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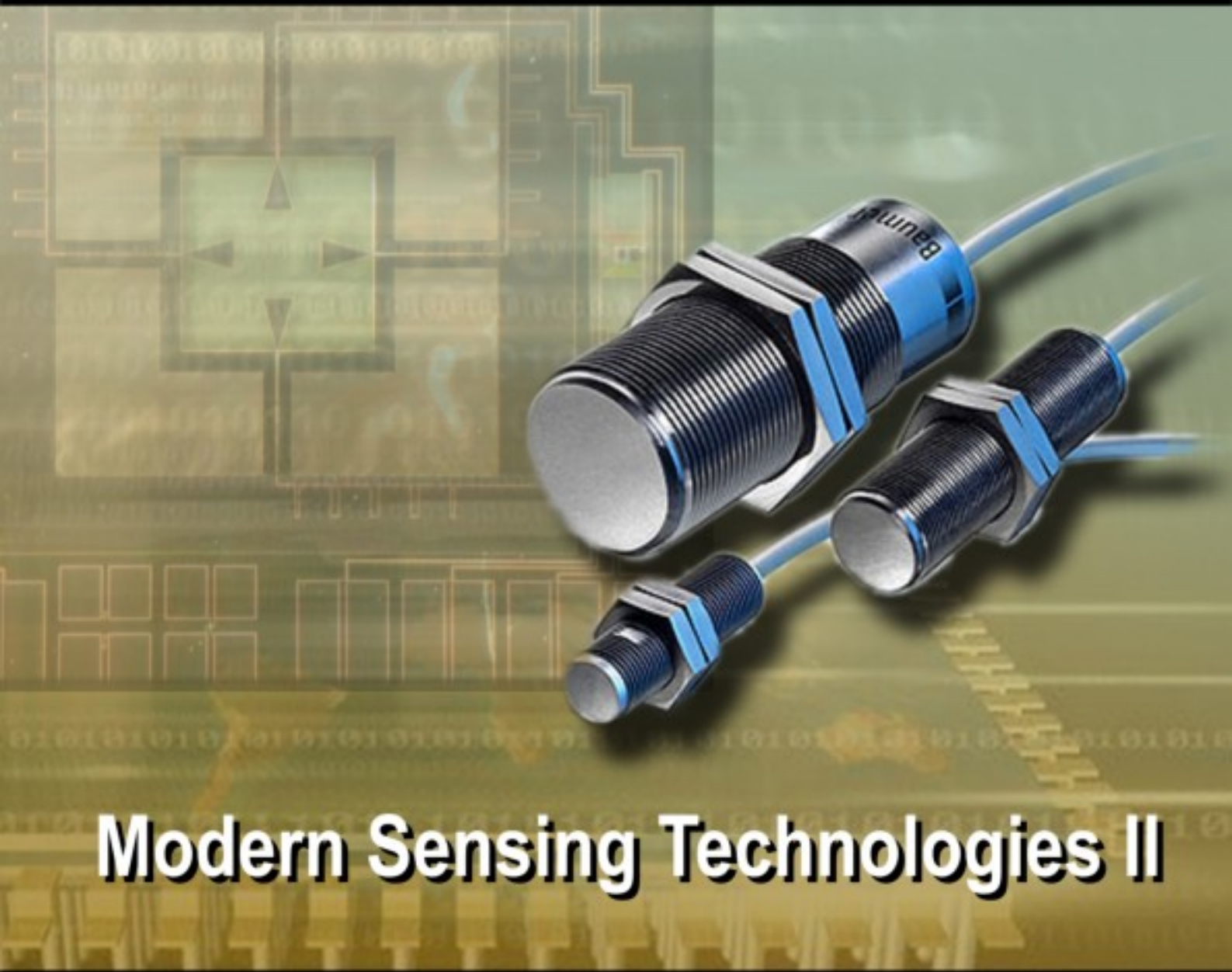

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# Effect of Organic Vapour on Porous Alumina Based Moisture Sensor in Dry Gases

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**Abstract:** A capacitive porous alumina based trace moisture sensor in the range of 50 to 500 ppm (V) was fabricated by low cost sol-gel technique. The cross-sensitivities due to the presence of organic vapours like ethanol, methanol, acetone and benzene were studied. The change in response and recovery time with ppm for moisture sensing was also calculated. The experimental results conclude that moisture sensor is responsive to the polar organic vapours but has almost negligible response to the nonpolar molecules like benzene. Response of the sensor to the organic vapours as compared to the moisture sensitivity is very less. The effect of ambient temperature was found to be negligible. *Copyright © 2009 IFSA.*

