



Remediation of Phenol-contaminated Aqueous Solutions by Synthesized Organoclay

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ABSTRACT

Phenol and its metabolites are among the hazardous organic compounds, due to their carcinogenicity, toxicity, long term persistence in the environment. The purposes of this study are the synthesis of organoclay as an adsorbent with high physicochemical stability, environmental compatibility and its application for remediation of aqueous solutions contaminated with phenol. In this research, organoclay was synthesized by the combination of sodium bentonite and Cetyl Trimethyl Ammonium Bromide (CTAB) surfactant with three different Cation Exchange Capacities (CECs). As well as, adsorption capacity of synthesized organoclay was determined during reuse until saturation. The results revealed that the removal efficiencies of phenol at concentrations of 100, 1000, 2000, and 3000 mg/L were 68, 84, 82, and 80%, respectively. In general, the use of organoclay can be an alternative to the complex and expensive systems for the removal of phenol from aqueous solutions with respect to its simplicity, high performance, and cost-effectiveness.

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INTRODUCTION

In recent years, because of population growth, industry advancements and global challenges the use of oil and fossil fuels is inevitable. It is one of the most important sources of energy for many countries, including Iran. Organic petroleum compounds, despite their positive properties, have many destructive effects caused by their toxicity and cumulative properties, have many destructive and undesirable effects on the health of humans, animals, and the environment [1]. Drilling and production of oil, the accident of oil tankers, the explosion of oil wells and pipelines, overflow of wells, refining of crude oil and its transport are the most important sources for the entry of oil contaminants into safe aquatic environment such as Caspian Sea [2].

Petroleum Hydrocarbons (PHCs) in the sea enter into the sedimentary environment through biological and human activities (bacteria, animals, algae, etc.) and pollute the environment [3]. One of the most important oil pollutants is phenol and its compounds that are entered into the surface waters via industrial activities by means of industrial effluents. Because of carcinogenicity, toxicity, persistence in the environment for the long term, high solubility in water and biodegradability, they are harmful to human health and other organisms and even are lethal at low concentrations [4]. The World Health Organization (WHO) has set a maximum

concentration of phenol in drinking water at 1 microgram per liter, and United States Environmental Protection Agency (USEPA) has established an allowable concentration of phenol in surface water (rivers and lakes) at 0.3 milligrams per liter [5]. Thus, prevention of polluting soil and groundwater located around the contaminated and hazardous areas is of the great importance [6].

Various biological, physical, and chemical approaches are used for the removal of phenol. The basis of the chemical method is oxidation-reduction (Redox). Chemical oxidizers such as H_2O_2 and ClO_2 are used in this method so that the organic pollutants can be oxidized and converted to non-toxic or biodegradable materials for biological method. The rate of this method is more than the biological method, but the reaction rate goes down when used with many oxidizers such as ozone and hydroxide [7]. The biological method cannot be used at high concentrations of phenol because of its toxic effect on microorganisms, and it requires more time compared with other techniques; while its efficiency declines [8]. Among the physical removal methods, adsorption is the most widely used approach, in which contaminants in the water bind to the surface of solid adsorbents such as activated carbon, and the physical and chemical bonding is achieved [9-10]. Activated carbon is considered as one of the most widely used adsorbents of organic compounds with high adsorption capacity, but the high price of the adsorbent material limits its

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applications [11]. Hence, recent investigations are seeking to find alternative adsorbents for the removal of low-cost and accessible organic pollutants [12]. Of these adsorbents, different clay minerals are desirable adsorbents because of their low cost, abundance in nature, high surface area, and high adsorption [13]. Clay minerals have the reconstruction capabilities during adsorption-desorption periods, which leads to minimization of the process costs [14].

Organoclay (organophilic clay) arises from the substitution of organic cations in the structure of clay minerals. Unlike clay minerals that are hydrophilic, organoclays can be strongly both hydrophobic and organophilic. Organoclays are hybrids that absorb organic molecules between the clay mineral layers or on its surface [15]. The distance between the layers in the commonly used clay is low, but the distance between the layers is increased, and the environment becomes hydrophobic in organoclay due to the addition of compounds called surfactants. Also, the water outflows during the adsorption process and contaminant is trapped. The surfactant stands for surface-active-agent, where the organic part of the material is made up of a polar head (hydrophilic) and a nonpolar head (hydrophobic) [16]. The study on the interaction between clay and organic compounds were commenced since the beginning of the 21th century and has developed in various subjects. Sharma and Reddy [17] in their study referred to organoclay as a synthetic engineered material and pointed to its various applications such as use in landfills. The organoclays to remove the organic contaminants from an aqueous medium have been highly welcomed that the high chemical and physical stability of these materials, as well as their compatibility with the environment, are the primary reasons for using such materials [18]. additional research has been carried out on the adsorption properties of clay and organoclay. For example, Banat et al. [19] and Rauff et al. [20] investigated the removal of phenol and copper from aqueous solutions by using natural bentonite. Irene et al. [21] tried to increase the adsorption capacity of bentonite relative to phenol by using suitable surfactant on bentonite. Koh and Dixon using Na-montmorillonite clay, zeolite, and three types of surfactants, including BDTDA, Hyamine, and BTMA, made organoclays that the methodology used was cation exchange and centrifuge. BDTDA for the adsorption of benzene, BTMA for the adsorption of toluene, and Hyamine for the adsorption of phenol were used that the adsorption rate was achieved 70, 66 and 50%, respectively [22].

