



Cysteine-grafted nonwoven geotextile: A new and efficient material for heavy metals sorption – Part B



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ABSTRACT

The development of a new material designed to trap heavy metals from sediments or wastewater, based on a polypropylene non-woven covalently grafted with cysteine, has been reported in a previous paper (Part A). The non-woven was first functionalized with acrylic acid (AA) which is used as spacer, and then cysteine was immobilized on the substrate through covalent coupling in order to obtain the so-called PP-g-AA-cysteine. Some preliminary heavy metals adsorption tests gave interesting results: at 20 °C for 24 h and in a 1000 mg/L heavy metals solution, PP-g-AA-cysteine adsorbs 95 mg Cu/g PP (CuSO₄ solution), 104 mg Cu/g PP (Cu(NO₃)₂ solution), 135 mg Pb/g PP (Pb(NO₃)₂ solution) and 21 mg Cr/g PP (Cr(NO₃)₃ solution). In this second part of the work, heavy metals sorption tests were carried out with Cu (II), Pb (II), and Cr (III) separately, in order to determine the sorption capacity of this new sorbent as a function of (i) the heavy metals concentration in the solution, (ii) the contact time with the solution, (iii) the pH and (iv) the ionic strength of the solution containing heavy metals. Moreover, the sorption capacity of PP-g-AA-Cysteine was studied using a polluted solution consisting of a mixture of these different heavy metals. An Electron Paramagnetic Resonance study was finally carried out in order to determine the coordination geometry in the environment of the copper trapped by the PP-g-AA-cysteine.

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1. Introduction

Currently, the accumulation of massive amounts of sediments leads to important economic and environmental issues. In order to maintain the depth of the navigational waterways, harbors and estuaries worldwide (Stronkhorst et al., 2003), and also to limit the remobilization of contaminants which are present in sediments (Eggleton and Thomas, 2004), dredging operations occur regularly. But most of these dredged sediments can be considered as waste as they contain harmful components such as organic compounds and heavy metals (Singh et al., 1998). Because of the high concentration in heavy metals such as Arsenic, Cadmium, Chromium, Copper, Mercury, Nickel, Lead, and Zinc (Gouzy and Ducos, 2008), these sediments must be put in storage centers. But as storage centers are

saturated, sediments have to be treated in order to remove heavy metals and to give them a second life.

Geotextiles are commonly used in filtration and drainage. The grafting of specific biomolecules provides heavy metals adsorption properties to the textile. Cysteine was chosen as a great number of chelating biomolecules (such as glutathione, phytochelatin and metallothioneins) contain a large amount of cysteine units (Cobbett, 2000; Klaassen et al., 1999; Maret et al., 1997; Zenk, 1996). The grafting of cysteine on polypropylene nonwovens (PP) was already carried out and optimized in our group and is detailed in the Part A of this study (Vandebossche et al., 2014).

In the present paper, a heavy metals sorption investigation is proposed, in order to determine the capacity of this new material, PP-g-AA-cysteine for short, to trap copper (II), lead (II), and chromium (III) in various experimental conditions, i.e. when initial metal concentration, contact time, pH and ionic strength vary. Electron Paramagnetic Resonance (EPR) analyses were also carried out on copper solutions and on PP-g-AA-cysteine – copper in order to determine the heteroatoms involved in the copper sorption.

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2. Materials and methods

2.1. Preparation of the optimized PP-g-AA-cysteine

Squares (5*5 cm²) were cut from polypropylene (PP) nonwoven INTN50 (50 g/m², provided by PGI nonwovens, France), washed and dried as already described in a previous paper (Vandebossche et al., 2013). Acrylic acid (purity 99.5%, provided by Acros Organics) was graft-polymerized on PP by a cold plasma process (PP-g-AA) also previously optimized and reported (Vandebossche et al. 2013). Then cysteine (purity ≥97%, provided by Sigma–Aldrich) was immobilized on PP-g-AA by a chemical coupling between the carboxylic acid group of acrylic acid and the amine group of cysteine using a carbodiimide: N-(3-dimethylaminopropyl)-N'-ethylcarbodiimide hydrochloride (purity ≥ 98%, provided by Sigma–Aldrich). The immobilization of cysteine was carried out according to the optimized conditions determined in the Part A (Vandebossche et al., 2014): PP-g-AA was first immersed in a solution containing the carbodiimide (1 h at 4 °C), and then immersed in a 0.229 mol/L cysteine solution for 28 h at 20 °C. Samples were then washed for 1 h in an ultrasonic bath, and for 6 h in a Soxhlet extractor with distilled water. Finally, PP-g-AA-cysteine samples were dried under vacuum for 16 h.

2.2. Preparation of the artificially contaminated solutions

Some artificially polluted solutions were prepared in order to determine the adsorption behavior of the PP-g-AA-cysteine as a function of the oxidation state, the ionic radius of the metal and the counter-ion. 1000 mg/L solutions of copper sulfate, copper nitrate, lead nitrate and chromium nitrate were prepared in a 1 L-volumetric flask, and the 5000 mg/L solutions were prepared in a 100 mL-volumetric flask. From the solutions containing 1000 mg/L heavy metals, various contaminated solutions were prepared, with the following concentrations: 100 mg/L, 250 mg/L, and 500 mg/L.

