Chemically activated hydrochars as an effective adsorbent for volatile organic compounds (VOCs)

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**Highlights**

- Chemically activated hydrochars showed good removal of acetone and cyclohexane.
- Sorption capacities of the activated hydrochars were 50.57 – 159.66 mg·g⁻¹.
- Surface areas of hydrochars and their adsorption capacities had a strong linear correlation.
- Physical adsorption was the dominant mechanism.
- Activated hydrochar showed good reusability.

**Abstract**

Hydrochars derived from hickory wood and peanut hull through hydrothermal carbonization were activated with H₃PO₄ and KOH to improve their performance as a volatile organic compound (VOC) adsorbent. Polar acetone and nonpolar cyclohexane were used as representative VOCs. The VOC adsorptive capacities of the activated hydrochars (50.57 – 159.66 mg·g⁻¹) were greater than that of the nonactivated hydrochars (15.98 – 25.36 mg·g⁻¹), which was mainly caused by the enlargement of surface area. The significant linear correlation (R² = 0.984 on acetone, and R² = 0.869 on cyclohexane) between BET surface areas of hydrochars and their VOC adsorption capacities, together with the obvious adsorption exothermal peak of differential scanning calorimetry curve confirmed physical adsorption as the dominating mechanism. Finally, the reusability of activated hydrochar was tested on H₃PO₄ activated hickory hydrochar (HHP), which had higher acetone and cyclohexane adsorption capacities. After five continuous adsorption desorption cycles, the adsorptive capacities of acetone and cyclohexane on HHP...
1. Introduction

The discharge of volatile organic compounds (VOCs) threatens the environment and human health severely because most of them are highly toxic, carcinogenic, and yield even more hazardous compounds such as secondary organic aerosol particles, which lead to photochemical smog, stratospheric ozone depletion, etc. Therefore, it is necessary to urgently develop effective and environmentally friendly VOC elimination techniques. Compared to common VOC control techniques such as absorption, regenerative thermal oxidizer, regenerative catalytic oxidation, etc. (Johnsen et al., 2016; Jia et al., 2017), Adsorption is one of the most effective and cost-efficient technologies; moreover, it can realize the recovery of VOCs (Tefera et al., 2014; Zhang et al., 2017a).

The selection of a suitable adsorbent is crucial for VOC adsorption technology. Among various adsorbents, carbon materials are widely used for their stable physical and chemical properties, high surface area, porosity, and regenerability (Guo et al., 2016; Son et al., 2016; Zhang et al., 2017b; Wan et al., 2018). Hydrochar produced by hydrothermal carbonization under mild conditions (180–250 °C) has been considered as a cost effective novel material (Guo et al., 2017; Fang et al., 2018). Compared with other carbonaceous materials such as activated carbon and biochar, hydrochar has some distinguished advantages: low energy consumption because of low production temperature and non-drying process; high yield because of low carbonization temperature; and low ash content because of the hydrothermal process (Fang et al., 2018).

Since the discovery of hydrochar, it has been widely used in soil fertility amendment and environmental remediation (Zhang et al., 2014; Gronwald et al., 2016; Fang et al., 2018; Song et al., 2018). However, the application of hydrochar in environmental remediation mainly focused on the adsorption of contaminants such as heavy metals and organics in aqueous solutions (Xue et al., 2012; Flora et al., 2013; Fang et al., 2016; Islam et al., 2017). To the best of the authors’ knowledge, little research has been done on air pollutant adsorption by hydrochar. Evaluating VOC adsorption onto hydrochar would thus expand the application of hydrochar.

