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Research article

Lead removal from aqueous solutions by raw sawdust and magnesium pretreated biochar: Experimental investigations and numerical modelling

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ABSTRACT

equilibrium model.

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1. Introduction

Among heavy metals, lead is considered as one of the most toxic substances. Indeed, even at low concentrations, children exposure to lead could damage their nervous system, kidneys, liver and reproductive system (Cechinel et al., 2013). Moreover, lead is a very persistent compound since it is not easily biodegradable and tends to be accumulated in living tissues which further endangers the health of human beings throughout the food chain. The toxico-kinetic behavior of lead(II) ion in human body mainly depends on its binding with the albumin protein. It was reported that the affinity of lead(II) for albumin protein is very high (Ayranci and Duman, 2004a). The binding of lead(II) to albumin protein is reversible and the unbound ions transfer easily from blood to tissue or organ. Therefore, lead(II) could cause several damages to some tissues and organs (Ayranci and Duman, 2004b).

Continuous efforts have been performed the last decades in order to ensure an effective heavy metals removal from natural and industrial wastewaters. Various technologies have been tested such as chemical precipitation, ion exchange, reverse osmosis and membrane filtration (Mlayah and Jellali, 2015). Even if these methods are relatively efficient in removing heavy metals under certain experimental conditions, they are found to be very expensive since they require high capital, operational and maintenance costs. For these reasons, adsorption onto natural as well as modified materials has been pointed out as an emerging, simple and economically feasible and attractive method for lead removal from wastewaters (Wang et al., 2015). This technology has also the main advantage of reducing the production of chemical and/or biological sludge and offering the possibility of the adsorbents regeneration and metal recovery for a further subsequent use. Several organic and mineral materials have been efficiently tested for lead removal from aqueous solutions such walnut sawdust (Bulut and Tez, 2007), activated carbon originated from cow bone (Cechinel et al., 2013), peanut shell's biochar (Wang et al., 2015), Fe- and Al-based water treatment residues and dead calcareous skeletons (Lim and Aris, 2014) and powdered marble wastes (Mlayah and Jellali, 2015).

Lead removal from aqueous solutions by raw cypress (Cupressus sempervirens L.) sawdust (RCS) and its

derivative magnesium pretreated biochar (Mg-B) was investigated under static and dynamic conditions

through batch and column assays. The Hydrus-1D model was used to estimate the transport parameters

of the lead measured breakthrough curves. The batch experiments results showed that Mg-B was very

efficient in removing lead compared to RCS and several other previously tested natural and modified materials. The column experiments results indicated that for both RCS and Mg-B, lead breakthrough curves and the related removal efficiencies were mainly dependent on the used initial concentration and

the adsorbents bed height. The use of Hydrus-1D showed that the two-site chemical non-equilibrium

model describes better the experimental lead breakthrough curves for both RCS and Mg-B as the



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Agricultural residues and sawdust have been identified as promising organic materials for heavy metals removal since they are available in abundance, renewable and low in cost (Abdolali et al., 2014; Chakravarty et al., 2010; Ofomaja et al., 2010). The heavy metals removal efficiencies of the raw lignocellulosic materials could be significantly increased if they are chemically and/or thermally treated (Calero et al., 2013; Cechinel et al., 2013; Do and Lee, 2013: Xue et al., 2012). In this context, biochars derived from the pyrolysis (in a closed container in absence of air) at relatively high temperatures (>500 °C) of various agricultural and forestry residues have been successfully used for the removal of both organic and inorganic contaminants from water (Hu et al., 2015; Zhang and Gao, 2013). Recent research works have demonstrated that the chemical pretreatment of biomasses (before the pyrolysis step) with specific reagents such as lanthanum hydroxides; lanthanum chlorides, magnesium chlorides, aluminum chlorides, highly improved the adsorption capacities of some pollutants such as arsenic, methylene blue and phosphates (Hu et al., 2015; Zhang and Gao, 2013). The improvement of the related adsorption capacity compared to the raw materials was mainly attributed to the deposition of nanomaterials on the adsorbents surface, the increase of their surface area, internal porosity and cation exchangeable capability (Wang et al., 2015).

On the other hand, the majority of lead removal studies from aqueous solutions by raw or treated lignocellulosic materials were carried out under static conditions (batch mode) with small scale devices. This kind of experiments, even if they permit to quantitatively determine the influence of some physicalchemical key parameters on the biomasses removal capacities, their results extrapolation to higher scale or to real sites is often very complicated (Bulgariu and Bulgariu, 2013; Long et al., 2014). Laboratory experiments under dynamic conditions using columns or continuous stirring tank reactors are more realistic and could be very helpful for the prediction of pollutants transport and propagation in real sites especially when using appropriate numerical models (Bulgariu and Bulgariu, 2013; Köhne et al., 2009).