**Keywords:** Sol-gel technique, Porous alumina, Traces moisture sensor, Cross-sensitivity, Response time, Recovery time

## 1. Introduction

Detection of gas moisture in the lower ppm(V) range using trace moisture sensor in a dry gas in the IC fabrication industries or SF6 gas (sulphur hexafluoride) used in switch gear circuit breakers is very much essential. A moisture sensor also finds application in monitoring the proper operation of power transformer where even a very small amount of moisture in air/gas used for cooling the transformer oil can lead to premature failure of the system [1]. A high humidity level may produce current leakage paths on the printed circuit board of the electronics circuit leading to device failures whereas low

moisture level can result in electronics discharge [1]. Conventional trace moisture sensors are based on organic polymer, optical hygrometer or porous alumina [2]. Conventional hygroscopic aluminium oxide or polymer based sensors are not stable causing shift in the output with respect to time needing frequent recalibration [3-5]. Recently, porous silicon has been utilized for measuring the moisture at ppm level. It has been confirmed that by slightly changing the anodisation parameters during fabrication of porous silicon, there can be a huge change in the morphology of the prepared sample. Also, by tuning the pore morphology of the porous silicon, it can be used as a trace moisture sensor but both the drift due to aging and the effect of ambient temperature are needed to be studied [6]. Thin film porous alumina (PA) has some key advantages because of which it has attracted much attention by the researchers for manufacturing the commercial moisture sensor because of its high stability, high sensitivity and almost negligible dependence on the ambient temperature [1-4]. However, they suffer from large response & recovery time and high cost and work properly only in clean and non corrosive environment [4, 5]. Effect of interchangeability is another issue that is of much concern [3]. In recent times, sol-gel technique has emerged as a powerful, simple, effective and low cost method for developing nanostructure films or coating of complex oxides of different pore morphologies for sensor based applications. It has been found appropriate for fabricating flexible, thick, crack free PA sheets that can be cut into different shapes and sizes by controlling the formation parameters [8-10]. For ohmic metal contact, the electrode can be easily screen printed on the sheet. The sol-gel and the tape casting processes have been exploited successfully by K. Sengupta and his group for developing a uniform mesoporous $\gamma$-$Al_2O_3$ film which has very large surface to volume ratio, and small pore dimensions in the range of few nanometers [5]. The small pore radius makes $Al_2O_3$ sensitive to very low water vapour pressure. There are several phases for $Al_2O_3$ whereas only two of them are common and used in humidity sensing: $\gamma$-$Al_2O_3$ (amorphous) and $\alpha$-$Al_2O_3$ (corundum). The former is more sensitive than the latter due to its high porosity, while the latter is most thermodynamically stable phase [11]. A capacitive trace moisture sensor in the range of 50-200 ppm(V) was fabricated by utilizing the mesoporous PA film. When the experimental results were compared with the commercial thin film PA moisture sensor manufactured by SHAW (UK) [2, 5], showed that mesoporous film prepared by cost effective sol-gel technique can also be utilized to develop commercial trace moisture sensor [7] with improved sensor parameters like response and recovery time. Detailed experimental results for sensitivity, response & recovery times, short term drift due to aging have been reported but the cross-sensitivity with other organic vapours like ethanol, methanol, isopropyl alcohol, acetone and nonpolar solvents like benzene and toluene have not been reported so far. The effect of ambient temperature and the drift due to long-term aging are needed to be explored. In order to analyze the commercial viability of the sensor, the present work studies the effect of cross-sensitivity due to the presence of organic vapours and the effect of working temperature on the sensor performance.

## 2. Principle of Moisture Sensing

When the nanoporous $\gamma$-$Al_2O_3$ alumina film sandwiched within parallel plate metal contact is exposed to the dry gas with certain moisture content in ppm(V), the water molecules get adsorbed on the surface and condensed inside the porous matrix. The rate of adsorption is proportional to the concentration of water molecules present in the dry gas and the exposed surface area, which in turn depends on the porosity and pore dimension of the porous layer. The equilibrium moisture content depends on the degree of void filling. Condensation of moisture takes place in all pores with a radius smaller than the Kelvin radius ($r_k$) [12] given by

$$r_k = \frac{2\gamma M_v \cos\theta}{\rho_v RT \ln(p_v / p_s)}, \quad (1)$$

where, γ is the surface tension, $\theta$ is the contact angle, $M_v$ is the molecular weight of the vapour, $\rho_v$ is the density of condensed vapour, $p_v$ is the instantaneous vapour pressure, $R$ is the universal gas constant, $T$ is the absolute temperature and $p_s$ is the saturation vapour pressure. Assuming the porous dielectric with the pore radius distribution $p(R)dR$ given by equation (2) between zero and $R_{max}$, the volume fraction of pores filled with condensed vapour (ϕ) can be approximately given by [12],

$$\phi = P \frac{\int_0^{r_k} R_p(R)dR}{\int_0^{R_{\max}} R_p(R)dR}, \tag{2}$$

where $P$ is the porosity of the alumina film. Considering adsorption, diffusion and condensation kinetics, the effective dielectric constant of the porous layer has been given by

