

Solvothermal Synthesis of Nanomagnetite-Coated Biochar for Efficient Arsenic Adsorption

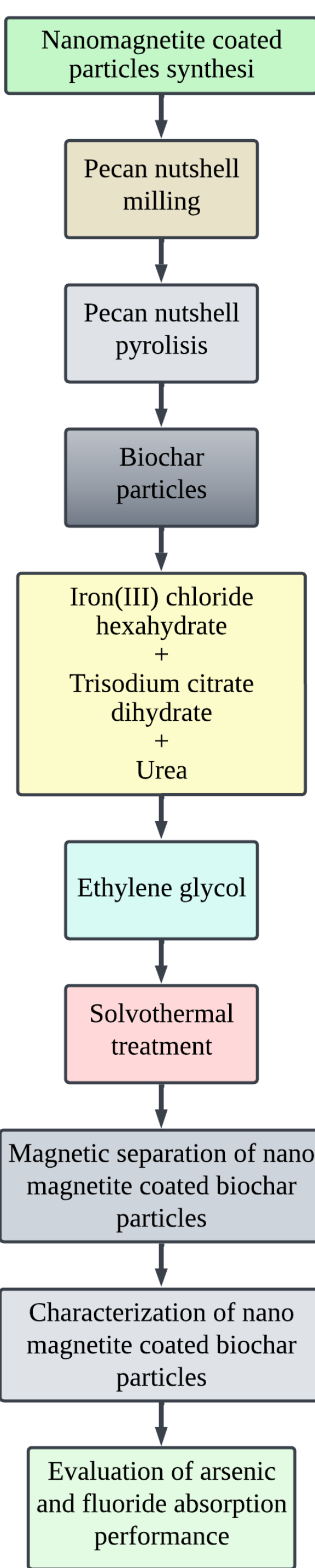
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INTRODUCTION & AIM

Arsenic contamination in groundwater poses a significant health risk, so it is essential to find alternative and effective removal methods. This research explores the alternative of nanomagnetite-coated biochar derived from pecan nut shells as an efficient arsenic adsorbent. By using solvothermal synthesis, magnetite nanoparticles are directly crystallized onto the surface of biochar particles. This method ensures a homogeneous coating with uniform size and requires short reaction times at low temperatures, eliminating the need for additional treatments. The resulting particles exhibit enhanced arsenic and fluoride adsorption capacity.

METHOD



The formation of nanomagnetite-coated biochar was carried out in a 50-mL Teflon-lined stainless-steel autoclave as follows (Figure 1):

Pecan shell particles were milled to sizes of 0.10 mm and 0.18 mm. Subsequently, they were pyrolyzed in a tube furnace at 700 °C for 1 h under nitrogen atmosphere to produce biochar particles with sizes of 0.18 mm and 0.38 mm.

The solvothermal process was carried out using a mixture of ethylene glycol ((CH₂OH)₂), trisodium citrate dihydrate (C₆H₅Na₃O₇·2H₂O), and urea CO(NH₂)₂ as solvothermal media, and Iron(III) chloride hexahydrate (FeCl₃·6H₂O) as iron ions precursor.

In this research, biochar with different particle sizes were employed, mixed with iron precursor in a ratio of 1:1 to Fe³⁺ ions, during solvothermal synthesis for 6 h, keeping the temperature constant at 200 °C to promote the crystallization of nanomagnetite on the surface of biochar particles to enhance arsenic removal.

Finally, the precipitated particles after the solvothermal treatments were washed with ethanol and distilled water to remove soluble byproducts. The remaining powders were magnetically separated and dried at 60 °C to finally be characterized.

In addition, fluoride kinetic adsorption experiments were carried out with different initial pH values (3 and 5), doses (20 mg/10 mL), and contact times (60–180 min).

RESULTS & DISCUSSION

The XRD results (Figure 2) confirmed the formation of magnetite at 6 h and 200 °C, as the obtained pattern correspond to the standard peaks of Fe₃O₄ (JCPDS 19-0629) magnetite crystalline structure. A sample without biochar was also analyzed for comparison. The sample called S11 showed higher peaks corresponding to a more defined crystalline structure, while the samples prepared using 0.18 mm biochar (S15, S16) exhibited lower intensity and broader peaks, attributed to the reduced particle size of the magnetite precipitated in the biochar.

