A novel analytical method for the in-depth study of the effects of humidity and temperature on the adsorption of volatile organic compounds

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Abstract: We further explored the effect of temperature and humidity on the adsorption of volatile organic compounds on activated carbon. The equilibrium adsorption capacity of methylene chloride and water on activated carbon at different temperatures and different absolute and relative humidity was determined by dynamic adsorption method using dichloromethane as the target. The temperature and humidity are controlled by the temperature regulation and humidity control process. Regression analysis was performed on dichloromethane concentration, temperature and absolute humidity. The regression analysis yielded the equation: \( Y=0.01138X_1-0.000968X_2-0.000326X_3+0.0492 \).

1. Introduction

VOCs (Volatile Organic Compounds, VOCs) are a general term for a class of organic compounds. Many processes in industrial production emit a large amount of exhaust gas containing volatile organic compounds, which cause harm to human health and serious pollution to ambient air[1-2].

VOCs treatment methods [3] mainly include adsorption methods, various types of thermal oxidation methods, condensation, room temperature photocatalytic oxidation, biological treatment technology and so on [4]. Activated carbon adsorption method is a main method for purification and recovery of organic waste gas. However, in the actual application process, the efficiency of adsorption of VOCs by activated carbon is reduced, and the operating cost is increased. This is due to the unreasonable adjustment of the humidity and temperature of the purified gas, or the insufficient drying of the activated carbon in the regeneration process, or the competitive adsorption from the water [5-6]. In recent years, many studies have been carried out on the effects of gas humidity on the adsorption equilibrium of activated carbon and the adsorption mechanism of activated carbon on water vapor. At present, there are two main theories about the adsorption mechanism of water on the surface of activated carbon: (1) the mechanism of capillary condensation; (2) cluster growth mechanism [7-8]. Juan [9] analyzed the water vapor adsorption mechanism of activated carbon with different pore size properties (narrow micropores, wide micropores, mesoporous and macroporous) and the content of functional groups on the surface.

Relative humidity is a composite variable of temperature and absolute humidity. And the adsorption equilibrium is also affected by temperature. Therefore, this study carried out a study on the combined effects of temperature and humidity (relative humidity, absolute humidity) on the adsorption of volatile organic compounds at different concentrations on activated carbon, in order to provide a basis for optimizing the adsorption process and parameters. In this study, dichloromethane was used as the
research object. Three concentrations of 200, 1000 and 5000 mg/m$^3$ were selected. The equilibrium adsorption capacity of dichloromethane and water was tested by dynamic adsorption method under different temperature and humidity conditions. Through the above methods, we studied the effects of temperature, humidity and adsorbate concentration on the adsorption of dichloromethane, and discussed how to improve the temperature regulation and humidity control process of the gas to be treated in the subsequent adsorption process of organic matter.

2. Material and Methods

2.1. Experimental apparatus of dynamic adsorption system

2.1.1 Experimental apparatus of adsorption. The adsorption equilibrium experimental apparatus for water and dichloromethane on activated carbon is shown in figure 1, and is improved by an experimental apparatus adopted by Li Zhaohai et al. It is mainly composed of a gas distribution system, an adsorption system, and a detection system[10]. The experiment used ambient air as the gas source, and the air after drying with silica gel (relative humidity RH≤5%) was used as blank reference in the experiment.

The gas distribution system is divided into: blower (Haley, ACO-9730), silica gel drying device, two sets of micro-injection metering pumps (LongerPump, LSP01-2A), two sets of heaters and a 2L mixing buffer chamber. The dried air is divided into two paths, which are adjusted by the rotor flow meter and mass flow meter (TSI, 4000Series) for accurate quantification. The two gases are mixed in the mixing buffer chamber and enter the adsorption system at a set concentration of dichloromethane, humidity and flow.

The adsorption system consists of a gas preheating and precooling line and a glass tube adsorption column. The adsorption column is a glass column with an inner diameter of 40 mm, a length of about 100 mm, a filling height of 60 mm, and a quartz sand cushion at the bottom. The gas preheating pipeline is concentric spiral coil with copper, and the gas precooling pipeline is a cooling system using ethylene glycol as a cold medium. During the experiment, the adsorption system was placed in a normal temperature environment of 23 °C, and the gas flow temperature was controlled by a gas pre-cooling line at 23 °C (± 1 °C). At temperatures of 30 °C, 40 °C and above, the adsorption system was placed in a constant temperature oven. And the temperature of the gas flow was controlled by a concentric spiral coil with copper to reach the experimental temperature (± 0.5 °C). A detection port is set before and after the adsorption system to detect the concentration, relative humidity and temperature of the experimental object in the gas path.

