

# Adsorption of toluene and acetone vapors on microwave-prepared activated carbon from agricultural residues: isotherms, kinetics, and thermodynamics studies

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**Abstract** Activated carbons produced with agricultural residues have attracted substantial attention in recent years. However, much work remains to be done. The current study determined the adsorption isotherms for toluene and acetone vapors for pinewood chip-derived activated carbon. The effects on the physical properties, such as the porous structures of the adsorbents, were also investigated using scanning electron microscopy (SEM). The results indicate that the activated carbon is a mainly microporous structure with a micropore volume of 0.701 cc/g (75.1 % of the total pore volume). The adsorption capacities of toluene and acetone can be as high as 0.71 and 0.57 g/g at room temperature, respectively. The adsorption isotherms for toluene and acetone vapors were obtained from 25 to 45 °C, with relative pressures between 0.01 and 0.9. As the temperature increased, the adsorption capacity decreased. This behavior indicates that the adsorption process is physical adsorption. The fitted isotherms using the Dubinin–Radushkevich adsorption models exhibited low mean total relative errors. The fitted isotherms obtained using the Dubinin–Radushkevich adsorption models had mean total relative errors of 3.3 and 4.1 % for the adsorption of toluene and acetone, respectively, at temperatures

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ranging from 25 to 45 °C. The results of this study may be highly significant because they provide a more accurate prediction of the adsorption capacities of the adsorbents, thus improving the design of adsorption systems.

**Keywords** Agriculture residues activated carbon · Adsorption isotherms · Organic vapors · D–R equation

## Introduction

Agricultural residues are disposed of by on-site combustion in the field, which results in the emission of air pollutants. In recent years, various agricultural residues have been recycled as activated carbon-producing materials for volatile organic compound (VOC; e.g., toluene and acetone) removal, such as wood sawdust, mangosteen peel [1, 2], biodiesel industry solid residue [3], pineapple peel [4], rice husks [5], jackfruit peel [6], durian shells [7], and *Pinus roxburghii* cones [8]. Activated carbons that are produced from agricultural residues have the advantages of low cost, high surface area, and extensive microporous adsorbents given their high volatile matter, lignocellulosic contents, and renewable nature [9]. Scholars have also studied the factors that influence the yield and adsorption capacity of agricultural residue-producing carbons [10, 11]. The adsorption mechanisms of activated carbons are another hot topic in related fields of interest.

The adsorption equilibrium and isotherms for VOC adsorption play important roles in determining the adsorption properties of activated carbons. The adsorption capacities of activated carbons of toluene/acetone adsorbed at equilibrium depend on the concentration of absorbable molecules in the phase surrounding adsorbent particles as well as the temperature and pressure [12]. Adsorption curves are typically measured to describe these adsorption equilibriums. The shapes of the curves are commonly classified using the Brunauer classification and are related to the physical structures of the adsorbents [13, 14]. In the case of toluene/acetone adsorbed on activated carbon, type I curves are the most common according to Brunauer–Deming–Deming–Teller (BDDT) classification [15, 16]. To further determine the adsorption equilibrium data of VOCs onto activated carbon, scholars have built models of the adsorption process and established classic adsorption isotherm models, such as the Langmuir model, the Freundlich model and the Dubinin–Radushkevich (D–R) equation [17–21]. Ramirez et al. [18] compared the adsorption isotherms of alkane, arene, and ketone steam compounds onto activated carbon fiber cloth and tire- and coal-derived activated carbon, and discovered that the adsorption isotherms for micropore adsorbents and meso-macropore adsorbents could be fitted using the Freundlich model and the D–R model. Cai et al. studied the adsorption properties and energies of three types of activated carbon using *n*-hexane and *n*-heptane as adsorbates. Their results indicated that the surface area and pore volume of activated carbon were the main factors affecting the adsorption properties. In addition, the adsorption behaviors of *n*-hexane and *n*-heptane were found to correspond to the Langmuir adsorption isotherm model [22]. Various studies have established relationships between the parameters involved in these

isotherm models and the properties of both the organic molecules and the adsorbent [23–25].