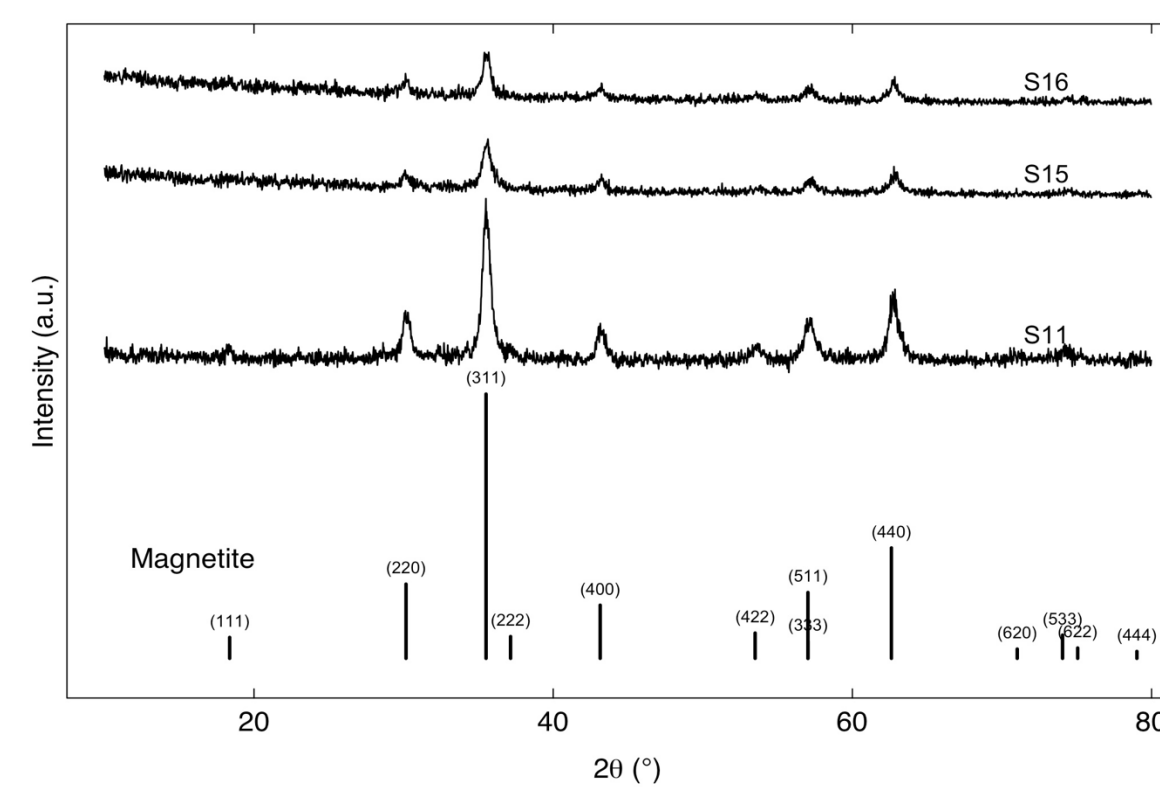


Figure 2 XRD patterns of nanomagnetite coated biochar powders prepared for 6 h at 200 °C employing: no biochar (S11), 0.18 mm (S15) and 0.38 mm (S16) biochar particles.

