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Research Article

Humidity Effect on Adsorption Kinetics of Aromatic and Chlorinated Hydrocarbon Vapors onto Fe_2O_3 Based Sensor

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ABSTRACT

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In this study, the influence of relative humidity on amorphous Fe2O3 thin film' sensing properties towards aromatic and chlorinated hydrocarbon vapor and their adsorption kinetics were examined systematically. The sensing results showed that the relative humidity level has a significant effect not only on the aromatic and hydrocarbon sensing performance of Fe2O3 films but also on the baseline currents of the sensors. It was found that sensitivity increased approximately twofold when the relative humidity was raised from 20% to 40% in the presence of 14% toluene vapor. A comprehensive evaluation of the sensing performance indicated that the Fe2O3 film offers promising potential as a sensing element for the detection of toluene (C7H8) vapor, even at relatively high humidity levels at room temperature. The adsorption kinetics of toluene and carbon tetrachloride (CCl4) vapors on Fe2O3 were modeled using the Pseudo-first-order equation, as well as the Elovich and Ritchie models, and the key parameters of each model were determined and analyzed. Results from regression analysis indicated that the sensing performance and adsorption kinetics are dependent on the molecular structure of the analyte molecules. The Elovich model was found to be to describe the adsorption kinetics of the CCl4 on Fe2O3. On the other hand, first-order equation most accurately described the adsorption kinetics of C7H8 vapors on the Fe2O3 thin film, The Elovich and Ritchie's kinetics models were not satisfactory.

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1. Introduction

Because of the increasing industrial activity, air quality pollution in the workplace and indoors caused by volatile organic vapors (VOCs) has become one of the most important threats to human health [1]. Studies show that VOC vapors have carcinogenic effects in both the short and long term and that there is even a direct relationship between VOC emissions and some types of cancer [2, 3]. Although VOC sensors are widely used in many different areas such as controlling and regulating industrial emissions [4] and monitoring indoor air quality [5], their use in the field of healthcare as biomarkers for early diagnosis and in food security are quite limited [6-8]. It is known that, the VOCs existing in the atmosphere, especially C_7H_8 and CCl_4, harm human health and also in terms of environmental pollution [9, 10].

As is known, C_7H_8 is an aromatic compound widely used in the furniture industry, paints, inks, and automobile fuels [11, 12]. It is also well known that long-term exposure to C_7H_8 vapor causes many diseases such as asthma and nasopharyngeal cancer due to its negative effects on the heart, kidneys, respiratory system, and nervous system, and it is listed as the third among cancer-causing VOCs by the World Health Organization [13, 14].
On the other hand, carbon tetrachloride is one of the chlorinated hydrocarbons found in the atmosphere as volatile organic vapor with a special odor and is used in many areas in the chemical industry such as organic solvents [15],

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degreasing agent, cleanout and dry-clean reagent in the chemical industry. Research has shown that exposure to carbon tetrachloride, one of the primary toxic pollutants, has significant negative effects on the liver, kidney, and central nervous system even at low concentration levels. [10, 16].

Due to the increasing use of C_7H_8 and CCl_4 in industry and their negative effect on the human body, intensive work is being done on the development of sensors for detecting low concentrations of these toxic gases. For this purpose, in addition to analytical methods such as gas chromatography and mass spectrometry [17], sensors with very different operating principles such as electrochemical, photoionization, capacitive, and surface acoustic waves have been developed [18-20]. Despite numerous efforts to enhance the VOC vapor sensing performance of single semiconductors, their sensing capabilities remain constrained by several limitations, including high operating temperatures, low sensitivity, poor selectivity, and stability issues [21].

