

# Research Article

# Adsorptive Removal of Trichloroethylene in Water by Crop Residue Biochars Pyrolyzed at Contrasting Temperatures: Continuous Fixed-Bed Experiments

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Biochar (BC) has attracted great attention as an alternative sorbent to activated carbon (AC). Objective of this study was to determine trichloroethylene (TCE) removal by soybean stover BC pyrolyzed at 300 (BC300) and 700°C (BC700) in continuous fixed-bed column. Columns packed with BC300, BC700, and AC reached breakthrough time in 1.1, 27.0, and 50.7 h, respectively. BC700 had higher TCE adsorption capacity than BC300 due to its higher surface area, nonpolarity, and aromaticity. The sorption capacities of AC (774.0 mg g<sup>-1</sup>) and BC700 (515.1 mg g<sup>-1</sup>) were 21.6 and 14.4 times higher than that of BC300 (35.9 mg g<sup>-1</sup>). The lower desorption rate of TCE from BC300 than BC700 and AC may be attributed to the strong binding/partition of TCE to the noncarbonized part of BC. Thomas model also adequately described the adsorption data indicating interphase mass transfer. Overall, AC showed best efficiency for removing TCE from water in column experiments. However, although sorption and desorption capabilities of BC700 were a little lower than AC, it is still a good alternative for AC to remove organic contaminants such as TCE from water due to its cost-effectiveness.

# 1. Introduction

Char, a solid material produced from carbonaceous biomass, is emerging as an alternative to activated carbon (AC) with lower cost and environmental advantages. Char commonly appears under uncontrolled natural conditions through partial or complete carbonization of biomass such as wood, manure, or leaves [1–3]. Biochar (BC) is a name developed in conjunction with soil science and related to carbon sequestration in soils [4–6]. Biochar means black carbon derived from biomass pyrolysis and closely resembles activated carbon with a structured carbon matrix and a medium-to-high surface area. Biochar has a wide range of chemical compositions

and surface properties depending on biomass type and pyrolysis temperature [7]. Several studies have already reported the effect of pyrolysis temperature on sorption properties of biochars [8, 9]. Higher temperature-pyrolyzed-biochar possesses high surface area, carbon content, aromaticity, and hydrophobicity, which lead to the increase of sorption capacity towards contaminants, especially for the nonionized chemicals [10]. Lower temperature-pyrolyzed-biochar was believed to be effective for polar organics and heavy metals due to abundant polar groups on biochar surface [10]. Because of its high efficiency and capacity to adsorb organic contaminants, it has also been spotlighted as an excellent adsorbent [10, 11] for water and wastewater treatment. In comparison with conventional activated carbon, BC may be economically preferable with less energy requirements and no pre- or postactivation processes during manufacturing. The estimated break-even price for BC is US \$246 t<sup>-1</sup>, which is approximately 1/6 of commercially available AC (~US \$1500 t<sup>-1</sup>) [11, 12]. It is also environmentally beneficial by converting/recycling of organic wastes via pyrolysis. However, substantial understanding is required to ensure efficiency of BC to remove organic contaminates from water/groundwater.

Trichloroethylene (TCE) is a widely used chlorinated solvent in industry that is released into the atmosphere as vapor [13]. It contaminates surface water or groundwater via direct discharge or leaching from disposal operations [14]. TCE has been identified as a prior environmental pollutant by the US Environmental Protection Agency [15]. Groundwater contamination by TCE commonly occurs worldwide in many industrial and urban areas. A severe level of TCE (1.52 mg L<sup>-1</sup>) has been detected from groundwater at the industrial complex in Wonju city, Korea, with typical values ranging from 0.01 to 1.52 mg L<sup>-1</sup> [16]. According to the Korea Ministry of Environment, the maximum permissible level (MPL) of TCE is  $0.03 \text{ mg L}^{-1}$  for residence and  $0.06 \text{ mg L}^{-1}$ for industrial areas. However, TCE concentrations in city groundwater are 50 times greater than the MPL [17, 18].