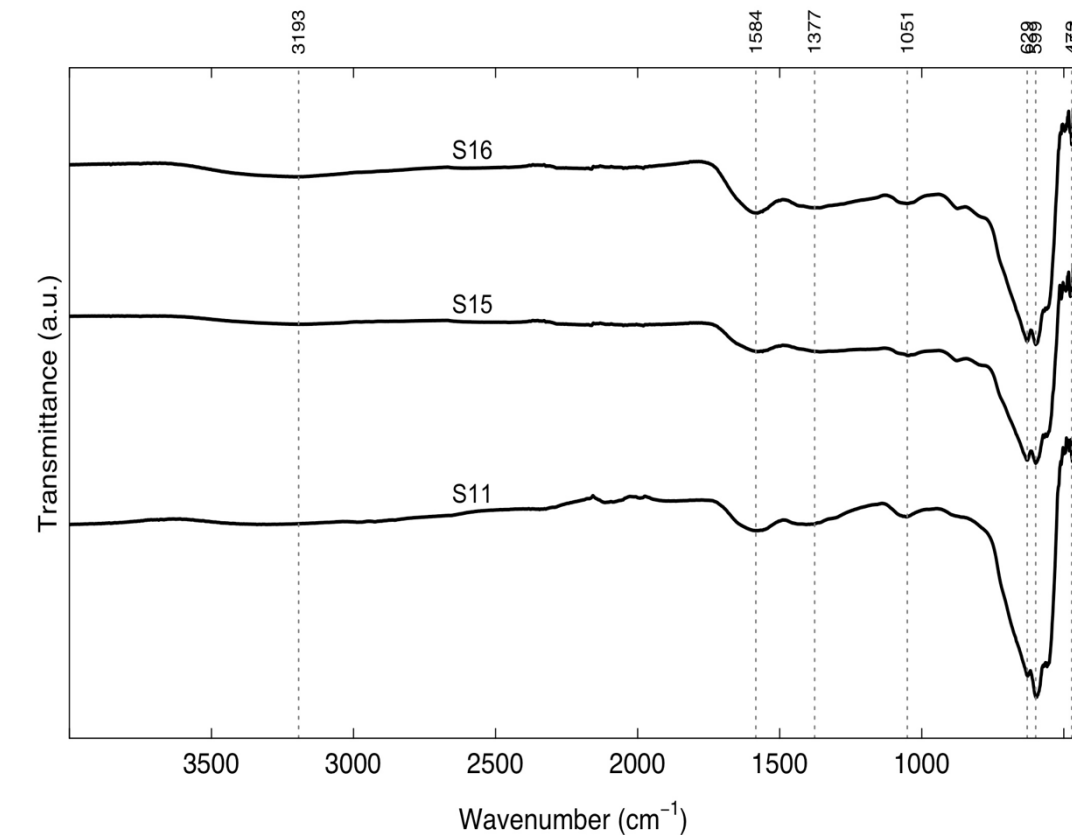


Figure 3 FT-IR spectra of nanomagnetite coated biochar powders prepared for 6 h at 200 °C employing: no biochar (S11), 0.18 mm (S15) and 0.38 mm (S16) biochar particles.

Similarly, FTIR analysis of nanomagnetite-coated biochar powders displayed absorption bands corresponding to the H-O-H and Fe-O bonds characteristic of magnetite (Figure 3).