In our previous study, we proposed pinewood chips as an efficient raw precursor for the preparation of activated carbon via microwave heating [9]. The physical characteristics related to iodine numbers, carbon yields and carbon-nitrogen-hydrogen (CNH) analysis of the activated carbon were investigated. The results demonstrate that the best activated carbons were obtained using a potassium hydroxide (KOH)/char ratio of 3.0, a microwave power of 600 W, a radiation time of 30 min, and a particle size of 0.1–0.42 mm in a humid environment [10]. However, the isotherms, kinetics, and thermodynamics of this activated carbon still need to be investigated because this information will be important for the design of adsorption systems in future industrial applications.

In this study, we extended the previous study to explore the effects of temperature on the adsorption isotherms of activated carbon for toluene and acetone. We also used the D–R equation to fit the isotherm data. The results of this study may provide technical references for the design of activated carbon-producing industrial tools.

## Materials and methods

### Theoretical background: Dubinin–Radushkevich (D–R) equation

The D–R equation, which is based on Polanyi's potential theory of physical adsorption, can be used to calculate the adsorption capacity for VOCs on microporous adsorbents, such as pinewood chip-based activated carbon [26]. The D–R equation can include the effect of temperature as well as the properties of both adsorbent and adsorbate. The D–R equation is given by [18]

$$q = W_0 \exp \left[ - \left( \frac{RT \ln(P_0/P)}{E} \right)^2 \right] \quad (1)$$

where  $q$  is the adsorption capacity (% , mass) of the adsorbate,  $P/P_0$  is the relative pressure (the ratio of the adsorbate vapor pressure to the saturated vapor pressure), and  $k$  and  $W_0$  are constants that depend only on the pore volume and surface area of the adsorbate, respectively. In this study,  $k$  and  $W_0$  can be calculated from the adsorption isotherm data. The objective of this investigation is to measure and fit the D–R model for the effect of temperature using a sorption analyzer at high concentrations.

### Preparation of pinewood-activated carbon

The pinewood chips were collected from Millar Western Forest Products Ltd. in Alberta, Canada. The samples were first air-dried for 1 day to achieve moisture balance and subsequently oven-dried overnight at 80 °C [9]. The preparation of the pinewood-activated carbon included carbonization and activation steps [9]. Before

activation, all of the samples were placed in a laboratory oven at 110 °C for 1 day to remove water vapor. KOH was dissolved in water and mixed with the pinewood char samples. Then, the mixture was dried in the oven at 110 °C for approximately 2 days until the weight was stable (less than 0.5 % change in 24 h). KOH (purity > 85.0 %, Wako Pure Chemical Industries, Ltd.) was used as the activation agent. After drying, 30 g of the char and KOH mixture (KOH:char ratio of 3:1 by weight) was placed in a glass reactor (diameter 2.6 cm, height 20 cm) and heated in a microwave activation setup (Supplementary files Fig. 1). The 2450-MHz microwave source (Goldstar Co., Ltd.) was used with a nominal power output of 600 W, and the mixture was irradiated for 30 min under a 0.5-L/min flow of nitrogen gas ( $N_2$ ) humidified by bubbling through water. Toluene (99.99 %) and acetone (99.5 %) were used as adsorbates.

After microwave activation, the KOH-impregnated char was cooled to room temperature under humid  $N_2$  at 0.5 L/min. The samples were then mixed with 0.1-N hydrochloric acid (HCl) and stirred for 1 h and then washed with deionized water until a pH of 7 was obtained. The activated char samples were dried at  $110 \pm 5$  °C for 24 h.

### **Brunauer–Emmett–Teller surface and SEM characterization of activated carbon**

The Brunauer–Emmett–Teller (BET) surface areas of the activated carbon samples were measured using  $N_2$  adsorption isotherms at 77 K with an automated gas sorption analyzer (Autosorb iQ2 MP, Quantachrome Instruments). Multipoint BET equations [27] were used to calculate the BET surface area using adsorption data recorded in the relative pressure range of 0.01–0.07. The total pore volume [27] was derived from the amount of  $N_2$  adsorbed at a relative pressure of 0.99, assuming that all of the pores were filled with liquid  $N_2$ . Micropore volume and surface area were determined using the t-plot method [28].