In general, hydrochar has relatively low surface area and pore volume, which would inhibit its application as an adsorbent (Fang et al., 2015, 2016, 2017). Thus, to improve the adsorption performance of hydrochar, modification is necessary. The common methods of biochar modification, such as CO2 steam, acids, bases, or salts, can significantly change the morphological structure of biochars, which would further influence their adsorption performance (Xue et al., 2012; Fang et al., 2016, 2017; Wang et al., 2017). It is anticipated that all those modification methods can also be used to improve the sorptive properties of hydrochar. Xue et al. (2012) have developed a chemical modification method to improve the adsorption performance of hydrochar through H2O2 oxidation and found that chemical modification greatly enhanced the sorption capacity of the hydrochar to lead in aqueous solutions. Fang et al. (2016, 2017) reported that physical and chemical activations can improve the ability of hickory and peanut hull hydrochars to sorb methylene blue, lead, copper, and cadmium from aqueous solutions. However, none of the previous studies have investigated the removal of air pollutants including VOCs by modified hydrochars.

In this work, hydrochars derived from the common feedstocks hickory wood and peanut hull, as well as their H3PO4 and KOH activated counterparts, were produced and used as VOC adsorbents. Polar acetone and nonpolar cyclohexane, which are among the most used organic chemicals, were selected as the representative VOCs to evaluate the adsorbents. Specific objectives of this work are to (1) determine the feasibility of hydrochar as a VOC adsorbent; (2) investigate the effects of H3PO4 and KOH activation on VOC adsorption by the modified hydrochars; and (3) elucidate the governing adsorption mechanisms of VOCs onto pristine and modified hydrochars.

2. Materials and methods

2.1. Materials

Pristine and activated hydrochars were prepared following the procedures of previous studies (Fang et al., 2015, 2017). Briefly, the hydrochar was prepared by the following process. The feedstocks of hickory wood and peanut hull were milled to particle sizes of 0.5–1 mm and added to a pressurizable lidded stainless-steel pot. Deionized (DI) water (18.2 MΩ, Nanopure water, Barnstead) was added to the same level. The pots were then sealed and heated on a hotplate to 200 °C for six hours. The resulting hydrochars were rinsed for one hour by submersion in tap water and ten minutes in DI water to remove water soluble matter, and then dried for 24 h at 70 °C. The activated hydrochar was prepared by the following procedure. The hydrochar was mixed in a 1:1 w/w ratio with either 50% KOH or 85% H3PO4 solution. The mixture was stirred for 2 h at 85 °C, and the resulting slurry was oven dried for 24 h at 100 °C. The dried slurry was then activated in a tube furnace for 1 h at 600 °C under N2 gas flowing at a rate of 150 mL·min⁻¹. After cooling to room temperature, the samples were washed with DI water in order to remove any residual chemicals. They were washed repeatedly with DI water until the pH of the rinse water stabilized. Hydrochar derived from hickory wood and peanut hull were abbreviated as HH and PH. Those modified by KOH and H3PO4 were abbreviated as HHH, PHH, HHP, and PHP, respectively. The physicochemical characteristics of the hydrochars have been characterized in previous studies (Fang et al., 2015, 2017) and are presented in Table 1. Acetone and cyclohexane purchased from Sigma-Aldrich, Inc. were chosen as adsorbates. The properties of the VOCs are shown in Table 2.

2.2. Adsorption and desorption processes

The adsorption experiments were performed by gravimetric method using a TGA (TGA/DSC 1 STAR System, METTLER TOLEDO). Approximately 10 mg sample was placed into the 70 mL Al2O3 vessel and degassed with 50 mL·min⁻¹ N2 at 105 °C for 2 h. After cooling to the adsorption temperature, the inlet line was switched from N2 to a 50 mL·min⁻¹ VOC vapor stream until the mass change reached an equilibrium. Desorption was performed by increasing the temperature from adsorption temperature to 150 °C at the rate of 10 °C·min⁻¹. A blank test run was performed to eliminate instrument and buoyancy influences from data. Duplicated tests were conducted for each experiment and mean values were reported. The adsorption data were described by the pseudo-first order (1)
and pseudo-second order (2) models:

$$ q_t = q_e \left(1 - e^{-k_1 t}\right) $$

(1)

$$ q_t = \frac{t - k_2 q_e^2}{1 + q_e t - k_2 q_e} $$

(2)

where $q_t$ (mg $\cdot$ g$^{-1}$) and $q_e$ (mg $\cdot$ g$^{-1}$) are the amount of VOC adsorbed at time $t$ (min) and equilibrium, respectively; $k_1$ (min$^{-1}$) and $k_2$ (g $\cdot$ mg$^{-1} \cdot $ min$^{-1}$) are adsorption rate constants.

