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Editorial

Adsorption Technology for Water and Wastewater Treatments

Hai Nguyen Tran [1,2]

[1] Center for Energy and Environmental Materials, Institute of Fundamental and Applied Sciences, Duy Tan University, Ho Chi Minh 700000, Vietnam; trannguyenhai@duytan.edu.vn or trannguyenhai2512@gmail.com
[2] Faculty of Environmental and Chemical Engineering, Duy Tan University, Da Nang 550000, Vietnam

Abstract: This Special Issue includes 12 research papers on the development of various materials for adsorbing different contaminants in water, such as Sb, Cr(VI), Cu(II), Zn(II), fluorine, phenol, dyes (indigo carmine, Congo red, methylene blue, and crystal violet), and drugs (dlevofloxacin, captopril, and diclofenac, and paracetamol). The commercial, natural, and synthetic materials used as adsorbents comprise commercial activated carbon, natural clay and montmorillonite, biosorbent based on sugarcane bagasse or algal, graphene oxide, graphene oxide-based magnetic nanomaterial, mesoporous Zr-G-C_3N_4 nanomaterial, nitrogen-doped core–shell mesoporous carbonaceous nano-sphere, magnetic Fe-C-N composite, polyaniline-immobilized ZnO nanorod, and hydroxy-iron/acid–base-modified sepiolite composite. Various operational conditions are evaluated under batch adsorption experiments, such as pH, NaCl, solid/liquid ratio, stirring speed, contact time, solution temperature, initial adsorbate concentration. The re-usability of laden materials is evaluated through adsorption–desorption cycles. Adsorption kinetics, isotherm, thermodynamics, and mechanisms are studied and discussed. Machine learning processes and statistical physics models are also applied in the field of adsorption science and technology.

Keywords: adsorption; mechanism; modelling; artificial intelligence; emerging pollutant; machine learning; statistical physics; water treatment

1. Introduction

The presence of pollutants (i.e., emerging contaminants, radionuclides, potential toxic metals, and dyes) in water environments has a negative impact on the environment and presents a potential health risk for inhabitants. Among the existing technologies (advanced oxidation process, membrane filtration, biodegradation, coagulation, and flocculation, etc.) for treating pollutants in water, adsorption has garnered great interest due to its low operational cost and fast removal. In particular, this method can effectively remove various pollutants at their low concentrations (trace levels) from water, if adsorbent materials exhibit excellent adsorption capacity and high adsorption affinity to pollutants. Unlike coagulation and flocculation processes, adsorption techniques do not generate by-products (i.e., sewage sludge). There is a remarkable and increasing amount of sewage sludge being discharged into the environment, which represents an enormous concern with regard to waste management aspects.

In recent times, the development of advanced materials and their application as potential adsorbents for water treatments have caught the attention of researchers. This Special Issue (SI) aims to establish the state of the art on adsorption and biosorption technologies for water and wastewater treatments.

Apart from traditional methods (i.e., response surface methodology), some machine learning processes are currently applied for estimating the adsorption capacity of the adsorbent (q_e) or the removal efficiency of the adsorption process (%R). Some typical examples of the machine learning are (1) the Gaussian process regression (GPR) coupled with the particle swarm optimization (PSO) model and (2) the nearest neighbor coupled with the gray wolf optimizer algorithm. The papers relating to the application of artificial

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intelligence in anticipating and optimizing operational conditions (strongly affecting the q_e or %R value) are given a major priority in this SI.

The adsorption isotherm plays an essential role in this field. In the literature, many isotherm models have been used to fit adsorption equilibrium datasets. They include the Langmuir and Freundlich models (the most widely used model), Lui model, Redlich–Peterson model, Hill model, Radke–Prausnitz model, Khan model, Toth model, Sips model, and Koble–Corrigan model. Unlike these models, several statistical physics models can help to well explain the adsorption phenomena and mechanisms. This SI also gives a priority for collecting this topic.

This SI belongs to the section "Wastewater Treatment and Reuse" of the journal (*Water*; SSN 2073-4441). After the Editor-in-Chief of *Water* (Dr. Jean-Luc PROBST) had approved this SI, "Adsorption Technology for Water and Wastewater Treatments", it was announced online on the website of the journal and opened for submissions from 20 September 2022 to 30 June 2023. The acceptance rate of this SI is approximately 63%. The impact factor (Web of Science), CiteScore (Scopus), and H-index (Scimago) values for this journal are 3.4, 5.5, and 85, respectively.