![Figure 1. Experimental apparatus of dynamic adsorption system](image)

2.1.2 Determination of equilibrium adsorption isotherms. The adsorption isotherm of water vapor on the adsorption unit was determined by dynamic adsorption method and verified by gravimetric method. The activated carbon is continuously supplied with clean air at a set temperature and relative humidity,
and then the temperature and relative humidity of the inlet and outlet of the adsorption unit are measured by a hand-held Thermo-hygrometers (VAISALA, HM70-MI70). The water content of activated carbon was titrated by Karl Fischer method before and after adsorption, and the water absorption of activated carbon under the corresponding temperature and humidity was obtained.

The adsorption isotherm of dichloromethane on the adsorption unit was determined by dynamic adsorption method and verified by gravimetric method. The activated carbon is continuously supplied with a set concentration of dichloromethane gas, and the concentration of the dichloromethane at the inlet and outlet of the adsorption unit is continuously detected. The continuous detection uses a portable PID (photo ionization detector, PID) (ppbRAE3000). When the inlet and outlet concentrations are the same, it is in equilibrium. Discontinuous measurements of inlet and outlet concentrations were analyzed using an ECD (electron capture detector, ECD) gas chromatograph (Tianmei Techcomp, GC7900). The adsorption amount of dichloromethane adsorbed by activated carbon under these conditions was obtained by curve integration. At the same time, the difference between the weight of the activated carbon before and after the adsorption and the difference in the weight of the change in the moisture content of the activated carbon by Karl Fischer method were used for the verification.

2.1.3 Percentage of attenuation of adsorption capacity. The percent adsorption capacity attenuation involved in this experiment is defined as the ratio of the equilibrium adsorption capacity of dichloromethane at each RH to the equilibrium adsorption capacity at 5% RH at the same temperature. The formula is as follows:

\[
\text{Percentage of attenuation of adsorption capacity} = \frac{\text{The equilibrium adsorption capacity of dichloromethane at each RH}}{\text{The equilibrium adsorption capacity at 5% RH}}
\]

2.2. Experimental materials and reagents
Adsorbent: coal columnar activated carbon (commercially available), diameter is 4 mm, CTC (carbon tetrachloride adsorption value, CTC) is 61.32%. The surface impurities on the activated carbon for the experiment were washed with distilled water before adsorption. Then, it was dried in a constant temperature drying oven at 105 °C for more than 4 h to constant weight to achieve the purpose of removing moisture in the activated carbon.

Adsorbate: commercially available chromatographically pure dichloromethane and distilled water.

3. Results and discussion
3.1. Characterization of activated carbon
The pore size distribution of the activated carbon used in the experiment is shown in figure 2. The activated carbon used in the experiment has a large number of micropores, and also contains a certain number of mesoporous, and the pore diameter is concentrated at about 0.5-2 nm. Table 1 is a table of characterization parameters of the pore structure of activated carbon. The micropore specific surface area accounts for 92.52% of the total specific surface area, and the micropore volume accounts for 81.13% of the total pore volume.

| Sample name  | Specific surface area (m²/g) | Total pore volume (cm³/g) | Micropore specific surface area (m²/g) | Micropore volume (cm³/g) |
|--------------|-----------------------------|---------------------------|---------------------------------------|-------------------------|
| Activated carbon | 973.43                      | 0.53                      | 900.64                                | 0.43                    |
3.2. Adsorption isotherm

3.2.1. Analysis of single component results of adsorption of dichloromethane. Figure 3 shows the adsorption isotherm of dichloromethane on activated carbon. It can be seen from the figure that the equilibrium adsorption capacity of dichloromethane decreases with increasing temperature at the same concentration. And the equilibrium adsorption capacity increases with the increase of the concentration of dichloromethane at the same temperature. At 40 °C, the equilibrium adsorption capacity of dichloromethane at a concentration of 0.2, 1, 5, and 10 (g/m³) was attenuated by 46%, 40%, 32%, and 26%, respectively, relative to 23 °C. The adsorption equilibrium data obtained by the experiment were fitted by Freundlich adsorption isotherm, and the results are shown in table 2. The R² of the Freundlich adsorption isotherm equation is all above 0.99.

