the compound during adsorption of the reducing gas **[21]**. Reactions that occur between C_2H_5OH gas and oxygen chemisorbed ions adsorbed in the surface are **[22, 23]** and the mechanism of the reaction is comparable to that mentioned by T. Tharsika et al. **[24]**:



Our $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ sensor response ($S = R_g/R_a$) confirms their use for ethanol sensing application. Indeed, $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ has been used on this application but in pellets forms by A. Benali et al. In addition, authors are used a very large amount of ethanol gas (250, 500 and 1000 ppm), but in this research paper we try to detect a small amount of ethanol reducing gas (5 ppm). Added to that the deposition of $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ in thin films forms leads to a decrease in the operating temperature from 280 °C **[2]** to 250 °C in case of our sample.

In the one hand, $BaSnO_3$ is a perovskite sensor which presents a higher response in presence of ethanol gas. Then, response is found to be equal to 5 at the operating temperature which is equal to 300 ppm for a concentration equal to 50 ppm **[25]**. In the other hand, our sensor presents a response equal to 2.040 in presence of very low concentration (5 ppm) of ethanol gas (**Table .2**). In addition, the mother compound $LaFeO_3$ presents a response equal to 3.8 in the existence of a concentration equal to 300 ppm which considered a very high concentration at 310 °C **[26]**.

Other important performance which is influenced by quantities of chemisorbed oxygen species is response time. Response time is defined as the time required to reach 90 % of the electrical resistance [27]. Response time values are gathered in **Table 2**, we can note that $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor shows low response time compared to other materials which are used for ethanol sensing application [28].

Conclusion:

A new perovskite $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ thin film has been prepared using drop-coating method for $\text{C}_2\text{H}_5\text{OH}$ -gas sensing application. The morphology and microstructure proved that our sensor can be usable for ethanol sensing application. The *XRD* pattern revealed that our sample crystallized in the orthorhombic phase with *Pnma* space group. The gas sensing properties of the double doped sensor LaFeO_3 by Ca and Pb showed that this captor could be a promising candidate for the detection of ethanol gas because of the detection of very low concentration equal to 5 ppm at 250°C. Then, 250°C corresponds to the best value of sensitivity that we have a maximum of chemisorbed oxygen at the surface.

Table 1: The Rietveld refinement results of X-Ray diffraction (XRD) data of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ compound.

	Space group	$a(\text{\AA})$	$b(\text{\AA})$	$c(\text{\AA})$	$V(\text{\AA}^3)$	$D_{w-H}(\text{nm})$
$\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$	Pnma	5.549 ₃	7.848 ₈	5.539 ₄	241.24	83.023
Fe_3O_4	Pbcm	11.868	11.851	16.752	589.03	-

Table 2: Response time of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor at 250°C compared to WO_3 sensor [28].

C (ppm)	5	10	20	30
S (R_g/R_a)	2.040	2.370	2.835	3.250
Response time (s) (PW)	200	190	183	146
Response time (s) (WO_3) [28]	580	-	-	-