Recently, ferric oxide functional films have found applications in diverse areas such as semiconductor materials, magnetic materials, sensitive materials, and catalysts, among others [22]. Researchers have reported the electrical responses of α-Fe_2O_3 nanostructures to various oxidizing/reducing gases and humidity [23, 24]. It has been demonstrated that factors like grain size, porosity, and operating temperature play a significant role in influencing the sensitivity of α-Fe_2O_3 to different gases and humidity levels [25]. Theoretically, the gas sensing mechanism of this type of sensor is based on a conspicuous conductivity change of oxide semiconductor, deriving from the adsorption of oxygen and the chemical reaction between oxygen species and target gas molecules. Consequently, the composition and structure of sensitive materials play crucial roles in their sensing performances. [26-28]. Stable alpha-iron oxide (α-Fe_2O_3), an *n*-type semiconductor with good sensitivity, environmentally friendliness, high corrosion resistance, and easy fabrication properties, has been extensively applied in many fields including gas sensors [29, 30]. However, interference from other components in the sensing environment often poses challenges for chemical sensors. Additionally, relative humidity (RH) in the operating environment is commonly overlooked in the design of high-performance and reliable sensing devices. While studies have shown that sensor efficiency in selective VOC vapor detection is promising, understanding the mechanisms governing the interaction between the sensitive layer and VOC vapor molecules has become increasingly necessary. In this context, adsorption kinetics, a fundamental aspect for analyzing the adsorption process, serves as an essential indicator for evaluating adsorption efficiency.

Numerous kinetic models have been developed to explain the adsorption process on solid surfaces in solid-gas interactions. These models can describe adsorption kinetics with two, three, or even more parameters. In this study, the influence of relative humidity on the sensing properties of Fe_2O_3-based sensors for detecting C_7H_8 and CCl_4 vapors was investigated. The obtained adsorption data were analyzed and discussed using four different kinetic models: Elovich equation, Ritchie equation, first-order equation, and second-order equation.

2. Experimental

The sensing film of Fe_2O_3 was deposited on interdigitated microelectrode (IDE) arrays by the dip coating method. The starting solutions for the deposition of Fe_2O_3 thin films were prepared by dissolving 0.27 gr $FeCl_3{\cdot}6H_2O$ in 20 ml de-ionized water (pH =1.50) by magnetic string at room temperature to obtain a clear solution. In a separated wesel, an aqueous solution containing 0.04 gr NaOH with a pH value of 10.8 was prepared. Then, the cleaned IDE arrays were immersed successively in $FeCl_3{\cdot}6H_2O$ and NaOH solutions and DI-water for 20 s. The process was repeated 70 times with the same solutions.

Here, the cycle number refers to the immersion of the transducer surface in $FeCl_3{\cdot}6H_2O$ containing solution. The obtained films were annealed in atmospheric conditions at 300° C for 2 hours to remove any hydroxide phase and postannealed at 500 °C for 3 hours to get a pure phase of α-Fe_2O_3 [31].

In order to comparatively test the sensing performances of the amorphous phase of the Fe_2O_3 film for C_7H_8 and CCl_4 vapors; the concentration of C_7H_8 varied between 2% and 14% and the concentration of CCl_4 varied between 1% and 4% and the sensor currents were measured using Keithley model 617 electrometer. After the coating of Fe_2O_3 on the surface of the transducer, the sensors were placed in a homemade sensing cell and exposed to carrier gas (99.9 % pure N_2 was used as carrier gas) flow until a stable baseline current was reached.

Afterward, the Fe_2O_3-based sensor surface was exposed to seven well-defined different concentrations of C_7H_8 and CCl_4 vapors for 10 min and the variations in sensor current were recorded. In order to test whether the changes in the sensor current were reversible, the sensor surface was exposed to only the carrier gas at the same flow rate for another 10 min. The real-time sensing test cycle for C_7H_8 and CCl_4 involves exposing the sensor surface to a gas flow with seven distinct concentrations of target molecules, followed by purging with a carrier gas.

Details of the VOC vapor sensing experiment were provided by Gümrükçü et al. [32]. Desired concentrations of C_7H_8 and CCl_4 vapor was obtained by bubbling the carrier gas through liquid C_7H_8 and CCl_4. For relative humidity dependent investigations, the carrier gas was divided into two parts using computer driven mass flow controllers (Alicat Scientific, Inc.). One of them was passed through deionized water and the other one liquid phase of the target molecules. To examine the effect of relative humidity on sensing performance and adsorption kinetics, level of the relative humidity inside the test chamber was varied between 0% and 40% throughout the C_7H_8 and CCl_4 sensing experiments and controlled with a commercially available humidity meter.

