Study on regeneration of waste adsorbent material in PVC industry

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Abstract
Activated carbon is an adsorbent material with pore structure, which is environmentally friendly and easy to operate. It is widely used in human production and life. However, the spent activated carbon has not been properly treated, and adsorption of harmful elements is highly likely to cause secondary pollution of the environment. Through the regeneration of waste mercury catalyst in PVC (Polyvinyl chloride) industry, the effects of different activators on the regeneration performance of spent activated carbon were studied. It was found that the regeneration effect of carbon dioxide on spent activated carbon was significant at 900 °C. The removal rate of harmful mercury-containing compounds from regenerated activated carbon can be reached 99.96%, and the specific surface area reaches 750.755 m$^2$·g$^{-1}$, which has basically met the adsorption performance index of activated carbon for wastewater treatment. This study is expected to provide theoretical support for the treatment of waste adsorbent material containing harmful elements and reveal the mechanism of regenerating activated carbon under different activator conditions.

1. Introduction
The research of adsorbent materials is of great significance to the production and development of various adsorbents. Adsorbents are widely used in human production and life [1–4]. Due to its excellent environmental protection and easy-to-operate properties, the adsorbent is favored by environmentalists and has outstanding performance in wastewater treatment and air pollution control [5, 6]. Adsorption properties and stability of adsorbents are also attracting attention in industrial production [7]. In China, in the process of preparing PVC (Polyvinyl chloride) by using calcium carbide, the activated carbon is used as a carrier, and the catalyst is supported on the surface of the activated carbon to prepare a mercury catalyst [8, 9]. These spent adsorbents are always incinerated or landfilled [10]. These adsorbents that adsorb harmful pollutants may cause secondary environmental pollution and cause irreversible damage to human living environment [11]. In the increasingly demanding form of environmental protection, depleted adsorbent regeneration is of great significance. China’s PVC industry is developing rapidly, and a large amount of mercury-containing waste adsorbents cannot be properly disposed [4]. Therefore, it is one of the important ways to recycle waste adsorbents by removing harmful pollutants from waste adsorbents through regeneration process and restoring the adsorption capacity of adsorbents.

Activated carbon is an adsorbent material with a porous structure [12]. Micropores ($\approx$ 3 nm) are the main adsorption sites, and relatively large molecules may be trapped in mesopores (3–60 nm) and macropores (60–10 000 nm), resulting in the adsorption capacity of activated carbon is reduced [13]. Therefore, in comparison with the regeneration of activated carbon and other adsorbents, suitable regeneration methods...
should be developed to meet environmental requirements and reduce the high cost and low efficiency caused by the disposal of waste activated carbon [14]. At present, the research on the regeneration method of activated carbon is quite fruitful. Sun et al prepared regenerated activated carbon products by solvent regeneration [15]. Wen et al obtained regenerated activated carbon through biological regeneration [16]. However, these methods only have an efficient desorption effect on organic pollutants, and the cost of desorbing and recovering the adsorbent is high.

Therefore, the purpose of this study is to activate and regenerate waste mercury catalysts in the PVC industry, compare the effects of different activation conditions on the regeneration of activated carbon, study the desorption effect of mercury, the experimental results will contribute to the reduction of secondary pollution in the PVC industry and provide theoretical support for restoring the performance of waste adsorbent materials, removing harmful pollutants and improving material recovery efficiency. Relieve the high cost pressure of waste adsorbent materials treatment.

2. Materials and methods

2.1. Materials

A plant in Yunnan province that produces polyvinyl chloride by calcium carbide method provides the waste activated carbon containing mercury (M-AC) used in this study. The element analysis of M-AC is shown in table 1. According to the production process, 1.82% of mercury is mercury chloride. The iodine adsorption value and specific surface area of M-AC are shown in table 2, the iodine number 354 mg g\(^{-1}\) and specific surface area 376.211 cm\(^3\) g\(^{-1}\). Figure 1. Shows the SEM analysis of M-AC. The surface of the activated carbon is loaded with a large amount of impurities, the pore structure is blocked, and the activated carbon basically loses its adsorption capacity.