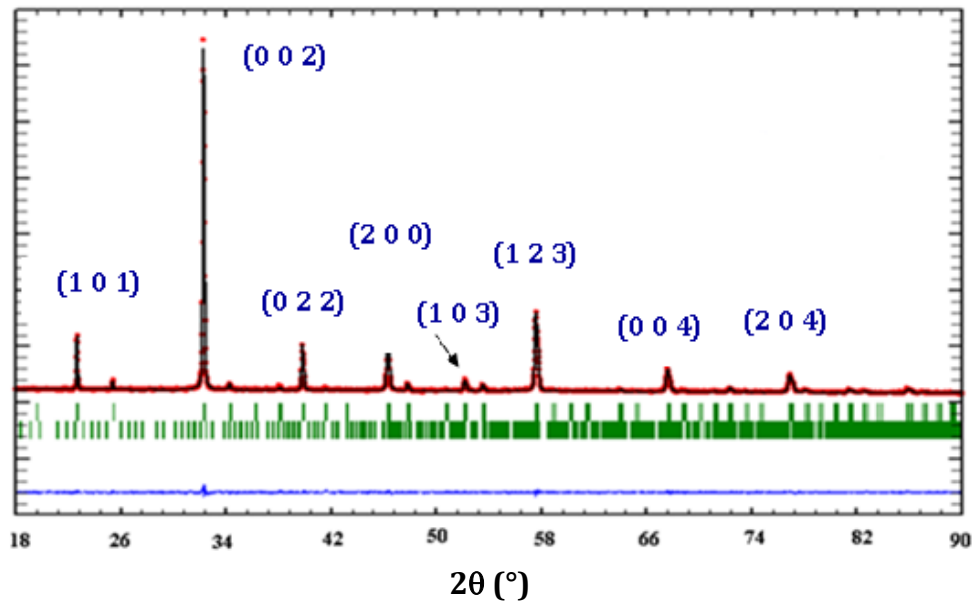


Figure. 1: Observed (red), Calculated (black solid line), their difference patterns (blue) and the Bragg positions (green) of $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ sensor and the secondary phase which is attached to the Fe_3O_4 impurity.

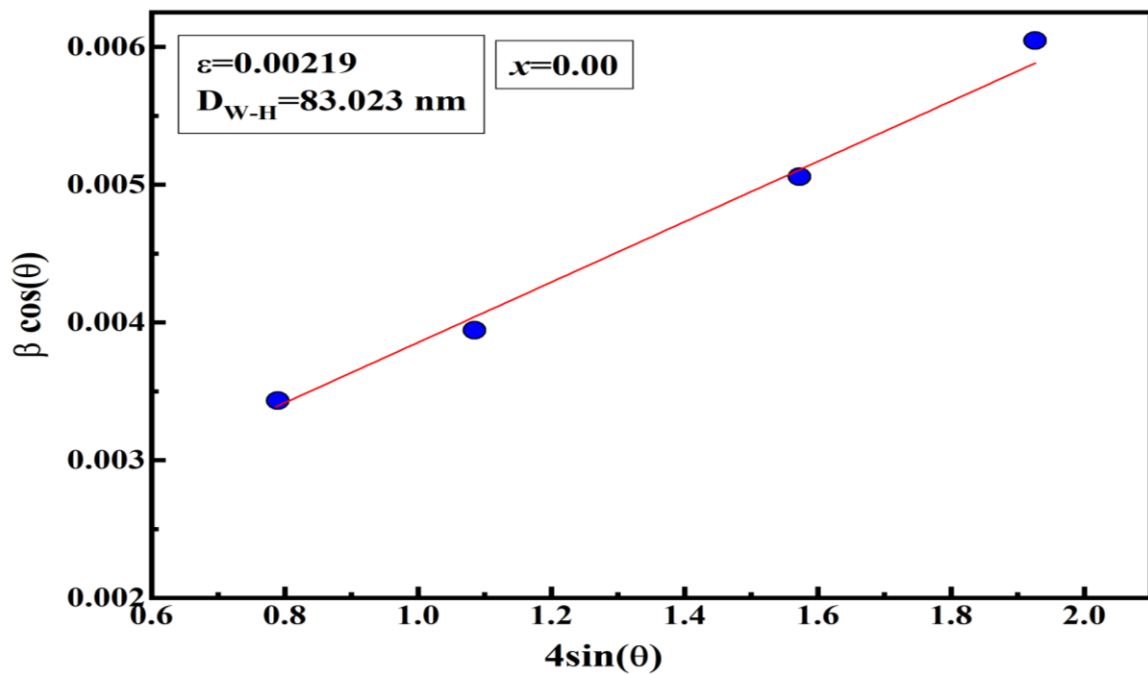


Figure. 2: Williamson-Hall plot of $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ compound prepared by the sol-gel method.

