# A Novel Nano Structured $LaCa_xCr_{1-x}O_{3-\delta}$ for Humidity Sensors

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**Abstract:** ***Nano structured $LaCa_xCr_{1-x}O_{3-\delta}$ have been synthesized by a novel sol-gel method, in different mole ratios with x=0 , 0.2,0.4,0.6,0.8 and 1.0 and characterized by X-ray diffraction (XRD), Scanning electron microscopy (SEM) EDX and FTIR were employed to study the structural phases. The compounds were sintered at 700°C for 6 hrs and were subjected to DC resistance measurements at RH 5-98% and temperature dependent studies. The change in surface conductivity as a function of applied field was measured using picoammeter (Keithely-6485). Response and recovery times were measured.***

**Keywords**: Chromites; Perovskite; Ceramic oxides; Humidity sensor; semiconductors

## 1. Introduction

Sensor [1] is a transducer that converts one form of energy concerning which information is sought into another suitable form amneable for further processing. Although sensors of a great variety of types are well established in process industries, agriculture, medicine and many other areas, still development of sensing materials with high sensing capabilities are proceeding at an unprecedented rate. There are different types of sensors RTD (Resistance temperature detector) sensors, inductive proximity sensors, photoelectric sensors, humidity sensors, radar and ultrasonic sensors. Humidity sensors have been used in an increasing number of applications in industrial processing and environmental control. Growing demands for controlling water vapor have led to considerable interest in the development of sensing materials [2-6].

Measurement and control of humidity in an environment is important in both human comfort and many of industrial processes [7]. There are some major requirements for a good humidity sensor: high-sensitivity, reversibility, fast response time, long life time, high-humidity selectivity and stability [8]. Many kinds of materials have been studied as sensing elements/ compounds in humidity sensors. The principle sensor groups of interest include ceramics [9, 10], conductive polymers [11, 12] and electrolyte [7].

Nano metal oxides [13] play a very important role in many areas of chemistry, physics and material sciences. In technological applications, oxides are used in the fabrication of microelectronic circuits, sensors, piezoelectric devices, fuel cells, coatings for passivation of surfaces against corrosion and as catalyst. Nano mixed metal oxides have shown advantages in terms of their mechanical strength, resistance to chemical attack and their thermal and physical stability.

In the present investigation a novel nano structured $LaCa_xCr_{1-x}O_{3-\delta}$ (x=0 to 1) which was prepared by sol-gel method reveals both excellent humidity and thermal sensitivity properties. The novelty of this material is achieved by preparing the mixed metal oxide has a single phase compound which is done by sintering the samples for 8 hours at 800°C in tubular furnace. This supported by the XRD reports. The material is characterized by SEM, XRD, and IR. The humidity dependent electrical properties of Perovskite oxide $LaCa_xCr_{1-x}O_{3-\delta}$ have been studied by preparing different mole ratios of $LaCa_xCr_{1-x}O_{3-\delta}$ by altering the addition of $Cr^{3+}$ with $Sr^{2+}$. It has been found in the humidity sensing studies that the material shows high sensitive factor by the substitution of $Cr^{3+}$ with $Ca^{2+}$ due to the change in the surface morphology of the material. Electrical conductance can occur through internal surface layers of adsorbed water [13], the tiny pores can fill with water as determined by the Kelvin equation [14] leading to electrolytic conduction through the moisture or the conductance of the semiconductor itself can change due to an interaction of its surface energy states with hydroxyl radicals, especially at the grain boundaries [15,16].

## 2. Literature Survey

Semi-conductive materials have been extensively used as the basis for humidity sensors, either as thin films, or porous pellets [17]. The monitoring of humidity in the air falls under another section of analytical chemistry which has a great impact on the highly industrialized world.

