Adsorptive Removal of Acetaldehyde from Propylene Oxide Produced by the Hydrogen Peroxide to Propylene Oxide Process

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ABSTRACT: Adsorption method was first introduced into the propylene oxide production via hydrogen peroxide process to remove the microimpurity in the propylene oxide (PO) product solution. It could replace the reactive distillation in separating acetaldehyde with less energy consumption and PO loss. A series of adsorbents (e.g., 3A, 4A, 5A, 10X, and Y) are first used to remove the impurity (i.e., acetaldehyde). It is found that 5A molecular sieves shows the best performance due to uniform porous channels with suitable pore size. Various techniques such as X-ray diffraction, scanning electron microscopy, thermogravimetric analysis, Fourier transform infrared, and N₂ physisorption are employed to investigate the structural properties of the adsorbent. Furthermore, effects of space velocity and temperature are also investigated. Cyclic desorption and adsorption tests indicate the PO yield is 92.2%, and 96.3% of acetaldehyde was removed. The acetaldehyde concentration of PO product was 0.0187%, indicating this method can produce industrial-quality PO that meets the first-level technical requirements.

1. INTRODUCTION

The conversion of hydrogen peroxide to propylene oxide (HPPO) has become more popular in recent years¹ and is considered a crucial method for the environment-friendly production of propylene oxide (PO)²,³ owing to the method’s safe reaction conditions, catalytic efficiency, and low production of polluting byproducts.⁴ A pilot plant designed to showcase this novel HPPO process via the production of 1500 tons per annum (t/a) was built, and propylene epoxidation was performed in methanol (CH₃OH) over TS-1 molecular sieves as catalysts.⁵ This facility has successfully been in operation since its establishment and produces fewer pollutants than plants using alternative methods.

The composition of the crude PO product being purified at the 1500 t/a pilot plant is illustrated in Table 1.

To recover the solvent (methanol), a distillation step was incorporated into the processing of the crude product (Table 1), and Table 2 shows the composition of the PO product following the solvent recovery process. When PO purity exceeded 99% in simple distillation, the relative volatility of methanol and PO was close to 1, indicating the need for a large reflux ratio to further reduce the methanol content. On one hand, the larger the reflux ratio, the more energy and investment are consumed. On the other hand, acetaldehyde has a boiling point of 20.8 °C, similar to that of PO (34 °C). It means that light components will accumulate at the top of the tower as the reflux ratio increases, which substantially increases the acetaldehyde concentration in PO.

Many U.S. patents⁶−⁹ have introduced various extractive distillation solvents for PO purification, and some U.S. patents¹⁰−¹³ include methods for separating methyl formate from PO. Although these patents include the removal of selected PO impurities, none addressed the removal of aldehydes, particularly acetaldehyde. Rueter¹⁴ disclosed the purification of PO-containing methanol by extractive distillation with a polar extraction solvent with a hydroxy functionality. The process also removes a portion of acetaldehyde present in the crude PO product. Rueter¹⁵ used methanol as the extraction solvent to remove acetaldehyde.

Table 1. Crude PO Product

| species               | wt %   |
|-----------------------|--------|
| propylene oxide       | 49.0−49.8 |
| CH₃OH                 | 50.1−50.6 |
| acetaldehyde (CH₃CHO) | 0.12−0.25 |
| H₂O                   | <0.25  |

Table 2. PO Product Composition

| species     | wt %  |
|-------------|-------|
| propylene oxide | >99.33 |
| methanol     | <0.07 |
| acetaldehyde | <0.5  |
| water        | <0.01 |

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from PO, but the solvent itself becomes a close-boiling PO contaminant. Koji and Toshio \(^{16}\) washed the PO product with water, extracted the aqueous phase with a hydrocarbon solvent, and then distilled the resulting solution. Because these techniques are impractical for the improvement of the existing plants, reactive distillation using additives \(^{17}\) was incorporated into the purification process to remove acetaldehyde from the PO product after recovering methanol (Table 2). However, during the actual operation, the results showed that the additives could react with PO, resulting in a PO yield of only 90% after reactive distillation. Schmidt \(^{18}\) believed that chemical reactions that interfere with the normal mass-transfer steps may be occurring during distillation, thereby producing anomalous results. It is therefore necessary to discover a novel separation method of removing aldehyde impurities to reduce the loss of PO during product purification.