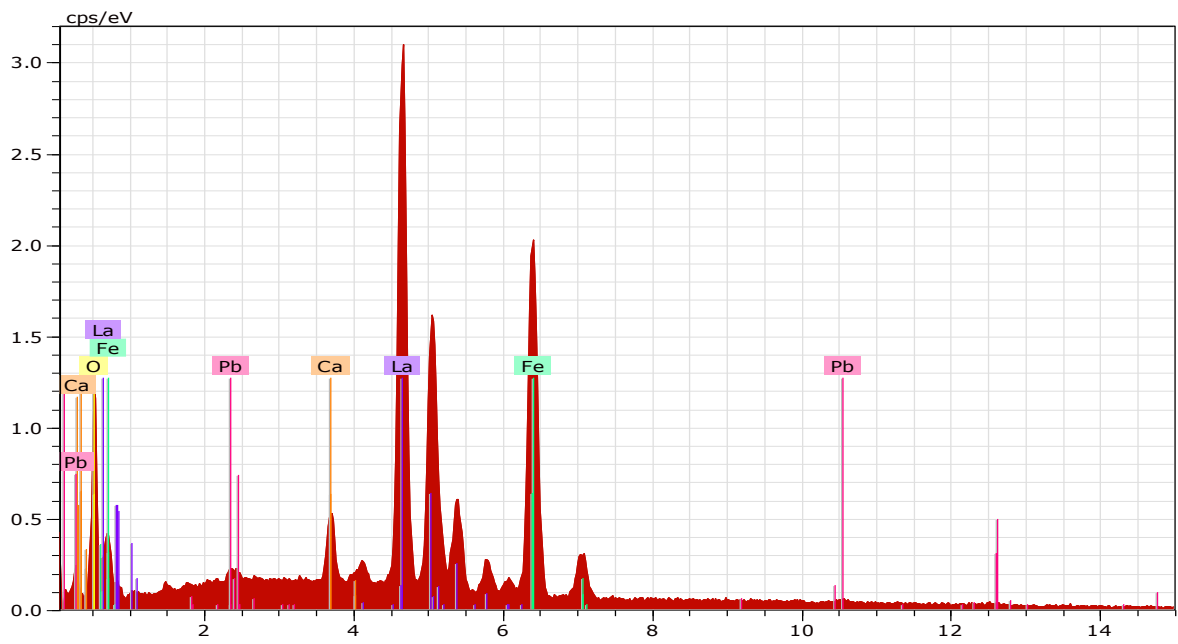


Figure.3: EDS-micrograph of chemical elements of $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ compound prepared by the sol-gel method.