$$\varepsilon_{eff} = f(\varepsilon_{vap,}\phi,\phi_p,\mathrm{T}), \tag{3}$$

where, $\varepsilon_{Vap}$ is the dielectric constant of the vapour, $\phi$ is the volume fraction of the absorbed vapour, $\phi_p$ is a parameter accounting for the degree of orientation and interconnectivity of the pores and $T$ is the temperature [13]. The condensed water vapour in the porous layer leads to a change in the dielectric constant of the porous layer. Hence the capacitance of the porous layer changes as a function of the moisture uptake, which is directly related to the moisture concentration present in the ambient gas [12], temperature and dielectric constant of the vapour. The parameters of the moisture sensor are a function of the pore morphology and thickness of the dielectric, the properties of the vapour molecules and geometry of the contacts [14]. In general, the capacitance $C_s$ of a porous alumina parallel-plate capacitor with sandwich-type contact structure having two rectangular contact pads can be written as [12]

$$C_s \approx \frac{\varepsilon_0 \varepsilon_{eff} A}{d}, \tag{4}$$

where, $\varepsilon_{eff}$ is the dielectric constant of the porous layer.

## 3. Experimental

### 3.1. Fabrication of the Sensor

The structure was prepared by sol-gel method as described by Yoldas [8]. A nanoporous alumina monolith was prepared by using sec-butoxy aluminium trioxide ($C_{12}H_{27}AlO_3$) as a starting material. The preparation was done by hydrolysis and subsequent refluxing techniques [7]. Hydrolysis was done by adding Al-sec-butoxide in excess water at a molar ratio of (100:1) at 90 $^0$C and stirring the mixture for 1 hr on a heating bath at constant temperature. A fixed amount of HCl (at a molar ratio 0.07:1) at the same temperature was added and the resulting mixture was stirred for another 1 hr for polymerization of the suspended solution. To obtain a 70-100 μm thick film of $Al(OH)_3$, the transparent sol that was formed by refluxing at 90-100 $^0$C for 16 hr was dried on a flat surface. The film was then cut into rectangular shape of dimension 10 mm x 8 mm and then annealed at 500 $^0$C for another 10 hr. The product obtained after annealing was porous $\gamma$-alumina ($\gamma$-$Al_2O_3$) with pore size of approximately 20 nm. Fig. 1 shows both schematic and an actual photograph of the fabricated PA sensor. The mesh structured contact electrodes were printed on both the surfaces of the PA using

Ag-Pd conducting paste. A mesh structure was used to improve the conductivity. The porous alumina with two electrodes on both sides of the sample formed a parallel plate capacitor. The Ag-Pd conductive paste was used for the formation of ohmic contact. It was prepared by mixing silver and palladium in a 4:1 ratio. The mixture was grounded for 5-6 hr and homogenized with an organic vehicle and thinner. The organic vehicle consisted of ethyl cellulose, terpenol, butyl carbetol acetate, surfactant and deflocculating agent. The paste so prepared was screen printed and dried in an air oven. It was finally cured at approximately 850 $^0$C. The thickness of the film was 40 – 50 μm.


(a) (b)

**Fig. 1.** (a) Schematic diagram of PA capacitive moisture sensor (b) Photograph of the sensor [13].

### 3.2. Testing of the Sensor

A robust measurement system using PC based data acquisition was utilized to obtain a rapid, accurate and reliable response of the sensor in presence of vapour, Fig. 2 shows the schematic diagram of the experimental setup. This setup consists of two chambers namely, vapour chamber and sensor chamber. The vapour chamber comprises of a bubbler which contains the vapour molecules to be sensed. Dry $N_2$ gas cylinder was used as a carrier gas at a constant flow rate of 1 L/min for carrying vapour under test from the vapour chamber to sensor chamber. The sensor chamber consists of a cylindrical glass chamber of 100 cm$^3$ volume. The chamber was fitted with inlet and outlet gas pipe line. The inlet of the chamber was fitted with a glass bubbler. The moisture sensor was placed inside this chamber and the sensor was connected with a shielded cable (length 2ft) to the Keithley C-V analyzer. The output of the C-V analyzer was interfaced with PC for online data acquisition with the help of Testpoint software. The capacitance change of the sensor with different moisture concentrations were studied by C-V analyzer, (Keithley, 590) at frequency of 100 kHz.

The experimental procedures for testing each vapour are as follows; first, the chamber was dried by passing dry nitrogen gas for several minutes to bring the capacitance of the sensor at the initial base line value (dry condition). The fixed volume liquid to be sensed measured by micropipette was poured into the glass flask and the liquid was vaporised by heating the flask. Then test vapour was carried by the dry gas into the chamber, the output of the C-V analyzer was recorded. The sensor was then refreshed to its original base line value by continuing the flow of dry gas for further several minutes. This way one cycle of measurement was completed. In the next cycle, the amount of liquid was increased by a fixed measured value. The same procedures were repeated for other vapours as well.