| Element | Na | Ca | K | C | H | S | Others |
|---|---|---|---|---|---|---|---|
| Content (wt%) | 2.37 | 0.76 | 0.63 | 36.32 | 4.28 | 4.81 | 53.82 |

| The iodine value (mg · g\(^{-1}\)) | Specific surface area (cm\(^3\) · g\(^{-1}\)) |
|---|---|
| 354 | 376.211 |

Figure 1. SEM analysis of M-AC.
2.2. Methods
Schematic diagram of experimental process is shown in figure 2. The activator flow rate is controlled by a gas flow meter, and the heat insulating material provides relatively stable temperature conditions for the reaction, and potassium permanganate, sodium sulfate and activated carbon constitute an exhaust gas absorption device for absorbing harmful mercury-containing vapor. The jar is made of quartz and can withstand temperatures up to 1200 °C. Take 20 g of the dried sample into a jar, and let the gas flow 200 L·h⁻¹ into the nitrogen for 15 min to ensure that the oxygen in the jar is discharged to prevent the activated carbon from burning. After the sample was heated to a certain temperature in a nitrogen atmosphere, the nitrogen flow was turned off, and the activator was applied at a rate of 10 L·h⁻¹ for 60 min. After the end of the activation, the activator was turned off and the nitrogen flow was resumed. The sample was removed from the jar and stored for further characterization as the oven was cooled to room temperature. The regenerated activated carbon prepared by using water vapor as an activator is called W-AC and using carbon dioxide is called C-AC. Since the mercury-containing vapor is discharged from the sample when the temperature exceeds 150 °C, the exhaust gas generated during the activation is purified before being discharged.

2.3. Analysis methods
The iodine number of activated carbon product was tested according to the Standard Testing Methods of PR China (GB/T12496.8-1999). The yield was the quality ratio of the activated carbon after regeneration to the mercury-containing spent activated carbon. The mineral composition of the samples was determined by x-ray diffraction (XRD, TTR III, Rigaku, Japan). The content of carbon phase was detected by using Raman spectra. The adsorption value of methylene blue is determined by GB/T12496.10-1999. Scanning electron microscopy (SEM) analysis was used to assess the surface morphology of sample. Nitrogen adsorption experiments on the raw material and activated carbon were conducted at 77 K using an automatic adsorption apparatus (AUTSORB-1, USA) at a relative pressure (P/P₀) ranging from 10⁻⁷ to 1. The sample was degassed at 573 K for 12 h. The BET equation was used to calculate the surface area of the samples and the pore size distribution was analyzed using the Non-localized density function theory (NLDFT).
Method for determining mercury content: 15 ml HCL, 5 ml HNO₃ was added to 20 ml of 100 g·l⁻¹ NaCl solution, the solution was set, the fixed solution volume was 500 ml. 1 g of the spent catalyst was digested for 15 min under slightly boiling conditions, and the digestion process was repeated twice, and the resulting solution was filtered. A blank test was also done. The concentration of Hg²⁺ in the solution is determined by Inductively coupled plasma atomic emission spectrometry (ICP-OES) [17].

3. Results
3.1. The influence of activation temperature on the regeneration effect
The yield of W-AC and C-AC were shown in figure 3. The yield of regenerated activated carbon decreases with increasing temperature, because regeneration temperature is the main factor impact on the regeneration process. To restore the adsorption capability of the waste carbon, the pollutant in the pore structure of the waste carbon was eliminated with the regeneration temperature increases. Iodine number of W-AC and C-AC were shown in figure 4. The sample activation temperature is in the range of 600 °C–900 °C, and the iodine value of the activated carbon continues to increase. With the temperature goes up, the iodine number goes down. It can be attributed to the accelerated reaction between
carbon and activation agent with the increase of activation temperature, and the more pores formed in a certain period of time, the more complicated pore structure. When the activation temperature exceeds 900 °C, the reaction rate too fast, some of the pores are ablation or collapse, making the pore size wider, resulting in the reduction of iodine adsorption capacity. The iodine adsorption value of CO₂ regenerated activated carbon is 744.5 mg g⁻¹ at 900 °C, the iodine number reflects the presence of microspores in the activated carbon. In the process of industrial wastewater treatment, the iodine adsorption value of activated carbon is required to be greater than 700 mg g⁻¹. The regenerated activated carbon prepared in this study can meet this requirement. In the research by Liu et al [7], the activated carbon was prepared by microwave, and the iodine adsorption value was 791.64 mg g⁻¹. The addition of microwaves enhances the adsorption capacity of activated carbon, but the requirements for experimental equipment and conditions are higher and the improvement is limited.