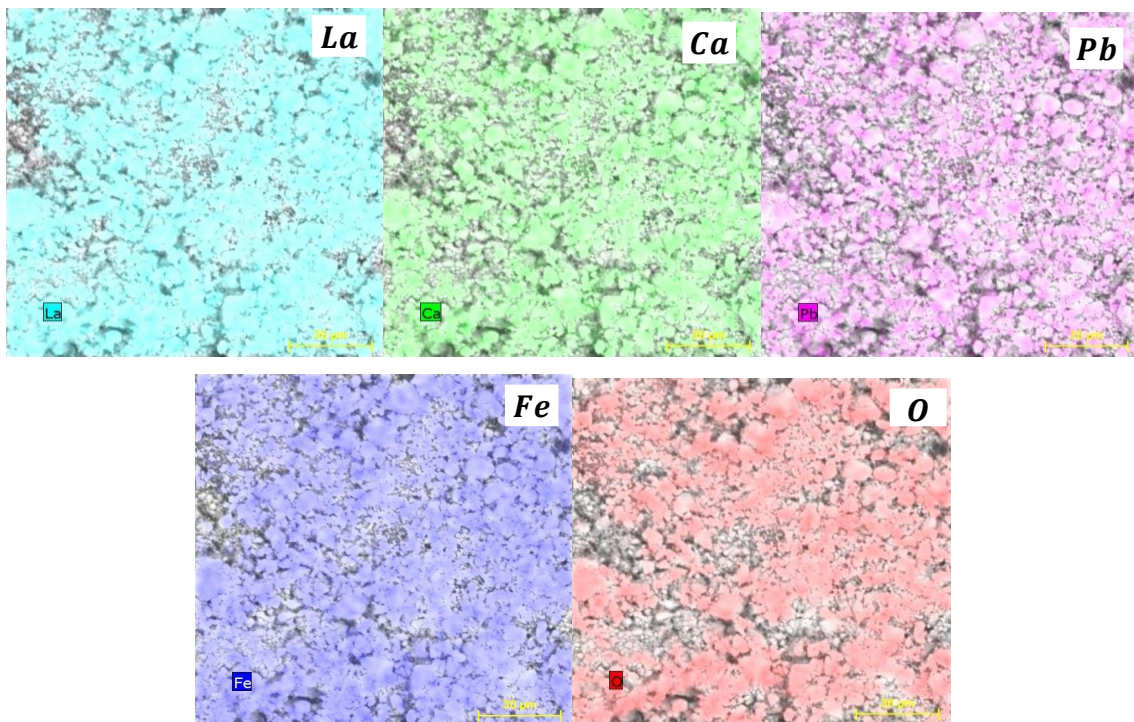


Figure. 4: EDS-mapping of chemical elements in $La_{0.8}Pb_{0.1}Ca_{0.1}FeO_3$ sample prepared by the sol-gel method.



Figure.5: Optical view after drop coating of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor.

