



Organophilic nickel phyllosilicate for reactive blue dye removal

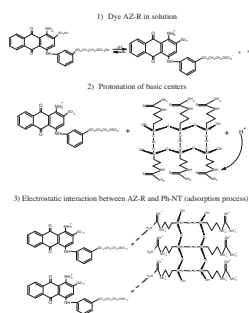
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HIGHLIGHTS

- The new organophilic nickel phyllosilicate was synthesized.
- The maximum adsorption occurred at pH 3, 300 min and 318 K.
- The capacity of adsorption was 179.9 mg g⁻¹.
- The data were better adjusted to the kinetic pseudo-second-order model.
- The adjustment was better to the model proposed by Langmuir.

GRAPHICAL ABSTRACT

Scheme of interaction of AZ-R dye by the phyllosilicate Ph-NT in an acid medium.



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ABSTRACT

A new organophilic nickel phyllosilicate (Ph-NT) was synthesized by sol–gel method, through the reaction with nickel nitrate and a new organosilane prepared from the reaction of 3-aminopropyltriethoxysilane and thiourea in a basic medium. The reaction effectiveness was confirmed by XRD, FTIR, elemental analysis, ¹³C and ²⁹Si NMR, and TG. The influence of contact time, pH temperature and concentration were investigated and the set of data have shown favorable results for employing the phyllosilicate as an adsorbent agent, and the maximum adsorption capacity using the optimized conditions was 179.9 mg g⁻¹ at 300 min, pH 3 and 318 K, which makes the novel functional surface a promising material for treating polluted aquatic environments. The experimental kinetics data were adjusted to pseudo second order model and the experimental isotherms data were better adjusted to Langmuir model.

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1. Introduction

In the last decades, environmental problems have become increasing critic and usual, mainly due to the population growth and intense industrial activity, being responsible for keeping the human activities based on natural resources under alert. This can be easily observed through the changes in the quality of soil, air and water [1].

In order to control the environmental pollution, especially regarding the contamination of water resources, the elimination of toxic chemicals present in effluent streams is one of the most important factors. Thus, is necessary to improve the growing research field in several areas, and we can highlight the chemistry and materials science to provide an efficient and low-cost alternative treatment for this issue [2]. The textile industry occupies a prominent position, since, depending on the process and equipment used, the quality of the final product and the type of fibers benefit, the water consumption can reach values between 25 and 250 m³ per ton of product, thereby generating large amounts of effluent [3,4].

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The fibers can be classified into two large groups called natural and synthetic fibers, and present different chemical structures and properties, which require a wide variety of dyes, and improved process for coloring fibers requires dyes with good features and well-defined properties [5]. Dyes can be classified by their chemical structure (anthraquinone, azo, etc.), or the method of fixing the textile fiber (reactive, direct, etc.). The interactions between dye molecules and the fiber is usually performed in aqueous media, where there are plenty of different chemical forces, such as ionic and covalent bonds, hydrogen bonds, and Van der Waals [6].

One of the major difficulties faced by the textile industry is centered in the control and removal of dyes present in wastewater [7]. According to a research conducted by the Industrial Ecotoxicology Association more than 90% of the 4000 dyes typically used in industrial processes show some degree of toxicity [8–10].

There are several methods of wastewater treatment for removal of textile dyes, such as coagulation or flocculation followed by sedimentation, photocatalytic oxidation, biodegradation, adsorption, among others [1,11–16]. The efficiency of each method are limited based on dye chemical structure to be removed, and this is the driving force for the constant development for new materials applied in physical, chemical and biological systems [1]. Among the several options mentioned for dye removal, the adsorption process is one of the powerful method that can be used for this purposes [15–18].

Adsorption can be defined as the enrichment of one or more components in the interfacial layer and may occur either as physisorption or chemisorptions [19,20]. Different materials are used as adsorbents, such as activated carbon, chitosan, cellulose, silicates, phyllosilicates, silicas, among others [21–26]. Usually, adsorbents are chemically modified, or functionalized, in order to increment their adsorption capacity [27–30].

A material is effective in the adsorption process not only when it exhibits a good affinity for the adsorbate, i.e. a great adsorption, but when it has a low application cost, is readily available, has simple operation and has an environmentally friendly approach. Phyllosilicates are examples of materials which have great ease in the adsorption process [31–33].

A set of properties makes silica-based having a large employment in the field of adsorption, such as thermal stability, anti-swelling, chemical inertia in organic solvent. Another possibility to increase the performance of this class of materials is the possibility of immobilize a variety of silylating agents for obtaining different functional groups, which becomes possible chemical surfaces tailoring, according to the chemical function and depending on the applied systems [34].