3. Result and Discussion

3.1. Structural analysis

Structural analysis of the Fe_2O_3 films used as the sensing element in the sensors produced was carried out by using the X-ray diffraction (XRD) method and observed spectra for the films obtained after 30, 50, and 70 cycles are shown in Fig. 1. The fact that no dominant peak was observed in the XRD spectrum (Fig. 1) clearly shows that the produced Fe_2O_3 films have an amorphous nature.

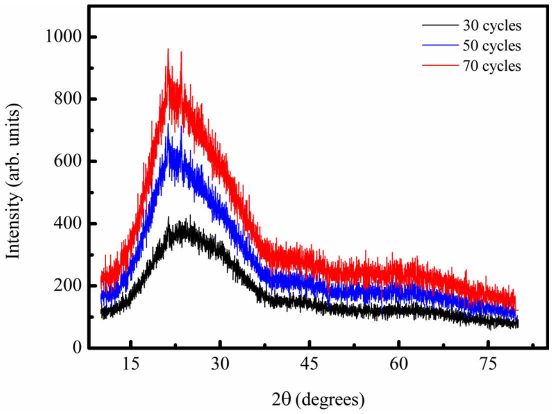

Figure 1. The effect of the number of the cycle on the XRD spectra of the Fe_2O_3 thin film

Additionally, as can be seen in Fig. 1, it was observed that by increasing the number of cycles from 30 to 70, the intensity of the broad peak in the XRD spectra increased. On the other hand, it was also observed that the width of the obtained XRD spectra decrease without any shift in its position. This behavior observed in the XRD spectra of the film was interpreted as an increase in the crystallization and grain sizes of the film with increasing cycle of numbers.

3.2. Sensing experiments

C_7H_8 and CCl_4 sensing performance of the Fe_2O_3 films were performed for seven different concentrations of target molecules at room temperature (297 K) and different relative humidity (RH) varying from 0% to 40%. These relative humidity values were obtained by passing the dry nitrogen gas used as a carrier through DI-water at different flow rates. The relative humidities produced in this way were measured and calibrated by using a hygrometer. In studies, to determine the effect of relative humidity on the sensors' C_7H_8 and CCl_4 sensing properties and adsorption kinetics, relative humidity levels were kept low to avoid condensation. Fig. 2 shows the effect of relative humidity on the sensors' current for concentrations ranging from 2% to 14% of C_7H_8 and 1% to 4% of the CCl_4. The rationale behind choosing different C_7H_8 and CCl_4 concentrations is based on the results obtained from preliminary studies [32].

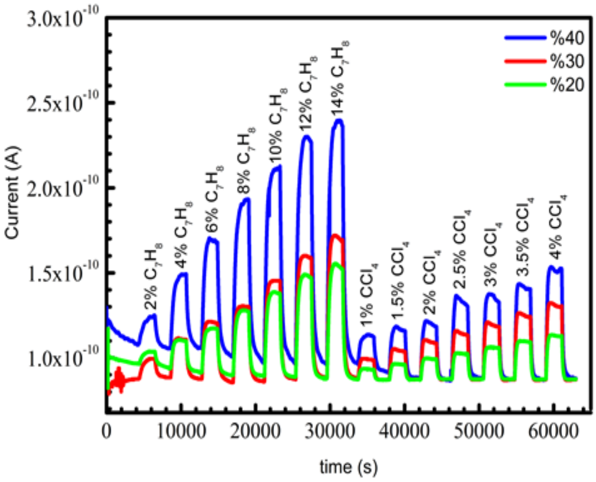

Figure 2. Relative humidity effect on C_7H_8 and CCl_4 response-recovery characteristics of the sensor

Fig. 2 clearly shows the effect of relative humidity level on both the baseline current of the sensors and the response-recovery characteristics towards C_7H_8 and CCl_4 vapors. It has been seen in Fig. 2 that the increase observed in the sensor baseline current with increasing relative humidity is compatible with similar studies in the literature. Studies on this subject showed that the resistance change in spinel-type metal oxides with RH level mainly is due to the adsorption of moisture molecules on the surface and capillary condensation [33]. Moisture adsorbed on the film surface used as the sensing unit may cause structural changes by forming hydrogen bonds with the sensing unit.