Comparing W-AC and C-AC, the activation temperature in the range from 600 to 800 °C, the iodine number is approximately equal. It can be attributed to the chemical reaction rate between carbon and the two different activation agents are approximately equal. But as the activation temperature continues to rise, the adsorption capacity of C-AC was better than W-AC. The reaction rate between carbon dioxide and waste carbon is faster, which is more conducive to the decomposition and gasification of pollutants on the surface of waste carbon, in this conditions, the activated carbon have better adsorption performance. Combined with the iodine number and yield of activated carbon determined 900 °C is the optimal activation temperature.

3.2. Pore structure and surface area analysis
Nitrogen adsorption experiments were carried out by preparing activated carbon from different activators at 900 °C. Nitrogen adsorption isotherm of M-AC, W-AC and C-AC were shown in figure 5. The adsorption isotherm of C-AC was found higher then W-AC, which indicates that there were a higher number of pores in C-AC. The pore size distribution is an important indicator of activated carbon that determines the fraction of the total pore volume that is accessible to molecules of a given size and shape. According to the division of International Union of Pure and Applied Chemistry classification [18], the adsorption isotherm of C-AC was
classified as type IV [19, 20]. When the relative pressure is low, the adsorption quantity increases sharply with the increase of relative pressure. When the relative pressure reaches 0.03, the adsorption quantity increases slowly with the continuous increase of relative pressure, indicating that adsorption is transitioning from monomolecular layer to multimolecular layer. When the relative pressure reaches 0.4, there is a significant hysteresis loop, which indicates that the pore structure of the adsorption isotherm of C-AC is affected by pressure and capillary condensation occur, the adsorption capacity of different adsorption layers is provided by each step. When the relative pressure is close to 1, the adsorption capacity continues to increase and tailing peak occur, which indicates that activated carbon is mainly composed of micro-pore and contains a certain amount of mesoporous and macropore. The adsorption isotherm of W-AC can be classified as type IV too. But the weak hysteresis loop occur when the relative pressure reaches 0.05, it suggests that the capillary condensation of W-AC is weak and adsorption capacity of different adsorption layers is lower than C-AC.

![Figure 5. Nitrogen adsorption isotherm of M-AC, W-AC and C-AC.](image)

| Sample preparation conditions | Specific surface area ($m^2 \cdot g^{-1}$) | Total pore volume ($cm^3 \cdot g^{-1}$) | Average pore diameter (nm) | Micro porous proportion (%) |
|-------------------------------|-------------------------------------------|-----------------------------------------|-----------------------------|-----------------------------|
| M-AC                          | 359.176                                   | 0.184                                   | 1.10                        | 41                          |
| W-AC                          | 540.372                                   | 0.242                                   | 1.95                        | 59                          |
| C-AC                          | 750.755                                   | 0.477                                   | 2.79                        | 63                          |

The pore structure parameters of M-AC, W-AC and C-AC were shown in table 3. The specific surface area of C-AC was 750.755 $m^2 \cdot g^{-1}$ and total pore volume was 0.477 $cm^3 \cdot g^{-1}$. These data have nearly doubled compared to M-AC. The specific surface area of W-AC was 540.372 $m^2 \cdot g^{-1}$ and total pore volume was 0.242 $cm^3 \cdot g^{-1}$. These data have increased nearly 0.5 times compared to M-AC. The results demonstrate that the total pore volume, average pore diameter and micro porous proportion of activated carbon after regeneration had high values. The average pore diameter of the activated carbon after regeneration by these two agents were 2.79 nm and 1.95 nm, the micro porous proportion were 63% and 59%, which indicates that the regenerated activated carbon were mesoporous because most of the pores and sharpest peak in the pore distribution are in the range from 20 to 50 Å (figures 6 and 7). These results suggest that the pore structure of the C-AC was more fully developed than W-AC, M-AC still has mesopores and mesopores (figure 8), but it is relatively dispersed, and most pores are blocked by impurities.