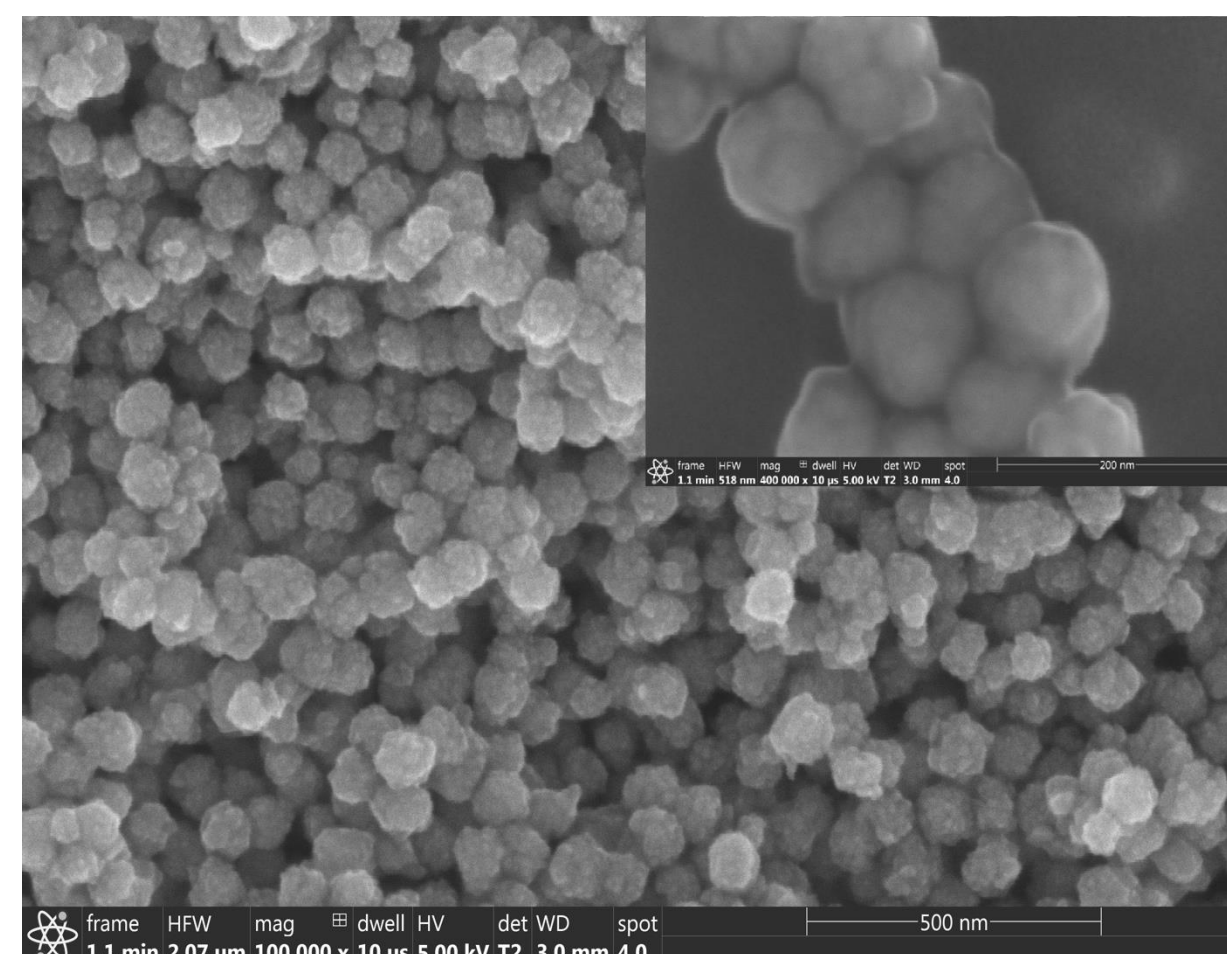


Figure 4 FE-SEM micrograph of nanomagnetite coated biochar powders prepared for 6 h at 200 °C employing 0.18 mm biochar particles (S15).

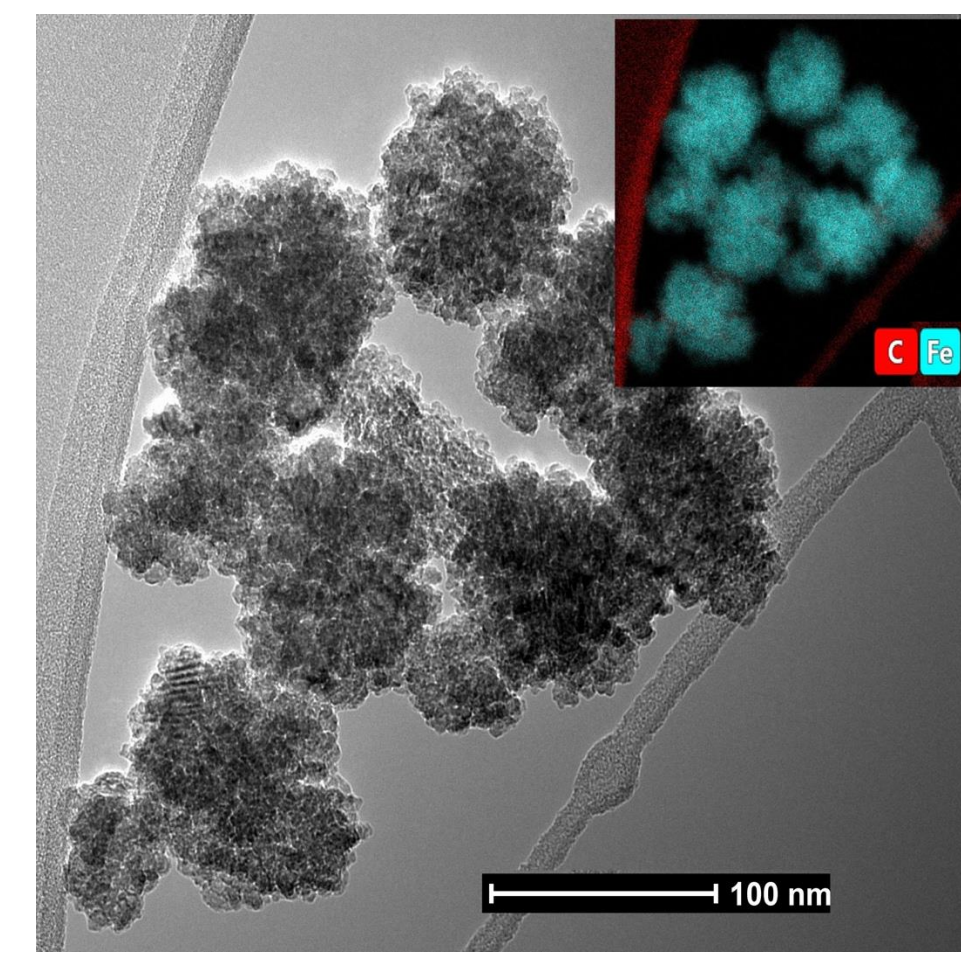


Figure 5 TEM image and chemical mapping of nanomagnetite coated biochar powders prepared for 6 h at 200 °C employing 0.18 mm biochar particles (S15).