Fig. 3 shows the dynamic response of the sensor at different moisture concentrations in the range of 50 to 500 ppm(V). It shows that the capacitance increases with an increase in moisture concentration. As the ppm level increases, the amount of vapour molecules also increases. This causes the dielectric constant of the porous layer to change and increase in a certain amount (equation 3). This leads to a rise in the capacitive value with higher ppm.

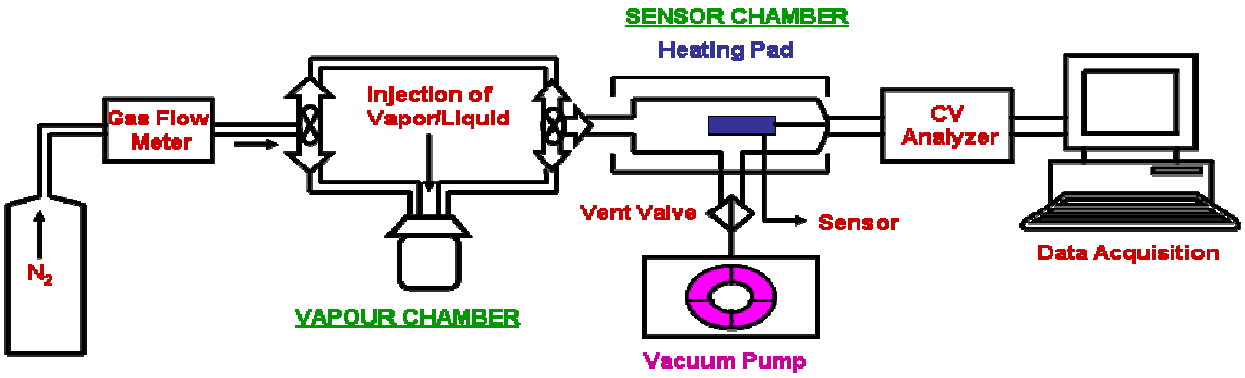


**Fig. 2.** Schematic diagram of the experimental setup.

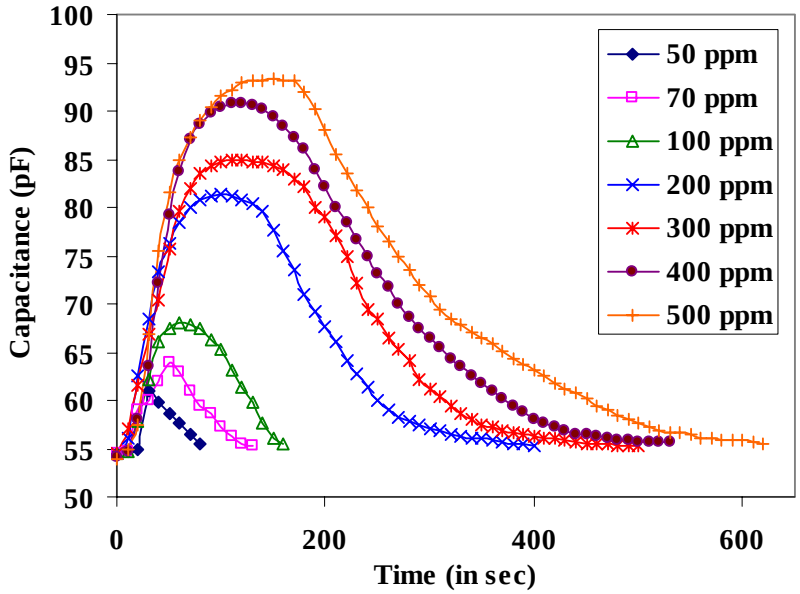


**Fig. 3.** Dynamic response of the moisture sensor at different moisture level.

Fig. 4 shows the capacitive response of the sensor at different moisture levels. The response is quasi linear. High sensitivity at the lower ppm results in better linearity over the full scale range which can provide improved accuracy when used with existing analog instrumentation. For each of the cycles shown in figure, the output of the sensor when refreshed reaches to its initial base line value. It shows that the response is highly repeatable.

To determine the response time of the sensor, the water molecules at a fixed concentration were exposed to the sensor (in dry vapour condition) placed in the sensor chamber. For recovery time, this sensor was refreshed by the flow of carrier gas so that the capacitance value reduces and reaches to its initial base line value. The time taken by the transient response curve for an increase of the output capacitance from 10 to 90 % of its maximum value is the response time while the time taken by the sensor to reach to 10 % of maximum value is the recovery time. Fig. 5 shows the response and recovery time calculated for the sensor exposed to water molecules at 200 ppm, which were around 48s and 117s respectively. Commercial Xentaur thin film sensor takes 90 s for 65 % step change in the moisture level while SHAW sensor takes much larger than Xentaur [3].