In order to prepare the solutions containing 1000 mg/L and 5000 mg/L of heavy metals, copper sulfate solution was diluted in distilled water (conductivity = 0.2 μS/cm) while copper nitrate, lead nitrate and chromium nitrate were dissolved (see supporting data, table S1) using these initial reagents: 0.1 M CuSO₄·5H₂O (provided by Sigma Aldrich), Cu(NO₃)₂·xH₂O (purity = 99.999%, provided by Sigma–Aldrich), Pb(NO₃)₂ (purity ≥ 99.0%, provided by Sigma–Aldrich), Cr(NO₃)₃ (purity = 99.0%, provided by Sigma–Aldrich).

Finally, a solution containing 1000 mg/L copper, 1000 mg/L chromium and 1000 mg/L lead was prepared by dissolving respectively 2.951 g of Cu(NO₃)₂, 7.700 g of Cr(NO₃)₃ and 1.600 g of Pb(NO₃)₂ in distilled water in a 1 L-volumetric flask.

2.3. Sorption studies

2.3.1. Chelation of heavy metals and sorption studies

One cysteine-grafted polypropylene sample was immersed for 24 h in 100 mL of the previously described heavy metal solution thermostated at 20 °C. The sample was then washed in a bath of 50 mL of ultrapure water (conductivity = 0.055 μS/cm) in order to remove heavy metals located at the surface which did not interact with cysteine or acrylic acid. Then the textile was digested with sulfuric acid at 95% and hydrochloric acid at 37% (provided by VWR BDH Prolabo) to remove heavy metals which interacted with the surface. Ultrapure water was added to the solution, and the concentration of the diluted heavy metal containing solution was determined using flame atomic absorption spectrometry (Thermo Solaar S4 AA Spectrometer, Thermo S Series, Multi-elements combined coded hollow-cathode lamp for Cr–Cu–Mn–Ni,

Thermo Scientific and Mono-elements coded hollow-cathodes lamp for Pb, Thermo Scientific).

2.3.2. Preparation of the solution for the calibration of the atomic absorption spectrometer

The determination of the concentration of heavy metals in the solution obtained after the digestion of the metals-chelated-textile was carried out by the standard addition method: the analysis was first made with a blank, and then with the solution to analyze of known dilution, and then with the same solution in which 1, 2, or 3 mg/L of heavy metals were added.

2.3.3. Influence of the metal concentration

Cysteine-grafted-polypropylene samples were immersed in 100 mL of 100 mg/L, 250 mg/L, 500 mg/L, 1000 mg/L, and 5000 mg/L contaminated solutions at 20 °C and pH = 4.5 for 24 h.

2.3.4. Kinetic studies

Cysteine-grafted polypropylene samples were immersed in 100 mL of 1000 mg/L contaminated solutions (Cu II, Pb II, Cr III) at 20 °C and pH 4.5. For each heavy metal, the amount of metal trapped by the functionalized textile was determined as a function of time. It was then possible to determine the kinetic law of adsorption followed by the grafted surface.

2.3.5. Influence of pH

Cysteine-grafted-polypropylene samples were immersed for 24 h in 100 mL of 1000 mg/L contaminated solutions at 20 °C. 1M HCl or 1M NaOH was added in order to reach the following pH: 2.7, 4.5, 6.0, or 8.0.

2.3.6. Influence of the sodium concentration

Cysteine-grafted-polypropylene samples were immersed in 100 mL of 1000 mg/L contaminated solutions in which NaCl (Sodium chloride for analysis EMSURE® ACS, ISO, Reag. Ph Eur, Merck) was added to reach the following concentrations: 10, 20, or 30 g/L. These experiments were carried out at 20 °C and pH = 4.5.

2.3.7. Influence of a mixture of heavy metals in the solution

Cysteine-grafted-polypropylene samples were immersed in 100 mL of an artificially contaminated solution containing 1000 mg/L of copper, 1000 mg/L of lead and 1000 mg/L of chromium (for all these heavy metals the counter-ion was NO₃⁻) at pH = 4.5, 6, 7 and 8 in presence of 30 g/L NaCl or without NaCl.

2.4. Physical characterizations of the grafted textile

2.4.1. Langmuir isotherm

According to the classification of Brunauer et al. (1940), the Langmuir isotherm is the first physical model of sorption (type I). It is an empirical sorption model (Foo and Hameed, 2010) which characterizes the adsorption of a monolayer of compounds on a microporous material. In this model, (i) the energy is assumed to be the same all over the surface, (ii) the trapped molecules are assumed not to interact together or migrate over the surface and (iii) at the maximum of sorption, only one monolayer can be formed on the free surface. In the case of the adsorption on a solid surface of a molecule from a solution, the Langmuir isotherm model can be expressed according to the following equation (Langmuir, 1918):

$$q = q_m * b * C / (1 + b * C) \quad (1)$$

where q (mg/g PP) is the sorption capacity, q_m (mg/g PP) is the maximum sorption capacity, C (mol/L) is the concentration of heavy metal in the solution, and b (L/mol) is the Langmuir constant. In this

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