Separation engineering relies heavily on liquid phase adsorption \(^{19,20}\). When screening adsorbents in the purification process, the properties of both adsorbates and adsorbents must be considered, with a focus on examining how operational conditions influence the dynamic liquid—solid adsorption.

2. RESULTS AND DISCUSSION

2.1. Adsorbent Screening. Physical adsorption is greatly influenced by the molecular diameter and polarity of the adsorbate. Table 3 illustrates the differences in molecular property between two adsorbates as simulated in Materials Studio 8.0 (MS).

### Table 3. Molecular Dimensions and Dipole Moments of Adsorbates

| Species          | Molecular Diameter ($10^{-10}$ m) | Dipole Moment (D) |
|------------------|----------------------------------|-------------------|
| Propylene oxide  | 6.0                              | 1.9               |
| Acetaldehyde     | 5.0                              | 2.7               |

As shown in the table, acetaldehyde is smaller and more polar than PO. Molecular sieves are suitable for adsorbing polar molecules and are able to sieve adsorbates based on the size and configuration of the molecules. Table 4 illustrates the pore sizes of several common molecular sieves.

### Table 4. Pore Size of Molecular Sieves

| Molecular Sieves | Pore Dimension ($10^{-10}$ m) |
|------------------|-------------------------------|
| 3A               | 2.1–3.2                       |
| 4A               | 4.1–4.6                       |
| 5A               | 4.9–5.6                       |
| 10X              | 8.0–9.0                       |
| 13X              | 9.0–10.0                      |
| Y                | 9.0–10.0                      |

Several common adsorbents shown in Table 4, as well as activated carbon, were selected as alternative adsorbents. Simulated PO product with 0.22 (wt %) CH\(_3\)CHO was prepared following the values given in Table 1. The static liquid—solid adsorption apparatus was used to examine the efficacy of alternate adsorbents. The mass of PO solution was 16 times that of the adsorbent. The adsorption results are given in Figure 1.

As shown in Figure 1, the 3A and 4A molecular sieves as well as the activated carbon adsorbed almost no acetaldehyde from the simulated PO product. However, the 10X, Y, and 5A molecular sieves effectively reduced the acetaldehyde content of the solution, and the 5A molecular sieves were most efficient. After 5 h, the 5A molecular sieve had adsorbed 100% of acetaldehyde, whereas the Y and 10X molecular sieves had adsorbed 72.0 and 58.1% of acetaldehyde, respectively. The 3A and 4A molecular sieves and the activated carbon removed less than 15% of acetaldehyde in 5 h because the pore sizes of 3A and 4A are smaller than acetaldehyde, which prevents adsorption. Activated carbon is nonpolar, making it unsuitable for adsorbing polar molecules.

The simulated PO product with 0.42 (wt %) CH\(_3\)CHO was prepared in accordance with the values given in Table 2. The static liquid—solid adsorption apparatus was used to further screen the adsorption of acetaldehyde from PO. The mass ratio remained 1:16, and the results are illustrated in Figure 2.

As illustrated above, 5A, Y, and 10X molecular sieves were somewhat effective at adsorbing acetaldehyde in the simulated PO product, though 5A sieves proved more effective than either Y or 10X. After 6 h of absorption, the 5A sieves had removed 100% of acetaldehyde, whereas the Y and 10X sieves had removed only 56.9 and 49.9%, respectively. This is because the diameters of the pores of the Y and 10X sieves are larger than those of PO and acetaldehyde molecules, meaning that not only the molecules of acetaldehyde but also PO can enter the adsorbents’ channels. As the concentration of PO is highly greater than that of acetaldehyde, plenty of porous space will be occupied by the molecules of PO, restricting the adsorption capacity of acetaldehyde on the Y and 10X sieves.
2.2. Effect of Space Velocity on Adsorption. The 5A molecular sieves were chosen as the primary adsorbent for separating acetaldehyde from the PO product. Liquid weight hourly space velocity (LWHSV) is a significant parameter of liquid adsorption. The dynamic liquid–solid adsorption apparatus was used to examine the effect of LWHSV at 20 °C. For an adsorbent to be considered effective, the amount of acetaldehyde remaining should be below 0.02 (wt %).