Sorption is one of the most popular and widely used technologies for depuration of groundwater [11, 19]. Various sorbents such as activated carbon, biomass, zeolite, and resins have been conventionally used to decontaminate water [15, 19–21]. However, there is a need to explore low-cost, effective, and environmentally friendly materials to purify contaminated groundwater. In this context, BC could be a strong candidate for TCE removal due to the advantages mentioned above. Most sorption studies have been confined to batch type equilibrium studies [11, 13]. However, data from batch type sorption experiments is insufficient particularly in column operations where contact time is inadequate to achieve equilibrium and may lead to low sorption efficacy of BC [11, 13, 20]. Therefore, sorption studies in columns should be performed to understand real application potential. Continuous fixed-bed column studies have been used very effectively for large-scale wastewater treatment operations [21, 22]. Other techniques involving destruction of TCE by ozonation, catalytic oxidation/reduction, and use of nanozero-valent metals are generally associated with the formation of daughter substances that may cause more negative impacts [23]. The objectives of this study were to evaluate the performance of BCs derived from soybean stover, pyrolyzed at different temperatures, for removing TCE from water using a fixedbed continuous flow column compared with AC. Thomas model was employed to evaluate the sorption and desorption properties of AC and the BCs.

#### 2. Materials and Methods

2.1. Biochar Production and Characterization. Soybean stover collected from a local agricultural field in Chungju city, Korea, was used as raw feedstock for producing the BCs. Ground feedstock was placed in a ceramic crucible with a lid and pyrolyzed in a muffle furnace (MF 21GS, Jeio Tech,

Seoul, Korea) increasing at 7°C min<sup>-1</sup> under limited oxygen conditions. Two different peak temperatures, that is, 300 and 700°C, which were selected as representatives of low and high pyrolysis temperatures, were adapted to carbonize each feedstock, and they were held for 3 h followed by cooling to room temperature inside the furnace. The BCs were denoted as BC300 and BC700 based on pyrolysis temperature. The commercially available AC was used in our study. BCs and ACs were ground and passed through 2 mm sieve prior to use. The modified proximate and ultimate analyses proposed by McLaughlin et al. [24] were employed to characterize the BCs and AC. The elemental composition including C, H, N, and O was determined by dry combustion using an elemental analyzer (EA1110, CE Instruments, Milan, Italy). These data were used to calculate molar ratios of H/C and O/C. Specific surface area was determined by the Brunauer-Emmett-Teller method of N<sub>2</sub> adsorption at 77 K (ASAP 2010 v 5.02 H, Micrometrics, Norcross, GA, USA) with 6 h degasification at 473 K prior to analysis.

2.2. Column Experiments. Fixed-bed continuous flow sorption experiments were conducted in a Plexiglass column with an inner diameter of 1.8 cm and length of 9.8 cm. 5 g of BCs and AC was placed in the columns. TCE was commercially purchased from Wako Pure Chemical Industries, Japan, with purity of 99.5%, and the TCE solution  $(100 \text{ mg L}^{-1})$ was pumped as influent through the column from top at 9 mL min<sup>-1</sup> with a peristaltic pump. Effluent samples were collected from the outlet of the column at different time intervals. The column was stopped when the effluent TCE concentration became nearly equal to the influent TCE concentration. After exhausting the column of TCE, the saturated sorbents were eluted with 25% methanol at relatively low flow rate (4 mL min<sup>-1</sup>) compared to the sorption experiment. Commercially available AC was also used for column sorption and desorption experiments to compare the efficiency of BCs in TCE removal.

2.3. Analysis of TCE. A high performance liquid chromatograph (SCL-10A, Shimadzu, Tokyo, Japan) equipped with an autosampler (SIL-10AD, Shimadzu) and UV-VIS detector (SPD-10A, Shimadzu) was used to analyze the aqueous TCE concentration. A reverse-phase Sunfire C18 column (Waters, Milford, MA, USA; 4.6 × 250 mm) was employed in a column oven (CTO-10AS, Shimadzu) heated at 40°C for the separation. The mobile phase was a mixture of 85:15 (vv<sup>-1</sup>) acetonitrile and water at a flow rate of 1.0 mL min. A 10  $\mu$ L sample aliquot was injected into the column, and absorbance was measured at 214 nm. The detection limit was 1.4 mg L<sup>-1</sup>, and the recovery of TCE was 98.13%.