The morphology of sample S15 was studied by scanning electron microscopy with focused ion beam (SEM/FIB). SEM images (Figure 4) confirmed the formation of homogeneous particles with a size ranging between 95 and 115 nm and a spherical morphology.

To verify the formation of magnetite nanoparticles on the biocarbon particles, a transmission electron microscopy (TEM) analysis was carried out, which allowed for chemical mapping that confirmed that the crystallization of the magnetite took place homogeneously over the entire surface (Figure 5).

CONCLUSION

The arsenic removal was studied (Figure 6) and the performance consistently exceeds 97%, with a stable adsorption capacity (q_e) of around 8 mg/g across all samples, indicating that nanomagnetite-coated biochar is effective for arsenic removal regardless of particle size.

In contrast, fluoride removal is more influenced by synthesis conditions, requiring further optimization. The best results are exhibited by the sample S15 which also shows the highest fluoride removal at approximately 32%. Overall, S15 is identified as the most effective option for removing both arsenic and fluoride.

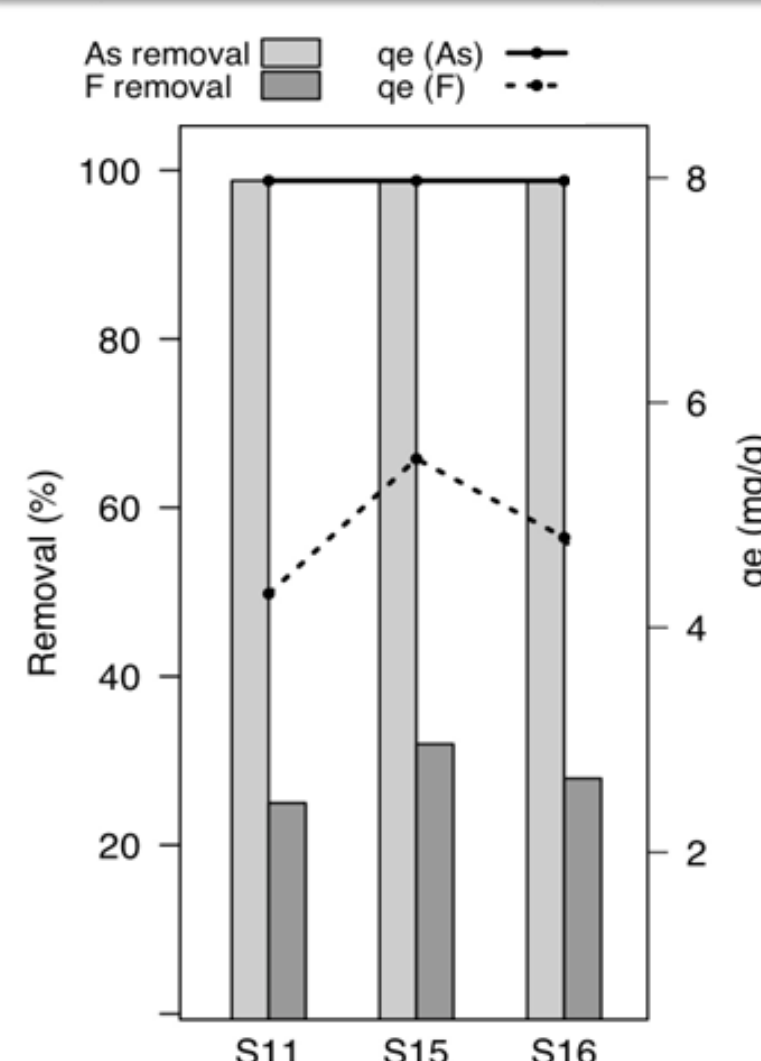


Figure 6 Arsenic and fluoride removal patterns of nanomagnetite coated biochar powders prepared for 6 h at 200 °C employing: no biochar (S11), 0.18 mm (S15) and 0.38 mm (S16) biochar particles.

Figure 1. Nanomagnetite coated particles synthesis process flowchart