The shapes and morphologies of the pinewood and wheat straw char were evaluated using SEM (Hitachi S-2500, Tokyo, Japan). The samples were coated with gold to ensure that the particles had suitable conductivity. For better image quality, the secondary electron images were selected. The acceleration voltage was set to 8 kV, and the magnification was 200-fold.

### **Adsorption experiments**

The adsorption isotherms were obtained gravimetrically using a sorption analyzer (TA Instruments, model VTI-SA) at 25, 35 and 45 °C using  $N_2$  as the carrier gas. The system logged the equilibrium weight of the pinewood chip-activated carbon sample (3–5 mg) in response to a step change in the concentration of the adsorbate (relative pressure range of 0.01–0.9) in the carrier gas. The equilibrium was assumed to be reached when the weight changed by less than 0.001 % in 5 min.

## Results and discussion

### BET surface area and pore size distribution

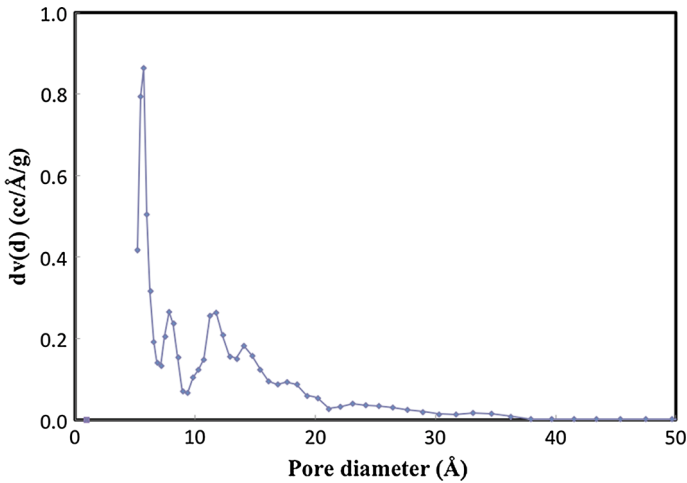
During the adsorption process of activated carbon, the corresponding adsorption energy can be determined. The magnitude of the adsorption energy is related to the specific surface area and pore structure of activated carbon, the nature (polar or non-polar) of the adsorbate molecules, and the distance between the adsorbate molecule and the adsorbent surface, which reflects the energy change during the adsorption process [12]. Therefore, to study the effect of the pore structure of activated carbon on toluene/acetone adsorption behavior, an automated gas sorption analyzer was used to analyze the microstructure of activated carbon, including the specific surface area and pore structure.

The main properties of the adsorbent are summarized in Table 1. The specific surface area of the activated carbon was 2044 m<sup>2</sup>/g, and the total pore volume was 0.9329 cc/g, of which the micropore volume reached 0.701 cc/g. These results are comparable with those of activated carbon prepared from other precursors, such as mangosteen peel, biodiesel industry solid residue, pineapple peel, rice husks, jackfruit peel, durian shell and coke [2–7] using KOH or potassium carbonate (K<sub>2</sub>CO<sub>3</sub>) microwave activation. For example, the activated carbon mixed with KOH achieved a higher BET surface area (1006 m<sup>2</sup>/g) than that mixed with K<sub>2</sub>CO<sub>3</sub> (680 m<sup>2</sup>/g) when using pineapple peels with microwave heating. Therefore, pinewood chips are a good precursor for the production of activated carbon with high surface area and pore volume. Additionally, V<sub>micro</sub>:V<sub>pore</sub> (0.75) indicates that the prepared pinewood activated carbon is microporous according to International Union of Pure and Applied Chemistry (IUPAC) classification. Pinewood chip char activated by treatment with KOH and microwave heating may develop micropores (pore size < 2 nm) that enhance the adsorptive capacity for small molecules, such as gases, according to the theory of the volume filling of micropores [9].