2.4. Analysis of Column Data. Fixed-bed continuous flow column performance was evaluated from the breakthrough curve expressed as the ratio of effluent TCE concentration  $(C_e)$  to influent TCE concentration  $(C_i)$  as a function of flow time (t) for a given bed height. Effluent volume  $(V_{ef})$  was calculated from

$$V_{\rm ef} = Q t_{\rm total},\tag{1}$$

where Q is the volumetric flow rate (mL min<sup>-1</sup>) and  $t_{total}$  is the total flow time (min).

Total adsorbed quantity of TCE  $(q_{\rm total})$  was calculated from

$$q_{\text{total}} = \frac{QA}{1000} = \frac{Q}{1000} \int C_{\text{ad}} dt,$$
 (2)

where A is the area under the break through curve that can be obtained by integrating the adsorbed concentration  $(C_{\rm ad})$  versus t plot.

The total amount of TCE sent to column  $(M_{\text{total}})$  is calculated from

$$M_{\text{total}} = \frac{C_i Q t_{\text{total}}}{1000}.$$
 (3)

Column performance was evaluated from total removal of TCE, which was calculated by

Total removal (%) = 
$$\frac{q_{\text{total}}}{M_{\text{total}}} \times 100.$$
 (4)

Column capacity or equilibrium TCE sorption  $(q_{eq})$  at the end of total flow time was calculated from

$$q_{\rm eq} = \frac{q_{\rm total}}{X},\tag{5}$$

where X is the weight of sorbent (g) used in the column.

The process parameters were also determined for the design of larger-scale column studies. The time at which  $C_e$  increases from a steady state, known as breakthrough time  $(t_b)$ , and the column exhaustion time  $(t_e)$  when  $C_e$  becomes equal to  $C_i$  were recorded and used to calculate the overall sorption zone  $(\Delta t)$  as follows:

$$\Delta t = t_e - t_b. \tag{6}$$

Critical bed length also known as length of the mass transfer zone  $(Z_m)$  was calculated from the following equation:

$$Z_m = Z\left(1 - \frac{t_b}{t_e}\right),\tag{7}$$

where Z is bed height (cm).

The Thomas model was applied to the column experimental data to model the breakthrough behavior of TCE sorption onto the BCs. The linearized form of the Thomas model is given as follows:

$$\ln\left(\frac{C_i}{C_e} - 1\right) = \frac{k}{Q} \left(q_0 X - C_i V_{\text{ef}}\right),\tag{8}$$

where k is the rate constant (mL min<sup>-1</sup> mg<sup>-1</sup>) and  $q_0$  is the maximum solid-phase concentration (mg g<sup>-1</sup>). k and  $q_0$  can be determined from a plot of  $\ln((C_i/C_e) - 1)$  versus  $V_{ef}$  at a given flow rate.

The amount of TCE desorbed  $(q_{total(desorbed)})$  was calculated from the area under the elution curve (desorbed concentration  $(C_{de})$  versus t), and elution efficiency (E) was calculated from

$$E (\%) = \frac{q_{\text{total}(\text{desorbed})}}{q_{\text{total}(\text{sorbed})}} \times 100.$$
(9)

TABLE 1: Proximate and ultimate analyses of soybean stover derived biochars at 300°C (BC300) and 700°C (BC700) and activated carbon (AC).

	BC300	BC700 <sup>a</sup>	AC
Proximate analysis			
Yield (%)	37.03	21.59	_
Moisture (%)	4.50	0.42	7.75
Mobile matter (%)	46.34	14.66	11.70
Resident matter (%)	38.75	67.74	42.97
Ash (%)	10.41	17.18	37.57
Ultimate analysis			
C (%)	68.81	81.98	87.31
H (%)	4.29	1.27	0.62
O (%)	24.99	15.45	6.65
N (%)	1.88	1.30	0.19
Molar H/C	0.74	0.19	0.085
Molar O/C	0.27	0.14	0.057
Surface area $(m^2 g^{-1})$	5.61	420.3	758.9
Pore volume ( $cm^3 g^{-1}$ )	_	0.19	0.33

<sup>a</sup>Data from Ahmad et al. [11].

