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Benzene and Toluene Vapor Sensing Properties of Sr(II)-added Barium Aluminate Spinel Composites

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Abstract: Pure and strontium(II)-added $BaAl_2O_4$ composites were prepared by sol-gel technique and was investigated for the sensitive properties of volatile organic compounds (VOCs) like benzene and toluene. X-Ray diffraction, scanning electron microscopy and nitrogen adsorption/desorption isotherms at 77 K was employed to identify the structural phases, surface morphology, vibrational stretching frequencies and BET surface area of the composites, respectively. The composites were prepared with the molar ratios of Ba: Sr as (1.0: 0.0, 0.0:1.0, 0.8:0.2, 0.6:0.4, 0.4:0.6 and 0.2:0.8) keeping the aluminium molar ratio as constant and were labeled as BaSA1, BaSA2, BaSA3, BaSA4, BaSA5 and SA respectively**.** The samples sintered at 900^{o}C for 5 h, were subjected to dc resistance measurements in the temperature ranging from 27-350^{o}C to study the VOC vapor sensing characteristics. The results revealed that the sensitivity factor (S_f) increased with temperature up to the optimum temperature at which maximum sensitivity is obtained and thereafter decreased. The sensitivity was found to increase with increase in concentration from 100-5000 ppm at their optimum temperature. Among the composites BaSA5 showed the maximum sensitivity towards benzene (S_f = 60±3) and toluene (S_f = 49±1) at an operating temperature of 150 and 175 ^{o}C respectively. *Copyright © 2008 IFSA.*

Keywords: Sol-gel growth, Composite materials, Ceramics, Electrical conductivity

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1. Introduction

There has been high demand to develop gas sensors to detect volatile organic compounds (VOCs), such as benzene and toluene due to their harmfulness to human health and environment [1, 2]. In order to monitor the dangerous and toxic VOCs, high performance sensors are required that can identify and measure various kinds and quantitities of VOCs. Several researchers have reported on the use of gas sensors using metal oxides, polymers etc. In recent years semiconducting oxides like zinc oxide, aluminum oxide, titanium oxide etc. have been studied extensively and have emerged as economical sensors for monitoring toxic gases and vapors [3]. The sensitivity of these sensors to gases depends on the microstructure and this can be achieved by adopting special techniques of preparation or by doping impurities [4]. The sol-gel technique is considered as the promising technique to prepare commercial ceramic sensors [5, 6] as it allows for high purity ceramics with homogeneous distribution of components on the atomic scale. Hence the development of a VOC sensor having higher sensitivity along with optimization of selectivity, durability and stability that can be operated at lower operating temperature is the currently undergoing research. Although metal aluminate spinels have been used for the purpose of humidity sensing [7-9] there are no literatures found regarding metal aluminates and Sr(II)-added metal aluminates for VOC sensing. Our previous work on barium aluminates and Sr(II) added barium aluminates for humidity sensing were reported in detail [10] and hence the present study is focused on the benzene and toluene vapor sensing properties of the same composites that was prepared by sol-gel technology. The main objective of the present work is to study only the effect of addition of Sr(II) on barium aluminates rather than the isomorphic substitution. In the present paper Sr(II)-added barium aluminate composites prepared by the sol-gel technique were characterized by X-ray diffraction, scanning electron microscopy, FT-IR spectroscopy and nitrogen adsorption/desorption isotherms at 77 K. The dependence of electrical response of these composites to the volatile organic compounds like benzene and toluene was studied.

2. Experimental Procedure

Pure and Sr(II)-added barium aluminate composites were prepared by a sol-gel route as reported in our earlier work [10] and the sample codes with corresponding molar ratio compositions are shown in Table 1. The structural studies were carried out using a Philips X'pert diffractometer for 2θ values ranging from 10 to 80 ^{o} using CuKα radiation at λ= 0.154 nm. The surface morphology of the sintered porous compacts was determined by a Leo-Jeol Scanning electron microscope at the desired magnification. The nitrogen adsorption-desorption isotherms of the composites were measured using an automatic adsorption instrument, Quantachrome Corp. Nova-1000 gas sorption analyzer at 77 K. The specific surface area (m^2/g) of the composites was calculated using BET equation. The pore size distribution was determined using the BJH method. In addition, the t-plot method [11] was applied to calculate the micropore volume and external surface area. The total pore volume was estimated as the liquid volume of the adsorbate at a relative pressure of 0.99.

Table 1. Sample code and activation energy for Sr(II)-added $BaAl_2O_4$ composites.

Sample code	Molar ratio of Ba:Sr:Al	E_a (eV)
BaSA1	1.0:0.0:2.0	0.33
BaSA2	0.8:0.2:2.0	0.31
BaSA3	0.6:0.4:2.0	0.28
BaSA4	0.4:0.6:2.0	0.24
BaSA5	0.2:0.8:2.0	0.22
SA	0.0:1.0:2.0	0.29

To measure the electrical conductance at room temperature, the samples were electrically connected to a dc power supply and a Keithley 485 picoammeter in series. Given the high resistivity of the materials under investigation, the potential inaccuracy due to contact resistance is assumed negligible. Conducting silver paste was employed to ensure the ohmic contacts. The temperature dependent conductance experiments were carried out to determine the activation energies of the samples using the linearised form of the expression $I = I_o\exp^{-Ea/kT,}$ where I was the current, E_a the activation energy, k the Boltzmann constant and T the temperature.