Additionally, it may cause swelling by diffusing within the sensing layer. The observed increase in baseline current with relative humidity can be attributed to the formation of a more ordered structure as a result of all these effects. A more ordered structure means higher charge mobility and higher electrical conductivity. The response-recovery characteristics presented in Fig. 2 show that when the sensor surface is exposed to different concentrations of C_7H_8 and CCl_4 vapors, it causes an initial rapid increase in the sensor current, and the rate of increase in sensor current slows down over time and tends to reach a steady state value.

As can be clearly seen from Fig. 2 when the sensor surface is exposed to the carrier gas, the sensor current starts to decrease again and reaches its initial value. This observation shows that the sensor response is reversible, thus the adsorption of the target molecules on the Fe_2O_3 film surface is physical adsorption. Although a satisfactory explanation of the interaction between metal-oxides and gas molecules has not been given so far, a reasonable explanation of the interaction between the Fe_2O_3 film surface and VOC vapor molecules, which causes an increase in the electrical conductivity of the films, can be given as follows.

Literature studies show that the electrical conductivity of metal-oxide thin films under atmospheric conditions is determined by reactions including charge exchange between oxygens in the atmosphere and the oxide [34]. Under atmospheric conditions, oxygen molecules in the environment are ionized as O^- or O_2^- by capturing the free electrons on the Fe_2O_3 film surface, and a depletion region is formed. This means that the charge carrier concentration and their mobility decrease, and as a result the sensor current also decreases. On the contrary, in the C_7H_8 and CCl_4 atmosphere, the reaction between C_7H_8 and CCl_4 molecules and oxygen species will release electrons. This process reduces the surface barrier and consequently, the electrical conductivity of the film increases.

The sensitivity, response, and recovery time of the gas sensor towards target odors are very important factors that relate directly to the rate of gas detection. The effect of the RH on the C_7H_8 and CCl_4 vapor sensing performance of the film of the Fe_2O_3 was compared in terms of sensitivity, response, and recovery time. Fig. 3 shows the variation of the C_7H_8 and CCl_4 sensitivity (S) of the sensor as a function of analytes (toluene and carbon tetrachloride) vapor concentrations for various humidity levels. It is worth noting here that sensor sensitivity, as usual, is defined as;

$$S = \frac{\Delta I}{I_0} \qquad (1)$$

In Equation (1), ΔI and I_0 show the changes in the sensor current when the sensor surface is exposed to analyte molecules and the baseline current, respectively. As can be seen in Fig. 3, a linear relationship was observed between both C_7H_8 and CCl_4 vapor concentrations and changes in the sensor current. Additionally, it is clearly seen that the sensor sensitivity increases as the RH level increases. For example, it has been observed that by increasing the relative humidity from 20% to 40%, the sensitivity of the sensor to C_7H_8 vapor at 14% concentration increases approximately two times.

A plausible explanation for the humidity dependence of the sensor sensitivity is as follows. Adsorbed water molecules on semiconductor oxides can alter the states and reactivity of adsorbed oxygen, which in turn affects the sensor's response to VOC vapor.

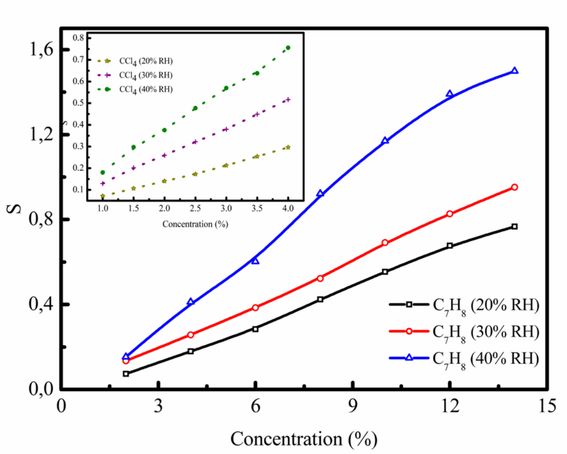

Figure 3. The variations of the sensor sensitivities as a function of target molecule concentrations

The response time is associated with the speed of change in the output on a stepwise change of the measurand. Fast response to a change of gas concentration is one of the key properties for assessing the suitability of sensors for VOC vapors safety. Response and recovery time are commonly used terms to define the speed of response of gas sensors. Response time is defined as the time it takes for the sensor to change its output signal from the initial state in the air to a certain percentage of the final value. The most common definition uses 90% of the final response.

