Preparation and Heavy Metal Adsorption Characteristics of Iron-Modified Hydrochar Derived from Waste Cigarette Filters

Jimin Myeong, Yunju Moon, Minyong Lee and Eun Jung Kim*

School of Architecture, Civil and Environmental Engineering, Mokpo National University,
1666 Yongsan-ro, Cheongye-myeon, Muan-gun, Jeollanam-do 58554, Republic of Korea
**Corresponding Author E-mail: ejkim@mnu.ac.kr*

ABSTRACT

Waste cigarette filters are among the most abundant and recalcitrant wastes worldwide, continuously releasing microplastics and hazardous substances that pose long-term threats to soil and aquatic environments. Converting such persistent waste into precursors for carbon materials offers an approach that can simultaneously reduce pollution and recover value through resource circulation. In this study, hydrochar and iron-modified hydrochar were prepared from waste cigarette filters via hydrothermal carbonization, with FeCl3 added at concentrations of 0.01, 0.05, and 0.1 M for iron modification. The resulting materials were characterized and evaluated for the adsorption of arsenic (As), chromium (Cr), copper (Cu), and lead (Pb) from single-component aqueous solutions. XRD analysis showed that hematite (α-Fe2O3) formed at all Fe concentrations, with markedly stronger reflections at 0.05 and 0.1 M than at 0.01 M. At an initial concentration of 25 mg/L, the hydrochar removed approximately 88% of Cu and 30% of Pb, whereas As and Cr were hardly removed. Cu and Pb removal was nearly independent of Fe loading, indicating that the adsorption of these cationic metals was likely governed mainly by the surface chemistry of the cigarette-filter-derived hydrochar, with only a limited contribution from the incorporated iron oxide. These results indicate the potential of cigarette-filter-derived hydrochar as a resource-circulating adsorbent for cationic heavy metals; the role of iron remains to be clarified by comparison with Fe-free hydrochar.

Keywords: Waste cigarette filters/ Iron-modified carbon/ Hydrothermal carbonization/ Heavy metal adsorption

1. INTRODUCTION

Every year, enormous quantities of waste cigarette filters are indiscriminately discarded into the environment, and the hazardous substances and microplastics released from them pose a serious threat to soil and aquatic environments.[1] Converting such recalcitrant waste into precursors for carbon materials via hydrothermal carbonization is a promising resource-circulation strategy, [2] and modifying the resulting carbon with iron has been reported to enhance surface reactivity and functional performance across various applications. [3] Cellulose acetate, the main component of cigarette filters, contains oxygen-containing groups that may serve as anchoring sites for metal ions during hydrothermal carbonization, making it a promising platform for the synthesis of composite materials. Building on this platform, the present study proposes the use of iron-modified hydrochar derived from waste cigarette filters as an adsorbent for removing heavy metals from aqueous systems. Surface iron modification is expected to alter the surface charge and increase the density of metal-binding active sites. [4] The objectives of this study are (i) to synthesize iron-modified hydrochar from waste cigarette filters via a one-pot hydrothermal carbonization process, (ii) to characterize its crystal structure, and (iii) to evaluate its performance as a heavy metal adsorbent in single-component solutions.

2. METHODOLOGY

2.1 Pretreatment and Preparation of Hydrochar Derived from Waste Cigarette Filters

Waste cigarette filters (CF) were used as the biomass feedstock. CF were collected on the campus of Mokpo National University, and after removing the ash and outer paper, they were rinsed 2–3 times with distilled water and then immersed in distilled water for 24 h. After immersion, only the filter

portion was separated and dried at 65°C. The pretreated CF were converted into iron-modified hydrochar through hydrothermal carbonization (HTC) using a stainless-steel hydrothermal autoclave reactor. Specifically, 3 g of CF and 90 mL of FeCl3 solution at concentrations of 0.01, 0.05, and 0.1 M were placed in the reactor. Both unadjusted pH and pH 5 (adjusted using 5 M NaOH) conditions were initially evaluated. Since preliminary tests indicated no significant differences in the physicochemical properties and adsorption performance between the two conditions, the hydrochar synthesized at pH 5 was selected as the representative sample for all subsequent characterizations and adsorption experiments. The mixture was stirred for 1 h and then reacted at 200°C for 6h to obtain iron-modified hydrochar (CF_Fe_HTC). After the reaction, the iron concentration was measured using a UV-vis spectrophotometer, and the crystal structure of the synthesized material was confirmed by XRD analysis.