Zhuyi Wang et al studied the effects of K- substitution at La-site of La1-xKxCo0.3Fe0.7O3-δ Perovskite on its structure and humidity sensing properties in detail.
Yong Zhang et al synthesized a humidity sensor Bi0.5Na0.5TiO3-Bi0.5K0.5TiO3 (BNT-BKT) powder by a metal-organic decomposition method and characterized by Fe-SEM, TEM &XRD. The results indicate that BNT-BKT powder is of potential applications for fabricating high performance humidity sensors [18].

B.C. Yadav et al deposited a nano crystalline (Mg-Zn-Ti) oxide films on an equilateral prism using sol-gel spin process. The humidity sensing properties of the films were

investigated at different angles of incidence. It was observed that the intensity of reflected light increased with an increase in relative humidity (RH %) in the range 10-95 % at a particular angle of incidence, which is quite significant for sensor fabrication purposes. Response and recovery time of the sensor were 18 & 30 s respectively.

Apart from humidity sensors, some of the mixed metal oxides have been used in the field of solid oxide fuel cells and catalysts. Furtado et al studied the development of lanthanum chromites-based materials for solid oxide fuel cell interconnects. The best ones results, in terms of densification and homogeneity characteristics, had been gotten through multiple doping with calcium and strontium, and in sintering temperature conditions lower that the normally considered to pure or mono-doped lanthanum chromite-based ceramics.

Israel E. Wachs has analyzed the recent conceptual advances in the catalysis science of mixed metal oxide catalytic materials. The most significant discovery has been that amorphous metal oxide phases are always present and are the catalytic active sites for many applications of mixed metal oxide catalysts. This has resulted in a significant paradigm shift as to how mixed metal oxide catalytic materials function for different applications. This article reviews the instrumental advances and the resulting conceptual advances that have evolved over the past 25 years of the catalysis science of mixed metal oxide catalysts.

## 3. Previous Work

In the past few years many researchers have synthesized the sensor material as composites of transition metals/alkali/alkaline earth mixed metal oxides or metal oxides. In the present investigation the mixed metal oxide were synthesized as nano compounds and not as composites, and sol-gel method is employed to synthesize these mixed metal oxide which was very rarely used in the previous work. One more advantage of the present work is rare earth mixed metal oxides as humidity sensor material was not studied in the previous work [19-22].

## 4. Experimental Procedure

### 4.1 Sample preparation

Sol-gel method: A known amount of citric acid and ethylene glycol (AR) were mixed well by keeping it over a magnetic stirrer for 10 minutes around 150° C and to this the dissolved nitrate solutions of La $(NO_3)_3$ .$6H_2O$, Cr $(NO_3)_3$ .$9H_2O$ and Ca $(NO_3)_2$ of appropriate mole ratios were added and stirred for 30 minutes and the mixture was heated to 150° C till powder form is obtained. The resulting LCaC compounds were compacted to pellet at a pressure of 4 ton/sq.inch. The diameter of the pellet is 13 mm and the thickness is 2mm. These solid pellet were sintered at 800°C for 8 hrs in ambient air atmosphere. The samples were cooled down to room temperature at the natural cooling rate of the furnace. As the concentration of strontium increased at particular level was shown to enhance the sensitivity of humidity sensors. Since our interest is to prepare a nano material the compounds were synthesized specifically by sol-gel method.

**Table 1**: Resistance at RH 5% and RH 98%, Sensitivity factor, Energy of activation of LCaC compounds.

| *Sample Codes* | *R(Ω), at RH 5%* | *R(Ω), at RH 98%* | *$S_f$ ($R_{RH\ 5\%}/R_{RH\ 98\%}$)* | *$E_a$(eV)* |
|---|---|---|---|---|
| LCaC -1 | $3.33x10^6$ | $5.00x10^6$ | 0.66 | 0.102 |
| LCaC -2 | $5.00x10^9$ | $1.00x10^7$ | 500 | 0.083 |
| LCaC -3 | $2.00x10^{10}$ | $4.60x10^7$ | 435 | 0.092 |
| LCaC -4 | $2.50x10^{10}$ | $3.33x10^6$ | 7510 | 0.062 |
| LCaC -5 | $2.80x10^{10}$ | $3.46x10^7$ | 809 | 0.071 |
| LCaC -6 | $5.00x10^9$ | $1.00x10^8$ | 50 | 0.098 |