The breakthrough curves shown in Figure 3 and the mass ratios of PO solution/adsorbent shown in Table 5 demonstrate that increasing the LWHSV could decrease not only the penetration time but also the adsorption capacity of the simulated solution. Although the penetration time when the LWHSV was 0.5 h⁻¹ was more than twice the penetration time at 1 h⁻¹, the treatment capacity of the PO solution and the adsorption capacity of acetaldehyde were both only slightly increased. Therefore, the optimal LWHSV should be 1 h⁻¹ in operation.

2.3. Effect of Temperature on Adsorption. The effect of adsorbing temperature was determined using the dynamic liquid–solid adsorption apparatus (LWHSV = 1 h⁻¹), and the results are illustrated in Figure 4 and Table 6.

When the temperature of the adsorbent bed was set between 15 and 20 °C, the concentration of acetaldehyde in the exudates stayed below 0.003 (wt %) over the course of the 10 h of adsorption period, and it ultimately reached 0.02 (wt %) after 14 h. When the adsorption temperature was increased to 25 °C, the concentration of acetaldehyde increased even though the penetration time was still 13 h. In addition, when the temperature was further increased to 30 °C, the penetration time only fell to 11 h, and the adsorption capacity of acetaldehyde continued to decrease.

As illustrated in Table 6, the treatment capacity of the simulated PO product decreased as the temperature increased, and lower temperatures proved most effective for adsorption. However, too low a temperature increases the cost of refrigeration, meaning the optimum temperature range for absorption is 15−20 °C.

By examining the effects of LWHSV and temperature on adsorption, the optimum operational conditions for separating acetaldehyde from PO are as follows: the LWHSV of the feed should be 1 h⁻¹ and the temperature should be 15−20 °C.

In separation processes meant for practical use, it is extremely useful to be able to reuse the adsorbents. Desorption can not only restore the adsorption capacity of adsorbent to the adsorbate but also recover any adsorbate adsorbed by the adsorbent. In this way, the desorption operation of the adsorbent is very important for the realization of adsorption and separation.

2.4. Exploration on the Desorption Process of Impurity Acetaldehyde. Water vapor desorption is one of the more common methods of molecular sieve desorption. However, in the preliminary desorption test, a certain amount of propylene glycol was detected in the desorption distillate. Some studies have reported that propylene glycol is produced by PO and water even in the absence of catalyst and hydrogen peroxide so that the propylene glycol in the desorption distillate was the residue in the adsorbent bed and the product generated from PO and high-temperature water vapor. Because the formation of propylene glycol affected the yield of PO, high-temperature inert gas was used for desorption.

After the adsorbent bed was penetrated, the adsorbed 5A molecular sieve was subjected to thermogravimetric analysis (TGA), and the results are shown in Figure 5.

The quality of the adsorbent decreased rapidly as the temperature began to rise, indicating that the adsorbed substances were blown off after the surface of the adsorbent was wetted by the solution. As shown in Figure 5, the quality of the adsorbent exhibited two apparent decline processes with increasing temperature. Between 50 and 80 °C, the mass of the adsorbent decreased by about 5.12%, which was because the adsorbed PO was gradually desorbed. Between 160 and 200 °C, the mass of the adsorbent decreased again, by about 9.56%.

