



Enhanced sulfamethazine removal by steam-activated invasive plant-derived biochar



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HIGHLIGHTS

- Steam activation of biochar enhanced sulfamethazine removal from aqueous media.
- Biochar properties varied drastically by the activation with steam.
- Solution pH and biochar properties had strong effect on sulfamethazine sorption.
- The steam activated biochar produced at 700 °C had the highest sorption capacity.

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ABSTRACT

Recent investigations have shown frequent detection of pharmaceuticals in soils and waters posing potential risks to human and ecological health. Here, we report the enhanced removal of sulfamethazine (SMT) from water by physically activated biochar. Specifically, we investigated the effects of steam-activated biochars synthesized from an invasive plant (*Sicyos angulatus* L.) on the sorption of SMT in water. The properties and sorption capacities of steam-activated biochars were compared with those of conventional non-activated slow pyrolyzed biochars. Sorption exhibited pronounced pH dependence, which was consistent with SMT speciation and biochar charge properties. A linear relationship was observed between sorption parameters and biochar properties such as molar elemental ratios, surface area, and pore volumes. The isotherms data were well described by the Freundlich and Temkin models suggesting favorable chemisorption processes and electrostatic interactions between SMT and biochar. The steam-activated biochar produced at 700 °C showed the highest sorption capacity (37.7 mg g⁻¹) at pH 3, with a 55% increase in sorption capacity compared to that of non-activated biochar produced at the same temperature. Therefore, steam activation could potentially enhance the sorption capacities of biochars compared to conventional pyrolysis.

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1. Introduction

Water contamination by antibiotics is becoming a worldwide environmental issue [1,2]. Sulfamethazine (SMT), which belongs to the sulfonamide group of antibiotics, is commonly used in veterinary medicine to control diseases and in livestock feeds for cattle

and swine [3]. Previous studies have shown that, SMT does not sorb to soil well and hence, can be detected at high concentrations in water [4]. Remediation of antibiotic-contaminated water has been achieved by sorption processes using various sorbents, including natural sorbents, clays, chitosan derivative, activated carbon, and more [5–8]. It has been shown that sorption of SMT by clays decreases with increasing pH of solution and increases with increasing ionic strength and surface charge density. Various SMT sorption mechanisms including cation exchange, hydrophobic partitioning, and surface complexation reactions (H-bonding and other polar interactions) between the functionalities (amino, carboxyl,

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and phenol) of the SMT molecules have been purposed [9–10]. Even though materials such as activated carbon are suitable for removing contaminants from water, they may not be economically feasible [11] due to the high energy requirements of their production.

Low-cost biochar, a solid material obtained from the carbonization of biomass in an oxygen-limited environment [12], has recently received major attention as a very effective environmental sorbent of many organic and inorganic contaminants in soil and water [11,13–16]. Biochar can be produced from various waste materials as a feedstock, which is advantageous as a waste management option [17]. Previous studies have focused on producing biochar from invasive plant species, thereby providing a useful way of eliminating them from the environment [18–20].

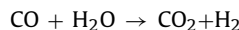
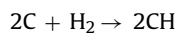
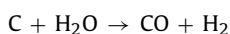
However, little work has been done to examine the mechanisms and effectiveness of SMT removal by biochars [6,9,21]. Previously, biochars produced by conventional slow pyrolysis showed potential as adsorbents of organic contaminants, although the reported sorption capacities were much lower than those of other adsorbents such as activated carbon (AC) for practical applications [22]. Hence, the production of biochar can be a good alternative to AC for environmental remediation, even though more studies are needed to enhance the removal efficiencies of biochars to match the sorption capacities with AC [23]. Thus, in this study, we developed a simple method for increasing the (maximum) sorption capacities of biochars by steam activation after slow pyrolysis. Ippolito et al. [24] recently demonstrated the potential of steam-activated pecan shell biochar to sorb excess Cu (inorganic metal ions) from waste waters. Despite this, the significance of steam-activated biochar in the sorption of organic compounds has not been studied in detail and requires continued research.

Here, we converted the biomass of an invasive plant species (Burcucumber; *Sicyos angulatus* L.) into biochar in order to simultaneously address two environmental issues i.e., (a) economically feasible removal of invasive plants will generate a valuable source of biomass for biochar production and (b) production of innovative and environmentally friendly material in pollutant removal. The physicochemical properties of steam-activated biochars were compared with those of conventional slow pyrolysis biochars. The potential applicability of steam-activated biochars for the removal of veterinary antibiotics was evaluated by conducting a series of batch experiments. The specific objectives of this study were (1) to produce invasive plant-derived steam-activated biochars, (2) to evaluate the effectiveness of steam-activated biochars on SMT sorption from water, and (3) to determine the factors governing SMT removal by biochars.