The sensitivity tests were carried out in a testing chamber designed for this study (Fig. 1) that measures the surface resistance of the samples. The volatile organic compounds such as benzene, toluene, acetone, hexane and acetic acid of AR grade were used for the sensing purpose. VOCs were injected by a micro syringe into the test chamber and the sensing characteristics of the sensor were observed by measuring the electrical resistance change of the sensor when the latter was exposed to VOCs. A typical injection of 0.3 ml of VOC corresponds to a gas concentration of about 100 ppm [12, 13]. Under the exposure of reducing gases such as benzene, toluene, hexane, heptane and acetic acid, its resistance decreases. The sensitivity factor S_f is defined as, $S_f = \frac{R_{air} - R_{gas}}{R_{gas}}$, where R_{gas} is the resistance of the sensor under gas exposure and R_{air} is the resistance of the sensor in dry air. Dry air from the columns containing $CaCl_2$ and P_2O_5 (RH 5%) was used throughout the study. Initially dry air was passed into the test chamber until the constant resistance (R_{air}) was reached. By injection of VOCs the resistance decreased and when it becomes constant it was noted as R_{gas}. Using the above formula, the sensitivity factor was determined. The resistances of the samples were measured at different temperatures in the range of 27-350 ^oC and different concentration levels (100 to 5000 ppm) of VOCs. All measurements were carried out in an air atmosphere. A dc power supply and Keithley 485 picoammeter was connected to the leads of the pellet through the chamber to read out the potential applied and the resistance respectively. Voltage was varied in the range of 2-30 V.

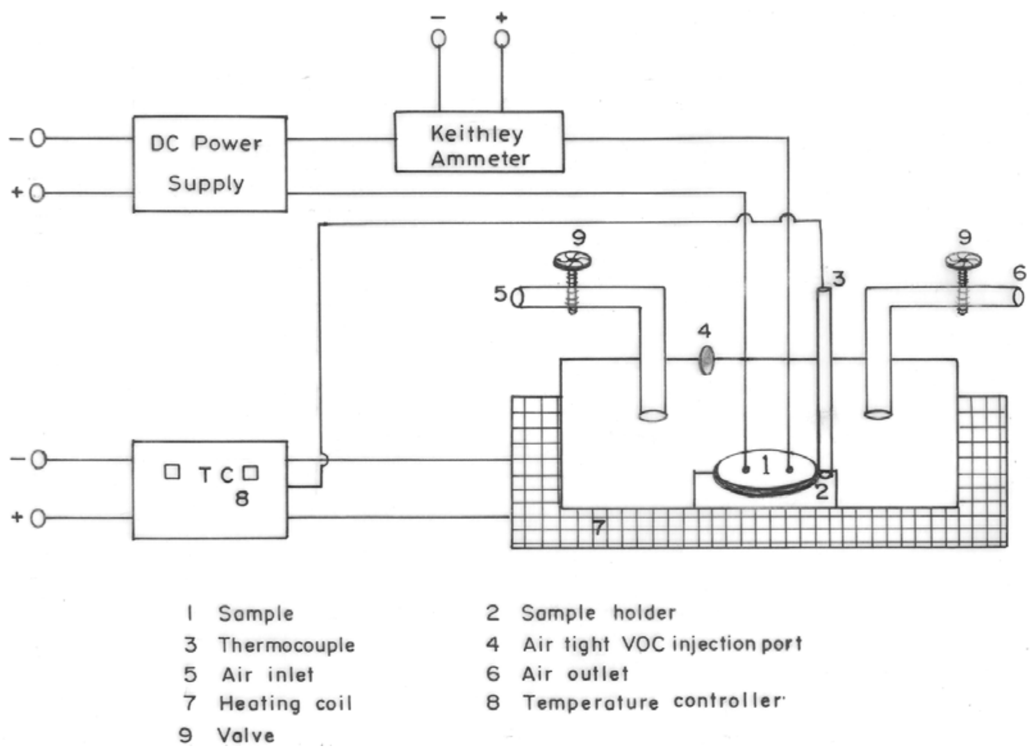


Fig. 1. Schematic diagram of the experimental set-up used to measure the VOC vapor responses.

The long-term stability of the maximum sensitivity composite BaSA5 has been evaluated by measuring the variation of resistance with time. The phenomenon of sensing VOC vapors depend on the surface, and the regeneration or deterioration of the surface plays a vital role in the sensing mechanism. This process was carried out for a total time period of 100 h. Also after a month the sensitivity to VOC vapors was measured and compared to indicate the stability and durability of the sensor [12, 13].

The response and recovery times are important parameters to characterize a sensor. The response time is defined as the time taken to reach 90% of the response when gas is introduced. The recovery time is the time taken to reach 90% of the recovery when gas is turned off [14]. Initially dry air from the columns containing P_2O_5 and anhydrous $CaCl_2$ was passed in to the chamber through the air inlet by opening the inlet valve and keeping the outlet also opened till the constant resistance (R_{air}) was obtained. This was then followed by injecting appropriate amount of VOC into the test chamber (Fig. 1) by keeping both the inlet and the outlet valves closed. The resistance dropped suddenly and reached a saturated level showing a constant resistance (R_{gas}), after a particular time interval, which is termed to be a response time. The outlet valve was again opened to release the vapors out by passing dry air continuously through the inlet valve until the original resistance was reached. The time taken for this process is termed as recovery time. The response and recovery characteristics were studied for the composites BaSA1 (pure barium aluminate), BaSA5 (mixed metal oxide composite with higher sensitivity) and SA (strontium aluminate) for at least three cycles to ensure repeatability.