![Figure 3. Breakthrough curves at various LWHSV.](image)

![Figure 4. Breakthrough curves under various temperatures.](image)

Table 5. Effect of LWHSV on Adsorption Capacity

| LWHSV (h⁻¹) | penetration time (h) | POsolution/adsorbent (mass ratio) | adsorption capacity (g(acetaldehyde)·g⁻¹(adsorbent)) |
|-------------|----------------------|-----------------------------------|-----------------------------------------------|
| 0.5         | 30                   | 15.0                              | 0.0742                                         |
| 1           | 14.5                 | 14.5                              | 0.0720                                         |
| 2           | 5.5                  | 11.0                              | 0.0541                                         |
| 4           | 2                    | 8.0                               | 0.0438                                         |
indicating that acetaldehyde was also desorbed by nitrogen blowing when the temperature was more than 160 °C.

The desorption experiment was carried out on the desorption apparatus. Nitrogen gas was introduced into the bed, and the temperature was gradually increased. The initial temperature was 40 °C and held for 15 min. The heating rate was 20 °C h⁻¹, and the desorption outflow was collected and cooled for each 20 °C temperature increment. The composition of the desorption outflow was analyzed by chromatography, and the results are shown in Figure 6. The substance with a retention time of about 1 min was acetaldehyde, and the substance with a retention time of about 3 min was PO.

As can be seen from Figure 6, when the desorption temperature was below 80 °C, the chromatogram of the desorption outflow only showed the peak of PO. As the temperature rose above 100 °C, the peak area of acetaldehyde gradually increased with increasing temperature. When the temperature exceeded 160 °C, the chromatogram of the desorption outflow only showed the peak of acetaldehyde. According to the results of TGA and the desorption experiment, the desorption conditions were as follows.

1. First, the adsorbent bed temperature was increased directly to about 70 °C, and the adsorption bed was purged with nitrogen to remove some of the adsorbed PO. Condensed liquid could be used as a refined PO product.

2. Then, the adsorbent bed temperature was increased directly to 160 °C, and the adsorption bed was purged with nitrogen to remove the acetaldehyde impurity adsorbed by the SA molecular sieve and the remaining PO. The condensed desorption outflow was a solution enriched with acetaldehyde impurity, which was subjected to a centralized adsorption and separation process.

### 2.5. Cyclic Desorption and Removal of Acetaldehyde Impurities

With a LWHSV of 1 h⁻¹ and a temperature of 15 °C, 120 g of SA molecular sieve was used to adsorb the acetaldehyde impurity from the simulated PO product solution containing 0.5% acetaldehyde, and the adsorption outflow was collected every 0.5 h to analyze its composition. The composition of the adsorption outflow collected in the sample collection tank was analyzed at every 0.5 h, and the feed was stopped when the average concentration of acetaldehyde in the adsorption outflow was close to 0.02 (wt %). After the adsorption outflow had flown for 3 h, the desorption of adsorbent was carried out, and then the desorbed SA molecular sieve was used to adsorb acetaldehyde in the simulated PO solution (the number of cycles: N = 8). The change in acetaldehyde concentration in the outflow from the cyclic desorption is shown in Figure 7.

| temperature (°C) | penetration time (h) | PO solution/adsorbent (mass ratio) | adsorption capacity (g(acetaldehyde)·g⁻¹(adsorbent)) |
|------------------|----------------------|-----------------------------------|--------------------------------------------------|
| 15               | 14.5                 | 14.5                              | 0.0719                                           |
| 20               | 14                   | 14                                | 0.0694                                           |
| 25               | 13                   | 13                                | 0.0642                                           |
| 30               | 11                   | 11                                | 0.0540                                           |

Figure 5. TG curve of adsorbed SA molecular sieve.

Figure 6. Chromatogram of desorbed exudate under different temperatures.

