



Trichloroethylene adsorption by pine needle biochars produced at various pyrolysis temperatures



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HIGHLIGHTS

- TCE removal by pine needle biochars produced at various temperatures was investigated.
- Pyrolysis temperature showed a pronounced effect on biochar properties.
- High surface area, micro-porosity and carbonized extent increased TCE removal.
- Prevailing TCE sorption mechanism on pine needle derived biochar is pore-filling.

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ABSTRACT

In this study, pine needles were converted to biochar (BC) at different pyrolysis temperatures of 300, 500, and 700 °C to sorb trichloroethylene (TCE), and the changes in BC properties with each temperature were evaluated. Pyrolysis temperature showed a pronounced effect on BC properties. Decreases in molar H/C and O/C ratios resulted from removing O- and H-containing functional groups with increasing temperature, and produced high aromaticity and low polarity BCs. BCs produced at higher temperature showed greater TCE removal efficiency from water due to their high surface area, micro-porosity, and carbonized extent. The performance of various BCs for TCE removal was assessed by the Freundlich, Langmuir, Temkin, and Dubinin–Radushkevich adsorption models, among which the Temkin and Dubinin–Radushkevich models best described TCE adsorption onto various BCs, indicating prevailing sorption mechanism as pore-filling.

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1. Introduction

Trichloroethylene (TCE) has been widely used as a chlorinated solvent in industry and contaminates the surface/ground water via direct discharge/leaching from disposal operations (Chiu et al., 2006). TCE has been identified as a priority environmental pollutant by the US Environmental Protection Agency (Klasson et al., 2009). In particular, groundwater contamination by TCE commonly occurs in many industrial areas worldwide. In Korea, a severe level of TCE was detected in groundwater at an industrial complex in Wonju, Gangwon province (Baek and Lee, 2011), showing >50 times of TCE level (Jo et al., 2010) as compared to the guidelines of Korean Ministry of Environment (the maximum

permissible level of TCE is 0.03 mg L⁻¹ for residential and 0.06 mg L⁻¹ for industrial areas).

Adsorption is one of the most popular and widely used technologies for groundwater depuration (Ahmad et al., 2012c). It is a simple technology showing a robust operating configuration and high reliability. Several adsorbents have been applied to remove TCE from groundwater, such as activated carbon (Ahmad et al., 2012c; Karanfil and Dastgheib, 2004), multi-walled carbon nanotubes (Naghizadeh et al., 2011), silica gel (Farrell et al., 1999) and organic mulch (Wei et al., 2010). The use of activated carbon or carbonaceous adsorbents is well-operated to purify the contaminated groundwater, but it is not a realistic option for large areas due to cost concerns.

Char, the solid material released from a carbonaceous material, is emerging as an alternative to activated carbon with cost-effective and environmental advantages. Char commonly appears under

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uncontrolled natural conditions through partial or complete carbonization of biomass such as wood, manure, or leaves (Chun et al., 2004). The term biochar (BC) is used in soil science and is related to carbon management in soils. Basically, it means a black carbon derived from pyrolysis of biomass and closely resembles activated carbon having a structured carbon matrix and a medium-to-high surface area. BC has a wide range of chemical composition and surface properties depending on the biomass type and pyrolysis temperature. BC is a multi-functional material that can be employed for carbon sequestration, metal immobilization, and fertilization to soils (Ahmad et al., 2012b; Awad et al., 2012; Uchimiya et al., 2010). Because of its high efficiency and capacity to adsorb organic contaminants, it has been spotlighted as an excellent adsorbent (Cao and Harris, 2010). In comparison to the conventional activated carbon, BC may be economically preferable with less energy requirement and no pre- or post-activation processes necessary during manufacturing. It is also environmentally beneficial by converting/recycling of organic waste materials via pyrolysis. However, a substantial understanding of BC is required to ensure its efficiency to remove typical contaminants such as TCE in groundwater. In our earlier study of Ahmad et al. (2012a), the BCs derived from soybean stover and peanut shells were used to depurate TCE contaminated water. According to the results of excellent performance of BCs derived from soybean stover and peanut shells as a TCE adsorbent, the necessity of discovering possible BC's materials or setting limit of proper pyrolysis temperatures is being emphasized to maximize its efficacy of TCE adsorption through the approach with various isotherm models.

Selecting the biomass as a source of BC is very important because it may determine the quality and economic feasibility. According to the Korean Forest Service in 2010, 6,368,843 ha of forest occupy 63.7% of the total Korea territory, and 2,580,629 ha (40.5%) of the total forest is covered by conifers. Among these, 35% of all coniferous forests are infected with pine wilt disease (Kim et al., 2010; Kwon et al., 2011). The pine wilt disease vector attacks the conifers to death and produces a large amount of waste. Pine tree waste can be a suitable source material for BC production and it promises not only to mitigate the waste disposal problem but also provide a low cost and efficient adsorbent for removing organic contaminants. Therefore, this study evaluated the effectiveness of pine tree BC produced at different pyrolysis temperatures on TCE adsorption from groundwater.