3. Results and Discussion

3.1. X-Ray Diffraction Studies

The X-ray diffraction patterns of barium aluminate and Sr(II)-added barium aluminate samples are shown in the Fig. 2a-f. The XRD pattern of Fig. 2a showed peaks corresponding to a barium aluminate spinel-like phase (JCPDS: 05-0669). The formation of mixed phases such as $5SrO{\cdot}4Al_2O_3$ (JCPDS: 09-38) and $SrO{\cdot}2Al_2O_3$ (JCPDS: 25-1208) in addition to SrO phase (JCPDS: 01-0886) were observed with higher amounts of Sr(II) added. The addition of Sr(II) retards the growth of the bulk barium aluminate phase on the surface and forms new phases $5SrO{\cdot}4Al_2O_3$ and $SrO{\cdot}2Al_2O_3$. This multiphasing was observed more notably in BaSA5 and was considered as the good criterion for VOC sensing.

3.2. Scanning Electron Microscopy (SEM)

Fig. 3a-c depicts the intergranular porous structure of the composite materials qualitatively. In the micrographs of barium aluminate (BaSA1) and strontium aluminate (SA) the size of the grains are larger than that of the Sr(II)-added barium aluminate (BaSA5). This suggests that the addition of a more amount of Sr(II) in the barium aluminate can reduce the grain size with the intergranular pores, leading to microporosity in addition to the presence of mesopores. It is also observed for BaSA5 composite that the grains connect each other and/or aggregate to some extent with well-developed porosity that is very important to VOC sensing.

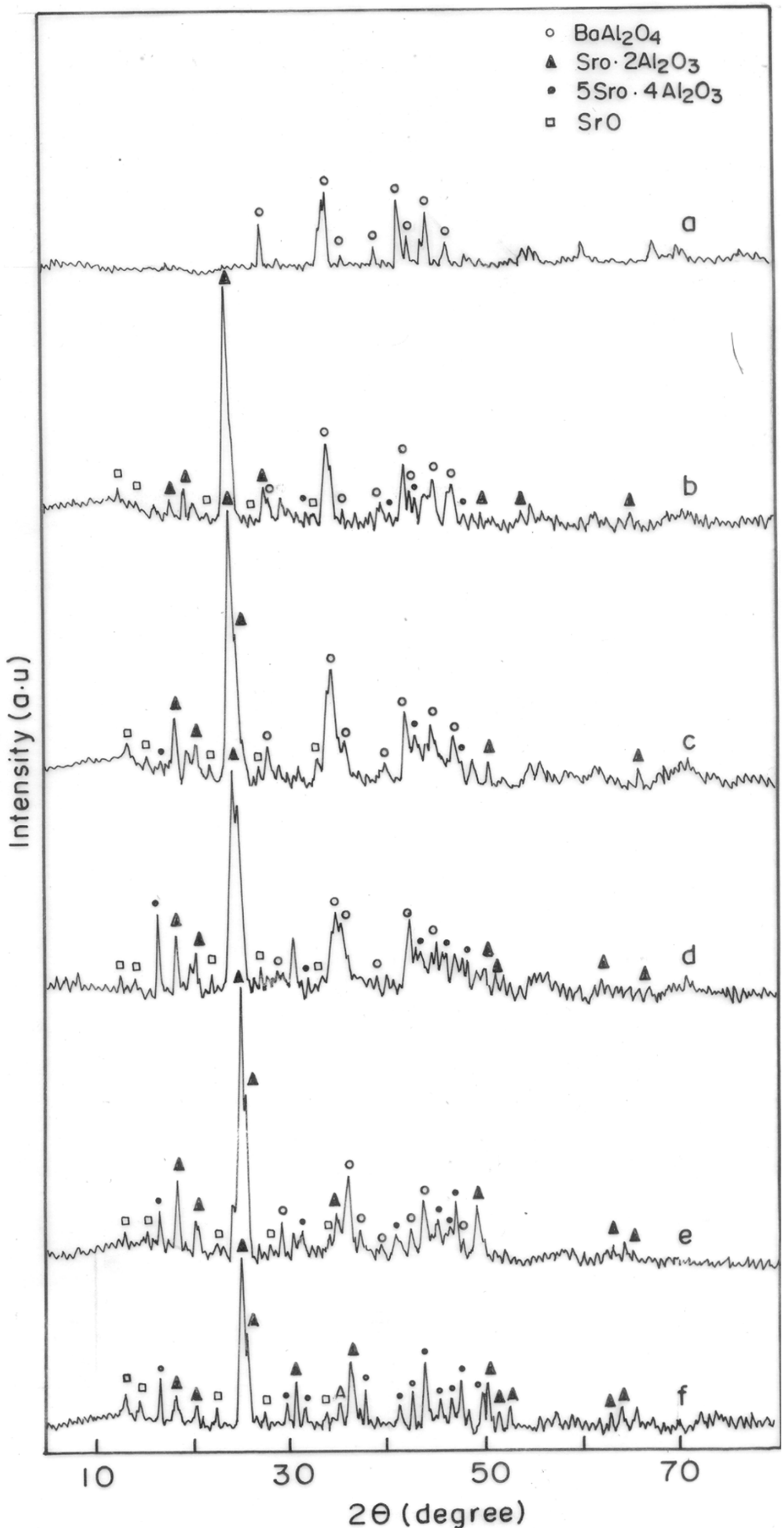


Fig. 2. X-Ray Diffraction pattern of a) BaSA1, b) BaSA2, c) BaSA3, d) BaSA4,e) BaSA5 and f) SA.