This SI successfully compiled 12 unique papers contributed by 75 authors from 12 countries (France, United Kingdom, Vietnam, China, Brazil, Colombia, Algeria, Saudi Arabia, Sudan, Morocco, Pakistan, and Sri Lanka). All of them [1–12] are of the "Article" type. In particular, one published paper was recommended as a "feature paper" by two academic editors (Andrea G. Capodaglio and Antonio Panico) of this journal. A Feature Paper is a substantial original article that presents "the most advanced research with significant potential for high impact in the field".

Various adsorbates that were investigated and published in this SI included antimony (Sb), hexavalent chromium, copper, zinc, fluorine, phenol, organic dye (indigo carmine, Congo red, methylene blue, and crystal violet), and pharmaceutical adsorbates (levofloxacin, captopril, diclofenac, and paracetamol). To provide considerable insights into the essential aspects of this SI, the Editor highlights some brief summaries on all the works published [1–12] in the next section.

2. Summary of Papers Published in This Special Issue

2.1. Enhanced Removal of Sb (III) by Hydroxy-Iron/Acid–Base-Modified Sepiolite: Surface Structure and Adsorption Mechanism

Zou and co-workers [1] developed a new composite derived from a sepiolite mineral (magnesium-enriched silicate clay) through two steps: treating sepiolite with acid–base (HCl and NaOH) and then impregnating it with $FeCl_3$.

The composite and the sepiolite were applied for adsorbing antimony (Sb) in an aqueous solution. The p*K*a of antimony is around 3. Antimony exists in water as $Sb(OH)_3$ (within a solution pH from 1.0 to 6.0) and as $Sb(OH)_6^-$ (pH from 1.0 to 14). At pH = 3, the percentage of $Sb(OH)_3$ and $Sb(OH)_6$—species was 50% and 50%. The batch adsorption experiments were conducted under different conditions: solid/liquid (m/V) ratio (0.5–3.0 g/L), solution pH (2–12), contact time (3–240 min), initial Sb(III) concentration (C_o = 10–150 mg/L), and temperatures (298–318 K).

The powder X-ray diffraction (PXRD) data indicated that the primary iron form present in the composite was iron(III) oxide-hydroxide FeO(OH). The BET (Brunauer–Emmett–Teller) specific surface area (S_{BET}) of the composite (152.4 m^2/g) was slightly lower than that of the sepiolite (160.1 m^2/g).

The maximum adsorption capacity (Q_{max}) calculated based on the Langmuir model of the composite (at 25 °C, 35 °C, and 45 °C) was 25.73, 26.89, and 32.45 mg/g, values that were higher than the corresponding values of the sepiolite (14.66, 14.87, and 16.42 mg/g, respectively). The adsorption mechanisms were evaluated via the X-ray photoelectron spectroscopy (XPS) technique.

2.2. Efficient Uptake of Angiotensin-Converting Enzyme II Inhibitor Employing Graphene Oxide-Based Magnetic Nanoadsorbents

Oliveira and colleagues [2] used graphite to generate a magnetic graphene oxide (M-GO) and applied M-GO as an adsorbent to eliminate captopril from water. The magnetic graphene oxide was prepared through the co-precipitation method of graphene oxide with $FeCl_2$ at pH 9.0.

The magnetization saturation (M_S) of M-GO was 45 emu/g. The results of Raman spectroscopy indicated that the I_D/I_G ratio of GO (0.96) increased to 1.22 after the magnetization procedure (M-GO).

Different conditions were applied for the batch adsorption processes such as solid/liquid (m/V) ratio (0.125–1.0 g/L), solution pH (2–9), NaCl (0.01–1 M), contact time (5–180 min), initial captopril concentration (C_o = 10–200 mg/L), and temperatures (20–40 °C). Regeneration and reuse studies were applied by using NaOH (0.25 mol/L) as a desorbing agent.