![Figure 2. Pore size distribution of activated carbon](image)

![Figure 3. Dichloromethane adsorption isotherms of activated carbon](image)

| Temperature/°C | Kf/Pa⁻¹ | n          | R²    |
|----------------|---------|------------|-------|
| 23             | 0.072237| 2.620545   | 0.9921|
| 40             | 0.048209| 2.390057   | 0.9901|
| 60             | 0.02869 | 2.23314    | 0.9995|
| 80             | 0.016661| 2.089864   | 0.9997|

According to the characteristics of adsorption isotherms of different concentrations of dichloromethane at 23 °C, 40 °C, 60 °C and 80 °C and the parameters of Freundlich adsorption
isotherm fitting, 23 °C and 40 °C were selected as experimental conditions for competitive adsorption of dichloromethane and water vapor. 31 °C was added to the two temperatures as experimental conditions for competitive adsorption of dichloromethane and water vapor.

3.2.2. Analysis of single component results of adsorbed water vapor. Figure 4 shows the water vapor adsorption isotherms of activated carbon at 23 °C and 40 °C. The abscissa is the relative humidity of gas and the absolute humidity of water vapor, respectively. It can be seen from the adsorption isotherm at 23 °C in Fig. 3(a) that the adsorption isotherm of water vapor conforms to the type V adsorption isotherm. When the RH(relative humidity, RH)<50%, the adsorption capacity of activated carbon to water vapor is less than 0.04 (g/g), and there is no significant difference between the two curves of 23 °C and 40 °C. This is basically consistent with the tendency of the isotherm to adsorb water in the ultramicropores of activated carbon by Lodewyckx, P [8]. It can be seen that the increase in adsorption of water by activated carbon is small when RH is increased to 25%. When RH>50%, as the RH increases, the adsorption equilibrium capacity of water vapor increases significantly. Converting the relative humidity to absolute humidity results in figure 4(b). As can be seen from Fig. 4, when the relative humidity is less than 50% and the absolute humidity is less than 10 g/m³, the equilibrium adsorption capacity of water vapor is very low. When the relative humidity exceeds 50% and the absolute humidity exceeds 10 g/m³, the lower gas temperature will lead to a significant increase in the water absorption of the activated carbon. However, it is generally considered that the adsorption capacity of organic matter increases as the temperature decreases. Therefore, the actual ability of activated carbon to adsorb organic matter involves complex changes in temperature and humidity.

![Figure 4. water vapor adsorption isotherms of activated carbon](image)

3.3. Competitive adsorption of different concentrations of dichloromethane and water vapor

The experimental results show that the adsorption equilibrium capacity of three concentrations of dichloromethane on activated carbon at 23 °C and 40 °C at a relative humidity of 5%, 25%, 50%, 75% is shown in figure (Fig. 5). The results show that with the increase of relative humidity, water has an inhibitory effect on the adsorption of different concentrations of dichloromethane. After more than 50%, the inhibition is more pronounced. When the temperature was increased from 23 °C to 40 °C, the adsorption equilibrium capacity of dichloromethane also decreased significantly under the same RH conditions. On the whole, it can be concluded that the temperature plays a dominant role in the Qc of the activated carbon competitively adsorbing methylene chloride. Under high relative humidity conditions, the increase of temperature has a great influence on the Qc of activated carbon adsorption of methylene chloride.

The equilibrium adsorption capacity of each concentration of dichloromethane at RH 5% was used as a baseline value to obtain a relative percentage of the equilibrium adsorption capacity of water to dichloromethane at different RH as shown in figure 6. The equilibrium adsorption capacity of dichloromethane at a concentration of 200 mg/m³ decreased to 82.82% of the baseline value, which
was much lower than that of the high concentration group. When RH increased from 50% to 75%, the three concentrations of dichloromethane adsorption equilibrium capacity decreased significantly. When RH exceeds 50%, the inhibition of the adsorption of dichloromethane by the humidity tends to increase.

