



Tuning the adsorptive properties of drinking water treatment residue via oxygen-limited heat treatment for environmental recycle



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HIGHLIGHTS

- Oxygen-limited heat treatment significantly increased SSA and TPV of DWTR.
- The treatment increased the amorphous Al/Fe contents in DWTR at 200–400 °C.
- The treatment induced organic matter in DWTR with higher aromaticity.
- The lability of heavy metals in DWTR tended to decrease after treatment.
- The Hg adsorption capability of DWTR treated at 200–400 °C significantly enhanced.

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ABSTRACT

As a by-product generated increasingly during potable water production, drinking water treatment residue (DWTR) recycling for environmental remediation can lead to a win-win situation. In this study, oxygen-limited heat treatment was applied to DWTR, attempting to tune the properties of DWTR for better recycling. The results showed that after the treatment, N₂ sorption capacity of DWTR was enhanced significantly: the specific surface area (SSA) and total pore volume (TPV) increased from 72.7 to 148–184 m² g⁻¹ and from 0.0746 to 0.189–0.201 cm³ g⁻¹, respectively. The treatment also relatively increased the amorphous Al/Fe contents in DWTR although Al/Fe aging and transformation from hydroxides to oxides with high stability were observed. The retained organic matter (OM) was dominant in humin, with higher aromaticity and lower aliphaticity as the treatment temperature increased. Further analysis suggested that aromaticity increase induced higher SSA, the combined effect of OM aromaticity increase, OM loss (e.g. C in COO⁻), and metal oxides formation resulted in larger TPV, OM inhibited Al/Fe aging, while Al/Fe oxides formation in turn enhanced OM aromaticity. Additionally, the maximum Hg adsorption capacity of DWTR treated at 200–400 °C (estimated by Langmuir model) increased from 53.5 to 69.9–147 mg g⁻¹, and the lability of heavy metals (e.g. Cu) in DWTR tended to decrease after treatment. The overall results demonstrated that oxygen-limited heat treatment sequestered C, making DWTR be a more reliable adsorbent.

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1. Introduction

Use of chemical immobilization to control pollutions has become a common method for environmental remediation [1]. Reasonably, chemicals that feature low-cost, environmentally friendly, and highly effective are with a promising developmental potential. In recent years, drinking water treatment residue (DWTR), an inevitable by-product generated increasingly during potable water production, has attracted worldwide interests to

be recycled for the chemical treatment [2]. Drinking water treatment residue can be classified into coagulant, groundwater or softening, natural and manganese residuals, while coagulant residuals constitute the majority of DWTR and are also focused mostly [3]. In conventional coagulation and filtration treatment process, raw water are purified by Al and Fe coagulants and other agents, and then producing the DWTR. According to the toxicity characteristic leaching procedure assessment method proposed by the US Environmental Protection Agency, the DWTR could be considered non-hazardous [4]. Drinking water treatment residue has been demonstrated to be an effective adsorbent of many contaminants, including P [5], hydrogen sulfide [6], perchloric acid [7], As [8], B

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[9], Cr [10], Cu [11], Hg [12], Pb [10], Se [13], Zn [14], antibiotics [15], chlorpyrifos [16], and glyphosate [17]. Moreover, DWTR has been recycled for environmental remediation [3,18,19]; in particular, it can be used as an amendment to reduce the P loss from P rich soils [20], as the main substrate in constructed wetlands to remove excessive P from wastewater [21], and as an inactivating agent to ameliorate lake internal P loading for eutrophication control [22]. The successful application of DWTR will lead to a win-win situation for environmental remediation.

To facilitate the widespread reuse, efforts were also attempted to tune the adsorptive properties of DWTR for better recycling. In general, the components of DWTR are closely related to raw water and applied water treatment processes. Because the raw water used for drinking water treatment plant has high quality, DWTR is mainly composted by inorganic substances, typically containing high concentrations of amorphous Al and Fe [3]. The Al and Fe, mainly from coagulants unitization, are the primary cause of the high adsorption capability of DWTR for many contaminants [2]. On the contrary, the limited organic substances, have often considered not only occupy the adsorption sites of Al and Fe in DWTR, but also form a diffusion-limiting layer on the surface of DWTR, restricting contaminants accessed by Al and Fe [23]. Thus, DWTR adsorptive properties tuning was commonly carried out by removing organic matter, e.g. heat treatment under atmospheric condition [23,24]. However, this modification may lead to the loss of some special adsorption capabilities of DWTR. The contained organic matters have been demonstrated to contribute to the high adsorption capability of DWTR for hydrophobic organic compounds (HOCs) [16]. To resolve the above dilemmas on the modification of DWTR, alternative strategy needed to be taken though maintaining the adsorption capability endowed by Al and Fe, as well as organic matters.