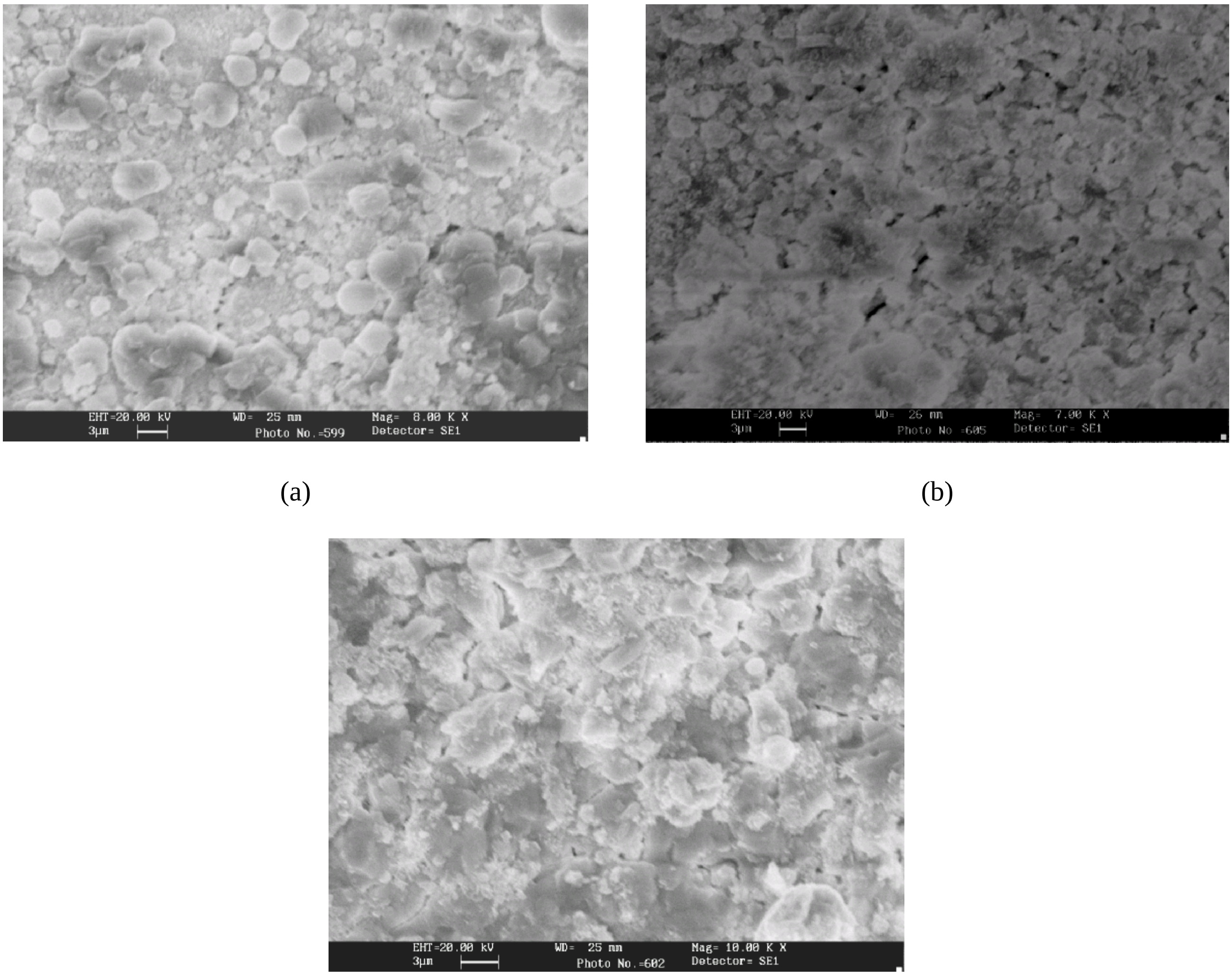


Fig. 3. SEM imag of a) BaSA1, b) BaSA5 and c) SA composite.

3.3. Nitrogen Adsorption/desorption Studies

Fig. 4 represents the nitrogen adsorption/desorption isotherms at 77 K of the barium aluminate (BaSA1), Sr(II)-added barium aluminate (BaSA5), which possessed maximum sensitivity, and strontium aluminate (SA) composites, respectively. The isotherms of BaSA1, BaSA5 and SA showed a hysterisis effect with the slope of the plateau increasing with a significant increase in the nitrogen uptake through the entire pressure range. The increase in uptake of nitrogen in the samples is a result of the major increase in porosity created within the barium aluminate, strontium aluminate and Sr(II)-added barium aluminate components. The composite material BaSA5 exhibited the most prominent hysterisis effect, which can be characterized by the formation of intergranular pores as a result of Sr(II) addition. The amount of nitrogen adsorbed increased to108 cm^3/g for BaSA5, while it was only 82 and 63 cm^3/g for BaSA1 and SA composites, respectively. The BET surface area of BaSA1 and SA composites was 85 m^2/g and 78 m^2/g, respectively, and it increased to 121 m^2/g for BaSA5 composite. The addition of Sr(II) retards the growth of the bulk barium aluminate phase, leading to an increase in the surface area, an enhancement in porosity and an increase in micro and mesopores. The increase in surface area is due to a decrease in grain size with the addition of Sr(II) in the barium aluminate composites as a result of non-isomorphic substitution.