Figure 5. The influence of temperature, RH and concentration on the equilibrium adsorption capacity of Dichloromethane

Comparing (a) and (b) of figure 6, it can be seen that the RH range which affects the adsorption capacity is larger when compared with the adsorption at 23 °C. When RH increases from 5%, it has a significant effect on the adsorption capacity, especially when the concentration of adsorbate is low. In the low concentration adsorption process at higher temperatures, the effect is significant when RH is above 5%. However, at lower adsorption temperatures, the effect begins to be significant when RH is above 50%.

3.4. Influencing factors of activated carbon competitive adsorption of methylene chloride and water balance

3.4.1. Effect of temperature and humidity on the adsorption of methylene chloride. Figure 7 shows the equilibrium adsorption capacity of dichloromethane at three temperatures of 23 °C, 31 °C, and 40 °C and relative humidity of 5%, 25%, 50%, and 75%. As the temperature increases, the equilibrium adsorption capacity of dichloromethane decreases. The decrease in the equilibrium adsorption capacity of the dichloromethane at different relative humidities at 31 °C to 40 °C was less than 23 °C to 31 °C decline in value. The effect of temperature change in the low temperature region on the equilibrium adsorption capacity of dichloromethane is greater than that in the high temperature region.

It can be seen from figure 7(b) that under the conditions of combined temperature and absolute humidity, the temperature plays a dominant role in the equilibrium adsorption capacity of water vapor and dichloromethane for competitive adsorption. With the increase of absolute humidity, the
equilibrium adsorption capacity of dichloromethane gas at a concentration of 1000 mg/m³ was higher than 31 °C at 23 °C. And with the increase of absolute humidity, the equilibrium adsorption capacity of dichloromethane gas at a concentration of 1000 mg/m³ was higher than 40 °C at a temperature of 31 °C. Therefore, when the equilibrium adsorption capacity of dichloromethane is taken as the main reference object, the lower the temperature, the more advantageous the adsorption.

Figure 7. The influence of temperature, relative humidity and absolute humidity on the equilibrium adsorption of Dichloromethane (1000mg/m³)

Combined with (a) and (b) of figure 7, it can be seen that at the same temperature, with the increase of relative humidity and absolute humidity, the equilibrium adsorption capacity of dichloromethane decreases. At the same humidity, with the increase of temperature, the equilibrium adsorption capacity of dichloromethane decreases. At a lower temperature, when the relative humidity is less than 50% and the absolute humidity is less than 10 g/m³, the equilibrium adsorption capacity of the dichloromethane is higher. When the relative humidity exceeds 50% and the absolute humidity exceeds 10 g/m³, the equilibrium adsorption capacity of the dichloromethane rapidly decreases. When the temperature is higher, the equilibrium adsorption capacity of dichloromethane decreases steadily as the humidity increases.

Similarly, when the relative humidity of each temperature is 5%, the adsorption equilibrium capacity of dichloromethane is 100% as the benchmark, and the curve of the relative percentage of adsorption equilibrium of dichloromethane with the change of relative humidity at different temperatures is shown in figure 7(c). It can be concluded from figure 6 that as the temperature increases, the percentage of inhibition of dichloromethane adsorption gradually decreases. It indicates that the inhibition increases with increasing temperature. With the increase of RH, the percentage of inhibition of dichloromethane adsorption at three temperatures tends to be consistent. It can be inferred from the figure that when the temperature is not more than 23 °C and the low relative humidity (RH < 50%), the presence of water vapor has little effect on the adsorption of methylene chloride. And as RH increases to not less than 75%, the equilibrium adsorption capacity of methylene
chloride is greatly reduced. As the temperature is raised to 40 °C, water vapor exhibits a significant inhibitory effect on the adsorption of methylene chloride under low relative humidity conditions. This rule has important guiding significance for the adsorption and treatment of VOCs such as methylene chloride by the activated carbon process in practical applications.