Biochar, a C-rich, fine-grained, porous substance, has high adsorption capability for many organic and inorganic contaminants [25] and is being recognized as a promising tool for long-term C sequestration, mitigating global warming [26]. Application of biochar can be a strategy to manage soil processes and functions, bringing both agricultural and environment benefits [27]. The production of biochar is mainly through pyrolysis/carbonization of plant- and animal-based biomass under a limited oxygen supply [28]. The thermochemical processes include slow pyrolysis, fast pyrolysis, flash carbonization, and gasification, while slow pyrolysis has the advantage that can retain up to 50% of the feedstock carbon in stable biochar [27,29]. The organic matter in DWTR is mainly from raw water formed by the breakdown of vegetable matter and resynthesis [3]. Here, we had a hypothesis that oxygen-limited heat treatment may make DWTR be a more reliable adsorbent through sequestering C, producing biochar-like substances, and increasing adsorption sites related to Al and Fe.

Given that hypothesis, oxygen-limited heat treatment was adopted to treat DWTR in this study. The contents for the properties assessment are specified below: (1) the N₂ gas sorption/desorption characteristics; (2) the chemical states and forms of Al and Fe; (3) the organic matter structures; (4) the adsorption capability assessment and (5) the lability of heavy metals. Based on the results, mechanisms leading to the changes of DWTR properties, as well as environmental implications for DWTR management and application were further discussed.

2. Materials and methods

2.1. Sample collection

Dewatered DWTR was collected from Beijing City No. 9 Waterworks in China in Apr. 2012, where reservoir and groundwater

were used as raw water sources. In this facility, poly Al chloride and polymerization Fe chloride were used as coagulants and activated carbon adsorption were used for advanced treatment. The moisture content of fresh dewatered DWTR was approximately 78%. The fresh dewatered DWTR was air-dried (within 2 months), and then were ground and sieved to a diameter less than 0.25 mm, because the air-dried samples were bulk materials. The air-dried samples, with moisture contents less than 2.5%, were used herein in Jul. 2014.

2.2. Oxygen-limited heat treatment

Oxygen-limited heat treatment used herein was mainly based on the methods described in Chen et al. [30] and Li et al. [31]. The DWTR (20 g) was placed in a quartz flask, covered with a quartz fitting lid, and heated under a N₂ atmosphere with a heating rate of 15 °C min⁻¹ to reach five settled gradient temperatures (200, 300, 400, 500, and 600 °C) for 4 h. The produced solids were washed by deionized water until the supernatant being colorless, and then were oven-dried at 50 °C. The cleaning procedure was applied to remove the dissolved organic matter from the DWTR [32] to avoid potential secondary pollution during application of the solids for environmental remediation, especially for water remediation. For simplicity, DWTR after oxygen-limited heat treatment under 200, 300, 400, 500, and 600 °C was designated as DWTR-200, -300, -400, -500, and -600, respectively.

2.3. DWTR characterization

The N₂ gas sorption/desorption by solids were first determined using the specific surface area (SSA) and porosity analyzer (NOVA4200e, Quantachrome, USA). The DWTR were also analyzed using Fourier transform infrared spectroscopy (FTIR, Nicolet iS5, Thermo Fisher, USA) to determine the main structures, using X-ray photoelectron spectroscopy (XPS, ESCALAB 250, Thermo Fisher Scientific, USA) to determine the surface elemental compositions and the chemical state of Al, C, Fe, and O, as well as using elemental analyzer (EA3000, Euro Vector, Italy) to determine the total organic C (TOC) contents.

For the studies on humic fractions in DWTR, a procedure used by Zbytniewski and Buszewski [33] for sewage sludge compost analysis with slight modification was applied. DWTR were extracted with a mixture of 0.1 M Na₂P₂O₇ + 0.1 M NaOH for 24 h for seven times, and the supernatant was separated by centrifugation (5000 rpm, 30 min). The extractions were combined and the contained TOC was determined by TOC analyzer (Torch, Teledyne Tekmar, USA) (corresponding to fulvic and humic acids--FA and HA, respectively). Part of the supernatant was acidified to pH < 2 with 6 M HCl, heated to 80 °C for 2 h, and allowed standing at room temperature overnight. Soluble FA was then separated from the precipitated HA by centrifugation, and the contained TOC was determined. The contents of HA and humin (HM) were calculated as (HA + FA) - FA = HA and HM = (TOC in DWTR) - (HA + FA), respectively.

The precipitated HA, the residuals after FA and HA extraction, and the DWTR before and after treatment were demineralized with 1 M HCl and 10% (v/v) HF at 1:5 solid/liquid ratio [34] for seven times. The demineralized DWTR, namely HA, HM, and total organic matter, were freeze-dried and then analyzed using FTIR, and the contents of C and H were determined for HM (because it was the dominate fraction in DWTR after treatment). In addition, the pH of DWTR was determined in the supernatant of a 1:2.5 (solid:suspension, g mL⁻¹) suspension by using CO₂-free water as matrix (pH-10, Sartorius, Germany). The surface charge properties were evaluated by zeta potential measurements, which were conducted using a Zetasizer (ZS MPT-2, Malvern, UK). Samples were

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