Several numerical models have been used for the prediction of inorganic and organic pollutants transport at laboratory scale, in real sites and for hypothetical pollution cases (Engelhardt et al., 2015; Jacques et al., 2008; Jellali et al., 2010; Köhne et al., 2009; Suarez et al., 2007). They demonstrated that the rates of pollutants migration depend on a wide range of physical and chemical porous media characteristics such as permeability, porosity, dispersivity, interconnectivity of macropores, mineralogy and organic constitution (Jacques et al., 2008; Köhne et al., 2009). The models that successfully predict the transport of the studied chemicals are found to be those taking into account the physical and/or chemical non-equilibrium processes related mainly to both heterogeneous flow and time dependent adsorption process (Hanna et al., 2010; Suarez et al., 2007).

The main objectives of the present study were: i) to experimentally quantify, in both static and dynamic conditions, the efficiency of raw cypress (*Cupressus sempervirens* L.) sawdust (RCS) and its derivative MgCl₂ pretreated biochar (Mg-B), for lead removal from aqueous solutions under different experimental conditions, ii) to evaluate the impact of the used lead(II) concentrations, feeding flow rates and bed heights on the experimental breakthrough curves and iii) to assess the non-equilibrium processes importance in lead transport through the studied adsorbents for all the experimental conditions cited above using the Hydrus 1D model.

2. Materials and methods

2.1. Used adsorbents and their characterization

In this study, the RCS wastes were collected from a wood carpentry located at Menzel Bouzelfa city, in the province of Nabeul (North East of Tunisia). These wastes were firstly sieved using a mechanical sieve with a mesh width of 2 mm in order to remove larger fragments. Then, they were washed with tap water and air dried for several days to a constant weight. The Mg-B was the result of the five following treatment steps: 1) preparation of magnesium chloride solutions by dissolving 200 g of MgCl₂·6H₂O in 1 L distilled water, 2) shaking of 40 g of RCS in 400 mL of the MgCl₂ synthetic solution during 4 h at 600 rpm using an AGIMATIC-S shaker, 3) drying the mixture of biomass and MgCl₂ in an oven at 60 °C during 24 h to remove the excess of water, 4) heating the dry mixture of biomass and MgCl₂ with a temperature gradient of 5 °C min⁻¹ up to 600 °C under N₂ flow for 1 h, 5) sieving the resulting solid matrix (Mg-B). Only the fraction with a diameter between 0.3 and 0.5 mm was used for the lead removal experiments in order to minimize the presence of residual ash particles.

The BET specific surface areas of RCS and Mg-B were determined using a Nova 4000-e, Quantachrome, Boynton Beach, Florida, USA. The pH of zero point charge (pH_{ZPC}) values of the two solid matrixes were performed according to the solid addition method (Jaouadi et al., 2014) using 0.01 M KNO₃ solutions, 1 g of solid matrix for initial pH values of 2; 4; 6; 8; 10 and 12.

2.2. Lead solutions preparation and analysis

Analytical grade of lead nitrate $(Pb(NO_3)_2)$ was used in this study as the source of lead ions. A stock lead solution of 1000 mg L⁻¹ was prepared with distilled water and used throughout this study. The analysis of lead concentrations either in batch or in column assays was achieved using an atomic absorption spectrometer (Perkin Elmer AAnalyst200). In order to avoid lead precipitation as Pb(OH)₂, the initial aqueous pH values of the used solutions were adjusted to 4.0 using 0.1 M HNO₃ (Blazquez et al., 2010). The pH and electrical conductivity measurements during the experiments were performed using a pH meter (692 pH/Ion Meter, Metrohm) and a calibrated specific electrode (Windaus LF538), respectively.

2.3. Batch experiments-isotherm modelling

Batch experiments consisted in analysing the effect of initial lead(II) concentrations on its removal from synthetic aqueous solutions at equilibrium. The used initial lead(II) concentrations were 10, 20, 30, 40 and 50 mg L⁻¹ for RCS and 200, 300, 400, 500 and 600 mg L⁻¹ for Mg-B. These experiments were conducted at $20 \pm 2 \,^{\circ}$ C in 120 mL capped flasks. During these assays, 0.1 g of RCS or Mg-B was shaken in 50 mL of lead solution during 90 min at 400 rpm using a Varimag-poly15 magnetic stirrer. On the basis of preliminary experiments (data not shown), this time was found to be enough to ensure an equilibrium state characterized by constant lead residual aqueous concentrations.

The amount of adsorbed lead at equilibrium (S (mg g^{-1})) was calculated based on the difference between initial (C₀) and final aqueous concentrations (C_e) as follows (Jellali et al., 2011):

$$S = \frac{(C_0 - C_e)V}{M} \tag{1}$$

where V is the volume of the aqueous solution (L) and M is the mass of the used adsorbent (g).

The graph giving the variation of the adsorbed lead amounts

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