Figure 1 illustrates the pore size distribution of the pinewood char activated with microwave heating. The sharpest peak of the pinewood-activated carbon occurred at pore diameters from 5 to 10 Å, which indicated that the vast majority of pores fell into the micropore range. Microporosity results from the

**Table 1** Main characteristics of pinewood chip-activated carbon

	Values determined using the VTI-SA analyzer
BET surface area (m <sup>2</sup> /g)	2043.7
Micropore area (m <sup>2</sup> /g)	1712.2
Micropore volume (cc/g)	0.701
Total pore volume (cc/g)	0.933
S <sub>micro</sub> /S <sub>p</sub>	0.84
V <sub>micro</sub> /V <sub>pore</sub>	0.75



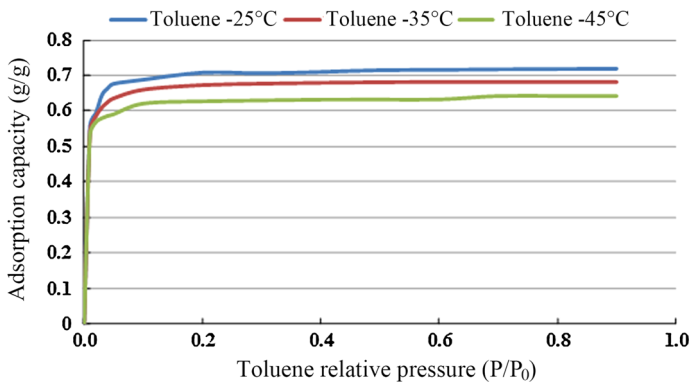
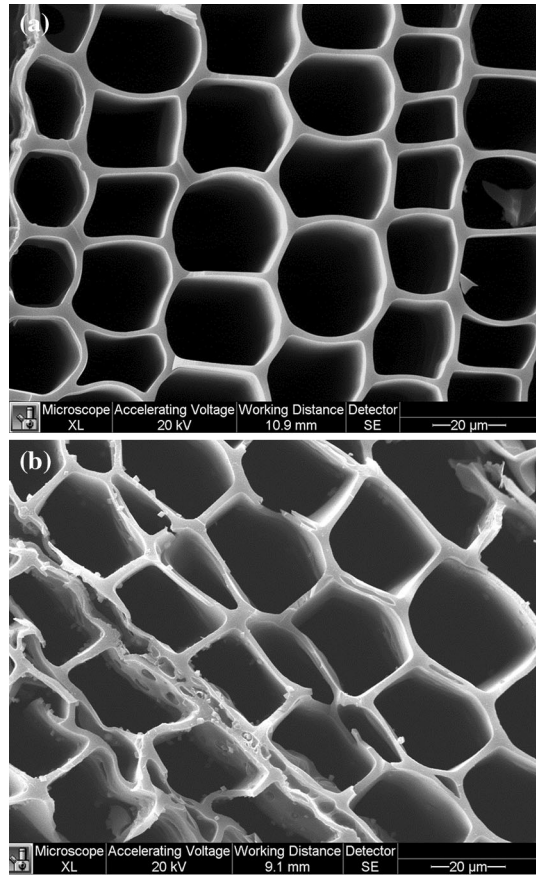
**Fig. 1** Pore size distribution of pinewood char-activated carbon with a KOH:char mass ratio of 3.0

initial porosity of the precursor material (e.g., the tracheids) that facilitated KOH impregnation into the char and from the chemical reactions between KOH and char during microwave heating. It was assumed that KOH was reduced to metallic potassium during the carbonization process and that metallic potassium formed during the gasification process would diffuse into the internal structure of carbon matrix, thereby widening the existing pores and creating additional ones [9].

### SEM micrographs

Figure 2a and b show SEM images at 2000 $\times$  magnification of raw pinewood char- and pinewood-activated carbon. The original pinewood char clearly contains honeycomb-shaped voids, which confirms that pinewood char maintains an unbroken and smooth cell wall shape after carbonization. These ordered holes provide channels for the adsorbate molecules, such as K or VOCs, to access the micropores and mesopores inside a carbon particle. The microwave-irradiated sample (Fig. 2b) exhibited a well-developed and uniform surface with an orderly pore structure. However, compared with the raw pinewood char, the pinewood-activated carbon (Fig. 2b) appears twisted because of the KOH used to activate the wall, which resulted in partial yield loss, inward hole contraction, and a smaller hole diameter. The surface of the pinewood-activated sample also contains some cavities resulting from the evaporation of impregnated KOH-derived compounds, leaving the space previously occupied by the reagent [29]. The surface of the wall can be seen to have abundant pores, which were developed by KOH activation associated with microwave irradiation.