Figure 7. Concentration of acetaldehyde in adsorbed exudate of cyclic desorption and adsorption.
The outflow of the nth adsorption was recorded as $A_{r,n}^{ald}$, the PO solution obtained by nitrogen purging and desorption at 70 °C was recorded as $D_{r,n}^{PO}$ and then the fine PO product after N times of desorption was $P_{n}^{PO} = \sum_{r=1}^{n}A_{r}^{ald} + \sum_{r=1}^{n}D_{r}^{PO}$. The outflow of the nth desorption at 160 °C was recorded as $D_{n}^{ald}$ and the solution enriched with acetaldehyde after N rounds of desorption was $S_{n}^{ald} = \sum_{r=1}^{n}D_{r}^{ald}$. The acetaldehyde concentration in $P_{r,n}^{PO}$ and $S_{n}^{ald}$ were measured, and the performance of using the cyclic desorption to remove acetaldehyde impurity from the simulated PO solution is shown in Table 7.

As shown in Table 7, under the optimized adsorption operating conditions, 1 g of fresh SA molecular sieve could be used to treat 17 g of simulated PO solution. The acetaldehyde impurity concentration in the resulting PO product was 0.0171%, and the PO yield was 92.9%. After the first desorption, 16.5 g of simulated solution could be processed by 1 g of adsorbent, and the processing capacity was 97.1% that of fresh adsorbent. After eight cycles, 15 g of simulated solution could be processed by 1 g of adsorbent in one adsorption, and the processing capacity could still reach 86.2% that of fresh adsorbent, suggesting that the SA molecular sieves had a good adsorption capacity after cyclic desorption. After eight cycles of desorption operation, the cumulative amount of simulated PO solution that could be processed by 1 g of adsorbent was 127 g, and the yield of PO was 92.2%. The condensate collected each time was enriched with the impurity acetaldehyde and could be used for centralized adsorption and separation treatment. Both PO and acetaldehyde were recovered as products, which was conducive to improving the yield of PO.

After eight cycles of desorption, the concentration of acetaldehyde impurity in the PO product was 0.0187%. The calculated removal rate of acetaldehyde was 96.3%, which met the technical requirement for the first level of aldehyde impurity in industrial PO. The acetaldehyde content in $S_{n}^{ald}$ was 7.74%, 15.5 times higher than that before adsorption. The effect of the impurity enrichment was significant. Because some of the PO was removed by the noncondensable gases in the process of condensing, desorbing, and outflowing, the concentration of acetaldehyde in $S_{n}^{ald}$ was slightly higher than the theoretical value.

### 2.6. Structural Characterization of Adsorbent.

After eight cycles of desorption operation, the durations of adsorption and desorption for SA molecular sieve were 127 and 48 h, respectively. The fresh SA molecular sieve sample was labeled Fresh sample and the SA molecular sieve sample after eight cycles of desorption was labeled as Desorbed sample. As shown from the scanning electron microscopy (SEM) images of the two samples in Figure 8, the crystallinities of the two samples were good and the crystal forms were similar and complete.

The X-ray diffraction (XRD) patterns of the fresh SA molecular sieve sample and the SA molecular sieve sample after cyclic desorption are shown in Figure 9. As shown in the figure, the characteristic diffraction peaks of the SA molecular sieve crystals after cyclic desorption did not change, indicating that the adsorption and desorption processes did not affect the crystal structure of the molecular sieve.

Figure 10 shows the adsorption isotherm of N$_2$ in fresh/desorbed SA molecular sieve samples. The equilibrium adsorption capacity of N$_2$ on desorbed SA molecular sieve was slightly lower than that of the fresh sample.

As shown in Figure 11, the pore size distribution of the adsorbent after cyclic desorption was similar to that of the fresh adsorbent. Based on the distributions, there were three types of pores in the sample: the intracrystalline pores sized around 0.5 nm, the intercrystalline pores sized 10–100 nm, and the pores sized 2–5 nm probably exist among the binder and zeolite grains.

The specific surface area, pore volume, and average pore size in Table 8 indicated that the specific surface area and pore volume of the SA molecular sieve after cyclic desorption were...
slightly decreased, and the average pore size was slightly increased and essentially recovered to the level of fresh adsorbent.