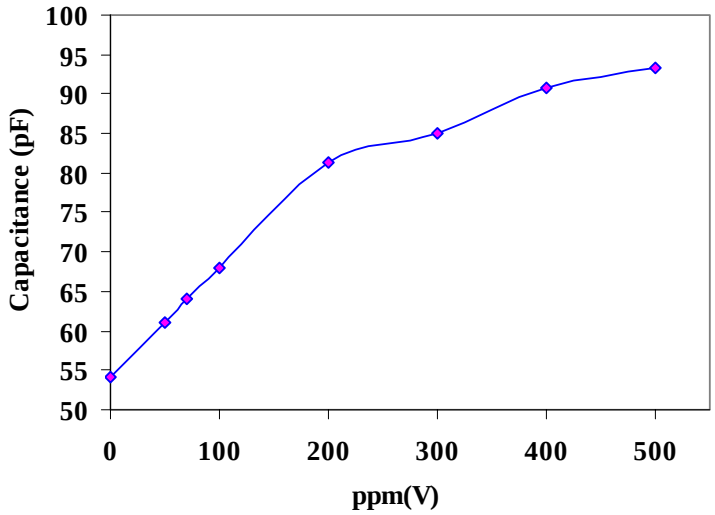


**Fig. 4.** Capacitive response of the moisture at different ppm level.

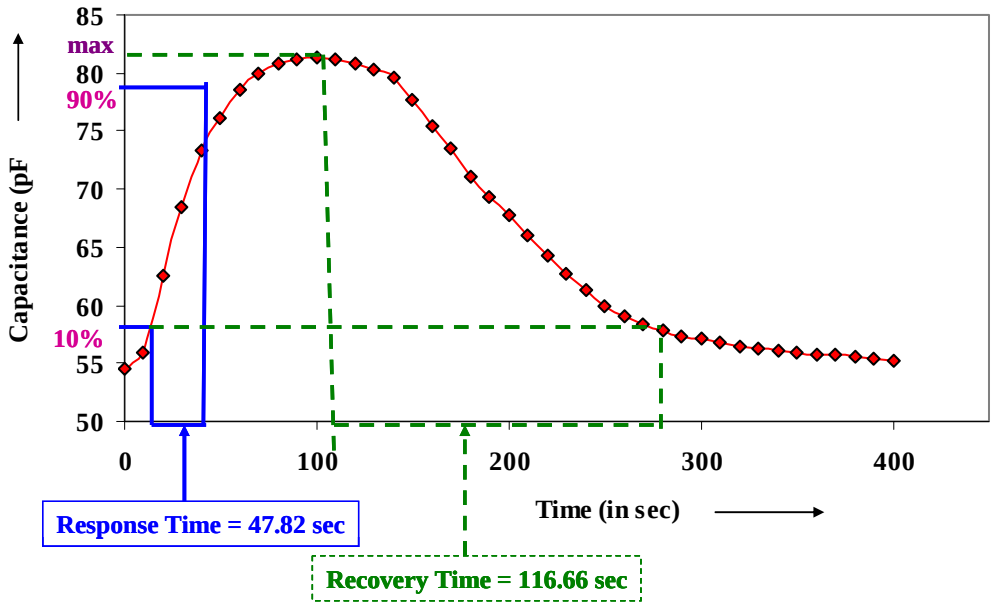


**Fig. 5.** Transient response of the sensor.

Fig. 6. shows the variation in response time at different ppm for sensing moisture. A linear increase in response time is observed at lower ppm however, at higher ppm the response time tends to become constant.

Similar kind of behaviour was observed with recovery time (Fig. 7). There is less amount of water molecules at lower ppm as compared to higher ppm. If the number of molecules is less, their movement is collision free and therefore it will take less time for them to enter the pores. It will also be convenient for them to get adsorbed, diffused and condensed in the porous matrix. The sensitivity of the PA layer is the change in dielectric constant of the layer after condensation of the water molecules. Also, the dielectric constant of the layer is vapour molecule concentration dependent. Therefore, less ppm corresponds to less response and recovery time.