#### 3. Results and Discussion

3.1. Characteristics of the BCs and AC. Table 1 presents the proximate and ultimate analyses results of the soybean stover derived BCs at two different pyrolysis temperatures compared with AC. Temperature strongly influenced the BC yields and properties. The decrease in the BC yield at high pyrolysis temperature was mainly due to a greater loss of volatile matter. Ahmad et al. [10] reported a decrease in BC yield with increasing pyrolysis temperature. This was further supported by the greater loss in mobile matter at 700°C compared to that at 300°C. In contrast to mobile matter, the resident matter, which indicates the fixed or nonbiodegradable matter, increased with increasing pyrolysis temperature.

Changes in the elemental composition of the BCs were also observed. Total C contents of the BCs increased with pyrolysis temperature. In contrast, H, N, and O contents decreased in BC700 than those in BC300. Compared with the BCs, AC has the highest C content and lowest H, N, and O contents. BC700 exhibited high aromaticity and lower polarity than BC300 as indicated by the low molar H/C and O/C ratios, which could be related to high carbonization and low hydrophilicity at high temperature [1]. AC had much lower H/C and O/C ratios and higher C content compared with those of the BCs, indicating higher aromaticity, hydrophobicity, and lower polarity. The higher temperature derived biochar (BC700) had higher surface area  $(420.3 \text{ m}^2 \text{ g}^{-1})$  and pore volume  $(0.19 \text{ cm}^3 \text{ g}^{-1})$  than BC300  $(5.61 \text{ m}^2 \text{ g}^{-1} \text{ surface area and } 0 \text{ cm}^3 \text{ g}^{-1}$  pore volume). We conclude that the loss of mobile matter in the soybean stover during pyrolysis at higher temperature created more empty space in the residue than that at lower temperature, which increased the surface area and pore volume of the BCs [25]. However, AC had the largest surface area and pore volume



FIGURE 1: Breakthrough curves for adsorption of trichloroethylene (TCE) onto biochar derived from soybean stover at 300°C (BC300) and 700°C (BC700) and activated carbon (AC). Solid line represents data predicted by the Thomas model.

among the sorbents used in this study, which may have been due to the activation process during its production.

3.2. Column Studies. The breakthrough curves of TCE sorption onto BC300, BC700, and AC are shown in Figure 1. The bed heights of the columns (Z) were 5.8, 4.8, and 3.5 cm for BC300, BC700, and AC, respectively, corresponding to 5 g of sorbent in each column. The sorption process parameters calculated from the breakthrough curve are presented in Table 2. It was predicted that  $t_h$  for BC300 was 1.1 h whereas it was 27.0 h for BC700. This indicated that the column packed with BC300 began to saturate much earlier than that of BC700 and reached exhaustion after 10.5 h, whereas BC700 column had an exhaustion time  $(t_e)$  of 66.3 h. The maximum  $t_b$  value of 50.7 h was observed for AC with  $t_e$  of 95.5 h. The adsorbent bed height (Z) affects the efficiency of a column [26]. However, the greater Z for BC300 (5.8 cm) compared to that of BC700 (4.8 cm) and AC (3.5 cm) did not enhance the efficiency of BC300. Consequently, the critical bed length  $(Z_m)$  required to obtain the breakthrough time was lower for BC700 (3.5 cm) than that of BC300 (5.2 cm), indicating the shorter mass transfer zone of TCE in the column packed with BC700. This further indicates that the BC700 column had greater capacity for cycling TCE sorption because of the greater difference between Z and  $Z_m$  (1.9 cm) compared to that of the BC300 (0.6 cm) and was similar to AC.