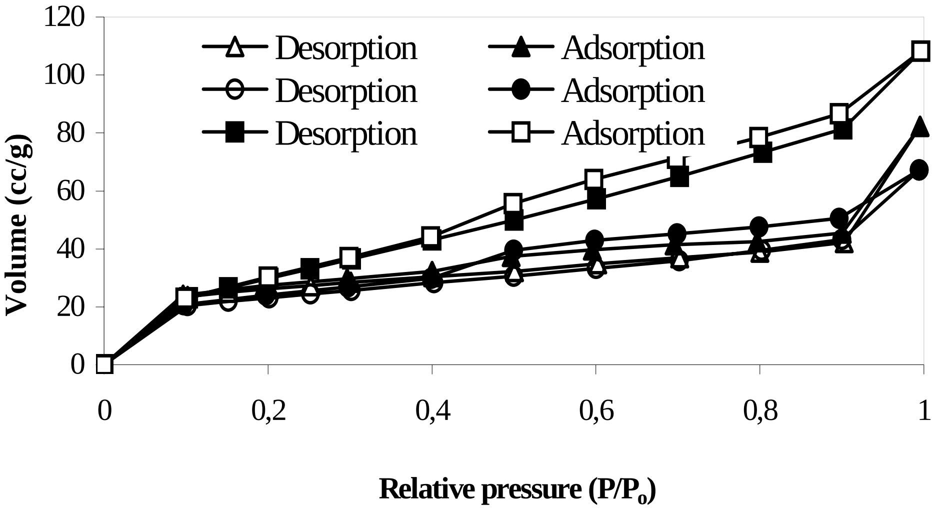


Fig. 4. Nitrogen adsorption/desorption isotherms of a) SA, b) BaSA1, and c) BaSA5 at 77K.

The pores could be generated within the matrices of three components of barium, strontium and aluminum oxide, and consequently pores of different dimensions are produced. The micropore surface area of BaSA1 and SA were 34 and 33 m^2/g, respectively, and BaSA5 possessed a comparatively high micropore surface area of 39 m^2/g. The mesopore surface area of BaSA1 and SA was 51 and 44 m^2/g, respectively whereas the mixed metal composite BaSA5 possessed comparatively a high mesopore surface area of 82 m^2/g. Thus the addition of Sr(II) led to a significant increase in micropore and mesopore surface area and also to the modification of the intergranular pores controlling the range of surface area.

Fig. 5 shows the pore size distribution of BaSA1, BaSA5 and SA composites. The total pore volumes of BaSA1 and SA were 0.13 and 0.10 cm^3/g, respectively, and in case of BaSA5 it is increased to 0.17 cm^3/g. The average pore diameters of BaSA1, BaSA5 and SA composites were 5.99, 4.99 and 5.34 nm, respectively. The average pore diameters obtained from the composites are due to the formation of intragranular pores within the combination of metal oxides. The decrease in the average pore diameter for BaSA5 composite can be attributed to the formation of a considerable amount of micro and mesopores due to non-isomorphic substitution of Sr(II) in the barium aluminate matrix. The presence of Sr(II) retards the growth of bulk barium aluminate phase, leading to an increase in porosity by introducing more micropores along with mesopores. The values of total pore volume, micro and mesopore volume and average pore diameter are given in Table 2.

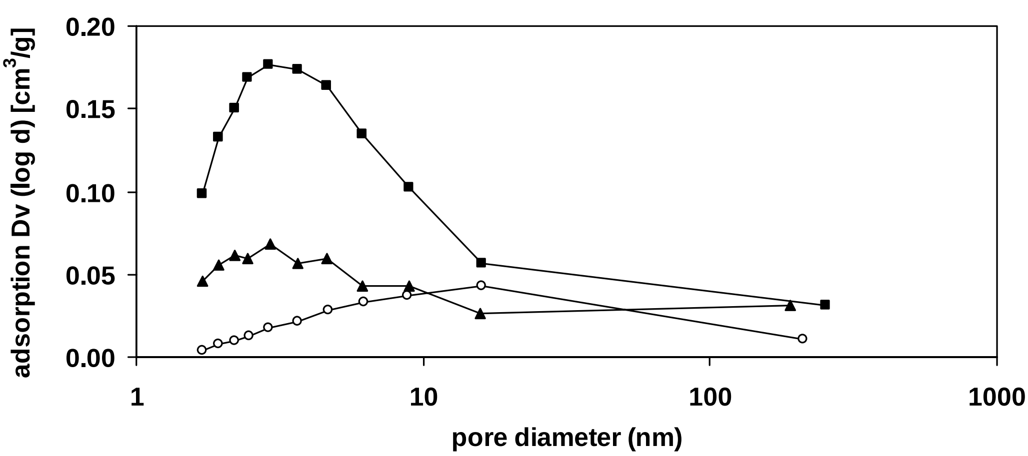


Fig. 5. Pore size distribution of a) BaSA1, b) SA and c) BaSA5 composites.

Table 2. Surface area parameters of BaSA1, BaSA5 and SA composites.

