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# Adsorption and photophysical properties of fluorescent dyes over montmorillonite and saponite modified by surfactant

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## HIGHLIGHTS

- Na-Mt and Na-Sap were prepared and exchange with cationic surfactant CTAB.
- The adsorption of Rhodamine, kiton red and sulforhodamine on these materials was studied.
- Whatever the material the fluorescent dyes are not located inside interlamellar space.
- The dyes are released in water as monomer even for the kiton red.

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## ABSTRACT

In the present study, the adsorption capacities of two intercalated smectites, CTAB-saponite and CTAB-montmorillonite with a cationic surfactant, were investigated with three fluorescent dyes namely Rhodamine 640 perchlorate rhodamine (Rho), sulforhodamine B (SR) and Kiton red 620 (KR). The adsorption isotherms fit well with the non-linear Langmuir isotherm model and the maximum adsorption capacities of all the composites are determined. The photophysical properties such as anisotropy and fluorescence lifetime of all the fluorescent dyes over the clay materials are determined. The set of experimental data based on X-Ray diffraction (XRD), transmission electron microscopy (TEM), Thermal analysis (TG-DTA) and fluorescence measurements allow highlighting the presence or the absence of interactions between the dyes and the modified clay minerals.

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## 1. Introduction

The adsorption of dyes have been largely investigated on clay minerals (Thangaraj et al., 2016; Fournier et al., 2016). The later have layered structure composed of octahedral and tetrahedral sheets consisting of fine particles of size <2 μm. Each tetrahedron consists of a cation, T, coordinated by four oxygen atoms and linked to adjacent tetrahedral by sharing three corners (the basal oxygen atoms, Oxb) to form an infinite two-dimensional 'hexagonal'. Each octahedron consists of a cation, O, coordinated by six oxygen atoms and linked to neighboring octahedral by sharing edges. They have been employed in several applications such as adsorbents, catalysts and host materials (Jaber et al., 2014). The surface properties of clay minerals were determined by the number of their active sites and the type of their exchangeable inorganic cations present in the interlayer space, their composition, the substitution ratio and its localization in the layers. These properties with the ability to swell in water and presence of both Bronsted and Lewis acidity make the clays possible excellent host materials for a wide range of organic and bio-molecules (Raha et al., 2009; Tanabe, 1981; Balme et al., 2013; Jaber et al., 2002; Lepoitevin et al., 2014). The Bronsted acidity is due to the formation of $H^+$ ions on the surface by dissociation of

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hydrated exchangeable metal cations of clays in water. This may also arise from a negative net charge on the surface by the substation of $Al^{3+}$ instead of $Si^{4+}$ at some tetrahedral positions and the net charge is balanced by $H_3O^+$ cations. Clays are acting as an electron acceptor in most of the electron transfer process and this Lewis acidity character arises due to the external surface of the clay build-up of outer surface and lateral surface (edges). In these cases mostly $Al^{3+}$ arising from rupture of Si-O-Al bond or $Al^{3+}$ at the edges, or through dehydroxylation of some Bronsted acid sites (Wang et al., 2014; Pankasem and Thomas, 1991). The edges and the faces of clay particles are responsible to adsorb cations, anions, nonionic and polar contaminants from natural water. Adsorption of pollutants over the surface of clay may occur through ion exchange, ion-dipole interaction or coordination process from either strong or weak interactions (Srinivasan, 2011; El Adraa et al., 2017).

Industries are main source of water pollution and it produces toxic pollutants that are harmful to humans, animals and water bodies. One of such important pollutant is dyes; colored compounds are widely used in many industries such as textile, leather, pharmaceutical, paper, tanning, pulp etc. Some of dyes are carcinogenic and not biodegradable (Vidhyadevi et al., 2013; Premkumar et al., 2013). Various physicochemical and biological treatment methods have been reported to remove organic dyes from wastewater. Adsorption is proven to be a practical way for the treatment of dye wastewater due to its efficiency, selectivity and cost effective nature (Vidhyadevi et al., 2014). The modification of clay minerals with organic molecules such as surfactant (He et al., 2010; Guegan, 2010) has been widely reported to increase the affinity of these materials with organic pollutants (Nourmoradi et al., 2012; Sanchez-Martin et al., 2006; Klumpp et al., 1993).

Among different types of dyes, molecules of Xanthene family are commonly used for their photophysical properties and have vast applications in sensors based thin films, in biological systems, in laser technology, in supramolecular chemistry etc. The three fluorescent dyes (Fig. 1) namely rhodamine 640 (Rho), sulforhodamine 640 (SR) and Kiton red 620 (KR) are selected for the current study due to their very good quantum efficiency and their photochemical stability.