2.2 Evaluation of Heavy Metal Adsorption Characteristics

Heavy metal adsorption experiments were conducted using the iron-modified hydrochar (CF_Fe_HTC). The adsorbent was added at a dosage of 0.5 g/L to single-component heavy metal solutions containing arsenic (As), chromium (Cr), copper (Cu), and lead (Pb), each at 25 ppm, and the initial pH was adjusted to various levels (e.g., pH 4–10) using 5 M HCl and 5 M NaOH. The mixture was then stirred at room temperature for 24 h to carry out the adsorption reaction. After the reaction, the supernatant was collected and filtered through a 0.45 µm syringe filter, and the residual concentrations of arsenic, chromium, copper, and lead were quantified using ICP-OES to evaluate the heavy metal removal efficiency according to iron concentration and initial pH conditions.

3. RESULTS AND DISCUSSION

3.1 Crystal Structure of Iron-Modified Hydrochar

XRD analysis of the iron-modified hydrochar synthesized at concentrations of 0.01, 0.05, and 0.1 M revealed clear differences in crystal structure depending on the concentration. At 0.01 M, only a broad amorphous scattering peak and weak-intensity peaks appeared near 10–30°, suggesting that the iron did not grow into a crystalline oxide but remained dispersed as a low-concentration amorphous phase. In contrast, at 0.05 M and 0.1 M, distinct crystalline peaks consistent with the standard diffraction pattern of hematite (α-Fe2O3) were observed, and the similar peak intensities at both conditions indicate that increasing the Fe input beyond 0.05 M did not further contribute to crystallinity. This shows that the FeCl3 concentration is the key variable determining the crystal structure of the hydrochar, and that 0.05 M is the most effective and economical concentration for forming crystalline hematite. Hematite is known to possess surface hydroxyl groups (FeOH) that are favorable for electrostatic attraction to heavy metal ions and for surface complex formation [5], and the pronounced formation of the hematite phase observed in this study is expected to contribute positively to the heavy metal adsorption performance discussed in the following section.

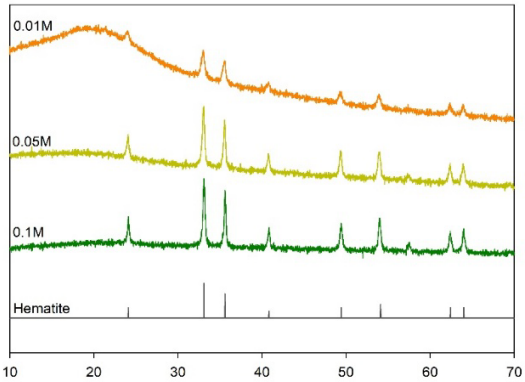

Figure 1. XRD analysis results of HTC at different iron concentrations derived from waste cigarette filters