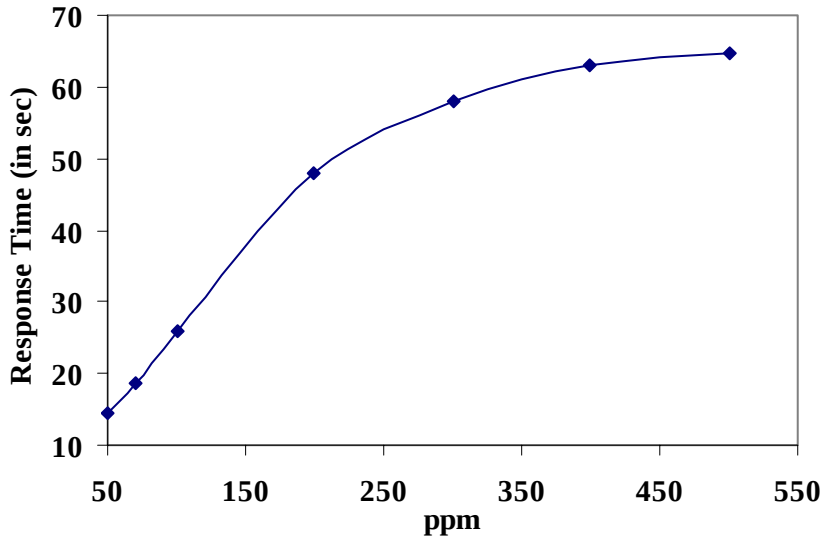


**Fig. 6.** Change in Response Time with ppm for Moisture.

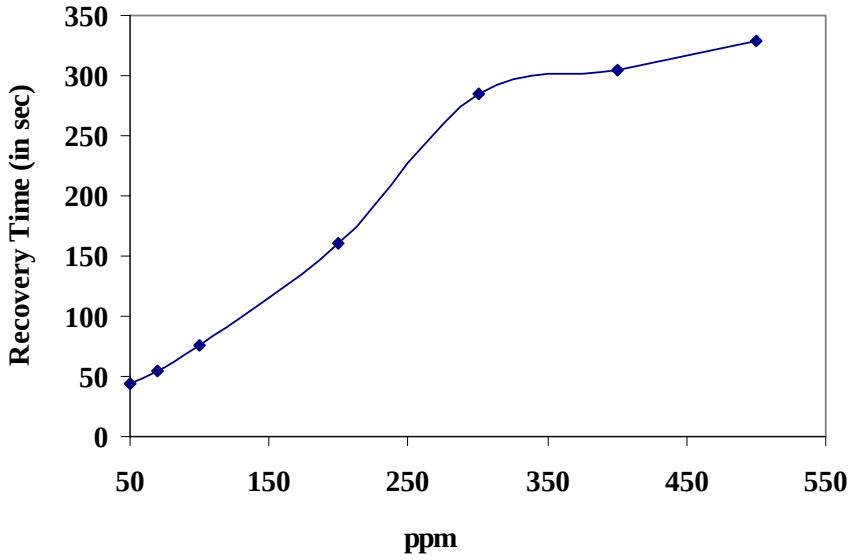


**Fig. 7.** Change in Recovery Time with ppm for Moisture.

At higher ppm, the water molecules are in large quantity and they restrict each other's movement because of collisions causing to slow diffusion of molecules into the pores of PA layer. This leads to a larger response time. Also, desorption process from the PA layer for large number of molecules is difficult causing slow recovery of the sensitive layer. The capillary condensation and chemisorption of water molecules are the key reasons for different times between adsorption and desorption processes. The capillary condensation occurs in all the pores with radius up to Kelvin radius, $r_k$ while desorption by a pore radius $2r_k$. The Kelvin radius of desorption is twice than that of adsorption, making former slower than the latter [15].

To select the proper signal frequency the experimental results with the variation of signal frequency are shown in Fig.8. The results show that at lower signal frequency capacitance change is much higher than at higher frequency [14]. The relation between capacitance and frequency for a capacitive-type sensor is similar to that for the resistive type sensor [16]. The capacitance of an ideal capacitor is independent of the frequency. When moisture level is low, little amount of water is adsorbed by the

porous layer. This can be treated as an ideal condition. After adsorbing moisture, the porous layer has leak conduction. The capacitance *C* of the material with leak conduction is given by

$$C = \varepsilon^* C_0 = \varepsilon_r - i\frac{\gamma}{\omega\varepsilon_0} C_0 , \tag{5}$$

where, $\varepsilon^*$ is complex dielectric parameter and $C_0$ and $\varepsilon_r$ are capacitance and relative dielectric parameters of an ideal capacitor, respectively; $\varepsilon_0$ is the permittivity in vacuum and $\gamma$ is the conductivity. So, the capacitance of the porous layer is proportional to $\gamma$ and inversely proportional to frequency $\omega$. Therefore, larger the frequency is, smaller the capacitance.