### 4.2 Characterization and Humidity Sensing Studies

Controlled humidity environments of relative humidity 5,31,51,79 and 98% were achieved by using anhydrous $P_2O_5$, saturated aqueous solution of Ca $Cl_2.6H_2O$, $Ca(NO_3)_2.4H_2O$, $NH_4Cl$ and $CuSO_4.5H_2O$ in a closed desiccators at an ambient temperature of 298K. Prior to the saturation of the pellets in the above buffers, the pellets were heated at 393K for 12 hrs to remove the adsorbed water. A degassed chamber of about 200 $cm^3$ was used for evaluating the response and recovery characteristics. This chamber has a provision for two-way inlet, one for transpiring the dry air and the other for moist air from a wet candle. Air drying was accomplished by transpiring the air stream through drying columns packed with anhydrous $CaCl_2$ and dry $P_2O_5$ connected in series. The resistance measurements in the dry air as well as in moist air alternatively helped to establish the response and recovery time of the compounds.

The DC electrical resistance at different relative humidity levels of the samples in the form of pellets was determined by a two- probe method as. The electrical contacts were made on the surface of the pellet by means of two thin copper wires affixed with silver paint. Given the high resistivity of the materials under investigation, the potential inaccuracy due to contact resistance is assumed negligible. The pellet was inserted in the middle of the Pyrex tube of 5cm diameter on which kanthal wire was uniformly wounded externally. The kanthal wire ends were connected to avarian to vary the temperature and a copper –constantan thermocouple kept at the pellet was used to measure the temperature of the sample. The electrodes were connected to DC power supply and the Keithley 6485 picoammeter in series. The temperature dependent conductance experiments in the temperature range of 120- 300°C under ambient conditions were carried out to determine the activation energies for electrical conduction of the samples using linearised form of the expression $I=Io\ exp^{-Ea/kT}$ where I is the current, Ea the activation energy , k the Boltzmann constant and T is the temperature.

The structural studies were carried out using a Philips X'pert diffractometer for 2θ values ranging from 10 to 80° using CuKα radiation at λ =1.54A°. The Fourier transform infrared (FT-IR) spectra were recorded with Perkin – Elmer spectrometer using KBr pellets whose thickness was about 1.3 mm. Each spectrum was collected at room temperature under the atmospheric pressure. The samples were dispersed

in spectroscopic grade KBr pellets and were scanned in the range of 4000-400 $cm^{-1}$. The surface morphology of the samples was observed on a Ultra 55 FESEM with EDS analyzer using gold coated samples.

## 5. Results and Discussions

### 5.1 X-Ray Diffraction Studies

The powder XRD patterns of LCaC - 1 to 6 compounds (Fig.1)showed the characteristic peaks corresponding to $LaSr_xCr_{1-x}O_{3-\delta}$ confirming the absence of impurities and presence of mixed oxide intermediate peaks of new phases in LCaC -2 to LCaC -5.The XRD pattern of LCaC -1 (JCPDS-75-0441) data corresponds to pure lanthanum chromite which is simple cubic system with 'a' value 3.880 whereas the XRD pattern of LCaC -6 (pure lanthanum calcium oxide) which is a known compound it does not match with the existing patterns. The XRD pattern of compounds LCaC -2 to LCaC -5 were studied by comparing LCaC -1 and LCaC -6.The uniqueness of these XRD patterns is a proof that the compounds LCaC -2 to LCaC -6 is of novel.

