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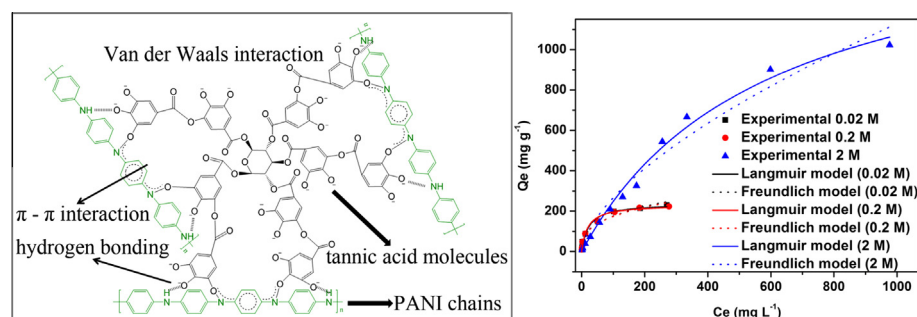
Adsorption removal of tannic acid from aqueous solution by polyaniline: Analysis of operating parameters and mechanism



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GRAPHICAL ABSTRACT



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ABSTRACT

Polyaniline (PANI) prepared by chemical oxidation was studied for adsorption removal of tannic acid (TA) from aqueous solution. Batch adsorption studies were carried out under different adsorbent dosages, pH, ionic strength, initial TA concentration and coexisting anions. Solution pH had an important impact on TA adsorption onto PANI with optimal removal in the pH range of 8–11. TA adsorption on PANI at three ionic strength levels (0.02, 0.2 and 2 mol L⁻¹ NaCl) could be well described by Langmuir model (monolayer adsorption process) and the maximum adsorption capacity was 230, 223 and 1023 mg g⁻¹, respectively. Kinetic data showed that TA adsorption on PANI fitted well with pseudo-second-order model (controlled by chemical process). Among the coexisting anions tested, PO₄³⁻ significantly inhibited TA adsorption due to the enhancement of repulsive interaction. Continuous flow adsorption studies indicated good flexibility and adaptability of the PANI adsorbent under different flow rates and influent TA concentrations. The mechanism controlling TA adsorption onto PANI under different operating conditions was analyzed with the combination of electrostatic interactions, hydrogen bonding, π - π interactions and Van der Waals interactions.

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1. Introduction

Tannic acid (TA) is a component of dissolved organic matter (DOM) resulting from the decomposition of plant biomass in natu-

ral systems [1]. It is a water soluble polyphenolic compound with relatively high molecular weight (average 1700 g mol⁻¹), which is commonly present in surface and ground water [2]. It is also identified in the wastewater of coir and cork process factories, plant medicine, paper and leather industries [3,4]. TA causes serious problems to the environment as it has been demonstrated to be toxic to aquatic organism and causes liver, kidney or central nervous system problems [5,6]. Moreover, TA is a precursor of carcinogenic disinfection byproducts (DBPs) in the drinking water

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supply and can seriously affect the health of human beings [7–9]. Therefore, it is of great importance to eliminate TA contamination in water.

The current technologies to eliminate TA contamination in water include coagulation, adsorption, ultrafiltration, electrochemical methods and biological methods [3,10–14]. Among these technologies, adsorption is one of the most common methods for removing TA from water with high efficiency and low cost. Various adsorbents, such as activated carbon, carbon nanotubes, natural and synthetic zeolites, resin, chitosan and clay minerals, have been reported for the removal of aqueous TA [7–9,14–16]. Among of them, amino-adsorbents have been found to have a strong affinity for aqueous TA owing to the strong interaction between the carboxyl and phenolic groups of TA molecules and the amino groups of adsorbent. For example, amino-functionalized magnetic mesoporous silica exhibits significant adsorption for TA molecules [8]. Amino-functionalized magnetic nanoadsorbent ($\text{Fe}_3\text{O}_4@\text{SiO}_2\text{-NH}_2$) has also been found to be very effective in removing aqueous TA [17].

Polyaniline (PANI), a well-known conducting polymer, has received a great deal of interest due to its simple preparation, controllable electrical conductivity, environmental stability, good redox reversibility, and low cost. Recently, PANI and its composites have been used effectively for the removal of inorganic and organic pollutants in wastewater [18–20]. They possess large amounts of amine and imine groups which can interact with heavy metals and organic pollutants, making them suitable for pollutant adsorption from aqueous solution. Previous studies have suggested that PANI and its composites are promising adsorbents for Cr(IV) [21], Hg(II) [22,23], anionic dyes [24], phenolic derivatives [25] and humic acids (HA) [26–28]. PANI showed high adsorption capacity for aqueous HA [26]. Wang et al. observed a significant improvement in the HA adsorption capacity when PANI was encapsulated on the surface of ATP [27]. Enhanced HA adsorption was associated with electrostatic interactions between the amine and imine groups of adsorbents and HA molecules in solution.