**Fig. 2** SEM images of raw pinewood char (a) and pinewood-activated carbon (b)



**Fig. 3** Pine-activated carbon adsorption isotherms for toluene at temperatures of 25–45 °C

### Impact of temperature on the adsorption isotherm of activated carbon

At different temperatures, the adsorption isotherms of toluene/acetone onto activated carbon are as indicated in Figs. 3 and 4. At temperatures of 25, 35 and 45 °C, the adsorption isotherms of toluene/acetone onto activated carbon exhibit the same variation tendency, namely, the adsorption capacity decreased as the temperature increased. Using the isotherm of toluene as an example, the adsorption capacity can be seen to decrease from 0.71 to 0.64 g/g when the temperature increased from 25 to 45 °C. In the case of acetone, the adsorption capacity ranged from 0.58 to 0.52 g/g, with as the temperature increased from 25 to 45 °C. This result is consistent with that of Ramirez et al. [18].

According to the IUPAC category, these adsorption isotherms were classified as type I; i.e., for the adsorption isotherms, minute changes of pressure might cause a drastic change in adsorption capacity at low relative pressure. At high relative pressure, the adsorption isotherm exhibited a horizontal shape or approximated a horizontal shape, indicating that the initial portion of the adsorption isotherm, in which the vast majority of pores occurred in the micropore range, represented the micropore filling of activated carbon. This result demonstrates that the adsorption capacity increased slowly at high concentrations. Specifically, the slope of the adsorption isotherm gradually decreased and tended to be flat. This adsorption isotherm shape can be attributed to the fact that although the concentration was low, the difference between the concentration inside and outside the micropore was quite small. Here, the micropores of activated carbon were extremely developed and could rapidly and effectively adsorb the toluene/acetone molecules. As a result, the adsorption capacity increased rapidly. At high concentration, although a large difference was noted between the concentration inside and outside the micropore, the micropores tended to exhibit adsorption saturation due to the limit of the adsorption capacity of activated carbon. Therefore, the adsorption capacity of VOCs increased slowly and gradually plateaued. At high relative pressure, the slope of the platform was caused by multilayer adsorption on the non-microporous surface. The

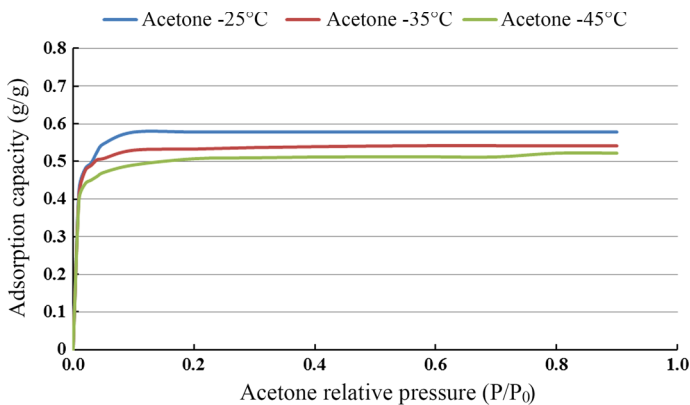


Fig. 4 Pine-activated carbon adsorption isotherms for acetone at temperatures of 25–45 °C



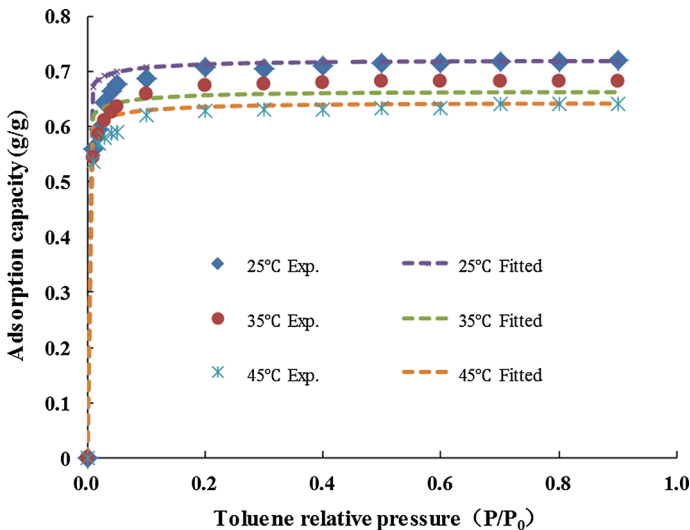
adsorption capacity of toluene/acetone decreased as the temperature increased, which indicated that the adsorption of VOCs onto pinewood-activated carbon was an exothermic process.