3. Results and discussion

3.1. Hydrochar and chemically activated hydrochar

The properties of hydrochar varied between both the feedstock and treatment condition (Table 1). The hydrochar derived from hickory wood had higher surface area and pore volume than peanut hull hydrochar, whereas it was the opposite after activation by KOH or H$_3$PO$_4$. Nonactivated hydrochars had low surface areas of less than 8 m$^2$·g$^{-1}$, whereas after modification by H$_3$PO$_4$ or KOH, the surface areas increased to 222–1436 m$^2$·g$^{-1}$. In addition, compared to KOH, the H$_3$PO$_4$ activation produced higher surface area on the hickory wood hydrochar, whereas it was the opposite after activation by KOH

| Sorbent | BET-SA (m$^2$·g$^{-1}$) | BJH-PV (mL·g$^{-1}$) | C (wt. %) | H (wt. %) | O (wt. %) | N (wt. %) | Ash (wt. %) | O/C (mol.%)
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<tbody>
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<td>8</td>
<td>0.121</td>
<td>68.7</td>
<td>4.6</td>
<td>26.1</td>
<td>0.2</td>
<td>0.4</td>
<td>28.5</td>
</tr>
<tr>
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<td>222</td>
<td>0.050</td>
<td>68.3</td>
<td>1.7</td>
<td>23.2</td>
<td>0.2</td>
<td>6.6</td>
<td>25.5</td>
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<tr>
<td>HHP</td>
<td>1436</td>
<td>0.028</td>
<td>62.8</td>
<td>1.9</td>
<td>28.7</td>
<td>0.2</td>
<td>6.4</td>
<td>34.3</td>
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<tr>
<td>PH</td>
<td>7</td>
<td>0.010</td>
<td>70.6</td>
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<td>21.9</td>
<td>1.9</td>
<td>0.5</td>
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</tr>
<tr>
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<td>1.5</td>
<td>4.5</td>
<td>27.9</td>
</tr>
<tr>
<td>PHP</td>
<td>1091</td>
<td>0.079</td>
<td>41.5</td>
<td>1.7</td>
<td>51.6</td>
<td>1.1</td>
<td>4.1</td>
<td>93.3</td>
</tr>
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</table>

SA = surface area and PV = pore volume.

Table 2
Main properties of VOCs.

<table>
<thead>
<tr>
<th>VOCs</th>
<th>Molecular formula</th>
<th>Molecular weight (g·mol$^{-1}$)</th>
<th>Boiling point (°C)</th>
<th>Dipole moment(D)</th>
<th>Kinetic diameter (nm) (Li et al., 2009)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Acetone</td>
<td>C$_2$H$_4$O</td>
<td>58.08</td>
<td>56.53</td>
<td>2.91</td>
<td>0.46</td>
</tr>
<tr>
<td>Cyclohexane</td>
<td>C$<em>9$H$</em>{12}$</td>
<td>126.20</td>
<td>80.74</td>
<td>0.00</td>
<td>0.60</td>
</tr>
</tbody>
</table>