Based on the premise that nitrogen atoms in PANI can interact with organic pollutants in aqueous solution, PANI is expected to be an efficient and economic adsorbent for TA removal. However, systematic studies of operating parameters on adsorption of TA onto PANI are still lacking, the associated mechanisms are yet to be elucidated, and continuous flow experiments are needed for the development of adsorption technology for effective treatment of TA-contaminated waters. In this study, PANI was synthesized by chemical oxidation to remove TA from aqueous solution, and its adsorption behaviors under different conditions were observed. The adsorption isotherms, kinetics and associated mechanism were discussed. A series of continuous flow experiments were also conducted to make it practical for application.

2. Materials and methods

2.1. Materials and reagents

TA (Sigma-Aldrich, USA) was used as received without further purification. Aniline (Merck) was purified by distillation under reduced pressure, kept in the dark, and stored in a refrigerator before use. Ammonium peroxodisulfate ($(\text{NH}_4)_2\text{S}_2\text{O}_8$ (>98%) was purchased from Fluka. Other chemicals like Na_2SO_4 , NaNO_3 , $\text{Na}_3\text{PO}_4 \cdot 12\text{H}_2\text{O}$, HCl, NaOH, NaCl (analytical grade) were purchased from Nanjing Chemical Reagent Co., Ltd (China). All solutions were prepared using ultrapure water ($18.25 \text{ M}\Omega \text{ cm}^{-1}$) at 25°C .

2.2. Preparation of PANI adsorbent

Polyaniline was synthesized according to our previous study with some modifications [22]. Briefly, 0.025 mol freshly distilled

aniline was dissolved in 250 mL of 1 mol L^{-1} HCl, to which 250 mL of 0.1 mol L^{-1} $(\text{NH}_4)_2\text{S}_2\text{O}_8$ with 1 mol L^{-1} HCl was slowly added under continuous stirring over a period of 5 h at room temperature. The insoluble dark-green precipitate formed was washed with large amounts of deionized water until the water became colorless, and then it was vacuum-dried at 80°C until it reached constant weight. The resultant polymer PANI was powdered in a mortar and sieved through meshes (70–150 screen mesh) to ensure that the particle size of the adsorbent was between 0.1 and 0.2 mm before use. Fig. S1 shows the SEM image and corresponding particle size distribution of the synthesized PANI, which has almost spherical shape with average particle diameter of 174.7 nm.

The specific surface area and pore structure of the PANI adsorbent were analyzed by nitrogen sorption (Micromeritics TriStar, USA) at 77 K on Tristar micromeritics surface area based on the BET method. Fig. S2 shows that the specific surface area based on BET data is $23.06 \text{ m}^2 \text{ g}^{-1}$, the BJH desorption cumulative pore volume is $0.196 \text{ cm}^3 \text{ g}^{-1}$ and the average pore diameter is 32.88 nm.

2.3. Batch experiments

Batch adsorption experiments were carried out in batch equilibrium mode. Briefly, desired amounts of PANI adsorbent was mixed with 10 mL TA solution with appropriate initial concentrations in glass tubes. The tubes were then shaken in an oscillation incubator at 150 rpm at 25°C for 4 h. Preliminary kinetic experiments showed that adsorption equilibrium of TA on PANI was completely achieved within approximately 4 h under the experimental conditions. At predetermined time intervals, the solutions were collected and then filtered using a syringe filter of $0.22 \mu\text{m}$ and the TA concentration after adsorption was determined by UV/VIS-spectrophotometer (Shimadzu UV-2550) at a wavelength of 278 nm (the procedure is described in sub-head 2.5 in detail). The amount of adsorption (Q_e) was calculated using the following equation:

$$Q_e = \frac{V(C_0 - C_e)}{m} \quad (1)$$

where Q_e is the adsorption capacity of TA on PANI (mg g^{-1}) at equilibrium, C_0 is the initial concentration of TA (mg L^{-1}), C_e is the equilibrium concentration of TA (mg L^{-1}), m is the mass of adsorbent used (g) and V is the volume of TA solution (L).

For determination of the adsorption isotherms, PANI adsorbent was added with various initial concentrations of TA at fixed ionic strength ($10\text{--}500 \text{ mg L}^{-1}$ TA for 0.02 and 0.2 mol L^{-1} NaCl, $10\text{--}2000 \text{ mg L}^{-1}$ TA for 2 mol L^{-1} NaCl), and other experimental conditions are noted in the figure caption. For kinetic experiments, PANI was added to TA solution with the initial concentration of 20 and 100 mg L^{-1} , respectively, with final pH at 10 and fixed ionic strength at 0.2 mol L^{-1} NaCl ($T = 298 \text{ K}$). The effect of solution pH was studied in the range of $0.5\text{--}12$. The solution pH was adjusted by addition of HCl and NaOH. Besides, coexisting anions with TA adsorption including Cl^- , SO_4^{2-} , NO_3^- , PO_4^{3-} were added when necessary.

2.4. Continuous flow experiments

Fig. S3 shows the experimental setup for continuous flow adsorption. Prior to loading into the columns, the adsorbent was initially wetted with ultrapure water. The column was wet packed with special care to avoid any air bubbles within the column. 0.2 g PANI particles were packed within the column between two filterable membranes at the top and bottom end to prevent the adsorbent from floating. The feed solution containing TA was pumped into the column in up-flow mode from a 1 L vessel of stock solution

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