### Fitting of the D–R adsorption isotherm

The adsorption isotherm reflects the adsorption mechanism of the adsorbent adsorbing the adsorbate; in addition, the equilibrium adsorption capacity and the adsorption isotherm equation mathematically describe the adsorption isotherm [12]. For different adsorption mechanisms, various adsorption isotherm equations could be used to fit the adsorption experimental results. In fact, no single model is suitable for all of the adsorbents and all of the adsorption isotherms. Generally, one model only has good prediction ability for a certain adsorption system within a certain pressure and concentration range. This experiment aimed to explore the adsorption and kinetic behaviors of agricultural residue-activated carbon at different temperatures (25, 35 and 45 °C). According to the measured pore structure parameters, the D–R equation was adopted for the fitting of the corresponding isotherms. This equation is mainly used in the microporous adsorption for porous adsorbents. Using the equation, the micropore volume can be estimated. The expression for the D–R equation is indicated in Exp. (1). Similarly, the Clausius–Clapeyron equation was adopted to calculate the adsorption heat as follows [12]:

$$\ln p = -\Delta H/(RT) + B, \quad (2)$$

where  $R$  is the gas constant,  $T$  is the adsorption temperature,  $p$  is the adsorption pressure, and  $\Delta H_m$  is the adsorption heat. According to the equation, by measuring

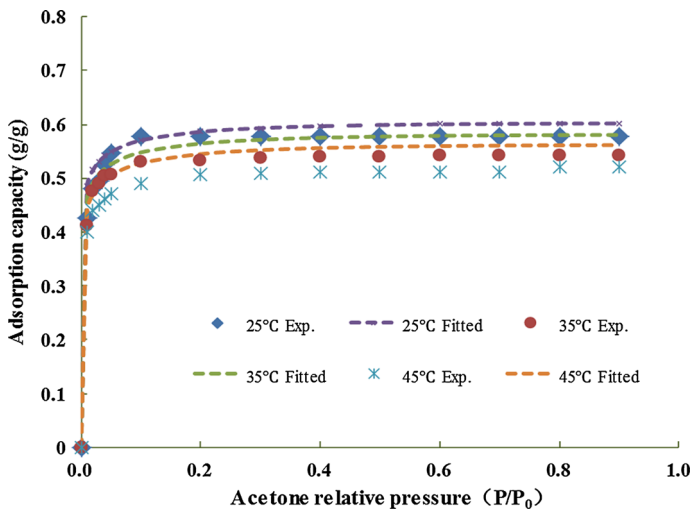


**Fig. 5** Pinewood-activated carbon adsorption isotherms and D–R model fitting for toluene at 25, 35 and 45 °C

two pressure values corresponding to the same adsorption capacity in the isotherms at two different temperatures, the isosteric heat of adsorption for the adsorption capacity could be solved. Specifically, using the Clausius–Clapeyron equation at the two temperatures, the isosteric heat of adsorption of toluene/acetone onto the agricultural residue-activated carbon can be derived. Using this approach, the adsorption potential energy  $E$  in Exp. (1) is solved.

The fittings of the adsorption isotherm D–R model of toluene/acetone and heat of adsorption during the adsorption process onto activated carbon are indicated in Figs. 5 and 6 and Table 2. For either polar or non-polar adsorbates, the D–R model can be adopted for good fitting of the experimental data of toluene/acetone adsorbed onto pinewood activated carbon, which is consistent with the predicted results. This agreement can be ascribed to the suitability of the D–R model to fit the microporous adsorption isotherm. The micropore volume of the activated carbon was 0.701 cc/g, which accounted for 75.1 % of the total pore volume. Using the fitting parameters in Table 2, the D–R equation fits the isotherm data of toluene adsorbed in the experiment with an average relative error of only 3.3 % and an  $R^2$  of 0.976. The heats of adsorption (Table 2) for toluene and acetone were calculated using the Clausius–Clapeyron equation. The values of  $\Delta H$  are 44 and 24 kJ/mol for toluene and acetone, respectively, which indicate that the process of adsorption is exothermic.