Control and data acquisition system

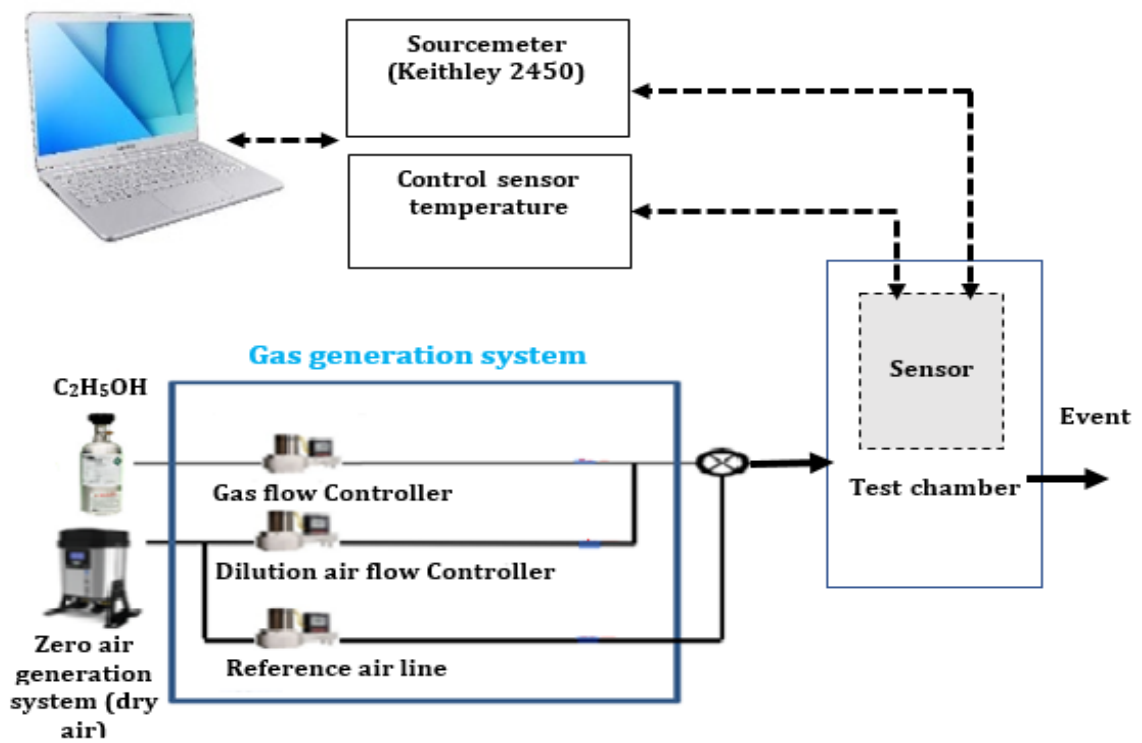
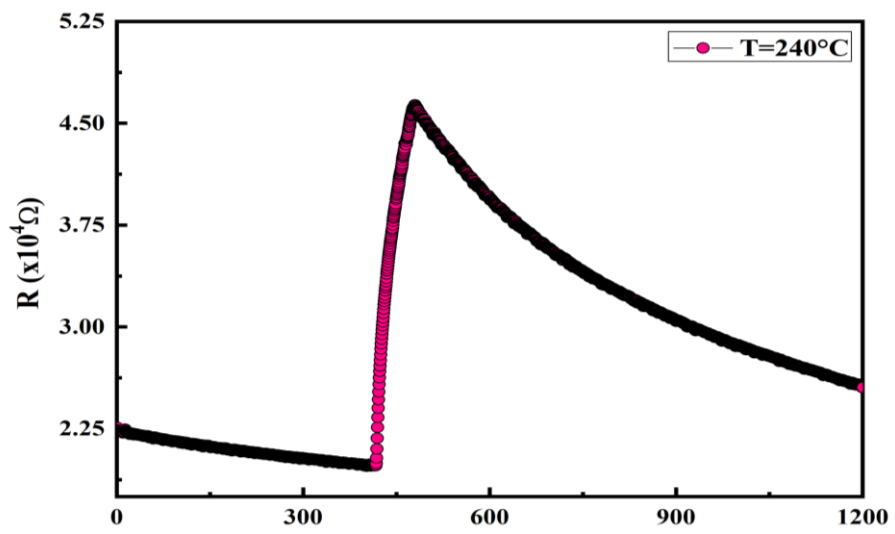
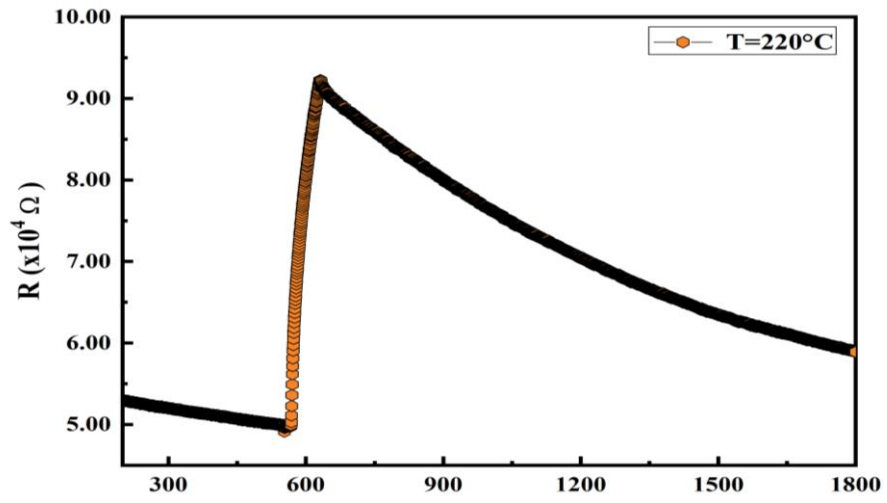


Figure 6: General diagram of measurements setup used to characterize ethanol microsensors.