The aim of the present work is to study the efficiency of different smectites in the adsorption of three fluorescent dyes *i.e.* Rho, SR, and KR. The smectites used were synthetic sodium montmorillonite (Na-Mt) and sodium Saponite (Na-Sap) and their equivalent modified with cetyltrimethylammonium bromide, a cationic surfactant (CTAB). The dyes location has been studied by XRD and TEM. The adsorption isotherms have been conducted to evaluate the adsorption mechanism and to evaluate the adsorption capacities of the clays. The photophysical properties of the adsorbed dyes were investigated in aqueous solution by the time resolved fluorescence and anisotropy of fluorescence.

## 2. Experimental section

### 2.1. Materials

Four different clays namely synthetic Montmorillonite (Na-Mt) and (CTAB-Mt), sodium Saponite (Na-Sap) and (CTAB-Sap) were used for the confinement of fluorescent dyes namely rhodamine 640 perchlorate (Rho), sulforhodamine 640 (SR) and Kiton Red 620 (KR) from aqueous solution. All dyes were purchased from Exciton.

### 2.2. Preparation of clays

#### 2.2.1. Montmorillonite synthesis

Hydrofluoric acid (HF, 40% w/w; Fluka), sodium acetate ($NaCOOCH_3$, 99%; Fluka), magnesium acetate ($Mg(COOCH_3)_2\ 4H_2O$, 99%; Fluka), boehmite ($Al_2O_3$, 75,278%, Pural SB1; Condea), and silica ($SiO_2$, 99.5%, Aerosil 130; Evonik) were used to prepare a hydrogel with the following chemical composition $1SiO_2$: $0.15{\cdot}Al_2O_3$: $0.2{\cdot}MgO$: $0.1{\cdot}Na_2O$: $0.05{\cdot}HF$: $96{\cdot}H_2O$ (Georgelin et al., 2013). The hydrogels were matured over a 2 h period at room temperature and then introduced into a PTFE-lined stainless steel autoclave. The autoclaves were heated at 200 °C for 72 h. After reaction, autoclaves were cooled to room temperature. Run products were then recovered by filtration, washed thoroughly with distilled water and dried at 60 °C for 12 h. The ideal formula per half unit cell is $Na_{0.2}(Si_4)(Al_{1.8}Mg_{0.2})O_{10}(OH,F)_2]$ with a theoretical octahedral substitution rate (*i.e.* the number of $Mg^{2+}$ to $Al^{3+}$ substitution per half-cell) of 0.2, yielding a cation exchange capacity of 0.382 meq $g^{-1}$ (Cornu et al., 2017).

Saponite was synthesized according to a previously published procedure having the following chemical formula: $Na_{0.4}[(Si_{3.6}Al_{0.4})(Mg_3)O_{10}(OH, F)_2]$. That is, the Al substitution rate expressed per full unit cell is x = 0.8 (Jaber and Miehe-Brendle, 2005). Hydrofluoric acid (HF, 40% w/w; Fluka), sodium acetate ($NaCOOCH_3$, 99%; Fluka), magnesium acetate ($Mg(COOCH_3)_2\ 4H_2O$, 99%; Fluka), aluminum basic diacetate (19%, sigma aldrich), and silica ($SiO_2$, 99.5%, Aerosil 130; Evonik) were used to prepare a hydrogel with the following chemical composition $1SiO_2$: $0.15{\cdot}Al_2O_3$: $0.2{\cdot}MgO$: $0.1{\cdot}Na_2O$: $0.05{\cdot}HF$: $96{\cdot}H_2O$. The hydrogels were matured over a 2 h period at room temperature and then introduced into a PTFE-lined stainless steel autoclave. The autoclaves were heated at 200 °C for 72 h. After reaction, autoclaves were cooled to room temperature. Run products were then recovered by filtration, washed thoroughly with distilled water and dried at 60 °C for 12 h.

#### 2.2.2. Cation-exchange montmorillonite and saponite

Na-Mt or Na-Sap was exchanged with cetyltrimethylammonium bromide (CTAB). The clay mineral was added in a large amount of the surfactant solution (0.1 mol $L^{-1}$) and kept under stirring. A ratio

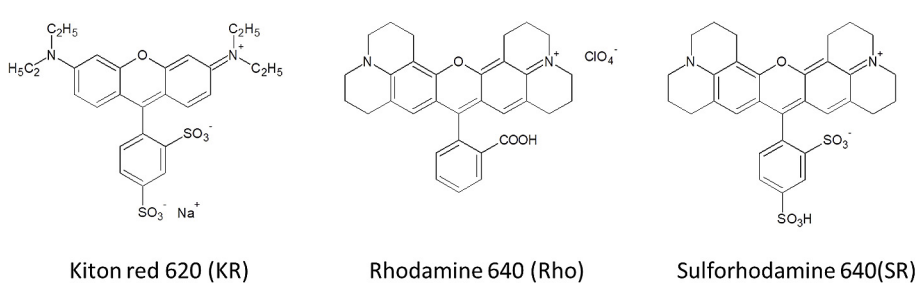


**Fig. 1.** Structure of dyes used in this study.

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