The result indicated that under the same adsorption conditions, M-GO (99.4 mg/g) exhibited a slightly higher adsorption capacity of captopril than GO (91.8 mg/g). The Q_{max} values of M-GO towards captopril at 20 °C, 30 °C, and 40 °C were 101.9, 101.5, and 99.3 mg/g, respectively. The standard enthalpy change ($\Delta H°$) of this adsorption process was −64.19 kJ/mol (exothermic in nature). The laden M-GO can be well reused after five adsorption–desorption cycles.

2.3. Predictive Model Based on K-Nearest Neighbor Coupled with the Gray Wolf Optimizer Algorithm (KNN_GWO) for Estimating the Amount of Phenol Adsorption on Powdered Activated Carbon

The application of machine learning in optimizing and predicting the amount of phenol (diameter: 0.56 nm) adsorbed by commercially available powdered activated carbon (PAC) under aqueous solution was examined by Zamouche et al. [3]. This paper has been recommended as a "feature paper".

The S_{BET} value and total pore volume (V_{Total}) of PAC were 463.4 m^2/g and 0.13 cm^3/g. Its strong acid carboxylic group and hydroxyl and phenol groups. The effect of several adsorption conditions on the phenol adsorption processes was explored, such as solid/liquid ratio (0.4–1.6 g/L), solution pH (2.1–12), stirring speed (200–600 rpm) contact time (5–60 min), initial phenol concentration (C_o = 10–200 mg/L), and temperature (20 °C–50 °C).

The Q_{max} values of PAC towards phenol were 156.3 mg/g at 20 °C, 150.0 mg/g at 30 °C, and 150.8 mg/g at 40 °C. The $\Delta H°$ of this process was −19.23 kJ/mol (an exothermic process).

The result of applying K-nearest neighbor (common method in machine learning) coupled with the gray wolf optimizer algorithm for predicting the phenol amount adsorbed by PAC demonstrated an advantage in providing highly precise values because of very low statistical errors (closing to zero) and high statistical coefficients (reaching unity).

2.4. Nitrogen-Doped Core–Shell Mesoporous Carbonaceous Nanospheres for Effective Removal of Fluorine in Capacitive Deionization

Zhao and coworkers [4] synthesized a mesoporous carbonaceous nanospheres (MCS) doped with nitrogen (NMCS) using a popular sol–gel method. The SEM images showed that MCS and NMCS exhibited a rough surface morphology with spherical shapes. Meanwhile, their core–shell structure was confirmed via transmission electron microscope (TEM). The Raman data provided a higher I_D/I_G ratio of NMCS (0.999) than MCS (0.982). The S_{BET} and V_{Total} of MCS (1058 m^2/g and 1.89 cm^2/g) decreased after doping with nitrogen NMCS (1049 m^2/g and 1.78 cm^2/g).

The presence of nitrogen-containing functional groups in NMCS was confirmed through a new band at around 3430 cm^{-1} (i.e., N–H) in its FTIR spectrum. The XPS data provided the information on the nitrogen content of MCS (0.31%; %atomic) and NMCS (4.30%; %atomic) as well as the fraction of nitrogen (%) in two samples. The high-resolution

N 1s spectrum of MCS and NMCS suggested their nitrogen-containing functional groups were graphitic-N (401 eV), pyrrolic-N (400 eV), and pyridinic-N (398 eV).

NMCS was applied for removing fluorine (F^- anions) from water. Its maximum electrosorption capacity to F^- anions (C_o = 1000 mg/L at 12 V) was 13.34 mg/g. The result of adsorption–regeneration cycles showed that NMCS exhibited an excellent repeatability, with its electrosorption capacity slightly decreasing after 10 cycles.

2.5. Montmorillonite-Based Natural Adsorbent from Colombia for the Removal of Organic Pollutants from Water: Isotherms, Kinetics, Nature of Pollutants, and Matrix Effects

The study of utilizing a natural clay (montmorillonite) as adsorbent for treating various organic pollutants in water was undertaken by Paredes-Laverde and colleagues [5]. The target adsorbates included traditional pollutants (Congo red (CR), indigo carmine (IC), crystal violet (CV), and methylene blue (MB) dye) and emerging pollutants (diclofenac and levofloxacin drugs).

The montmorillonite was characterized through thermogravimetric analysis (TGA), Fourier transform infrared spectroscopy (FTIR), scanning electronic microscopy (SEM), XRD, and BET analyzer. Its S_{BET} and V_{Total} were 85.5 m^2/g and 0.004 cm^3/g.