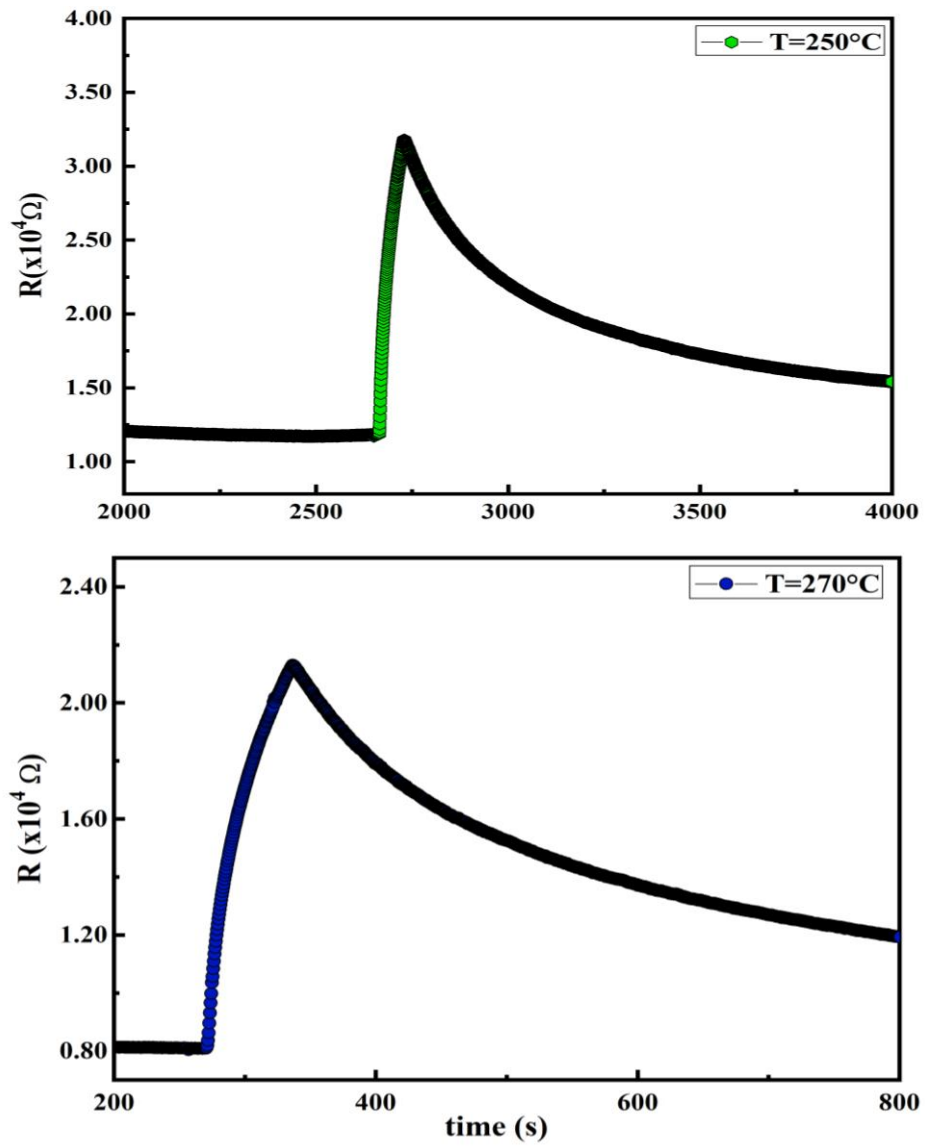


Figure. 7: Ethanol gas response of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor as a function of time at several temperatures ($T = 220^\circ\text{C}$, $T = 240^\circ\text{C}$, $T = 250^\circ\text{C}$ and $T = 270^\circ\text{C}$).