As can be seen from figure 7(d), the equilibrium water content per unit of activated carbon was 0.006 and 0.022 higher than that at 31 °C and 40 °C when the concentration of methylene chloride was constant at 1000 mg/m³ and the absolute humidity was less than 10 g/m³ at temperature of 23 °C. At this time, the decrease in temperature had little effect on the equilibrium adsorption capacity of water during competitive adsorption on activated carbon. When the absolute humidity is greater than 10 g/m³, the equilibrium water content per unit of activated carbon at 23 °C is greatly increased. At this time, the equilibrium water content of activated carbon at a temperature of 23 °C is much higher than 31 °C and 40 °C. Therefore, when the activated carbon adsorption process is comprehensively considered, it should be considered that the water absorption of the activated carbon under the conditions of low temperature (23 °C) and high relative humidity is greatly increased. Otherwise, the subsequent regeneration process of desorption and condensation recovery of activated carbon may cause adverse effects such as increased energy consumption, reduced solvent recovery rate, and increased wastewater volume.

Combined with the above analysis, it can be concluded that under the condition of constant concentration of dichloromethane of 1000 mg/m³ and absolute humidity of less than 10 g/m³, the lower the temperature, the more favorable the equilibrium adsorption of methylene chloride. When the absolute humidity is between 10 and 25 g/m³, there is an optimum adsorption temperature which enables the equilibrium adsorption capacity of methylene chloride and water to be controlled at ideal values at any relative humidity. When the absolute humidity is greater than 25 g/m³, the equilibrium adsorption capacity of methylene chloride on activated carbon is lower at any temperature and relative humidity. In this case, it is not suitable for treatment directly by activated carbon adsorption.

3.4.2. Regression analysis of factors affecting adsorption of methylene chloride on activated carbon. Table 3 shows the equilibrium adsorption capacity of 10 cm fixed bed activated carbon to methylene chloride at different temperatures, absolute humidity and dichloromethane concentration.

| Number | Dichloromethane concentration g/m³ | Temperature °C | Absolute humidity g/m³ | Activated carbon adsorption capacity g/g |
|--------|-----------------------------------|----------------|------------------------|----------------------------------------|
| 1      | 1.0                               | 23             | 1.0265                 | 0.0480                                 |
| 2      | 1.0                               | 23             | 15.3975                | 0.0357                                 |
| 3      | 1.0                               | 23             | 20.35                  | 0.0272                                 |
| 4      | 1.0                               | 40             | 1.938                  | 0.0288                                 |
| 5      | 1.0                               | 40             | 19.38                  | 0.0226                                 |
| 6      | 1.0                               | 40             | 38.76                  | 0.0198                                 |
| 7      | 0.2                               | 23             | 1.0265                 | 0.0163                                 |
| 8      | 0.2                               | 23             | 10.265                 | 0.0135                                 |
| 9      | 0.2                               | 23             | 15.3975                | 0.0087                                 |
| 10     | 0.2                               | 40             | 1.938                  | 0.0088                                 |
| 11     | 0.2                               | 40             | 9.69                   | 0.0055                                 |
| 12     | 0.2                               | 40             | 38.76                  | 0.0031                                 |
| 13     | 5.0                               | 23             | 1.0265                 | 0.0963                                 |
| 14     | 5.0                               | 23             | 10.265                 | 0.0931                                 |
| 15     | 5.0                               | 23             | 15.3975                | 0.0728                                 |
| 16     | 5.0                               | 40             | 1.938                  | 0.0588                                 |
LINEST quantitative regression analysis was used to analyze the effects of dichloromethane concentration, temperature and absolute moisture on the adsorption capacity of activated carbon for methylene chloride. The results are shown in Tables 4 and 5.

Table 4. regression statistics

| Regression statistical coefficient name                              | value          |
|---------------------------------------------------------------------|----------------|
| Complex correlation coefficient R (Multiple R)                       | 0.94616729     |
| Complex coefficient R²(R Square)                                    | 0.895232541    |
| Adjusted post-measurement coefficient R²Adjusted R Square           | 0.881567221    |
| Standard error                                                      | 0.008611143    |
| Observations                                                        | 27             |

Table 5. Variance analysis table

|                          | df | SS      | MS      | F         | Significance F   |
|--------------------------|----|---------|---------|-----------|-----------------|
| Regression analysis      | 3  | 0.014573| 0.004858| 65.51127  | 2.02843E-11     |
| Residual                | 23 | 0.001705| 7.42E-05|           |                 |
| Total                   | 26 | 0.016279|         |           |                 |

