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FTIR determination of adsorption characteristics for volatile organic compounds removal on diatomite mineral compared to commercial silica

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Abstract

Gas phase FTIR measurement has being used to determine adsorbents characteristics such as adsorption capacity and isotherm. The experimental approach was applied to compare adsorption performances in terms of volatile solvent (*o*-xylene) adsorption and desorption for low cost diatomite mineral and commercial silica. The Langmuir equation was suggested to correlate the experimental isotherm. This assumption allowed an estimation of adsorbed molecular density within a monolayer, on the basis of the agreement obtained in *o*-xylene vapor pressure range where Langmuir model holds. ***To cite this article: H. Zaitan, T. Chafik, C. R. Chimie 8 (2005)***.

Résumé

Une méthode expérimentale basée sur la mesure de spectres IRTF en phase gazeuse enregistrés dans des conditions dynamiques (sous flux de gaz à la pression atmosphérique) a été utilisée pour la détermination des capacités et des isothermes d'adsorption. L'approche a été appliquée pour comparer les performances d'un minerai diatomite avec celles d'une silice commerciale en terme d'adsorption et de désorption d'un composé organique volatil de type *o*-xylène. Une estimation de la monocouche de l'*o*-xylène adsorbée a été faite en se limitant au domaine de pressions où le modèle de Langmuir s'applique. ***Pour citer cet article : H. Zaitan, T. Chafik, C. R. Chimie 8 (2005)***.

Keywords: Dynamical adsorption; Breakthrough curves; Adsorption isotherms; FTIR analysis; Diatomite; Silica; *o*-Xylene

Mots clés : Adsorption dynamique ; Courbes de percées ; Isothermes d'adsorption ; Analyses IRTF ; Diatomite ; Silice ; *o*-Xylène

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1. Introduction

The treatment of volatile organic compounds' (VOCs) emissions in industrial plant is subject of great concern because of its harmful effect on human health and environment [1]. Removal technology based on adsorption is chosen mainly because of high elimination efficiency as well as economical advantages such as solvent recovery for reuse and low energy cost [2,3]. For such applications, activated carbon is widely used regarding its properties [4]. Nevertheless, such adsorbents become inappropriate if the adsorbed solvents to be recovered are flammable [5]. In this case materials like zeolites, alumina or polymers become of interest [6–9]. Hence continuous efforts are focusing on investigating adsorbent material that associate efficiency and low cost.

The present work aimed at investigating a local mineral, known as diatomaceous silica or kieselguhr, and valorizing it as a VOC adsorbent. This material gave promising results during its application for removing water pollutants such as atrazine, organophosphorus pesticides or silver [10,11]. In this study *o*-xylene was chosen as a representative of VOC, regarding its multiple uses in many chemical industries. The adsorption performances of the mineral were compared to those of silica, which was the main component of diatomite. The mineral presented an economic advantage in terms of a lower cost of 0.25 \$ kg^{-1} [12], as compared with a price of about 60 \$ kg^{-1} for silica aerosil.

The main objective of this work was to illustrate the suitability of an easy and efficient method for adsorption characteristics' investigations. The experimental approach is based on gas phase FTIR monitoring during dynamical adsorption and desorption. The obtained adsorption capacities yield to adsorption isotherm. The latter allowed, furthermore, an estimation of the adsorbed *o*-xylene monolayer in the range of *o*-xylene partial pressures where Langmuir equation holds.

2. Experimental

2.1. Experimental apparatus and methods

Dynamic adsorption/desorption experiment were carried out using a home-made device consisting of an assembly (Fig. 1) that permits (*i*) to prepare model gas mixture and flow rate control, (*ii*) to perform in situ pre-treatment, adsorption and desorption tests in quartz micro-reactor, (*iii*) to monitor *o*-xylene concentration in the gas flow at the reactor outlet using FTIR spectrometry or gas chromatography.

The experiments were carried out under dynamic conditions at atmospheric pressure, with respective masses of 1 and 0.1 g of diatomite and silica. Nitrogen was used as carrier gas to make *o*-xylene/N_2 mixture. The latter, was sent into the quartz reactor through six way valve V_{6H}. The concentration of *o*-xylene in the mixture was obtained using a saturator (S1) associated with a condenser (C1) immersed in a thermostatically controlled bath. This temperature was permanently checked so as to maintain constant *o*-xylene vapor pressure and consequently keep the *o*-xylene concentration unchanged. Attention was paid to hold always the *o*-xylene partial pressure lower than the vapor pressure value at adsorption temperature in order to avoid *o*-xylene condensation in gas phase. In addition, all tubes, valves and connections of the experimental apparatus were permanently heated.

Prior to each set of adsorption–desorption experiment, the solids were treated under air flow in the quartz micro-reactor. The treatment consists in gradually heating ($5\ K\ min^{-1}$) until 473 K for diatomite and 573 K for silica, and maintaining the final temperature for 1 h before cooling to room temperature. Following this pretreatment, the adsorption step was carried out with a flow of $100\ cm^3\ min^{-1}$ of *o*-xylene/N_2 model mixture. Once the solid saturated, the gas mixture was shifted again to N_2 flow to proceed with isothermal desorption until the *o*-xylene response reached zero. Subsequent desorption under a progressive heating was necessary to achieve complete regeneration of the solids. During all these steps (1 cycle), the response of the system was monitored by measuring the gas mixture composition (X_{out} *o*-xylene molar fraction or partial pressure) at the reactor outlet. Note that *o*-xylene molar fraction X_{in} before contact gas/solid correspond to P/P_0, where P is vapor pressure of *o*-xylene and $P_0 = 760$ Torr. The *o*-xylene concentration in the gas stream at the reactor outlet was monitored with a FTIR Jasco 410 spectrometer (resolution of $4\ cm^{-1}$) using a Pyrex gas cell equipped with CaF_2 windows. This was achieved owing to multiples advantages offered by FTIR instrumentation such as rapid spectra acquisition and easy quantitative analysis, especially in case when they are no IR