Activated carbon fiber cloth (ACFC), tire-derived activated carbon (TDAC) and coal-derived activated carbon (CDAC) are adsorbents that exhibit similar properties given that the fitted and D–R models had a mean total relative error between the experimental and modeled results of only 5.6 % for acetone between 20 and 50 °C [18]. Lashaki et al. [12] investigated the effect of the kinetic diameter (KD) of the reference adsorbate on the accuracy of the D–R equation for predicting the



**Fig. 6** Pinewood-activated carbon adsorption isotherms and D–R model fitting for acetone at 25, 35 and 45 °C

**Table 2** D–R model constants for toluene and acetone adsorption on pine-activated carbon

Sample	Adsorbate	$E$ (kJ/mol)	$W_0$ (cc/g)	Mean relative error (%)	$R^2$	$\Delta H$ kJ/mol
Pinewood-activated carbon	Toluene	44.2	0.764	3.3	0.976	44
Pinewood-activated carbon	Acetone	22.8	0.710	4.1	0.956	24

adsorption isotherms of organic vapors on microporous activated carbon. The isotherm results of this study are consistent with those of previous studies.

However, at low pressure, the simulated result of the D–R equation deviated somewhat from the experimental values mainly because the D–R equation itself could not regress to Henry's law at low pressure. In contrast, when the pressure gradually increased, the simulation effect of the D–R equation improved. Observation of the adsorption isotherm of toluene onto activated carbon at 25, 35 and 45 °C revealed that the response of activated carbon follows the micropore filling theory. In 1949, Pierce and Dubinin et al. arrived at the following conclusions based on their research. The micropore adsorption mechanism cannot be attributed to the surface covering of adsorbate onto the pore wall but is instead due to micropore filling, which exhibits similarities and differences with capillary condensation. The structure of activated carbon is mainly microporous. When micropores are filled, increasing the pressure only slightly increases the adsorption capacity (because of the multilayer adsorption occurring on the surface); therefore, a plateau appears in its adsorption isotherm [25–29].

The short activation time (30 min) and simplicity of the activation process and this fitting demonstrates that microwave-assisted activation is a promising method to convert agricultural residues into a useful adsorbent with a high adsorption capacity. In our future work, a more complete study will be conducted that includes more adsorbents/adsorbates to test the D–R model fitting for the adsorption capacities of VOCs to predict the adsorption isotherms of any compound. Meantime, we will attempt to use COMSOL software to predict the adsorption isotherms of VOCs on the activated carbon derived from agricultural residues via microwave heating.

## Conclusions

This research focused on measuring the adsorption isotherm of toluene/acetone onto pinewood-activated carbon prepared by the microwave method using an automated gas sorption analyzer and explored the adsorption behavior of toluene/acetone onto activated carbon at different temperatures (25, 35 and 45 °C). The D–R equation was adopted to fit the adsorption isotherm data obtained in the experiment. Based on the results of this study, the following conclusions are reached:

1. At different temperatures (25, 35 and 45 °C), toluene and acetone exhibit similar trends regarding the adsorption isotherm of pinewood-activated carbon.

- In addition, their adsorption capacity decreases as the temperature increases, indicating that the adsorption of VOCs onto activated carbon is an exothermic process and that the adsorption of toluene/acetone is physical adsorption.
- At different temperatures (25, 35 and 45 °C), the D–R model can be adopted for quite good fitting of the adsorption isotherm of toluene/acetone onto agricultural residue-activated carbon.

These results are important because they can be used to accurately predict the adsorption isotherms of pollutants and hazardous compounds and to design a reliable system for their abatement. The D–R model fit the adsorption capacities of the organic vapors and microporous-activated carbons tested herein, and the fitting procedure is easier, simpler, and less expensive than conducting experiments at various temperatures. Moreover, this fitting demonstrated that microwave-assisted activation is a promising method to convert agricultural residues into a useful adsorbent with a high adsorption capacity. This paper provides basic data for studying mixtures of VOCs and activated carbon obtained from agricultural residues via microwave heating.

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### Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflicts of interest.

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