and micropores by carbonization (Yorgun and Yildiz, 2015). As for KOH, the intercalated KOH can react with carbon at high temperature. The generated potassium carbonate and potassium oxide can further interact with carbon and broaden the structure, which hence leads to the formation of mesopores (Romanos et al., 2011; Wang and Kaskel, 2012; Thubsuang et al., 2017). The ash content on the original hydrochar was less than 0.5%, while after activation by H$_3$PO$_4$ or KOH, the content increased to 4.1–6.6%. The increase in ash content may be attributed to residual activating agents. As for the O content, H$_3$PO$_4$ activated hydrochar PHP and HHP had high oxygen contents of 51.6% and 28.7%, respectively, which were higher than that of original and KOH activated hydrochars (21.9–26.1%). Since most of the oxygen groups such as carboxyl, hydroxyl, carbonyl, anhydride and lactone are the source of surface acidity on carbon materials, the O/C molar ratio of carbon materials can reflect their polarity (Abe et al., 2000; Uchimiya et al., 2010). Thus, hydrochar with higher O/C ratio has superior potential for hydrophilic VOC adsorption (Li et al., 2011). H$_3$PO$_4$ activated hydrochars possess higher polarity than original or KOH activated hydrochars, as the O/C ratio in PHP and HHP were 93.3% and 34.3%, respectively, which are higher than 23.3–28.5% in original and KOH activated hydrochars. Therefore, the PHP and HHP have higher acetone adsorption capacities.

3.2. Adsorption properties

The adsorptive capacities of acetone and cyclohexane on pristine hydrochars were very low, which were in a range of 15.98–25.36 mg·g$^{-1}$, whereas after activation by H$_3$PO$_4$ or KOH, the adsorption capacities of hydrochar increased significantly to 50.57–159.66 mg·g$^{-1}$ (Fig. 1). Generally, most of the H$_3$PO$_4$ activated hydrochars had higher adsorption capacities than that of KOH activated hydrochars. The acetone adsorptive capacities on HHP and PHP were 147.77 mg·g$^{-1}$ and 113.94 mg·g$^{-1}$, which were higher than that on HHK and PHK (50.57 and 61.26 mg·g$^{-1}$, respectively). Much more acetone was sorbed onto H$_3$PO$_4$ activated hydrochar than that of KOH, which may be caused by the following two reasons. On one hand, H$_3$PO$_4$ activated hydrochar had higher surface area, which would provide more adsorption sites for VOC molecules. On the other hand, the H$_3$PO$_4$ activation generated plenty of oxygen functional groups on the surface of hydrochar, which can enhance the adhesion of hydrophilic VOCs onto hydrochar (Li et al., 2011). The similar phenomena of acid modified carbon adsorbents having superior performance toward polar VOCs adsorption was observed by Kim et al. (2006), who found the polar methanol, ethanol and i-propanol adsorptive capacities on H$_3$PO$_4$...
modified AC are greatly improved compared with those of AC, even though the surface area decreased from 892 m²·g⁻¹ to 719 m²·g⁻¹.

However, the presence of oxygen groups has negative effects on the hydrophobic VOC adsorption by inhibiting the interactions between hydrophobic VOCs and π-electron rich regions of adsorbents (Lillo-Rodenas et al., 2005; Subramanian et al., 2013). Nevertheless, alkali treatment can remove the oxygen groups (Li et al., 2011; Niu et al., 2017). This may be the reason why when modified by KOH, the PHK had a slightly higher cyclohexane adsorptive capacity of 105.71 mg·g⁻¹ than PHP (101.04 mg·g⁻¹), even though the surface area of latter was almost two times higher than the former. Similar observations have been reported in the literatures, such as the nonpolar VOCs o-xylene, benzene and carbon tetrachloride have lower adsorptive capacities on acid modified ACs than on pristine adsorbents (Bansode et al., 2003; Li et al., 2011).

The cyclohexane adsorptive capacity on most of the tested hydrochar was higher than that of acetone. This may be related to the properties of VOC molecules, such as the molecular weight and boiling point. The molecular weight of VOCs can impact their adsorption onto an adsorbent, which was confirmed by Tsai et al. (2008), who compared the adsorption of CH₃CN, C₂H₅O and CHCl₃ on sludge-derived carbonaceous adsorbents and found the diffusion coefficients of higher molecular weight VOCs were significantly higher than that of lower molecular VOCs. In the current study, the cyclohexane had a higher molecular weight (84.16 g·mol⁻¹) than acetone (58.08 g·mol⁻¹), which may be a reason why the former had a higher adsorption capacity than the latter. In addition, the VOC adsorption onto porous adsorbent is similar to vapor-liquid phase transitions and liquid-like condensation, where the boiling point VOCS would be preferentially adsorbed for their stronger intermolecular forces with the adsorbent (Chiang et al., 2001; Guo et al., 2013). Therefore, the cyclohexane with a boiling point of 80.74 °C had higher adsorption capacity on hydrochar than that of lower boiling point acetone (56.53 °C). Similar observations have been reported on VOC adsorption by ACF (Giraudet et al., 2014), AC (Oh et al., 2010; Dobre et al., 2014), and other porous adsorbents (Qu et al., 2009). Although the adsorption performance of high boiling point VOCs is superior to those of low boiling point VOCs, it is more difficult for them to desorb due to their strong affinity with adsorbent. The different desorption processes will be investigated in the following section.