The montmorillonite exhibited a relatively low Q_{max} value for dyes: CV (18.2 mg/g), CR (16.2 mg/g), MB (14.1 mg/g), and IC (1.07 mg/g) under aqueous solutions. The montmorillonite can remove 99% CV dye under an aqueous solution and 85% CV dye under real textile wastewater. The result of adsorbing pharmaceuticals in urine indicated that the montmorillonite can remove approximately 90% of levofloxacin and 8% of diclofenac after a 60 min contact.

2.6. Mesoporous Zr-G-C3N4 Sorbent as an Exceptional Cu(II) Ion Adsorbent in Aquatic Solution: Equilibrium, Kinetics, and Mechanisms Study

Khezami et al. [6] slightly modified an one-step ultrasonication method to synthesize a mesoporous nanomaterial (Zr-G-C_3N_4) that was graphitic carbon nitride (g-C_3N_4) doped with ZrO_2.

The XRD data confirmed the existence of both g-C_3N_4 and ZrO_2 peaks. The textural parameters of Zr-G-C_3N_4 were 95.7 m^2/g (S_{BET}) and 0.216 cm^3/g (V_{Total}).

Adsorption kinetic of Cu(II) by Zr-G-C_3N_4 reached an equilibrium at around 500 min. The studies on the effect of pH solution (pH = 1.0–8.0) indicated that the optimal pH value for the Cu(II) adsorption process was established at 5.0. The adsorption isotherm (C_o = 5–200 mg/L) provided the Q_{max} value of Zr-G-C_3N_4 was 144.1 mg/g. After four continuous cycles of the Cu(II) adsorption–desorption processes using 0.1 M NaOH as a desorbing agent, the removal efficiency was still higher than 80%. The XPS data confirmed that the primary adsorption mechanism was surface complexation.

2.7. Applying Linear Forms of Pseudo-Second-Order Kinetic Model for Feasibly Identifying Errors in the Initial Periods of Time-Dependent Adsorption Datasets

Tran [7] studied the kinetic process of paracetamol adsorption onto commercial activated carbon (its S_{BET} = 1275 m^2/g, and V_{Total} = 0.670 cm^3/g) under experimental conditions: C_o = 3.45 mmol/L; pH = 7.0; 25 °C; m/V = 1 g/L. The contact time was 1, 5, 15, 30, 45, 60, 90, 120, 180, 240, 300, 360, 720, 1440, 2880, and 4320 min. Some adsorption kinetic models were applied for modelling time-dependent adsorption datasets, such as the pseudo-n^{th}-order, pseudo-first-order, pseudo-second-order (PSO), Elovich, and Avrami models. The best-fitting model was evaluated through the highest value of determination coefficient (R^2) and adjusted determination coefficient (adj-R^2) and the lowest value of chi-squared (χ^2), reduced chi-square (red-χ^2), and Bayesian information criterion (BIC).

Among those models, the PSO model is the most commonly used in this field. Six linear forms (Types 1–6) and one non-linear form of the PSO model were used for exploring the error points under the initial periods (potential outliers). The results indicated that although the use of a non-linear optimization method is often recommended to minimize error functions, it cannot help to identify errors in the initial periods of time-dependent

adsorption datasets. Among six forms of the PSO model, those error experimental points (i.e., very high q_t values at 1 and 5 min) can be observed though Types 2–5.

In the literature, the Type 1 is often used for fitting the experimental data of time-dependent adsorption because of very adj-R^2 (i.e., 0.9999). However, in many cases, this high adj-R^2 or r^2 indicates a spurious correlation. Therefore, the estimation of the rate constant k_2 of time-dependent adsorption processes based on the Type 1 should be avoided. After removing the outliers, the experiential data were well described by the following models: Avrami > PNO > PSO > PFO > Elovich.

2.8. Factorial Design Statistical Analysis and Optimization of the Adsorptive Removal of COD from Olive Mill Wastewater Using Sugarcane Bagasse as a Low-Cost Adsorbent

Sugarcane bagasse that was utilized as a biosorbent for treating COD (chemical oxygen demand) from real olive mill wastewater was published by Elayadi and colleagues [8]. The authors applied the 2^{5-1} fractional factorial design (using Minitab 18) for optimizing the effects of various operational conditions on the COD removal efficiency.