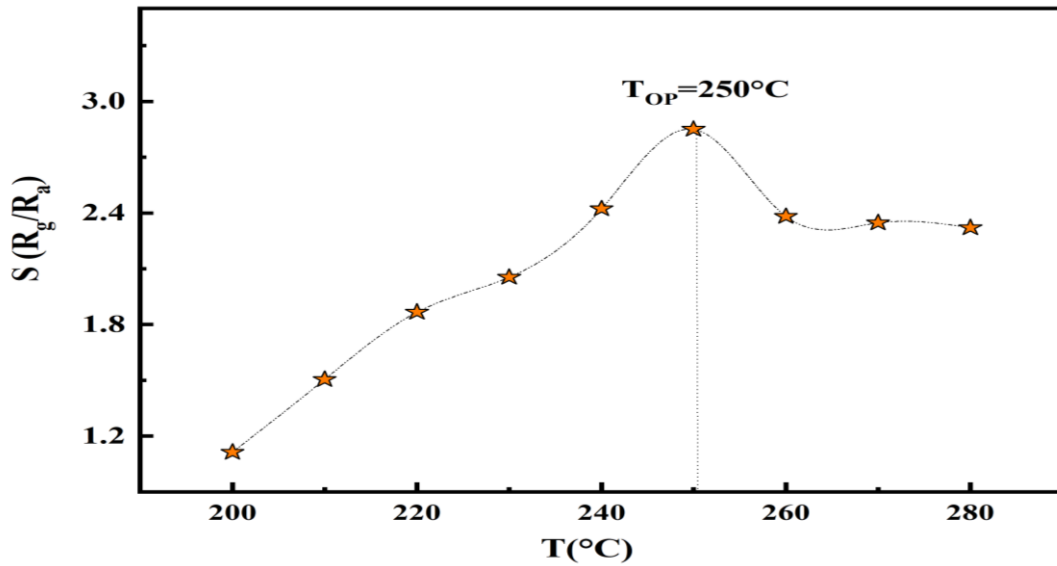


Figure.8: The relationship between response ($S = R_a/R_g$) vs. temperature (T) of $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ based sensor during ozone exposing time of 1 min for a concentration equal to 30 ppm.