3.3. Adsorption kinetics and mechanisms

Adsorption kinetics were comparatively described by pseudo-first order model and pseudo-second order model. Generally, the latter matched the experimental data better than the former, whereas the VOC adsorption onto HHP which had the largest surface area was an exception (Fig. 2). The pseudo-second order model fit the VOC adsorption on carbonaceous adsorbents better than pseudo-first order model, which was consistent with other literature. For benzene and toluene adsorption on coconut shell based porous carbon, the adsorption capacity deviation between experimental results and calculated values by pseudo-second-order kinetic model were relatively lower than that of pseudo-first-order kinetic model (Nasri et al., 2014). The model parameters were present on Table 3. The coefficient of determination ($R^2$) values of pseudo-second order model were in a range of 0.879–0.999, which were higher than that of pseudo-first order model (0.877–0.986). This result suggested that most of the VOC adsorption on hydrochars were controlled by more than one rate-determining step or mechanism (Creamer et al., 2014). Generally, the high adsorption rate constants of $K_1$ and $K_2$ were present on the activated hydrochars other than original hydrochars. In addition, the hydrochar with higher surface area, such as HHP, had larger adsorption rate constants of up to 0.439 min⁻¹. Many studies have widely revealed that the specific surface area of carbonaceous adsorbents are primarily responsible for the physical adsorption of organic compounds (Gupta and Saleh, 2013; Son et al., 2016). Therefore, higher surface area hydrochar had larger adsorption rate constants implied the physical adsorption may be the dominating mechanism for the high surface area hydrochar.

The linear relationship between surface area and adsorption capacity was investigated. As shown in Fig. 3a, significant positive correlation was observed with $R^2 = 0.869$ (P = 0.0067) on cyclohexane, and $R^2 = 0.984$ (P = 0.0009) on acetone. The higher correlation coefficient confirmed the effect of surface area on VOC adsorption. Besides, the typical differential scanning calorimetry curve of acetone adsorption onto HHP was present on Fig. 3b. An obvious exothermic peak can be observed accompanying the adsorption process, which further indicate the VOC sorption on biochar is primary physical exothermic process. This result is consistent with the findings of a previous study that the adsorption of acetone, cyclohexane and toluene on biochars is an exothermic process (Zhang et al., 2017b).

3.4. Reusability

Reusability is one of the most crucial factors determining the possibility of a sorbent to be used commercially. To investigate the reusability of hydrochar, the desorption process was determined through batch desorption experiments conducted on VOC saturated hydrochar via TGA with a $10$ °C min⁻¹ heating rate from $20$ °C to $150$ °C. As shown in Fig. 4a, all the acetone and cyclohexane adsorbed on pristine hydrochar HH were entirely desorbed at temperatures less than $45$ °C, while that on the activated hydrochar had higher desorption temperatures. The desorption temperatures of cyclohexane on HHP and HHK were 142 °C and 148 °C, respectively, which were higher than that of acetone (106 °C and 83 °C, respectively). The higher desorption temperature of activated hydrochars than that of pristine hydrochars may be related to their
porosity and abundant chemical functional groups, which would inhibit the release of adsorbed VOCs. As reported by Lashak et al. (2012), who investigated automotive painting VOC adsorption-desorption on beaded activated carbon, the VOCs adsorbed in the narrow micropores need higher regeneration temperatures. Also, the higher boiling point of cyclohexane than that of acetone may be the reason why cyclohexane was much more difficult to be desorbed compared to acetone. As reported, the high boiling point VOCs had strong affinity to the sorbent. Thus it is more difficult to be desorbed compared to the low boiling point VOCs (Lashaki et al., 2012; Son et al., 2016). Shah et al. (2014) compared the process of thermal regeneration between acetone and methyl ethyl ketone (MEK) and found 95% acetone adsorption capacity on activated carbon can be restored at 80 °C, while that was only 90% for MEK at 120 °C, which had a higher boiling point. The temperature of complete desorption of acetone on HHP was higher than that of HHK. This may be caused by the abundance of oxygen functional groups on the surface of H3PO4 modified hydrochar, which improve the adhesion force with hydrophilic acetone.