The experimental variables (their low and high levels) included biosorbent dosage (10 and 60 g/L), stirring speed (80 and 300 rpm), adsorption time (1 and 24 h), pH (2 and 12), and solution temperature (20 °C and 60 °C).

The result showed that the optimal conditions reached at 10 g/L, 80 rpm, 1 h, pH 12, and 60 °C, with the COD removal efficiency being 55.1%. The authors reported that the Q_{max} value of the sugarcane bagasse obtained from the adsorption isotherms was 331.9 mg/g.

2.9. Comparison of Phenol Adsorption Property and Mechanism onto Different Moroccan Clays

Dehmani and coworkers [9] investigated the adsorption capacity and mechanisms to phenol in aqueous solutions using two natural clay materials (RCA and RCG). Those clays were characterized via X-ray Fluorescence (XRF), XRD, FTIR, TGA, and SEM–EDS. The pH_{PZC} and CEC values of RCA were 8.60 and 10.6 meq/100 g, and those of RCG were 8.60 and 16.6 meq/100 g, respectively. The clay samples (RCA and RCG) exhibited low values of S_{BET} (51.41 and 25.31 m2/g) and V_{Total} (0.13 and 0.03 cm/g).

The adsorption process occurred fast and reached equilibrium after 30 min of contact. Adsorption isotherms were conducted at 30 °C, 40 °C, and 50 °C. Isotherm modeling was evaluated through physical–statistical approaches such as the monolayer model with a single energy site, monolayer model with two energy sites, double-layer model with one energy site, double-layer model with two energy sites, and multilayer model. The best fitting of models was evaluated based on R^2, adj-R^2, minimum squared error, BIC, and average relative error.

The result showed that the adsorption equilibrium data were satisfactorily described by the monolayer model with two different energy sites. The primary adsorption mechanism of phenol onto RCA was van der Waals, and those onto RCG were van der Waals and hydrogen bond.

2.10. In Situ Polyaniline Immobilized ZnO Nanorods for Efficient Adsorptive Detoxification of Cr(VI) from Aquatic System

Alharthi and colleagues [10] applied an in situ free radical polymerization method to synthesize a PAni-ZnO composite that was zinc oxide nanorod (ZnO-nanorod) immobilized with polyaniline. Some techniques that were used for characterizing the materials (ZnO-nanorod, polyaniline, and PAni-ZnO composite) included XRD, TGA, FTIR, SEM–EDS, and TEM.

The S_{BET} value and the average pore width of PAni-ZnO composite were 113.5 m^2/g and 7 nm. The analysis result of zeta potentials of the composite indicated its isoelectric point (IEP) at pH = 4.32 (its pH_{IEP} = 4.32).

The adsorption process of Cr(VI) by the PAni-ZnO composite was conducted at different operational conditions such as pH, contact time, adsorbent dose, initial Cr(VI)

concentration, and solution temperature. The effect of the nanorod sizes (10.5, 19.0, 32.6, and 52.2 nm) on the Cr(VI) adsorption behavior of the composite was also evaluated.

The result of the absorption isotherm demonstrated that the Q_m values of the composite increased (142.3, 219.2, and 310.4 mg/g) within an increase in solution temperatures (25 °C, 35 °C, and 45 °C, respectively). The value ΔH° of this adsorption process was 32.01 kJ/mol, suggesting dominantly physical adsorption.

2.11. Algae and Hydrophytes as Potential Plants for Bioremediation of Heavy Metals from Industrial Wastewater

Biosorption of potential toxic metals from real industrial wastewater using algae (*Zygnema pectiantum* and *Spyrogyra* species) and hydrophytes (*Typha latifolia* and *Eicchornia crassipes*) for 40 days was studied by Farid and coworkers [11]. The industrial wastewater (IWW) discharged from various industries (steel, textile, marble, chemical, ghee, and soap and oil).