3.2 Heavy Metal Adsorption Characteristics of Iron-Modified Hydrochar

Figure 2 presents the adsorption capacities (mg/g) of the pure hydrochar (0 M FeCl3) and the iron-modified hydrochars with 0.01, 0.05, and 0.1 M FeCl3 for heavy metals in single-component solutions at an initial concentration of 25 mg/L. The adsorption behavior varied markedly depending on the target metal. Arsenic (As) and chromium (Cr) exhibited negligible adsorption, whereas appreciable adsorption was observed for the cationic metals Cu and Pb. Specifically, Cu (Fig. 2a) showed adsorption capacities of approximately 43–45 mg/g, corresponding to about 88% removal, while Pb (Fig. 2b) exhibited adsorption capacities of approximately 15–16 mg/g, corresponding to about 30% removal. Cu and Pb adsorption capacities were nearly identical for the pristine and iron-modified hydrochars, despite the increased formation of hematite (α-Fe2O3) with increasing FeCl3 concentration observed by XRD. This result indicates that iron modification did not substantially enhance the adsorption of Cu and Pb under the tested conditions. Instead, their adsorption was likely governed mainly by the surface chemistry of the cigarette-filter-derived hydrochar, particularly oxygen-containing functional groups generated or retained during hydrothermal carbonization, with only a limited contribution from the incorporated iron oxide [2,6]. Although surface hydroxyl groups associated with iron oxides can participate in metal complexation [5], the absence of a clear Fe-loading-dependent increase in adsorption suggests that such interactions were not the dominant uptake mechanism in this system. In contrast, As and Cr predominantly exist as oxyanionic species under typical aqueous conditions. Their negligible adsorption may therefore be attributed to unfavorable electrostatic interactions and the limited affinity of the prepared hydrochar surfaces toward anionic species under the experimental conditions [4].

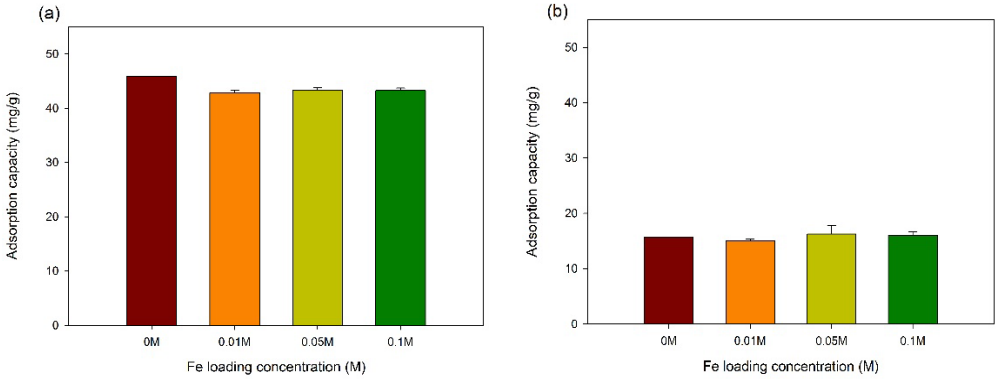

Figure 2. Comparison of adsorption capacities for (a) Cu and (b) Pb in single-component solutions by hydrochars synthesized under different iron precursor concentrations (0–0.1 M) (Synthesis condition: pH 5.0; Adsorption conditions: Adsorbent dosage = 0.5 g/L, Initial concentration = 25 mg/L, Contact time = 24 h, Initial pH = 7.0). As and Cr were hardly adsorbed.

4. CONCLUSIONS

Based on laboratory experiments and data analysis in this study, the following conclusions were drawn:

1. Fe concentration was found to be the key variable governing the crystal structure of the Fe modified hydrochar derived from waste cigarette filters. At 0.01 M, an amorphous phase was dominant, whereas at 0.05 M and above, hematite exhibited high crystallinity, confirming the optimal synthesis condition.

2. In the single-component adsorption evaluation, the synthesized hydrochars demonstrated significant adsorption capacities for the cationic heavy metals Cu (approx. 88% removal) and Pb (approx. 30% removal), while As and Cr were hardly adsorbed. Notably, the pure hydrochar (0 M) and the iron-modified hydrochars showed nearly identical adsorption capacities. This indicates that the uptake of metal cations is primarily driven by the surface characteristics (e.g., functional groups) of the hydrochar matrix itself rather than the hematite.

3. This study confirms the potential of utilizing waste cigarette filters, a recalcitrant waste, as a resource-circulating adsorbent material effective for controlling cationic heavy metals. However, to clearly demonstrate the effects of surface iron modification, detailed comparative analyses with pure hydrochar and elucidation of the surface chemical mechanisms remain as future work.

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