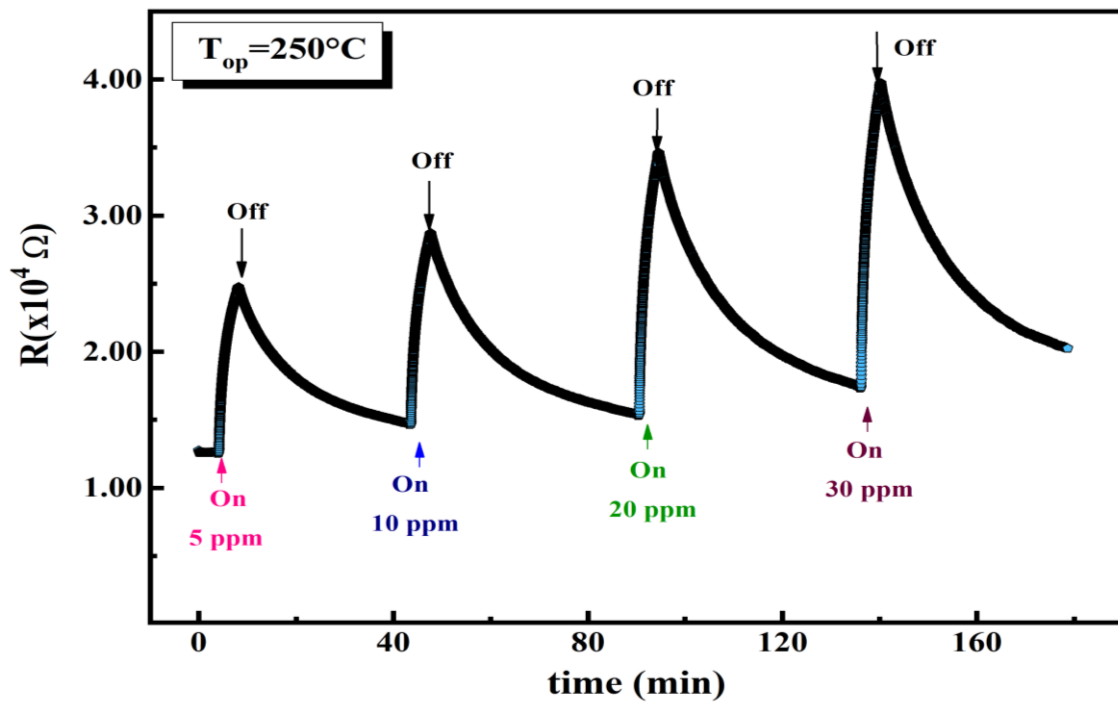


Figure.9: $\text{La}_{0.8}\text{Pb}_{0.1}\text{Ca}_{0.1}\text{FeO}_3$ sensor response (S) to different concentrations (5,10, 20 and 30ppm) of ethanol gas ($\text{C}_2\text{H}_5\text{OH}$) at $T_{op} = 250^\circ\text{C}$ and for an exposure time equal to 4 min.

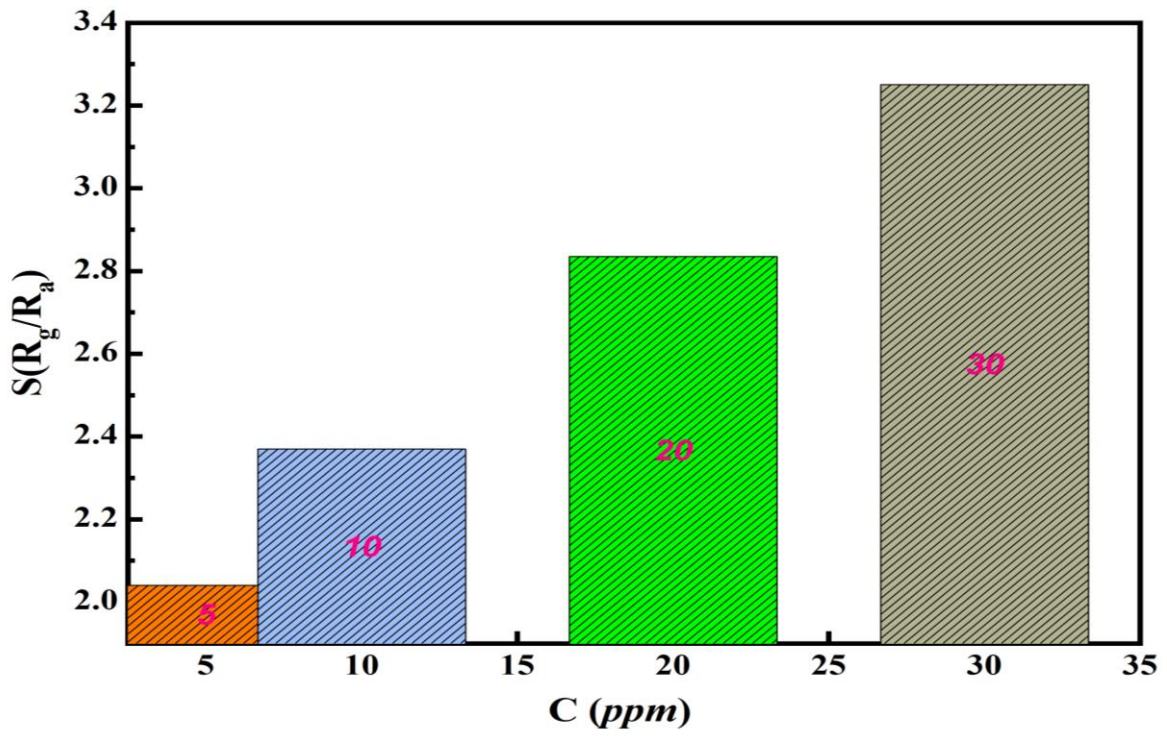


Figure. 10: Response ($S = R_a/R_g$) vs. ethanol-concentrations (5, 10, 20 and 30ppm) at the working temperature ($T_{op} = 250^\circ C$).

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