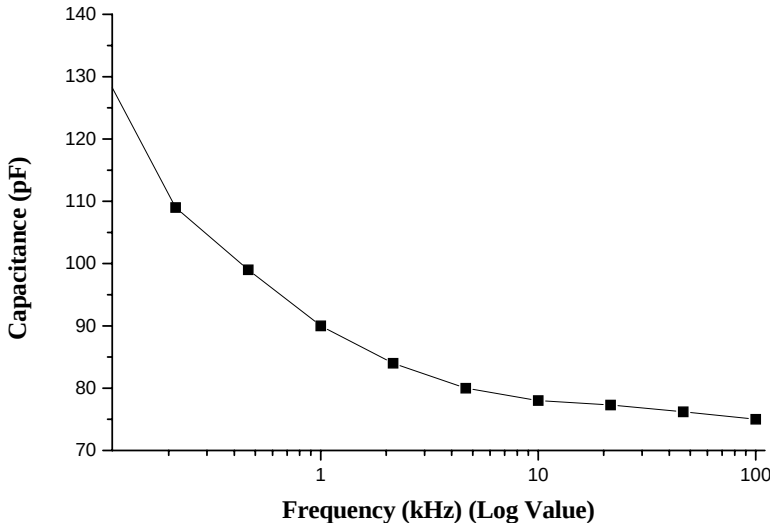


**Fig. 8.** Variation of capacitance with signal frequency at constant moisture of 50 ppm.

Experiments have also been conducted to evaluate the effect of the ambient temperature. The sensor was placed inside a temperature controlled chamber. At a constant moisture level in ppm, the temperature of the chamber was varied from 10 to 50 $^0$C and the capacitance value was noted. The results are shown in Fig. 9 which depicts the change in capacitance value is insignificant as the temperature rises. Also, the effect of temperature due to moisture on the capacitance is negligible.

As the temperature rises, the thermal movement of water molecules becomes strong and the increase of polarization results in the increase of capacitance value [16]. Also, the leakage conductivity γ is a function of temperature T. The expression for the same is given by

$$\gamma = Ae^{-B/T} , \tag{6}$$

where, A and B are constants. As the temperature rises, $\gamma$ increases and leading to a slight rise in capacitance (equation 5).

### 3.3. Response of the Sensor to the Organic Vapour

The sensor was exposed to the molecules having different dielectric constants, polarisabilities and dipole moments, to analyze the cross-sensitivity of the porous alumina based moisture sensor in presence of organic solvents, The solvents were separated into two groups, polar and nonpolar

molecules. The polar sovents consisted of ethanol, methanol and acetone while the non polar solvents included benzene. The properties of the solvents are shown in Table 1.

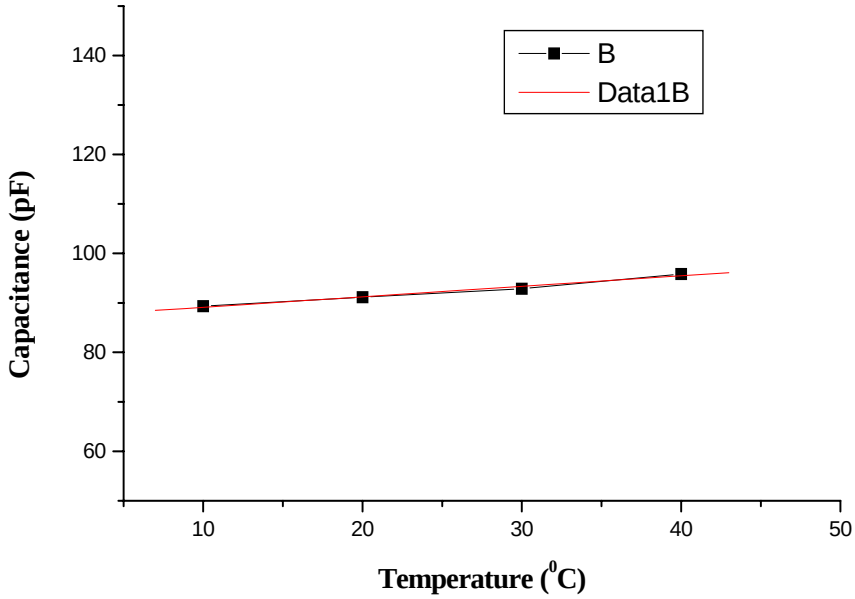


**Fig. 9.** Variation of capacitance with ambient temperature at constant moisture of 50 ppm.

**Table 1**: Properties of different solvents.

| Name | Dielectric Constant (at 77°F) | Dipole Moment | Electronic Polarisability (x $10^{24}$) | Bond Character |
|---|---|---|---|---|
| Benzene | 2.27 | 0 | 10.32 | Non Polar |
| Ethanol | 24.5 | 1.69 | 5.41 | Polar |
| Methanol | 33.6 | 1.7 | -- | Polar |
| Acetone | 20.7 | 2.85 | 6.33 | Polar |
| Water | 80 | 1.82 | 1.45 | Polar |

The concentration of the vapour molecules were varied from 0 to 500 ppm(V). Fig. 10 shows the dynamic response of the sensor to moisture, ethanol, methanol, acetone and benzene vapours. In the dynamic response, sensor output increases with increase in vapour concentration and reaches a steady state condition. Among four different organic vapours, ethanol shows maximum sensitivity and benzene shows almost negligible response. It is observed that the dielectric constant, dipole moment and also the nature of polarization of the solvents play significant role in the sensitivity of the vapour molecules. A vapour needs to polr in nature for it to get sensed by PA sensor. The polarity of the binding molecule helps it in getting adsorbed onto the porous surface. Thus though the electronic polarisability of benzene is much higher than other polar organic vapours, the response of the sensor for benzene is minimum. If we compare the sensitivity of the sensor for water molecules at the concentration of 300 ppm, the capacitance change is almost 30 pF but the highest sensitivity for ethanol vapour at the same concentration is only 6 pF. This may be due to the fact that the dielectric constant of water is much higher than other polar molecules. Also water is the most polar solvent among all the solvents being sensed. This gives a reason for giving the maximum sensitivity of water among all vapours under detection.