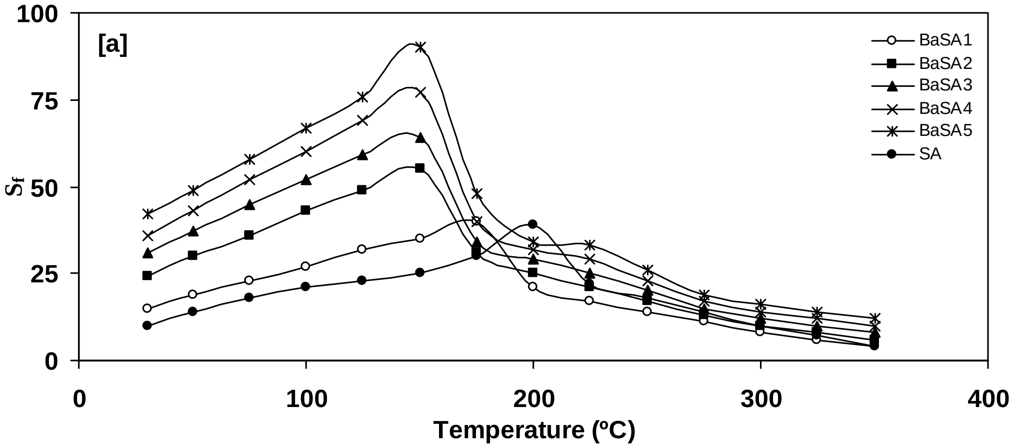
Surface area parameters	BaSA1	BaSA5	SA
S_{BET} (m^2/g)	85	121	78
S_{mic} (m^2/g)	34	39	34
S_{meso} (m^2/g)	51	82	44
Total pore volume (cm^3/g)	0.13	0.17	0.10
Micropore volume (cm^3/g)	0.02	0.02	0.02
Mesopore volume (cm^3/g)	0.11	0.15	0.08
Average pore diameter (nm)	5.99	4.99	5.34

S_{BET} BET surface area, S_{mic} micropore surface area, S_{meso} mesopore surface area

3.4. Gas-sensing Measurements

To determine the optimum working temperature of the sensors, the sensitivities were examined as a function of temperature ranging from 27-350^oC at a concentration of 1000 ppm of benzene and toluene as shown in Fig. 6 (a-b). 1000 ppm of VOC concentration was selected to be the intermediate concentration in the studied range of concentration from 100 to 5000 ppm. Each of these curves showed a maximum sensitivities corresponding to an optimum heating temperature. The sensitivity increases with increase in temperature from 27^oC to a particular temperature at which the maximum sensitivity is obtained and it was fixed as the optimum temperature and thereafter decreases for all the composites. It was observed that the sensitivity of the pure $BaAl_2O_4$ was lesser than the Sr(II)-added composites for both benzene and toluene vapors. It is observed that the sensitivity of the BaSA5 towards VOCs is the highest which is consistent with the low activation energy of that composite. The sensitivity at 1000 ppm of benzene increases with operating temperature, and attains maximum at 150°C, and decreases further for BaSA5 composite. Hence the optimum temperature for detecting benzene vapor was fixed as 150°C. Similarly the optimum temperature for BaSA1 and SA for the detection of benzene vapor was found to 175 and 200°C respectively. For the detection of toluene vapor, the optimum temperature for BaSA1, BaSA5 and SA was found to be 175, 175 and 225°C respectively. The maximum sensitivity of the composite BaSA5 towards benzene and toluene at their corresponding optimum temperature was 60±3 and 49±1 respectively. The increase in sensitivity with operating temperature can be attributed to the fact that the thermal energy obtained was high enough to overcome the activation energy barrier of the reaction, while the reduction in sensitivity above 150°C was due to the difficulty in exothermic vapor adsorption.

It is clear from the Table 3 that varying in the concentration of benzene and toluene from 100-5000 ppm at their respective operating temperatures, the sensitivity increases up to 1000 ppm with a higher rate of increase in sensitivity and thereafter slows down reaching near equilibrium. At low concentration sensitivity has a linear relationship with concentration because there may be sufficient number of pores for benzene or toluene vapor adsorption. At higher concentration range the rate of adsorption decreases due to less assess of these vapors in to the filled pores leading to less increase in sensitivity values. The increase in porosity with increase in Sr(II) content as evidenced from SEM image and BET studies confirm the presence of more sites for VOCs adsorption, which produces more charge carriers for electrical conduction. In addition the SEM photographs also reveal that increase in the strontium addition produces fine grains with smaller dimensions compared with the other indicating that smaller the grain size higher will be the surface energy and the adsorption capacity.



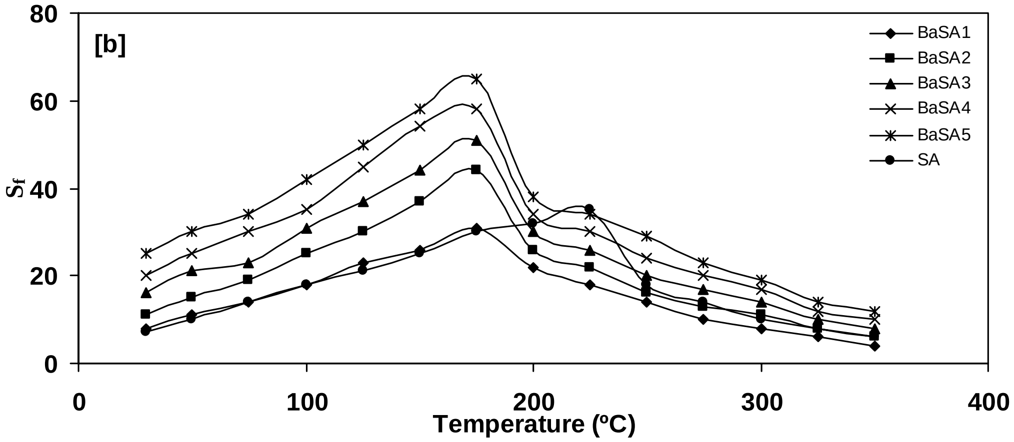


Fig. 6. Sensitivity of composites at different temperatures to a) 1000 ppm of benzene and b) 1000 ppm of toluene.