Therefore, one of the most important parameters in gas sensors is the response time. In order to determine the influence of relative humidity on the response times of the sensors for C_7H_8 and CCl_4 vapors, a parameter denoted by τ_{90} has been defined. Here, as is customary in the literature, τ_{90} refers to the time required for the response (in our case sensor current) of the sensor to reach 90% of its maximum value when the sensor surface is exposed to the target molecules. Another important parameter for a gas sensor is the recovery time which is usually denoted as τ_{10}. The recovery time is defined as the time required for the sensor response to decrease to 10% of its initial value. Fig. 4 depicts the variations of the estimated response time for C_7H_8 and CCl_4.

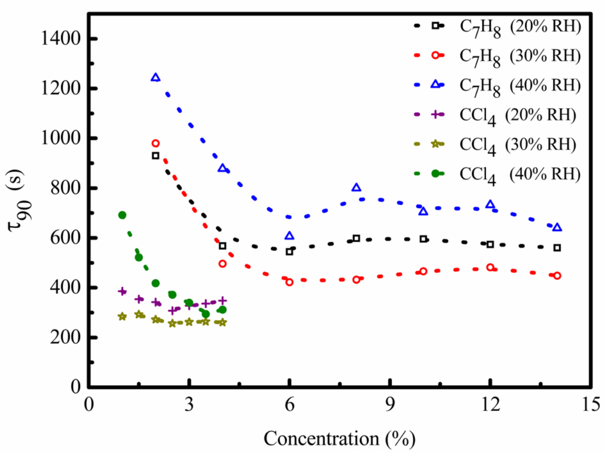

Figure 4. The variations of the estimated response time for C_7H_8 and CCl_4

On close analysis of response time plots Fig. 4 for these films, it becomes clear that the response time is decreasing function for low concentrations of C_7H_8 and CCl_4 vapors. This behaviour can be atributted to the ratio of the number of the gas molecules and the active adsorption sites. It is also clear from Fig. 4 that the response time of the sensor remains unchanged for higher concentrations of the analyte molecules.

In order to make clear the effect of the RH on the C_7H_8 and CCl_4 sensing performance of the Fe_2O_3 film, recovery times were also evaluated from the measured response and recovery characteristic of the sensor. The recovery time (τ_{10}) is defined as the time require the sensor current return to 10% below its base line value after exposure carrier gas and its concentration dependence is shown in Fig. 5. As can be seen from the Fig. 5 that, in general, the recovery time is a decresing function of C_7H_8 and CCl_4 vapor concentrations. It should also be mentioned here that the recovery time is strongly dependent on the RH level. The lowest value of the recovery time was observed for 4% of the CCl_4 at 30% RH level. On the other hand, the sensor showed the quickest recovery for 14% ppm C_7H_8 at 20% RH. An overall evaluation of the response and recovery times, it was observed that these parameters are dependent on the RH level and the gas concentration.

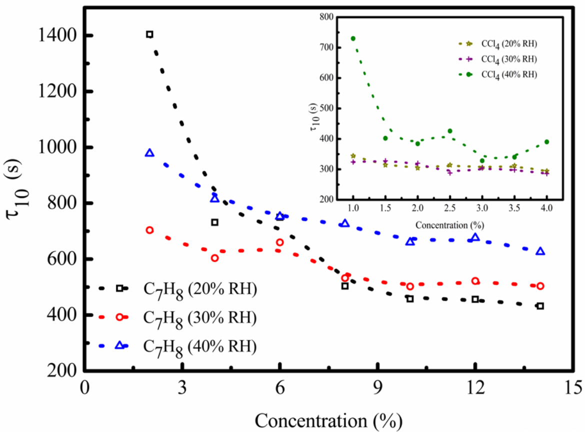

Figure 5. The effect of the relative humidity on the recovery times on Fe_2O_3-based sensors for C_7H_8 and CCl_4

4. Adsorption Kinetics

The best way to examine the interaction between adsorbent and adsorbate during the adsorption process is to examine adsorption kinetics. For this purpose, many models such as the first and second-order rate equation, Ritchie's and Elovich's equations have been developed to elucidate the gas adsorption mechanism on the solid surface. However, the number of studies in the literature comparatively examining the effect of relative humidity on C_7H_8 and CCl_4 adsorption kinetics onto the solid surface using these models is quite limited.