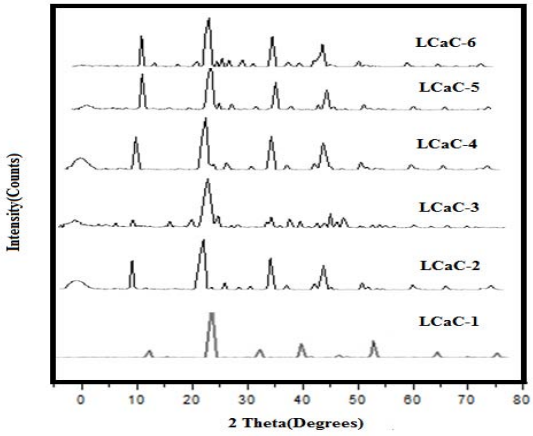


**Figure 1:** XRD patterns of LCaC-1 to LCaC-6 compounds

### 5.2 FT-IR Spectroscopy

The FT-IR spectra of LCaC -1 to 6 compounds (see Fig. 2) exhibit a common broad band near 3400 $cm^{-1}$ due to the OH-stretching vibrations of free and hydrogen-bonded hydroxyl groups, and a second typical absorption region at 1630 $cm^{-1}$ is assigned to the deformative vibration of water molecules which is most probably due to water adsorption during the compaction of the powder specimens with KBr [23, 24]. The metal–oxygen stretching frequencies in the range 400–1000 $cm^{-1}$ are associated with the vibrations of La-O,Cr-O and Ca-O bonds which is existing has mixed metal oxides. FT-IR spectra of the compounds showed sharp band around 625 and 512 $cm^{-1}$ which are characteristic of metal chromite spinels [25]. From the FT-IR spectra of the LCaC -1 to 6 compounds it is evident that the band near 3400 $cm^{-1}$ and 1600 $cm^{-1}$ are broader for LCaC-4 compound. This indicates that the surface adsorption of water on the ceramic surface is more in LCaC -4 compound than in other compounds.

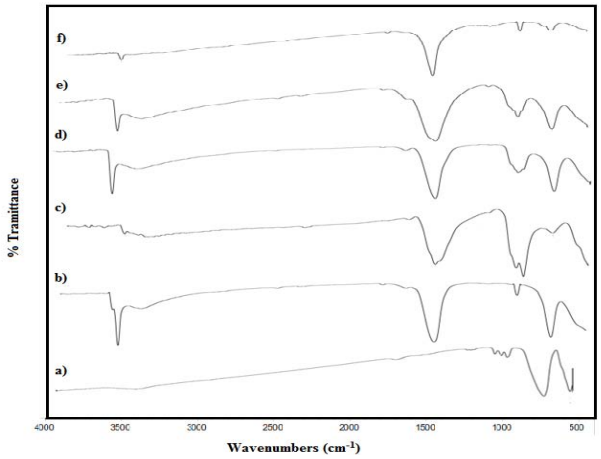


**Figure 2:** FT-IR spectrums of a) LCaC -1,b) LCaC -2, c) LCaC -3,
d) LCaC -4, e) LCaC -5 & f) LCaC -6

### 5.3 Scanning Electron Microscopy (SEM)

The SEM micrographs of LCaC-1 & 6 (Fig.3. a & d) is of the pure lanthanum chromite with the particle size 150 – 200 nm and pure lanthanum calcium oxide with the particle size 1 µm, which doesn't show much of porosity. The LCaC-4&5 (Fig.3.b&c) is of the mixed metal oxides of lanthanum chromium and calcium oxide in the mole ratio of 1: 0.6:0.4 & 1:0.8:0.2, which is formed as a compound by the sol-gel method. The particle size of LCaC-4 is 85 -100nm and LCaC-5 is 200- 280 nm. On comparing the SEM micrographs, LCaC-4 shows a larger grain size and decrease in the particle size than LCaC-1 & LCaC - 6.The surface morphology is well defined in the LCaC -4 compound than in LCaC -1 & LCaC -6. The well developed porosity is found in LCaC -4 compound which plays a very important role in humidity sensing studies.