There were two reasons for the change in microstructure take place through the separation. On one hand, the acid sites of 5A molecular sieve could catalyze the ring opening and polymerization of PO, and the retention of polymers blocked parts of pores in smaller size. On the other hand, partial binder, which was broken under high temperature, led to the variation in pores sized \((2−5) \times 10^{-9} \text{ m}\), and it could also block some micropores.

According to the microstructure analysis results, the micropores of 5A sample sized \((4.9−5.6) \times 10^{-10} \text{ m}\) were smaller than the molecular diameter of PO \((6 \times 10^{-10} \text{ m})\) but larger than that of acetaldehyde \((5 \times 10^{-10} \text{ m})\). In this case, the acetaldehyde molecules could enter the microscale pores of 5A but the PO molecules could not (Figure 12). Therefore, acetaldehyde could be filtered from PO by uniform zeolite channels with high separation efficiency. When the samples were processed by the variable temperature desorption by purging with N\(_2\), the macro- and mesoscale pores benefitted the diffusion of PO molecules with low resistance, whereas acetaldehyde in the micropores could not be desorbed easily.

The results of SEM, XRD, and microstructural data showed that the adsorption and desorption operations did not generally affect the microstructure of 5A molecular sieves, which exhibited a good recovery of the adsorption performance after desorption and retained a consistent microstructure with the fresh adsorbent, showing that the adsorption and desorption operations did not affect the performance of 5A molecular sieve and the adsorbent could be reused multiple times.

**3. CONCLUSIONS**

Based on the results of static liquid–solid adsorption experiments, 5A molecular sieves were chosen to separate acetaldehyde from PO. The conditions for adsorbing acetaldehyde were optimized using dynamic liquid–solid adsorption experiments, and the ideal conditions were found to be an adsorption temperature between 15 and 20 °C with an LWHSV of 1 h\(^{-1}\).

The desorption conditions were determined according to the results of thermogravimetric analysis. First, the adsorbent bed was purged with nitrogen at 70 °C, and the condensed

### Table 8. Brunauer–Emmett–Teller (BET) Results of Fresh Sample and Desorbed Sample of 5A Molecular Sieve\(^a\)

| samples       | \(S_{\text{BET}} (\text{m}^2 \cdot \text{g}^{-1})\) | \(V_p (\text{cm}^3 \cdot \text{g}^{-1})\) | \(d_p (\text{nm})\) |
|---------------|---------------------------------------------|---------------------------------|-----------------|
| fresh sample  | 551.66                                      | 0.246                           | 0.558           |
| desorbed sample | 520.67                                     | 0.230                           | 0.571           |

\(^a\)Test range for \(d_p\): 0.3–200 nm. \(S_{\text{BET}}\): specific surface area; \(V_p\): pore volume; \(d_p\): pore diameter.
liquid could be used as a refined PO product. Then, the adsorption bed was purged with nitrogen at 160 °C to remove the acetaldehyde impurity and the remaining PO.

After eight cycles of adsorption and desorption, 96.3% of the acetaldehyde had been removed. The concentration of acetaldehyde impurity in the PO product was 0.0187%, which meets the first-level technical requirements for aldehyde content in industrial PO. The characterization of the adsorbent showed that the adsorption and desorption procedures did not affect the structure of the 5A molecular sieves, and the adsorbent could be reused multiple times.

4. EXPERIMENTAL SECTION

4.1. Calculating Molecular Diameter and Dipole Moment. This study uses simulations carried out in the Materials Studio 8.0 (MS) software package published by Accelrys Software, Inc. Density functional theory (DFT) using Dmol3 code was used to estimate the dipole moment of the molecules. Molecular diameter simulations were then carried out using Forcite code under the following conditions: force field: COMPASS II,21 ensemble: NPT and NVT, time step: 1 fs, total time: 300 ps. Ewald and atom-based summation methods were used for electrostatic interactions and van der Waals forces, respectively, and all atom–atom interactions were added together to a 15.5 Å direct cutoff distance.