In this study, the adsorption data obtained during the interaction of amorphous Fe_2O_3 films with C_7H_8 and CCl_4 vapors at different relative humidity rates were analyzed using the Ritchie equation, Elovich model and pseudo-first-order equation, and the obtained results were discussed.

4.1. Ritchie equation

To explain the kinetics of solid-gas interactions, a model was developed by Ritchie in 1977 [35] based on the assumption that the adsorption rate at any time t depends solely on the fraction of unoccupied sites on the solid surface. Under these assumptions, the linear form of the Ritchie's equation can be expressed as,

$$\frac{1}{q_t} = \frac{1}{\alpha q_e t} + \frac{1}{q_e} \qquad (2)$$

where q_t and q_e are the amount of gas adsorbed at time t and after an infinite time, respectively. If the adsorption of C_7H_8 and CCl_4 on the Fe_2O_3 surface occurs by the Ritchie model, according to equation (2), the plots of $1/q_t$ versus 1/t should give a straight line. Fig. 6 shows $(1/q_t)$ vs.1/t plot for various C_7H_8 vapors in 20% RH at room temperature. As can be seen from the Fig. 6, $(1/q_t)$ vs.1/t plots for all concentrations of C_7H_8 exhibit a non-linear behaviour. The same type of behavior was observed for other RH levels investigated.

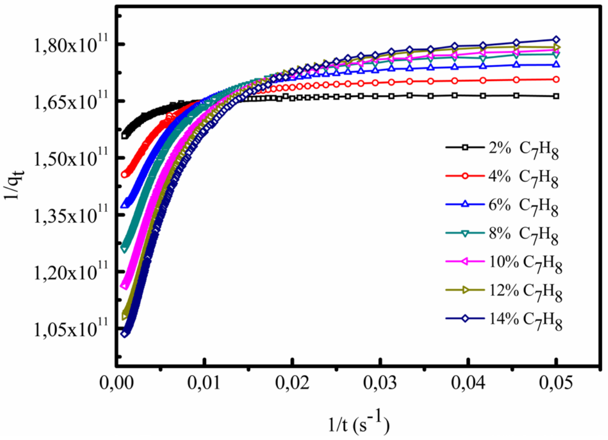

Figure 6. The plots of C_7H_8 adsorption kinetics for various C_7H_8 concentrations at 20% RH conditions

Whether the Ritchie equation was a suitable model to represent the adsorption kinetics of C_7H_8 vapor at different concentrations on the Fe_2O_3 surface, regression analysis was performed. The obtained regression analysis results showed that the correlation coefficient is in the range of 0.574 – 0.740. Additionally, the analysis of CCl_4 adsorption kinetics onto Fe_2O_3

surface according to the Ritchie's model, indicated that the correlation coefficient varies between 0.428 and 0.682 for all concentration and relative humidity examined. An overall evaluation of the regression analysis reveals that the Ritchie equation is not a suitable model to represent the adsorption kinetics of neither C_7H_8 nor CCl_4 vapor on the amorphous Fe_2O_3 surface.

4.2. Elovich model

Elovich's equation is another rate equation based on the adsorption capacity. Elovich equation was first developed to describe the kinetics of the chemisorption of gases on solids [36, 37]. According to the Elovich model, it is assumed that the rate of adsorption decreases with time because of the increase in surface coverage on the solid surface, and under this assumption, the integrated form of the Elovich equation is given by Eq. (3),

$$\theta = \left(\frac{1}{\beta}\right)\ln(\alpha\beta) + \left(\frac{1}{\beta}\right)\ln(t) \tag{3}$$

In this equation, θ represents the adsorption capacity at any time t. On the other hand, since $d\theta/dt$ approaches α when θ approaches zero, the constant α in the Elovich equation is regarded as the initial adsorption rate. If it is assumed that the changes in the electrical conductivity of the sensing uni939t when the sensor surface is exposed to the target molecules are proportional to the surface coverage, according to Eq. (3) the plots of θ against ln (t) should be a straight line. A set of θ vs. ln (t) plots the adsorption of C_7H_8 and CCl_4 vapors on the Fe_2O_3 film surface are shown in Fig. 7 (a) and (b), respectively. The validity of the Elovich kinetic model is tested by the magnitude of the regression coefficient R^2.