The physicochemical characteristics of IWW were pH (6.85), EC (electrical conductivity; 6.35 mS/cm), COD (chemical oxygen demand; 236 mg/L), BOD (biological oxygen demand; 143 mg/L), DO (dissolved oxygen; 114 mg/L), TDS (total dissolved solids; 559.67 mg/L), TSS (total suspended solids; 89.30 mg/L), Cu (7.13 mg/L), and Zn (5.73 mg/L). The authors concluded that the concentrations of COD, BOD, Cu, and Zn of the raw IWW were higher than the corresponding values currently published by the Pakistan Environmental Protection Agency (maximum permissible limits).

The results demonstrated that the removal efficiencies of both hydrophytes and algae towards various selected parameters were EC (74.01–91.18%), DO (13.15–62.20%), BOD (21.67–73.42%), COD (25.84–73.30%), TDS (14.02–95.93%), and TSS (9.18–67.99%). In particular, they can effectively remove approximately 85% of Cu ions and 82% of Zn ions from IWW. Namely, the removal efficiency of Cu and Zn was 85.13% and 71.55% (using *Typha latifolia*), 80.78% and 62.12% (*Eicchornia crassipes*), 81.20% and 82.19% (*Zygnema pectiantum*), and 81.90% and 60.90% (*Spyrogyra* species).

2.12. Adsorption–Reduction of Cr(VI) with Magnetic Fe-C-N Composites

Liu et al. [12] reused an iron-enriched coagulant sludge collected from a dye chemical wastewater plant to develop magnetic Fe–C–N composites through a direct calcination process at different temperatures (200, 400, 500, and 600 °C). The developed material was then used for studying the adsorption of Cr(VI) anions.

The results of the materials' properties showed that the highest magnetization saturation (M_S = 4.3 emu/g) was recorded for the FCN-500 sample (prepared by calcinating the sludge at 500 °C). A high M_S value resulted from the existence of Fe_3O_4 nano-particles in this material that was confirmed via XRD analysis. FCN-500 can be classified as a non-porous material because of its low S_{BET} value (47.77 m^2/g). The XPS survey data indicated that FCN-500 contained mainly carbon (41.24%; %atomic), oxygen (34.74%), iron (12.8%), and nitrogen (11.22%). The XRF data demonstrated that FCN-500 and the sludge had a high percentage of iron (74.01% and 68.57%, respectively).

The Langmuir maximum adsorption capacity of FCN-500 towards Cr(VI) anions in aqueous solutions (adsorption isotherm conditions: 35 °C, pH = 2.0, 120 min, m/V = 0.8 g/L, and C_o = 10–50 mg/L) was 52.63 mg/g. The study of consecutive adsorption–desorption cycles (using 1 M NaOH as a desorbing agent) confirmed that FCN-500 exhibited an excellent reusability, with its adsorption capacity decreasing by approximately 20% after the five cycles.

The adsorption mechanism was considered at pH 2.0. The Cr 2p XPS spectrum of FCN-500 after adsorbing Cr(VI) indicated the dominant species of Cr(III) in the laden FCN-500, suggesting adsorption-coupled reduction mechanisms. Cr(VI) anions in solution were reduced to Cr(III) ions by contacting the nitrogen groups on the surface of FCN-500. The reduced Cr(III) ions were adsorbed in FCN-500 by mainly chelating with aprotic nitrogen atoms in its surface. Another mechanism was surface precipitation that was confirmed by

the appearance of a peak at around 42° (involved in the crystal plane of Cr–N–Fe species) in the laden FCN-500.

Notably, under real electroplating wastewater conditions (C_o = 117 mg/L, 25 °C, m/V = 2.4 g/L, and pH = 3), FCN-500 can effectively treat Cr(VI) in this wastewater sample, with its removal efficiency reaching 99.93% after a 120 min contact.

3. Conclusions

Different materials were synthesized and used as potential adsorbents for removing a variety of adsorbates: inorganic and inorganic pollutants (especially some emerging pollutants) from aqueous solution, synthetic wastewater, and real wastewater samples. Various operational parameters affecting adsorption processes were instigated. Adsorption mechanisms were discussed and proposed.

However, all adsorption experiments [1–12] were conducted via a batch technique. With regard to real applications, adsorption phenomena under column conditions need to be continuously investigated. Current data for a leaching test and waste management after adsorption processes are missed. Further studies should be carried out to estimate the cost preparation of materials. Techno-economic analyses for treating pollutants from water via adsorption-based techniques are also suggested.

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