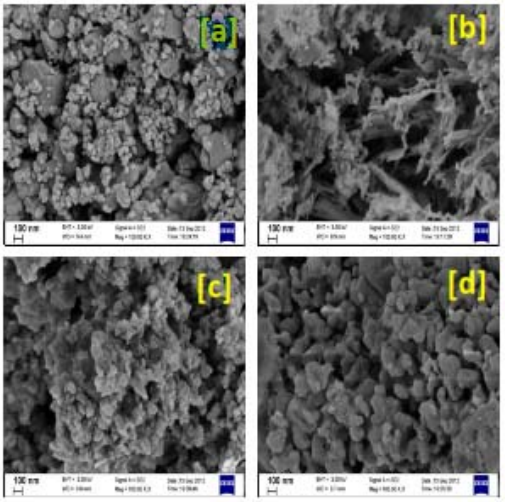


**Figure 3:** SEM micrographs of a) LCaC -1, b) LCaC - 4, c) LCaC -5 & d) LCaC -6

### 5.4 Humidity Measurements and Temperature dependent Studies

In humidity sensing measurements, all the $LaCa_xCr_{1-x}O_{3-\delta}$ (x=0 to 1) compounds showed a decrease in resistance with an increase in % RH. The resistance changes in porous Perovskite type oxides with increasing of the humidity level occur because of adsorption and capillary condensation of

water. The optimum concentration of x=0.8 mole ratio shows good sensor towards humidity. The LCaC -4 compound showed a highest humidity sensitivity of 7510 with a resistance of $2.50x10^{10}\Omega$ at RH 5 % and $3.33x10^{6}\Omega$ at RH 98 %. At low humidity levels, chemisorptions takes place, leading to formation of free surface hydroxyls with the charge transport occurring by the hopping mechanism [26]. While at high humidity levels, water is physisorbed on the top of the chemisorbed layer. As a result, the condensation of water in the capillary like pores leads to a liquid like layer leading to electrolytic conduction. The increase in porosity as evidenced from SEM images confirms the presence of more sites for water adsorption in LCaC -4 compound. The SEM micrographs also reveal that the LCaC -4 compound produces fine grains with maximum porosity compared with the other, indicating that the smaller the grain size, the higher the surface energy and the adsorption capacity. The sintered porous semiconductor has a large internal surface area for the adsorption of water vapor. The sensitivity of the compounds with various molar ratios of LCaC -1 to LCaC -6 is shown in Table 1.

Further the coordination of water molecule to $Cr^{3+}$ ions in LCaC -1 to LCaC -6 compounds increase the acidity thereby release of $H^{+}$. Good linearity in the log R versus RH % plot is an important criterion for good humidity sensitivity material (see Fig. 4). The results suggest that the more linear the plot, the better the response, recovery and sensitivity of the material. The LCaC -1with the humidity sensitivity factor of 0.66 has a resistance of $3.33x10^{6}\Omega$ at RH 5 % and $5.00x10^{6}\Omega$ at RH 98 % .The LCaC -6 with the humidity sensitivity factor of 50 has a resistance of $5.00x10^{9}\Omega$ at RH 5 % and $1.00x10^{8}\Omega$ at RH 98 % due to fewer sites for water adsorption on the ceramic surface compared to that of LSF-4 compound.