Column performance was evaluated based on the total TCE removal percentage and total TCE uptake  $(q_{eq})$  by the sorbents. BC700 resulted in 68.4% removal of TCE compared to 30.0% removal by BC300 (Table 2). Similarly, the  $q_{eq}$  value was extremely high for BC700 (515.1 mg g<sup>-1</sup>) than that of BC300 (35.92 mg g<sup>-1</sup>). These sorption process parameters for the BC packed columns clearly indicate the high performance efficiency of BC700 compared to that of BC300. In contrast,

TABLE 2: Sorption process parameters for trichloroethylene (TCE) adsorption onto biochar derived from soybean stover at  $300^{\circ}$ C (BC300) and  $700^{\circ}$ C (BC700) and activated carbon (AC).

	<i>t</i> <sub>b</sub> (h)	t <sub>e</sub> (h)	$\Delta t$ (h)	Z (cm)	$Z_m$ (cm)	Removal (%)	$q_{\rm eq} \ ({ m mgg}^{-1})$
BC300	1.1	10.5	9.4	5.8	5.2	30.0	35.9
BC700	27.0	66.3	39.3	4.8	2.9	68.4	515.1
AC	50.7	95.5	44.8	3.5	1.6	72.1	774.0

TABLE 3: Thomas model parameters for trichloroethylene (TCE) sorption onto biochars derived from soybean stover pyrolyzed at  $300^{\circ}$ C (BC300) and  $700^{\circ}$ C (BC700) and activated carbon (AC).

	Regression mode	$k \pmod{(\mathrm{mLmin}^{-1}\mathrm{mg}^{-1})}$	$\begin{array}{c} q_0 \\ (\mathrm{mgg}^{-1}) \end{array}$	$R^2$
BC300	Linear	0.091	45.81	0.972
	Nonlinear	0.056	78.02	0.965
BC700	Linear	0.018	682.8	0.908
	Nonlinear	0.011	961.4	0.977
AC	Linear	0.009	1212	0.863
	Nonlinear	0.009	1436	0.987

the column packed with AC outperformed the BCs with a removal efficiency of 72.1% and 774.0 mg g<sup>-1</sup> uptake of TCE. This could be explained by the presence of a more noncarbonized fraction in BCs than in AC, which could lower the sorption of TCE onto the relatively less carbonized fraction in the BCs [27]. Moreover, higher surface area and pore volume of BC700 and AC was one of the reasons for the higher sorption capacity. However, higher hydrophobicity of BC700, indicated by the lower molar ratio of O/C and higher C content, also resulted in higher sorption capability to the relatively hydrophobic TCE by BC700 [11].

3.3. Determination of Sorption Rate Constants. The behavior of a sorption column was modeled using the simpler and more tractable Thomas model. This model is frequently used because of its simplicity, the lack of numerical simulations, and immediate practical benefits [22]. The rate constant (k), maximum solid-phase concentration  $(q_0)$ , and the correlation coefficient  $(R^2)$  values for BC300, BC700, and AC are presented in Table 3, while the predicted nonlinear regressions of the Thomas model are shown in Figure 1. The  $R^2$  values calculated from the nonlinear regressions of the Thomas model were 0.965, 0.977, and 0.987 for BC300, BC700, and AC, respectively, indicating that the experimental data was well fitted to the Thomas model. The k value was higher for BC300  $(0.091 \,\mathrm{mL\,min^{-1}\,mg^{-1}})$ for linear and  $0.056 \,\mathrm{mL\,min^{-1}\,mg^{-1}}$  for nonlinear regression) than for BC700  $(0.018 \text{ mLmin}^{-1} \text{ mg}^{-1}$  for linear and  $0.011\,mL\,min^{-1}\,mg^{-1}$  for nonlinear regression) and AC (0.009 mL min<sup>-1</sup> mg<sup>-1</sup> for both linear and nonlinear regression), indicating that BC300 achieved the maximum sorption of TCE within a short time. These predictions are in agreement with the relatively low  $t_b$  value in BC300 than those



FIGURE 2: Elution curves for trichloroethylene (TCE) desorption from biochar derived from soybean stover pyrolyzed at 300°C (BC300) and 700°C (BC700) and activated carbon (AC).