### 3.3. SEM analysis

The microstructure of activated carbon prepared by different activators at 900 °C was studied. The SEM analysis of W-AC and C-AC are shown in figures 9 and 10. There were still some impurities on the surface of W-AC and the pore structure was not clear enough. The impurities were removed and a clear pore structure was clear observed. The pore structure was cleaned due to the volatilization of mercury compounds. These results suggest that the regenerated activated carbon had a large pore structure and surface area. In particular, the regeneration effect of carbon dioxide preparation condition is most significant.
3.4. Analysis on the effect of mercury removal

The mercury removal effect of W-AC and C-AC is shown in table 4. Mercury content in regenerated activated carbon decreases with increasing temperature. When the activation temperature exceeds 900 °C, the mercury content in the regenerated activated carbon are about 0.006 mg g⁻¹, which suggests that mercuric compound almost completely pulled away from activated carbon [7]. The mercury removal effect is remarkable, and the removal rate reaches 99.96%.

Figure 6. The pore diameter distribution of W-AC.

Figure 7. The pore diameter distribution of C-AC.

Figure 8. The pore diameter distribution of M-AC.
3.5. Phase analysis of regenerated activated carbon

Regenerated activated carbon prepared with different activators at 900 °C was used for phase analysis. The XRD diffraction patterns of M-AC, W-AC and C-AC are shown in figure 11. The diffraction peaks of M-AC are relatively irregular. The diffraction angles contain a large amount of organic impurities near 25.13° and 43.89°.

### Table 4. Mercury removal effect of W-AC and C-AC.

| Temperature(°C) | Mercury content in the filtrate(µg l⁻¹) | Mercury content in regenerated activated carbon(µg g⁻¹) | Removal rate(%) |
|----------------|----------------------------------------|-----------------------------------------------------|-----------------|
|                | W-AC  C-AC                      | W-AC  C-AC                      | W-AC  C-AC |
| 600            | 0.126  0.116                      | 0.063  0.058                      | 99.65  99.68  |
| 700            | 0.100  0.092                      | 0.050  0.046                      | 99.72  99.74  |
| 800            | 0.058  0.042                      | 0.029  0.021                      | 99.84  99.88  |
| 900            | 0.016  0.016                      | 0.008  0.008                      | 99.95  99.95  |
| 1000           | 0.012  0.014                      | 0.006  0.007                      | 99.96  99.96  |
and the peaks of HgCl₂ crystals are obvious. The peak of organic impurities in the regenerated activated carbon is significantly weakened, and a large amount of organic impurities are converted into gas removal along with the activation process. Combined with SEM analysis, it is known that the impurities adsorbed on the surface of the regenerated activated carbon are significantly reduced, and the adsorption capacity of the activated carbon is restored. Moreover, HgCl₂ crystals could not be found in the regenerated activated carbon, and the activation process had excellent Hg²⁺ removal effect, which was consistent with the conclusion of 3.3.

Combined with the study of 3.2–3.4, it is speculated that the activation mechanism is shown in figure 12. Process I shows the initial stage of regeneration of spent activated carbon with water vapor as an activator. It can be seen that at the initial stage of activation, some impurities are still present on the surface of the activated carbon. When the impurities are separated from the activated carbon, a fresh reaction interface occurs, and the water vapor and the activated carbon itself generate a pore-forming reaction. As the reaction continues, the activation process enters the final stage (Process II), impurities are substantially detached from the surface of the activated carbon, and the fresh reaction interface is exposed.
Processes III and IV show the initial and final stages of regeneration of spent activated carbon using carbon dioxide as the activator. After the fresh reaction interface is exposed, carbon dioxide and the activated carbon itself produce a pore-forming reaction $\mathcal{\hat{O}}$.