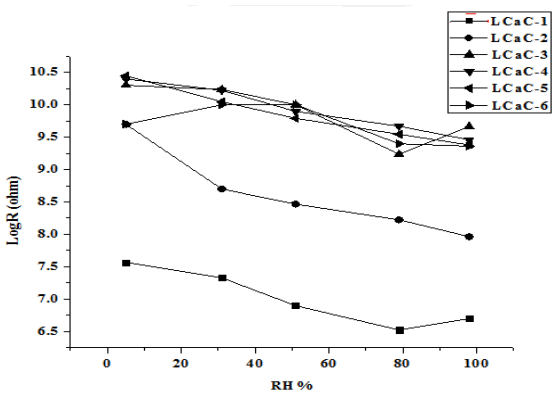


**Figure 4:** Plot of log R vs RH% for LCaC -1 to LCaC -6 compounds

### 5.5 Temperature Dependant Studies

The electrical conductance measurements of LCaC -1 to LCaC -6 compounds at room temperature prior to relative humidity measurements signified that the current increased linearly with the applied voltage, indicating the ohmic contact of the electrodes. The temperature dependence of the electrical conductance carried out in the temperature range 120 °C to 350 °C suggested that the current (I) increased with an increase in temperature (T). The activation energies calculated from the temperature dependence of conductance data are also shown in Table 1. The activation energy for electrical conduction in polycrystalline materials generally involves the combination of the energy required to raise the carriers from the dominant levels to their corresponding transport bands and the energy required to create the carriers in the dominant levels [27]. The low activation energy of LCaC -4 compound predicts that the small polaron conduction dominates in the studied temperature range.

### 5.6 Response and Recovery Characteristics

The response and recovery time obtained from the plot of Log R vs Time (Fig.5.) for LCaC -4 were found to be 200 s and 90 s, respectively. The longer time taken for the restoration of the resistance to that in dry air could be understood by the fact that these experiments are conducted at 25 ° C at which temperature the desorption kinetics is expected to be slow thus evidencing a surface controlled phenomena. For LCaC -4 the invariant resistance in dry air is in the order of $10^{10}\Omega$. The resistance drops (see Fig. 5) by four orders of magnitude to reach the constant value of approximately $10^{6}\Omega$ within 270 s on purging the moist air. The time taken for the restoration of the original signal is approximately 90 s.

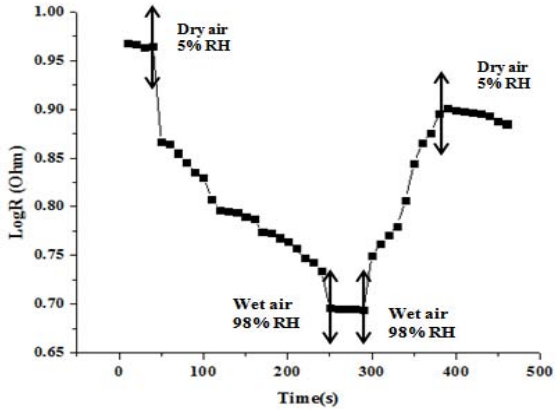


**Figure 5:** Plot of log R vs. time of LCaC-4 compound

## 6. Conclusion

The LCaC ($LaCa_xCr_{1-x}O_{3-\delta}$) compounds with different mole ratios x= 0, 0.2, 0.4, 0.6, 0.8 &1.0 were prepared by sol-gel method and their purity was confirmed by XRD analysis. The intensity of XRD peaks shows a variation from LCaC -2 to LCaC -5 due to the presence of $La^{3+}$, $Cr^{3+}$ and $Ca^{2+}$. The novelty of the compound has been proved by the XRD patterns which is different from the existing phases. The morphology of the compounds was studied by the SEM micrographs. FT-IR study showed the characteristic metal oxide vibrational frequencies. The LCaC -4 ($LaCa_{0.6}Cr_{0.4}O_{3-\delta}$) compounds showed the highest humidity sensitivity factor of $7.510x10^{4}$. Temperature dependent studies showed the low activation energy of the entire compound confirming the involvement of small polaron hopping mechanism in the conduction. The good response and recovery time of LCaC -4 compound and its highest sensitivity factor might be a promising humidity sensing material for practical application.

## 7. Future Scope

This material apart from humidity sensing property it also has a very good application in the field of gas sensors, solid oxide fuel cells and catalyst [28-30]. Thereby the future scope of this material is to analyse for gas sensing properties and to study its catalytic activities for some of the organic synthesis related to the field of green chemistry.

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