Table 3a. Sensitivity of the composites to benzene vapor at their respective operating temperature and at different concentrations.

Composites	100 ppm	500 ppm	1000 ppm	2500 ppm	5000 ppm
BaSA1	15	25	40	46	50
BaSA2	21	32	45	50	55
BaSA3	27	39	48	55	60
BaSA4	32	44	52	60	66
BaSA5	38	49	60	66	70
SA	19	24	39	44	49

Table 3b. Sensitivity of the composites to toluene vapor at their respective operating temperature and at different concentrations.

Composites	100 ppm	500 ppm	1000 ppm	2500 ppm	5000 ppm
BaSA1	8	15	29	35	40
BaSA2	12	21	33	39	45
BaSA3	18	27	40	45	50
BaSA4	24	34	42	49	55
BaSA5	29	39	49	55	61
SA	15	20	35	40	45

It is commonly known that the major adsorption occurs in the micropores (diameter $d < 2$ nm) while weaker adsorption occurs in the mesopores ($2 < d < 5$ nm). The results from our studies infer that the composite that possessed a mixture of micro and mesopores (BaSA5) showed the higher sensitivity towards VOCs. In this case the adsorption process would have proceeded through a sequence of diffusion steps from the bulk phase in to the mesopores and then to the micropores. It is observed that the sensitivity of the composites towards VOC vapors is in the order benzene > toluene that can be explained on the basis of their molecular size. The molecular size of benzene and toluene is about 0.49 and 0.54 nm [15] respectively and the micropores ($d < 2$ nm) would be a suitable site for the adsorption of benzene and toluene vapors. Since the molecular size of benzene is less than that of toluene, more adsorption of benzene would have occurred in the pores rapidly leading to quite higher sensitivity factor than that of toluene. In addition a pure microporous or mesoporous material would not have favored higher sensitivity towards VOCs. Hence for the BaSA5 composite used in the present work, the presence of considerable mesopore volume behaves as an entrance for the movement of VOC molecules from the bulk phase to the inner micropores without any hindrance. Thus during the diffusion process the presence of mesopore is of great advantage enabling easy assess to the micropores not only for accelerating the diffusion within micropores but also increasing the equilibrium coverage of micropore surface. If the composite would have possessed microporosity alone then the pore blockage might have occurred at the mouth of the pores due to aggregation of adsorbate molecules or due to the smaller cross sectional area of the micropores. Hence if the diffusion process were longer then there would be a possibility of pore blocking that could lead to a reduced adsorption and desorption would be enhanced if mesopores are alone present where the pores are larger than the molecular size of benzene and toluene. Thus the high sensitivity of BaSA5 can be explained by the fact that the composite containing considerable mesopore volume leading to the shortening of the diffusion path of micropores for benzene or toluene adsorption to get easy assess to the composite interior.

3.5. Resistance Stability of BaSA5 Composite

To investigate the resistance stability of the maximum sensitivity composite BaSA5, we continuously measured the resistance towards 1000 ppm of benzene and toluene at their respective operating temperature for 100 h. The stability of the composite MSA5 towards benzene and toluene is shown in Fig. 7. It was found that its resistance was stable except a small variation of about 1×10^6 Ohm within the studied duration for the composite. Also after few months the composites sensitivity to VOCs was nearly the same with very low variations in sensitivity factor ($S_f \pm 2$-4), indicating good stability and durability of the sensor prepared [14, 16].

3.6 Response and Recovery Characteristics

Fig. 8 (a-b) presents the transient response-recovery characteristics, which indicated quick dynamic response of the maximum sensitivity composite BaSA5. The times of response/recovery are defined as the time required for adsorption and desorption of oxygen ions from the sensor surface to reach saturation, or to reduce the conductivity back to the initial conductance [16]. The response and recovery time of BaSA1, BaSA5 and SA towards 1000 ppm of benzene and toluene is given in Table 4. The quicker response and recovery time of BaSA5 composite may be due to the presence of higher Sr(II) content in copper aluminate matrix and the high sensitivity of the composites [14]. Thus by the addition of Sr(II) to pure barium aluminate, the response and recovery time for sensing of VOCs is reduced considerably.