in BC700 and AC (Table 2). As expected, the  $q_0$  value was higher for AC (1212 mg g<sup>-1</sup> for linear and 1436 mg g<sup>-1</sup> for nonlinear regression) and BC700 (682.8 mg g<sup>-1</sup> for linear and 961.4 mg g<sup>-1</sup> for nonlinear regression) compared to BC300 (45.81 mg g<sup>-1</sup> for linear and 78.02 mg g<sup>-1</sup> for nonlinear regression), which also appeared in the observed experimental  $q_{eq}$ values given in Table 2. The fitness of the Thomas model to the experimental sorption data presumed that the TCE sorption onto BCs was controlled by interphase mass transfer, but axial dispersion may be an important factor determining the rate limiting step [22].

The greater efficiency of the column packed with AC and BC700 for removing TCE from water could be related to its specific properties such as high surface area (758.9 m<sup>2</sup> g<sup>-1</sup> for AC and 420.3 m<sup>2</sup> g<sup>-1</sup> for BC700) and low molar H/C (0.085 and 0.19) and O/C (0.057 and 0.14) compared to those of BC300 (Table 1). High aromaticity (as indicated by low molar H/C) and low polarity (as indicated by low molar O/C) of BC700 control TCE sorption from water [11].

3.4. TCE Desorption. After the columns were exhausted from TCE, they were eluted with 25% methanol to desorb the TCE from column to evaluate the recovery rate of each sorbent. Figure 2 shows the elution curves for TCE desorption from saturated BC300, BC700, and AC at the eluent flow rate of  $4 \text{ mL min}^{-1}$ . A relatively low flow rate was applied for the desorption process compared to sorption to impart more contact time of the eluent with TCE and also to obtain maximum desorption with less eluent consumption. A sharp increase in TCE desorption was observed in the beginning for all sorbents, which then gradually decreased with time. A similar trend was reported in several other desorption studies [21, 28]. Elution efficiency was 57.9% for BC300, 69.2% for BC700, and 82.8% for AC. The relatively low elution efficiency for the BCs compared to AC may presumably be due to the strong

binding of TCE to the BCs [11]. As mentioned above, low temperature pyrolyzed BCs contain a larger noncarbonized fraction than high temperature pyrolyzed BCs and AC, which led to the multiple sorption mechanisms. Chen et al. [27] found that sorption of organic contaminants to biochar consists of two parts: partition to noncarbonized fractions and adsorption to the carbonized fraction of the biochar. As a result, sorption of TCE on AC and BC700 may have been predominantly contributed by surface adsorption as they had much higher surface areas and lower noncarbonized fractions. Therefore, TCE was much easier to be contacted by eluent and easier to remove. Sorption of TCE on BC300 may have also significantly contributed by partitioning to the noncarbonized fractions, which was not easy to contact with eluent and harder to remove.

Overall, AC showed best efficiency for removing TCE from water in column experiments. Although sorption and desorption capabilities of BC700 were a little lower than AC, it is still a good alternative for AC to remove organic contaminants such as TCE from water due to its cost-effectiveness.

## 4. Conclusions

Biochar derived from soybean stover pyrolyzed at 700°C and AC outperformed biochar pyrolyzed at 300°C for removing TCE from water in continuous fixed-bed columns. High surface area, low polarity, and high aromaticity were involved in the greater efficiency of AC and BC700 than that of BC300. The sorption capacities of AC and BC700 were 20.5 and 13.3 times higher than that of BC300. The Thomas model well described the column sorption data, indicating that the sorption of TCE onto BCs and AC is controlled by interphase mass transfer. A comparison of the efficiency of the BCs for removing TCE from water suggests that BC700 is costeffective comparable to AC. The lower desorption rate of TCE from BC300 than BC700 and AC may be attributed to the strong binding/partition of TCE to the noncarbonized part of BC. Future studies, such as BC surface activation and modification, are needed to further improve the sorption capacity of BCs.

### **Conflict of Interests**

The authors declare that there is no conflict of interests regarding the publication of this paper.

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