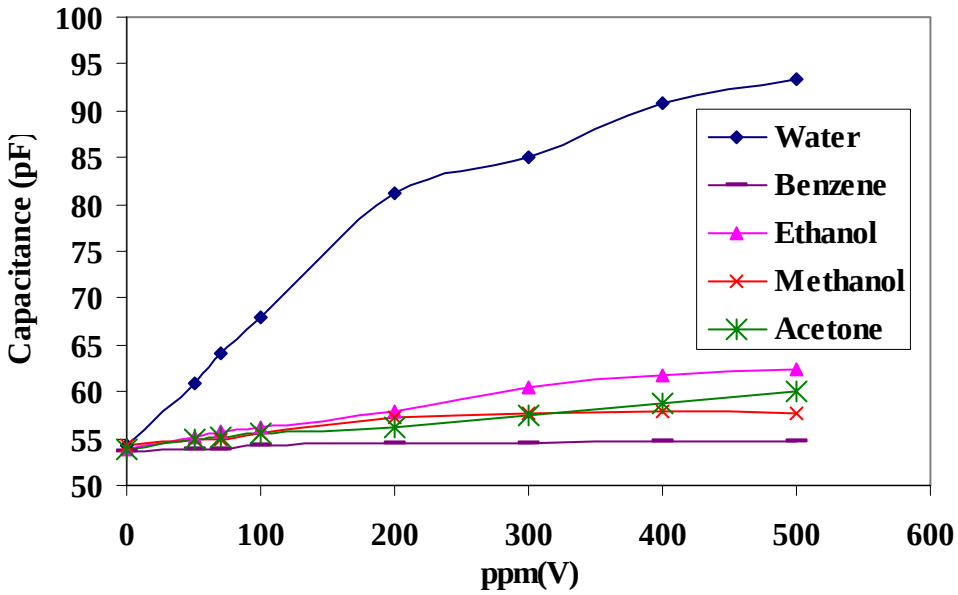


**Fig. 10.** Response of sensor to moisture and other organic vapours.

## 4. Conclusions

The cross-sensitivities of the PA based trace moisture sensor prepared by sol-gel technique with respect to organic vapours of both polar and nonpolar type have been studied in the range of 50 to 500 ppm(V). The experimental results were observed and analyzed for various sensor parameters like sensitivity, response and recovery time for detecting moisture in the dry gases. The results conclude that nanoporous alumina film was extremely sensitive to moisture whereas less sensitive to organic vapour molecules. The behaviour of both the response and recovery time was analyzed with an increase in ppm of water molecules. Both show a linear change at low ppm while tend to become constant at high ppm. The response of sensor towards polar organic molecules like ethanol, methanol and acetone was more as compared to nonpolar molecule like benzene for which the response was extremely negligible. In comparison to moisture sensitivity, the cross-sensitivity of the sensor for organic vapours was confirmed to be negligible. They also proved that in case of moisture sensing the main parameters are the dielectric constant and polarity which brings out a huge change in capacitive value with moisture.

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# Guide for Contributors

## Aims and Scope

*Sensors & Transducers Journal (ISSN 1726-5479)* provides an advanced forum for the science and technology of physical, chemical sensors and biosensors. It publishes state-of-the-art reviews, regular research and application specific papers, short notes, letters to Editor and sensors related books reviews as well as academic, practical and commercial information of interest to its readership. Because it is an open access, peer review international journal, papers rapidly published in *Sensors & Transducers Journal* will receive a very high publicity. The journal is published monthly as twelve issues per annual by International Frequency Association (IFSA). In additional, some special sponsored and conference issues published annually. *Sensors & Transducers Journal* is indexed and abstracted very quickly by Chemical Abstracts, IndexCopernicus Journals Master List, Open J-Gate, Google Scholar, etc.

## Topics Covered

Contributions are invited on all aspects of research, development and application of the science and technology of sensors, transducers and sensor instrumentations. Topics include, but are not restricted to:

- Physical, chemical and biosensors;
- Digital, frequency, period, duty-cycle, time interval, PWM, pulse number output sensors and transducers;
- Theory, principles, effects, design, standardization and modeling;
- Smart sensors and systems;
- Sensor instrumentation;
- Virtual instruments;
- Sensors interfaces, buses and networks;
- Signal processing;
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- Microsystems;
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