With water vapor as the activator, in contrast to carbon dioxide, impurities are removed during the activation process. At the same time, the activator reacts with the activated carbon itself. However, since the rate of occurrence of pore-forming reaction $\mathcal{O}$ is higher than that of pore-forming reaction $\mathcal{\hat{O}}$, the yield of activated carbon under water vapor conditions is higher than that of carbon dioxide (figure 3). On the other hand, when the temperature exceeds 800 °C [21], the pore-forming reaction $\mathcal{O}$ occurs sharply, resulting in significant collapse and ablation of the pore structure of the activated carbon, resulting in a smaller specific surface area of the recycled activated carbon. The pore-forming reaction $\mathcal{\hat{O}}$ occurs sharply more than 950 °C [22]. Therefore, with carbon dioxide as the activator, the regeneration activity specific surface area is larger than the water vapor condition (table 3), and the adsorption capacity recovery effect is more remarkable (figure 4).

3.6. Analysis of adsorption properties of methylene blue
The adsorption properties of methylene blue of regenerated activated carbon under different activator conditions at different temperatures are shown in figure 13. The methylene blue adsorption value of C-AC is 210.07 mg g$^{-1}$ at 900 °C. The adsorption ability of methylene blue reflects the presence of mesopores to assess its suitability to adsorb large dye molecules. The change law of methylene blue adsorption effect is basically consistent with the change law of iodine adsorption value. The activated carbon regenerated with carbon dioxide as the activator has basically reached the adsorption index of pollutants such as methylene blue in the treated wastewater. In the research of Xu, H et al [23], it is pointed out that the research on advanced catalysts in the PVC industry is still in the laboratory stage, and the emission of waste catalysts is huge. The research on the activation and regeneration mechanism of spent catalysts is of guiding significance for promoting the regeneration of spent catalysts.

4. Discussion
In this study, the activated product of the waste product in the PVC industry was activated and regenerated, and the effects of different activators on the activation process were studied. The harmful elements in the waste adsorbent materials are removed efficiently, which has reference significance for the purification of the same type of waste adsorbent materials. The entire activation process is relatively simple and provides feasibility for industrialization. The activation process can further optimize the process, adding microwaves and other heating methods to study the activation effect. On the other hand, it is possible to study the organic impurities in the waste adsorbent materials and propose more effective utilization methods.

5. Conclusions
In this study, the activated mercury regeneration catalyst produced in the process of PVC production was activated and regenerated. The activation regeneration conditions were determined by iodine adsorption performance. The specific surface area of the activated carbon was determined by nitrogen adsorption. The
microstructure was studied by SEM. Combined with mercury removal rate, XRD analysis, Raman analysis and methylene blue adsorption effect analysis, the conclusions are as follows:

1. The optimum temperature for the regeneration of spent activated carbon is 900 °C. The performance of activated carbon prepared by carbon dioxide as an activator is better than that of water vapor. After regeneration, the recovery of activated carbon pore structure is remarkable, and the specific surface area reaches 750.755 m²·g⁻¹. The regeneration process has excellent mercury removal effect, and the removal rate can be reached 99.96%.

2. The activation mechanism of two different activators is summarized. During the activation of carbon dioxide, the rate of pore formation is lower than that of water vapor. At 900 °C, the pore-forming reaction of water vapor occurs violently, resulting in collapse and ablation of the pore structure. After regeneration, the activated carbon has a specific surface area lower than that of carbon dioxide.

3. The regeneration effect of activated carbon is remarkable, the whole activation process is relatively simple, and the adsorption performance of regenerated activated carbon basically meets the requirements of people’s domestic sewage treatment. This study provides an alternative way for prolonging the lifetime of catalysts.

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**Author Contributions**

Conceptualization, Junjing Tang; Data curation, Junjing Tang and Ping Guo; Formal analysis, Junjing Tang; Funding acquisition, Libo Zhang; Methodology, Ping Guo, Tu Hu, Libo Zhang, Shiwei Li, Song Cheng, Jian Liu and Chao Liu; Writing—original draft, Junjing Tang; Writing—review & editing, Tu Hu and Song Cheng.

**Conflicts of Interest**

The authors declare no conflicts of interest.

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