4.2. Static Liquid–Solid Adsorption. Figure 13 illustrates the setup used for determining the static liquid–solid adsorption. The apparatus featured a temperature control mechanism capable of keeping the system within ±1.0 °C. A magnetic agitator was used in the main body of the apparatus. The feed pump transferred PO product into the vessel, and a condenser was placed at the connection point.

Simulated PO product containing a predetermined percentage of acetaldehyde was prepared. A predetermined quantity of adsorbent was added to the vessel before it was sealed. The temperature was set to 20 °C. Once the temperature stabilized, the feed system was activated and the timer was started. The samples were gathered from the condenser outlet.

4.3. Dynamic Liquid–Solid Adsorption Experiments. The simulated PO product containing 0.5 (wt %) acetaldehyde was prepared. A water jacket was used to control the reaction temperature, the condenser was situated at the outflow connection point. Once the temperature stabilized, the pump was activated, and the solution was fed into the adsorbent bed at controlled liquid weight hourly space velocity (LWHSV). The timer was activated when the solution first contacted the bed. Figure 14 illustrates the dynamic liquid–solid adsorption apparatus. The adsorbent bed was 20 mm in diameter, and 120 g of adsorbent was used. The samples were taken every 30 min.

4.4. Desorption. The adsorption bed was purged with nitrogen at different temperatures at a volumetric space velocity of 60 h⁻¹ (Figure 15).
4.5. Components Analysis of Feed Solutions and Outflow Samples. The solution composition was determined using HP-1 gas chromatography (GC) with a flame ionization detector and a capillary column measuring 50 m × 0.32 × 1.05. The parameters for GC analysis were as follows: column temperature: 35 °C; vaporizer temperature: 150 °C; detector temperature: 150 °C; and injection volume: 0.2 μL.

4.6. Properties of Adsorbent. Molecular sieves were purchased from SUOP Co., Ltd. (Shanghai, China), and activated carbon was purchased from Xinhui Activated Carbon Co., Ltd. (Shanghai, China). The physical properties of the adsorbents are given in Table 9.

4.7. Characterization of Adsorbent. X-ray diffraction (XRD): The crystal structure was determined by a Rigaku D/max-2550 VB/PC apparatus using Cu Kα radiation. Scanning electron microscopy (SEM): The crystal morphology of the product was analyzed on a Micromeritics ASAP 2020 system, and using atmospheric nitrogen. Thermogravimetric analysis (TGA): The product was examined using a JEOL Model JSM-6360 LV electron microscopy (SEM): The crystal morphology of the product was analyzed using a Micromeritics ASAP 2020 system, and using atmospheric nitrogen. The physical properties of the product were recorded on a Toledo TGA/SDTA 851e (Mettler) (Japan) system.

The microstructure of the product was analyzed on a Micromeritics ASAP 2020 system, and using atmospheric nitrogen. Thermogravimetric analysis (TGA): The product was examined using a JEOL Model JSM-6360 LV electron microscopy (SEM): The crystal morphology of the product was analyzed using a Micromeritics ASAP 2020 system, and using atmospheric nitrogen. The physical properties of the product were recorded on a Toledo TGA/SDTA 851e (Mettler) (Japan) system. The microstructure of the product was analyzed on a Micromeritics ASAP 2020 system, and using atmospheric nitrogen.

Table 9. Physical Properties of Adsorbents

| adsorbent            | average diameter (nm) | crush strength (N) | bulk density (kg·m⁻³) | true density (kg·m⁻³) |
|----------------------|-----------------------|--------------------|-----------------------|-----------------------|
| 3A                   | 3                     | 58                 | 858                   | 2750                  |
| 4A                   | 3                     | 67                 | 870                   | 2790                  |
| 5A                   | 3                     | 48                 | 820                   | 2630                  |
| 10X                  | 3                     | 56                 | 757                   | 2430                  |
| Y                    | 3                     | 50                 | 830                   | 2670                  |
| activated carbon     | 3                     | 45                 | 650                   | 2090                  |

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