



Static sorption of phenol and 4-nitrophenol onto composite geomaterials based on montmorillonite, activated carbon and cement



Mohamed Houari^{a,b}, Boualem Hamdi^c, Omar Bouras^b, Jean-Claude Bollinger^{a,*}, Michel Baudu^a

^a Université de Limoges, Groupement de Recherche Eau Sol Environnement (GRESE), Faculté des Sciences & Techniques, 123 avenue Albert Thomas, 87060 Limoges, France

^b University of Blida, Faculty of Technology, Department of Chemical Engineering, P.O. Box 270, 09000 Blida, Algeria

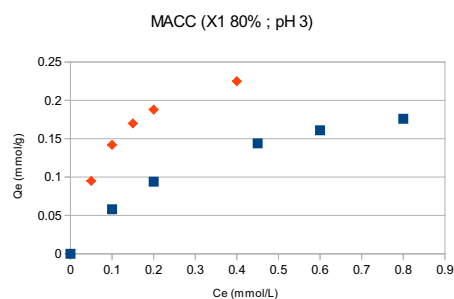
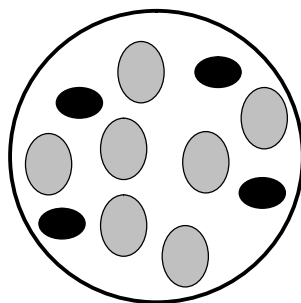
^c Laboratoire d'Etude Physico-Chimique des Matériaux et Application à l'Environnement (LEPCMAE), Faculté de Chimie, Université des Sciences & de la Technologie Houari Boumediene (USTHB), BP 32 El Alia, 16111 Alger, Algeria

HIGHLIGHTS

- Solid sorbents were produced by mixing montmorillonite, activated carbon and cement.
- These mesoporous sorbents prove to be efficient for phenol and 4-nitrophenol (4NP).
- These materials show enhanced sorption capacity relative to their pure components.
- According to the Langmuir model, sorption capacity for 4NP is higher than for phenol.

GRAPHICAL ABSTRACT

Schematic view of MACC composites, produced by mixing montmorillonite (white), activated carbon (black) and cement (grey); they are efficient sorbents for phenol (blue squares) and 4-nitrophenol (red diamonds).



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ABSTRACT

This paper studies the sorption of phenol and 4-nitrophenol (4NP) onto solid sorbents derived from mixtures of montmorillonite, activated carbon (AC) and cement, denoted herein MACC. These materials are mesoporous and some of their physicochemical properties have been measured and discussed. The weight fraction X_1 (%) of montmorillonite in the mixtures strongly influences the sorbate removal rate. The sorption isotherms were experimentally established by batch testing on geomaterials with various X_1 values at 20 °C and at different pH values (from 3 to 8). The Langmuir model correctly fits the sorption isotherm data ($R^2 > 0.95$). The results show increased sorption behavior for both phenol and 4NP on the composite geomaterials compared to the pure components, yielding the order: MACC > AC > montmorillonite.

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1. Introduction

In industry as well as day-to-day life, substituted phenolic compounds are widely used. Due to their high stability, high toxicity

and carcinogenic nature however, they have caused considerable damage and threaten ecosystems both in water bodies and for human health. The efficient elimination of phenolic compounds in wastewater has thus become an urgent need. Conventional wastewater treatment techniques include photocatalytic degradation processes, chemical oxidation and biological digestion, in which sorption onto activated carbon constitutes one of the most

* Corresponding author. Tel.: +33 624 307 201; fax: +33 555 457 203.

E-mail address: jean-claude.bollinger@unilim.fr (J.-C. Bollinger).

widely studied technologies. Recent research has focused on the types and modification of clay-based sorbents, the corresponding sorption principles or mechanisms, and the factors influencing both sorption rate and kinetics [1,2].

Phenol, a small but important organic molecule, appears as an end or side group in a number of polymers. Furthermore, phenol is a frequent and toxic byproduct of industrial processes and thus causes concern from an environmental perspective. The use of activated carbon to remove phenol from aqueous solutions, for industrial and environmental applications, has been primarily motivated by a physisorption mechanism due to dispersive forces [3,4].

Phenol was chosen as the study target herein, given its status as an intermediate product in the oxidation pathway of higher molecular weight aromatic compounds. Mononitrophenols can be classified as compounds exhibiting moderate to high toxicity in the aquatic compartment [5]. Based on information gathered, 4-nitrophenol (4NP) will enter the environment during its production and then be used as an intermediate product in the manufacturing of parathion, methyl-parathion and N-acetyl-*p*-amino-phenol [6]. For this reason, it has been selected as a comparative model molecule in the present study.

Swelling 2:1 clay minerals play an important role in a number of engineering and environmental processes. These clays consist of negatively charged silicate layers; their negative charges are compensated by interlayer counterions, which along with the charged clay mineral surfaces strongly interact with polar solvents, most notably water. This interaction results in the well-known swelling of smectites and vermiculites. Clay swelling can lead to the sorption of organic molecules and an exchange of cations, two processes that are of environmental concern. The swelling properties of clay minerals are utilized in applications for their role as an adsorbent, ion exchanger and molecular sieve catalyst [2,7].