Table 6. Regression parameter table

|                        | Dichloromethane concentration X₁ | Temperature X₂ | Absolute humidity X₃ | Constant c         |
|------------------------|---------------------------------|----------------|----------------------|-------------------|
| Coefficient            | 0.011378931                     | -0.000968472   | -0.000325964         | 0.049198353       |
| Standard error         | 0.000872                        | 0.00022        | 0.000155             | 0.007022          |
| T-Stat                 | 13.0503                         | -4.39438       | -2.10355             | 7.006466          |
| P-value                | 4.07E-12                        | 0.000211       | 0.046557             | 3.86E-07          |

It can be seen from Table 4 that R²=0.8952 and standard error=0.0086, indicating that the regression equation of this experiment has a good correlation. The correlation between the three factors and the equilibrium capacity of activated carbon for the adsorption of methylene chloride requires a comparison of the significant results of each factor.

Table 5 shows the variance analysis table. The regression test was used to determine the regression effect of the regression model. The Significance F=2.02843E-11 obtained in this experiment is much smaller than the significant level of 0.05, indicating that the regression equation has a significant regression effect.

Table 6 is a regression parameter table, according to which the regression analysis can be obtained that the equation is: Y=0.01138X₁-0.000968X₂-0.000326X₃+0.0492. The P-value is the P value of the t statistic of the three influencing factors. The P values of X₁, X₂, and X₃ were all less than the significance level of 0.05, indicating that the three independent variables were significantly correlated with Y.

4. Conclusion

For methylene chloride gas with a concentration of 1000 mg/m³, the effect of temperature change in the low temperature region on the equilibrium adsorption capacity of methylene chloride is greater than that in the high temperature region. When the absolute humidity is less than 10 g/m³, the temperature decreases has little effect on the equilibrium water absorption when the activated carbon is competitively adsorbed. When the absolute humidity is greater than 10 g/m³, the equilibrium moisture content of activated carbon at 23 °C is greatly increased.

Under the same temperature conditions, the concentration of methylene chloride plays a leading role in the equilibrium adsorption capacity (Qc) of methylene chloride when the activated carbon competes for adsorption of methylene chloride and water. At lower temperatures (23 °C), the inhibition of RH above 50% began to be significant. For methylene chloride gas with a concentration
of 1000 mg/m³, the effect of temperature change in the low temperature region on the equilibrium adsorption capacity of methylene chloride is greater than that in the high temperature region. When the absolute humidity is less than 10 g/m³, the temperature decreases during the competitive adsorption of activated carbon has little effect on the equilibrium water content. However, when the absolute humidity is higher than 10 g/m³, the equilibrium water content of activated carbon at 23 °C is greatly increased.

Suggestions for the application of activated carbon competitive adsorption of methylene chloride and water in the dehumidification pretreatment of adsorption process. Under the conditions of constant concentration of dichloromethane at 1000mg/ m³, absolute humidity less than 10g/m³, and any relative humidity, the lower the temperature, the more advantageous for equilibrium adsorption of methylene chloride. When the absolute humidity is between 10 and 25 g/m³, there is an optimum adsorption temperature which enables the equilibrium adsorption capacity of methylene chloride and water to be controlled at ideal values at any relative humidity. When the absolute humidity is greater than 25 g/m³, the equilibrium adsorption capacity of methylene chloride on activated carbon is lower at any temperature and relative humidity. In this case, it is not suitable for treatment directly by activated carbon adsorption. It is recommended to lower the temperature by means of cooling, condensation, etc., in order to reduce the water content before adsorption.

Three factors influencing the adsorption capacity of active competitive adsorption of methylene chloride and methylene chloride in water were analyzed by regression analysis of dichloromethane concentration, temperature and absolute humidity. The regression analysis yields the equation: Y = 0.01138X₁-0.000968X₂-0.000326X₃ + 0.0492. The P-value is the P value of the t statistic of the three influencing factors. The P values of X₁, X₂, and X₃ are all less than the significance level of 0.05. The three influencing factors were significantly correlated with the methylene chloride adsorption capacity. However, the composition of the actual VOCs is more complicated. The effects of temperature, relative humidity and concentration of VOCs on the adsorption time and saturation time of activated carbon remain to be further studied.

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