2. Experimental

2.1. Biochar production

Fallen pine needles were collected from the green space at Kangwon National University in Cheonchun, Gangwon province, Korea. The material was oven-dried overnight at 60 °C to reach <10% moisture content, and then crushed and sieved to obtain <1.0 mm particle size. The powdered biomass (BM) was pyrolyzed at three different temperatures of 300, 500 and 700 °C using a muffle furnace (MF 21GS, Jeio Tech, Seoul, Korea). Heating rate was adjusted to 7 °C min⁻¹ and then the targeted (or peak) temperature was maintained for 3 h for complete carbonization under limited oxygen condition. The BC was cooled to room temperature and its yield was determined by proportion of BC to BM.

2.2. BM and BC characterization

2.2.1. Proximate analysis

The moisture contents of BM and BC samples were determined using a drying-oven at 105 °C to constant weight. Mobile matter, also known as volatile matter, was measured by heating BM or

BC in a covered porcelain crucible at 450 °C for 1 h. Ash content was also determined by heating the samples at 750 °C for 1 h using an open-top porcelain crucible. Resident matter as an indicator of fixed matter was determined by the difference in moisture, ash, and mobile matter. All measurements were performed in at least duplicates.

2.2.2. Ultimate analysis

Elemental composition, including C, N, H, S, and O, for BM and BCs was determined by combusting the material at 1100 °C using an elemental analyzer (Flash EA 1112 series, CE Instruments, Wigan, UK). Molar ratios of H/C and O/C were calculated. The surface structure of each sample, which was degassed at 473 K for 6 h, was characterized by N₂ adsorption at 77 K using an adsorption apparatus (ASAP 2010 v 5.02 H; Micromeritics, Norcross, GA, USA). The specific surface area, pore volume, and pore diameter were determined by the Brunauer–Emmett–Teller and the Barret–Joyner–Halender methods.

2.2.3. Morphological and spectral analyses

Surface physical morphology and microstructure of BM and BCs were determined by field emission scanning electron microscopy equipped with an energy dispersive spectroscope (SU8000, Hitachi, Tokyo, Japan). The spectral characteristics were measured by Fourier-transform infrared spectroscopy (FT-IR) (Excalibur 3000MX; Bio-Rad, Hercules, CA, USA). A wavelength range of 600–4000 cm⁻¹ was used to obtain the FT-IR spectra with 32 successive scans at a resolution of 4 cm⁻¹. The spectra were baseline-corrected and normalized for difference spectroscopy using Essential FT-IR software (v 2.00.045).

2.2.4. Acidic and basic surface functional groups distribution

The oxygenated acidic and basic surface functional groups of BM and BCs were determined using the Boehm's titration method (Goertzen et al., 2010). Briefly, 0.5 g of sample was suspended in 50 mL of 0.05 M HCl, NaHCO₃, Na₂CO₃, or NaOH and equilibrated for 24 h. After equilibration, the sample was filtered and neutralized with excess 0.05 M HCl. The sample was then back titrated with 0.05 M NaOH using an auto titrator (Mettler Toledo G20, Solon, OH, USA). The concentrations of acidic and basic functional groups were determined by assuming that NaHCO₃ neutralizes strong acids (primarily carboxylic), Na₂CO₃ neutralizes moderate acids (primarily low pKa phenols and hydrolysis products of lactones), and NaOH neutralizes weak acids (primarily high pKa phenols). HCl neutralizes all basic ketonic, pyronic, and chromenic groups (Chun et al., 2004; Goertzen et al., 2010).

2.3. Adsorption experiments

TCE (100 mg L⁻¹; 99% purity; Wako Pure Chemical Industries, Osaka, Japan) stock solution was prepared with ultrapure water (18.2 MΩ cm⁻¹ resistivity at 25 °C; Sartorius Stedium Biotech, Glade Bach, Germany). A series of TCE working solutions ranging from 5.0–50 mg L⁻¹ was prepared and then buffered with 1 mM phosphate buffer at pH 7.0 (0.5 mM Na₂HPO₄·H₂O and 0.5 mM NaH₂PO₄) for batch type adsorption experiments. The BCs produced at three different temperatures of 300, 500, and 700 °C (i.e., BC300, BC500 and BC700, respectively) and BM were applied at a rate of 0.3 g L⁻¹ as adsorbents. The adsorption experiments were carried out in Teflon-lined screw-capped glass-vials with no headspace to minimize the volatile loss of TCE. The added adsorbents were equilibrated at 50 rpm for 48 h on a horizontal shaker at 25 °C. After equilibrium, an aliquot from each vial was filtered through a 0.45-μm syringe filter and the TCE concentration was determined by high performance liquid chromatography system (SCL-10A, Shimadzu, Tokyo, Japan) equipped with an auto-sampler

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