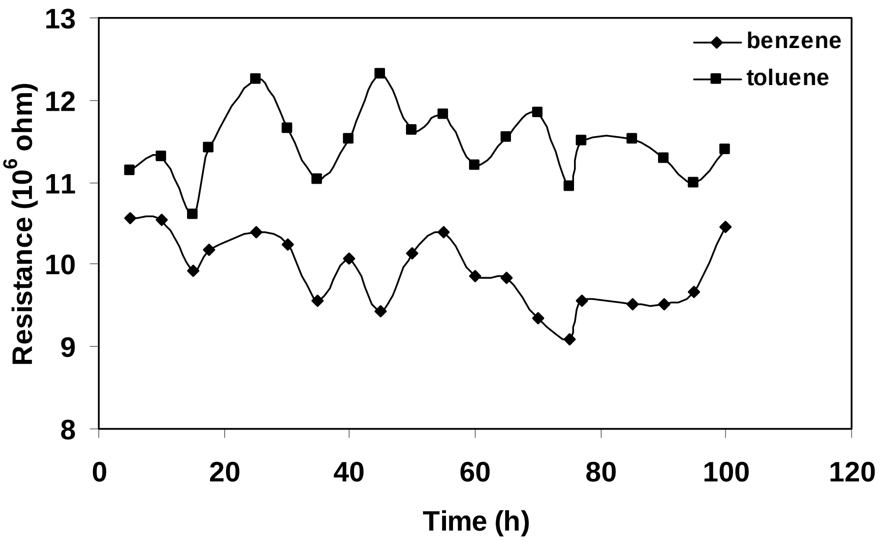
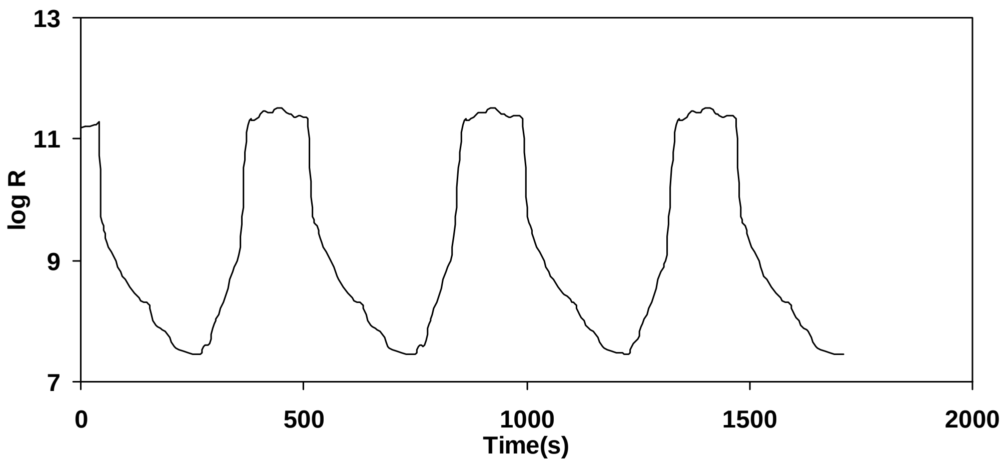
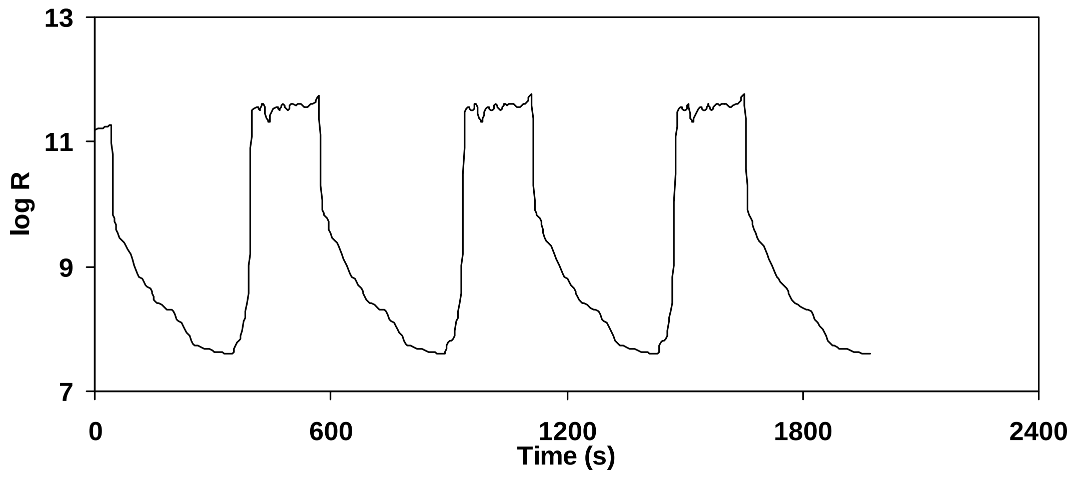


Fig. 7. Resistance stability of BaSA5 composite to benzene and toluene.



(a)



(b)

Fig. 8. Response and recovery plot of BaSA5 composite to a) 1000 pm of benzene and b) 1000 ppm of toluene.

Table 4. The Response and recovery time of BaSA1, BaSA5 and SA composites towards 1000 ppm of benzene and toluene.

composite	Benzene		Toluene	
	Response time (s)	**Recovery time (s)**	**Response time (s)**	**Recovery time (s)**
BaSA1	280	270	330	300
BaSA5	260	230	310	240
SA	310	290	340	310

4. Conclusion

A study on the VOC sensing characteristics of pure and Sr(II)-added $BaAl_2O_4$ composites prepared by sol-gel technique has been carried out. The results suggest that increase in the concentration of strontium can increase the porosity resulting in higher sensitivity values. The XRD patterns suggested the formation of single phase for barium aluminate composite while multiphasing was observed in the strontium added barium aluminate composites. It was observed that the sensitivity of the composites to VOCs was higher at their optimum temperature. The sensing properties of the composites were studied for different concentrations at their respective operating temperatures and found to be increasing with increase in VOCs concentration. The increase in surface area and porosity as shown by nitrogen adsorption-desorption isotherms and pore size distribution, with the presence of both micro and mesopores, makes the composite BaSA5 to be a better composite among the other composites prepared to detect benzene and toluene.

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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.

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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