In recent years, reusable organo-inorgano-clay sorbents have been increasingly introduced for their capacity to efficiently remove phenols and many micropollutants from wastewater, as well as for their good sorbent properties and mechanical stability [8–25]. This heightened interest stems from the adsorption properties and high specific surface area of clay, the high volume of micropores and the broad range of surface functional groups introduced into clay structures.

With dependable availability in nature, clay is able to remain inexpensive. The purpose of the present investigation is to synthesize geomaterials (MACC) based on two sorbent materials with very distinct properties (namely, Na-montmorillonite and activated carbon) and a binder, i.e. cement [26–28]. These constituents were chosen in order to associate a convenient mechanical stability (contributed by the cement and partially by the clay) with the complementary sorptive properties of both the hydrophilic clay and the lipophilic activated carbon.

This combination of adsorptive capacities for clay and activated carbon has been examined by assessing the extent of sorption and by removing two model phenolic compounds: phenol and 4NP were chosen herein given their status as two of the most challenging priority pollutants to be eliminated from waste streams and groundwater.

The porous nature of these geomaterials and their high internal surface area constitute favorable properties for sorption. A more extensive investigation of the effect of the montmorillonite weight fraction on sorption kinetics has also been undertaken. An attempt has been made herein to apply a simple sorption model to predict both the sorption isotherms and kinetics.

2. Materials and methods

All reagents were of analytical grade and used without further purification. The various solutions were prepared in high purity

de-ionized water (HPW) (Milli-Q system: resistivity of 18.2 M Ω cm, TOC < 10 μ g/L). All glassware and containers were previously soaked in 10% (v/v) nitric acid for at least 24 h and then rinsed with HPW.

2.1. Sorbates

Phenol ($\geq 99\%$) and 4NP ($\geq 99\%$), purchased from Sigma-Aldrich, were chosen as the sorbates; they were used without further purification. A number of useful physicochemical properties are listed in Table 1 [29,30].

2.2. Sorbents: starting materials

Bentonite from the Maghnia deposit (western Algeria), obtained from the ENOF Company (Algeria), was used as a starting material; it is composed essentially of montmorillonite (75%) with minor impurities (quartz, feldspar, calcite, etc.). Its cation exchange capacity of 86.5×10^{-5} eq/g was determined according to the sodium acetate saturation method [31], applicable to materials containing appreciable amounts of clays. Moreover, its chemical composition and purification method were described previously [27] and will be briefly summarized herein. The raw bentonite was dispersed in 1 mol/L NaCl solution in order to obtain homoionic Na-montmorillonite and then separated from the solution and washed several times with high-purity water (HPW). A 2 wt.% suspension was placed in graduated cylinders to allow particles >2 μ m to settle. The suspension at a depth of 10 cm was collected with an Andreason pipette. This operation was repeated several times until the suspension became almost transparent at the 10-cm depth. The particles collected smaller than 2 μ m were recovered by centrifugation, washed with HPW and finally dialyzed to eliminate the excess chloride ions. After drying 48 h at 40 $^{\circ}$ C, the sample was pulverized to pass through a 200- μ m sieve. This fraction was used in the geomaterial preparation process.

The activated carbon used is a commercially available activated wood coal (Fluka) with a microporous structure; it was ground and sieved at <20 μ m before use. The cement (produced at the Chlef plant, in western Algeria) is a fine powder containing 94.8% w/w SiO₂. The main properties of these raw solid sorbents are shown in Table 2.

2.3. Preparation of geomaterials

The composite geomaterials (MACC) were prepared by mixing the 3 constituents Na-montmorillonite ($X_1\%$ w/w), activated carbon (X_2) and cement (X_3) with water. Three different weight fractions of Na-montmorillonite ($X_1 = 60\%$, 70%, 80%) were mixed with a fixed amount $X_2 = 7\%$ of activated carbon and complementary fractions of cement (i.e. $X_3 = 33\%$, 23%, 13%, respectively) under mechanical mixing at medium speed (40 rpm) (W49121 powder

Table 1
Main physicochemical properties of the sorbates.

Compound	Phenol	4-Nitrophenol
Molar mass (g/mol)	94.11	139.11
Solubility in water (g/L) ^a	84.0	15.6
Log P_{ow} ^b	1.50	1.91
pK _a ^c	9.92	7.15
V_{mol} (mL/mol) ^d	77.5	94.9
λ_{max} (nm) ^e	269	285

^a At 25 $^{\circ}$ C; from Achard et al. [29].

^b Lipophilicity in octanol/water; from Abraham et al. [30].

^c Acidity constant; cited in Achard et al. [29].

^d McGowan's characteristic molar volume; from Abraham et al. [30].

^e Characteristic wavelength for detection; this study.

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