As can be clearly seen from Fig. 7 (a), the plots of θ vs. ln (t) for C_7H_8 adsorption deviates greatly from linearity for all concentrations of C_7H_8 investigated. The results obtained suggest that this model is not a suitable model to represent C_7H_8 adsorption on the Fe_2O_3 surface. On the other hand, a comparison of the correlation coefficients R^2 (R^2 is in the range of 0.994–0.998 for the carbon tetrachloride adsorption) shows that the Elovich model fits better the experimental data for the adsorption of carbon tetrachloride vapors onto Fe_2O_3 thin film.

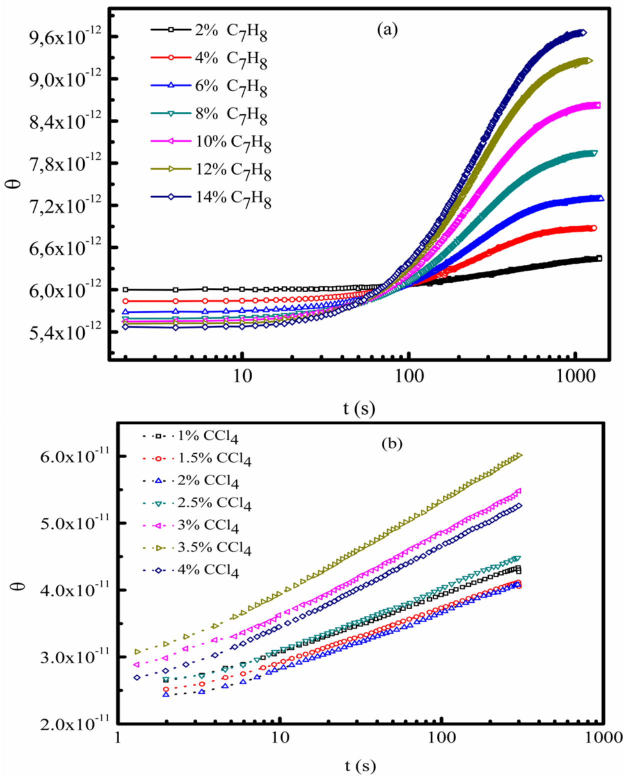

Figure 7. The plots of C_7H_8 (a) and CCl_4 (b) adsorption kinetics according to the Elovich model in a 20% RH environment

4.3. The Lagergren's first-order rate equation

The pseudo-first-order equation, also known as Lagergren's first-order rate equation, is one of the first models developed to understand the adsorption capacity of the adsorbent and the kinetics of the interaction between the adsorbent and adsorbate during the adsorption process. In this model, it is assumed that the adsorption rate on the sensor unit surface is directly proportional to the number of sites unoccupied by target molecules. It is expressed mathematically as follows [37].

$$\frac{dq_t}{dt} = k_{ads}(q_e - q_t) \tag{4}$$

where q_t and q_e represent the adsorbed target molecules at any time during adsorption processes and at equilibrium, respectively. k_{ads} is the first-order adsorption rate constant. Under this assumption, the linearized form of the Eq. (4) can be expressed as,

$$\log(q_e - q_t) = \log q_e - \frac{k_{ads}}{2.303} t \quad (5)$$

If the plot of Log (q_e-q_t) versus t was found to be linear with a good correlation coefficient, indicating that Lagergren's equation is appropriate to VOC adsorption on Fe_2O_3 film. To quantify the applicability of the first-order model, the correlation coefficient was calculated from the response-recovery characteristics of the sensor. A set of Log (q_e-q_t)- t plots for room temperature adsorption of C_7H_8 and CCl_4 with various concentrations on Fe_2O_3 surface are shown in Figs. 8 and 9 for 20% RH, respectively.