The silicates present an extremely stable structure, composed by tetrahedral units, represented by SiO_4 , depending on the arrangement they are grouped into Nesosilicates, Sorosilicates, Cyclosilicates, Inosilicates, Phyllosilicates and Tectosilicates [35]. Phyllosilicates are basically constituted of two types of sheet, the octahedral and tetrahedral ones, and each to them is composed of planes of atoms self-arranged one another [31–33,35]. There are slight variations of the physical and chemical properties of phyllosilicates, which are due to those structural arrangements and to electrostatic effects arising from chemical substitutions [35]. A typical example of phyllosilicate is talc, represented by anhydrous magnesium silicate with the formula $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$, consisting of on the octahedral layer with Mg^{+2} ions around two layers of silicate, this may be replaced by various other divalent cations such as nickel, which does not have any work described in the literature, with the silicon occupying tetrahedral sites [36]. These talc-like structures allow to modify the surface layer by inserting molecules in the lamellar space during the functionalization reaction, which consists in the intercalation reaction [37].

The insertion process of organic molecules in empty space of a lamella leads to formation of an inorganic–organic hybrid material,

which usually improves the adsorption properties. Talc-like phyllosilicate-hybrid materials have a trioctahedral 2:1 structure, with a structure similar to the natural talc, and present general formula $\text{Si}_8\text{Mg}_6\text{O}_{20}(\text{OH})_4$ [36,37].

The present work aims the synthesis of an organophilic nickel phyllosilicate with a new silylating agent, obtained from the reaction between the silane 3-aminopropyltriethoxysilane and thiourea through the sol–gel process; The sample was characterized by XRD, FTIR, elemental analysis, TG and ^{29}Si and ^{13}C NMR, and it was applied to adsorption of the reactive blue dye. Some parameters, such as pH, time, temperature and concentration were investigated in order to obtain the optimal conditions. Finally, the set of data were studied by different physical–chemical models, concerning the kinetics, thermodynamics and isotherms fitting.

2. Experimental

2.1. Materials

Silylating agent 3-aminopropyltriethoxysilane (N) (Aldrich), thiourea (T) (Synth), $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Vetec), NaOH and ethanol PA (Synth), HCl (Sigma), $(\text{NH}_4)_2\text{SO}_4$ (VERTEC), KNO_3 (VERTEC), Toluene (Sigma), reactive blue dye RN (RB) (Dystrar Ltd.), deionized water. Fig. 1 below shows the structure of the dye. All materials were used without prior purification.

2.2. Organophyllosilicate preparation

A new silylating agent was prepared by reacting 3-aminopropyltriethoxysilane (N) with thiourea (T), and was named NT. The organosilane NT was obtained from the addition of 10 mL of N silane (42 mmol) and 1.63 g of thiourea (21 mmol) in a flask with magnetic stirring, in the presence of 0.030 g of ammonium sulfate $(\text{NH}_4)_2\text{SO}_4$ [38–40]. The system was kept at 423 K for 15 h in a sand bath and the reaction took place under nitrogen flux. The reaction progress was monitored by the silica color change, from blue to black, kept in the drying tube, and the product yielded a yellow viscous liquid.

The nickel phyllosilicate was prepared by dissolving 8.2 g of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ in 75 ml of ethanol and mixing with a solution of 10 ml of NT dissolved in 80 ml of ethanol. Both solutions were placed together under stirring and 128 ml of a solution of sodium hydroxide 0.50 mol L^{-1} were added. The suspension was kept under stirring at 323 K and all the basic solution was added. After the addition of sodium hydroxide, the stirring was suspended and the solution was left to rest for 48 h [41]. Finally, the final solid was washed with deionized water, filtered and air dried for 24 h and under vacuum drying for 12 h. The nickel phyllosilicate containing the modified silane was named Ph-NT.

2.3. Point of zero charge (PZC)

In order to evaluate the optimum pH for dye adsorption, the point of zero charge of the sample was determined by the method of solid addition [42]. For this, 50 mL of KNO_3 0.10 mol L^{-1} were added to several beakers of 100 mL, numbered from 1 to 12. The

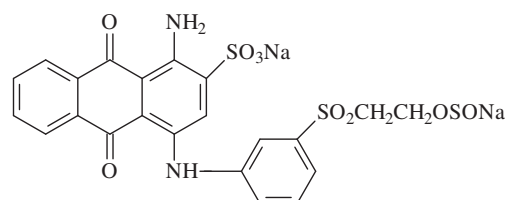


Fig. 1. Structure of reactive blue RN dye.

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