The highest acetone and cyclohexane adsorption capacities were obtained on HHP. HHP was then chosen to do the recycling experiments, and the results are presented in Fig. 4b. Both the acetone and cyclohexane adsorption capacities on HHP decreased slightly during the 5 times repeat tests. The adsorptive capacity of

<table>
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<tr>
<th>Adsorbate</th>
<th>Model</th>
<th>pseudo-first order model</th>
<th>pseudo-second order model</th>
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<tr>
<td></td>
<td>Equation</td>
<td>$q_t = q_e(1 - e^{-k_1t})$</td>
<td>$q_t = \frac{t_k 2_q^2}{1 + t + 1 + 2_q^2}$</td>
</tr>
<tr>
<td>Parameters</td>
<td>$q_e($mg g$^{-1})$</td>
<td>$k_1($min$^{-1})$</td>
<td>$R^2$</td>
</tr>
<tr>
<td>Acetone</td>
<td>HH</td>
<td>25.22</td>
<td>0.077</td>
</tr>
<tr>
<td></td>
<td>HHK</td>
<td>48.81</td>
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<tr>
<td></td>
<td>PHP</td>
<td>98.83</td>
<td>0.151</td>
</tr>
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</table>

Fig. 2. Adsorption kinetics of acetone onto pristine and activated (a) hickory wood hydrochar and (b) peanut hull hydrochar at t 20 °C; and cyclohexane onto pristine and activated (c) hickory wood hydrochar and (d) peanut hull hydrochar at 20 °C.

Table 3
Best-fit model parameters of VOC adsorption onto the adsorbents at 20 °C.
acetone on HHP decrease 6.2%, while that of cyclohexane decreased 7.8%. Additionally, the largest decline appeared in the first cycle, where the acetone and cyclohexane decreased 5.1% and 5.3%, respectively. Thereby, there were only 0.9% and 2.5% decline during the last four cycles on acetone and cyclohexane. The cyclohexane had higher reduction than acetone during the cycles, which may be caused by its stronger affinity with adsorbents, thus a little cyclohexane was retained under the desorption temperature of 150 °C.

4. Conclusions

Hydrochars derived from hickory wood and peanut hull, as well as their H3PO4 and KOH modified counterparts, were investigated for their performance on VOC adsorption. Their adsorption and desorption performance on polar acetone and nonpolar cyclohexane were tested by TGA. After modification by H3PO4 and KOH, the BET surface area of activated hydrochar increased significantly, while H3PO4 activation created larger surface area than that of KOH activation. The adsorption capacities of acetone and cyclohexane on nonactivated hydrochars were lower than 25.36 mg·g⁻¹, while that on the activated hydrochars increased significantly up to 159.66 mg·g⁻¹. Furthermore, the H3PO4 activated hydrochar had higher adsorptive capacity than that of KOH activated hydrochar. The higher adsorption capacity can be attributed to their high surface area, which was confirmed by the significant linear correlation between surface area and VOC adsorption capacity. At last, a slight decline in VOC adsorption capacity on activated hydrochars after 5 times recycle tests confirmed the reusability of the activated hydrochar.

Acknowledgments

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