Fig. 8 reveals that the plots of Log (q_e-q_t) versus t are linear for C_7H_8 adsorption, indicating that the interaction obeys the first order equation. However, for the adsorption of carbon tetrachloride vapor, the experimental data deviated considerably from the theoretical model (see Fig. 9). Therefore, the adsorption of C_7H_8 on Fe_2O_3 was more favorable by the pseudo-first-order kinetic model.

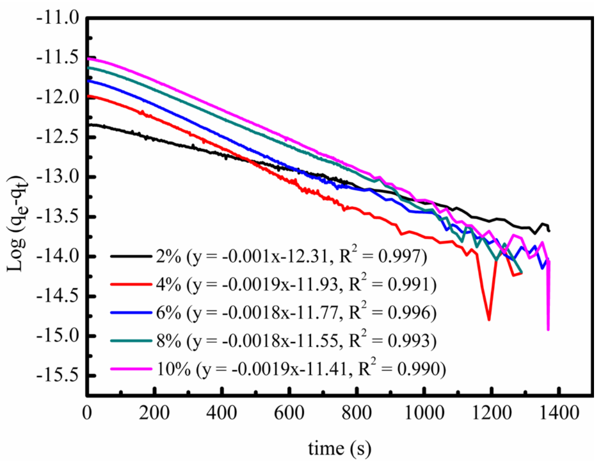

Figure 8. The dependency of the adsorption kinetics on the C_7H_8 concentration concerning Lagergren's first-order rate model at 20% RH

5. Conclusion

Fe_2O_3 films were grown on IDE structures by dip coating method. The sensing properties of these films for two different groups of volatile organic vapors, one aromatic (toluene) and the other chlorinated hydrocarbon (carbon tetrachloride) in different relative humidity environments were investigated as a function of relative humidity by measuring the changes in their electrical conductivity. The influence of relative humidity on the baseline conductivity and adsorption kinetics of C_7H_8 and CCl_4 vapor on Fe_2O_3 thin film has also been examined. Studies have shown that the gas sensing mechanism in Fe_2O_3 thin film is influenced by surface reactions, with humidity interference enhancing sensor sensitivity.

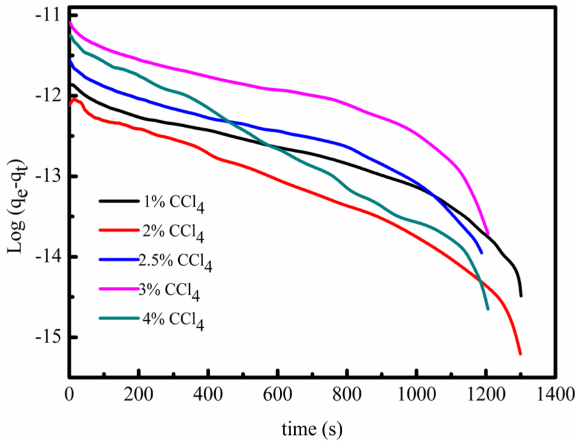

Figure 9. The dependency of the adsorption kinetics on the CCl_4 concentration with respect to Lagergren's first-order rate model at 20% RH

Observations confirmed that VOC vapor detection is feasible using the Fe_2O_3 film, even at room temperature. Experimental data were analyzed using the Pseudo-first-order, Ritchie's equation, and Elovich models. A comparison of regression coefficients (R^2) indicated that the Elovich model and first-order model best describe the adsorption of CCl_4 and C7H8 vapor, respectively. With this strategy, we hope that Fe-based materials such as Fe_2O_3, the most abundant and cheapest material in the earth's crust, will become the key material for next-generation gas sensing technologies. It was found that the physical properties of the analyte molecules are important parameters in VOC adsorption kinetics onto Fe_2O_3. Overall findings indicate that ferric oxides a promising material for IDE based VOC sensor applications at room temperature.

Article Information Form

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The Declaration of Conflict of Interest/ Common Interest

No conflict of interest or common interest has been declared by the author.

The Declaration of Ethics Committee Approval

This study does not require permission from the ethics committee or any special permission.

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