2. Experimental

2.1. Preparation of biochars and steam activation

Burcucumber plants were collected from Chuncheon city in Gangwon province, Korea. The dried plants were crushed and ground to <1.0 mm particle size. Portions of this biomass were pyrolyzed at two temperatures (300 and 700 °C) in a pyrolyzer (N11/H Nabertherm, Germany) under a limited supply of air. Slow pyrolysis was achieved at a rate of 7 °C min⁻¹. A holding time of 2 h was applied for each peak temperature for complete carbonization of biomass. Then, the pyrolyzed samples were steam-activated at a flow rate of 5 mL min⁻¹ for 45 min, under the peak temperature (i.e., 300 or 700 °C) [25,26]. Steam may change the properties of biochar by removing the trapped products of incomplete combustion during pyrolysis [27]. The typical reactions involved in the steam activation [28] are as below:



Steam oxidizes the C surface generating H₂ and CO₂ which may activate the surface of biochar. In other words, the rate of gasification is inhibited due to these water-gas shift reactions. Burcucumber biochars (BBC) produced at 300 and 700 °C without steam activation were denoted as BBC-300 and BBC-700, whereas biochars produced by steam activation were designated as BBC-300S and BBC-700S.

2.2. Characterization of biomass and biochars

Proximate analysis, including moisture, mobile matter, ash, and residual matter contents, were determined according to the modified thermal analysis methods of McLaughlin et al. [29]. Briefly, moisture content was calculated based on weight loss after heating biochars at 105 °C for 24 h to a constant weight. Mobile matter content was determined by calculating weight loss after heating in a covered crucible at 450 °C for 30 min, whereas ash content was determined as the residue remaining after heating at 700 °C in an open-top crucible. The pH levels of produced biochars were estimated in a suspension of 1:5 BBC/de-ionized water using a digital pH meter (Orion, Thermo Electron Corp., Waltham, USA). Elemental (C, H, N, S, O) analyses were conducted by dry combustion using an elemental analyzer (EA1110, CE Instruments, Milan, Italy).

Structural analysis was performed using Fourier transform infrared spectroscopy (FTIR) (Bio-Rad Excalibur 3000MX spectrophotometer, Hercules, CA, USA). The spectra were obtained at 4 cm⁻¹ resolution by measuring the absorbance from 400 to 4000 cm⁻¹ using a combined 190 scans. Surface areas of sorbents were measured by nitrogen adsorption isotherms at 77 K using a gas sorption analyzer (NOVA-1200; Quantachrome Corp., Boynton Beach, FL, USA). Specific surface areas were determined from adsorption isotherms using the Brunauer–Emmett–Teller (BET) equation. The Barret–Joyner–Halender (BJH) method was used to determine pore volume and pore diameter from the N₂ adsorption data.

2.3. Batch sorption studies

Ultrapure water with a resistivity of 18.2 MΩ was used for all procedures. SMT was obtained from Sigma–Aldrich Chemical Co. (99% purity). Acid dissociation constants and physical properties of SMT are given in Table S1 of the supplementary data. Stock solution (100 mg L⁻¹) was prepared in 1% methanol and was used to obtain the starting SMT concentrations in batch sorption studies. Batch sorption isotherm studies were carried out in the concentration range of 2.5 ~ 50 mg L⁻¹ at pH 3, 5, 7 and 9 (10 mM ammonium phosphate for pH 3, 7 and 9, and 10 mM ammonium acetate for pH 5) [10]. A sorbent dose of 1 g L⁻¹ was used for all sorption experiments at an ionic strength of 0.1 M (adjusted by ammonium chloride). During the experiments, the pH values were checked and readjusted using conjugated acids and bases if required. The mixtures were shaken at 100 rpm in an incubator shaker (Jeio Tech, SI-600R) for 72 h at 25 °C.

After equilibrium, an aliquot from each vial was filtered through a 0.45 μm syringe filter, after which the SMT concentration was determined by high performance liquid chromatography (HPLC; SCL-10A, Shimadzu, Japan) equipped with an auto-sampler (SIL-10AD, Shimadzu, Japan) and UV–vis detector (SPD-10A, Shimadzu, Japan). A reverse-phase Sunfire C18 column (4.6 mm by 250 mm; Waters, USA) was employed in a column oven (CTO-10AS, Shimadzu, Japan). Mobile phase A was composed of HPLC grade water

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