The removal of phenol was investigated in several studies conducted using the modified clay that the HDTMA surfactant was used in most of them. Furthermore, the effect of other surfactants such as DDTMA, DDDMA and Hyamine in the removal of phenol were studied, but CTAB surfactant was rarely used in investigations and its ability to remove phenol at very high concentrations was not evaluated. Therefore, in this study, the efficiency of organoclay synthesized with CTAB surfactant was assessed because of its convenient preparation and high potential in increasing hydrophilicity and adsorption of aromatic and phenolic compounds at various Cation Exchange Capacity (CEC). The independent variables of contact time, different concentrations of phenol (low, medium, and high) were also

examined, and the capacity and adsorption process of organoclay for reuse until saturation were determined.

MATERIALS AND METHODS

Clay

In this study, sodium bentonite was used for synthesis of organoclay and the initial rate of swelling (ASTM D854) and amount of CEC were 34 mL/2g and 85 meq/100g, respectively. To perform the CEC test, the soil sample was mixed with a large amount of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. As a result, the sample was washed with isopropyl alcohol. An ammonium acetate solution then added, which replaces the adsorbed sodium with ammonium. The sodium concentration of the displacement was determined by atomic absorption, emission spectroscopy, or an equivalent means.

Synthesis of organoclay

Initially, for the synthesis of organoclay, 5g clay was added to 400 mL of distilled water. Meanwhile, the value of CTAB was calculated according to the formula (molecular weight of surfactant \times g/mol clay value g \times CEC value \times 100%) and poured into 200 mL distilled water. Both of them were placed on the stirrer to well form a uniform solution in the distilled water. These two solutions were mixed and placed on the stirrer for 4 h. After that, the product was passed through a sieve and washed three times with distilled water and then with ethanol. Eventually, it was placed in a furnace for 48 hours. After the drying process, it was in the form of powder [23]. All three clays with different CEC percentages of 100, 200 and 300% were made in the same way, with the difference that the amount of surfactant for clays with a capacity of 200 and 300% CEC, was determined to be 2 and 3 times, respectively. The physicochemical properties of the initial and synthesized clay are illustrated in Table 1 and Figure 1, respectively.

TABLE 1. The physicochemical properties of the studied soils

Properties	Clay	Organoclay
Fine content % (<# 200 sieve)	90	86
Organic surfactant type	-	CTAB
Liquid limit	320	65
Specific gravity	2.41	1.88
Specific surface area (m ² /g)	380	161



Figure 1. The applied soil: clay (Left) and organoclay (Right)

Materials

All chemical compounds used in this research (Table 2) were prepared from Merck (Darmstadt, Germany).

Construction and commissioning of reactors and test methods

To create the contact of contaminant with the organoclay and investigate the removal rate at different times, along with its various components the experimental set up is shown in Figure 2. A 10 mL contaminant was mixed with 0.4 g organoclay (with different CEC) and poured into the tubes. In this study, the removal rates of concentrations of 100, 1000, 2000, and 3000 mg/L were investigated at 2, 30, 60, 120, 180, and 360 minutes. All tests were carried out in triplicate.

Measurement of phenol concentration

Phenol concentration was Measured using Section 5530D and standard methods tests [24]. After performing the test, the sample is placed in a centrifuge at 9000 rpm for 20 minutes, so that the suspended materials can be deposited and supernatant is taken to measure the removal rate of phenol. At the first, 5 mL of sample reached to 100 mL of volume with distilled water. The production of color in the sample represents the presence of phenol as the colorant (color-producing material). This procedure requires phosphate buffer solution, potassium ferricyanide solution, 4-Amino Antipyrine and ammonium hydroxide solution. The adsorption rate was determined by spectrophotometer at a wavelength of 500 nm, and the phenol concentration was determined using the calibration curve.

TABLE 2. Material properties

Materials Name	Chemical formula
Hexadecyltrimethylammonium bromide	$C_{19}H_{42}BrN$
Ethanol	C_2H_5OH
Phenol	C_6H_5OH
Potassium dihydrogen phosphate	KH_2PO_4
Dipotassium hydrogenphosphate	K_2HPO_4
Potassium ferricyanide	$K_3Fe(CN)_6$
Ammonium hydroxide solution	NH_4OH
4-Aminoantipyrine	$C_{11}H_{13}N_3O$

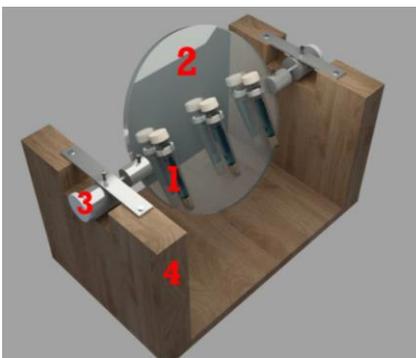


Figure 2. Experimental set up: 1- test tube, 2- test tube grip rack, 3- Gearbox motor, 4- leg

RESULTS AND DISCUSSIONS

Phenol removal rate

The results of phenol removal using organoclay with 100% CEC are illustrated in Figure 3. While using the organoclay, most of the contaminants will be removed in the early stage after 2 minutes. So that the removal efficiencies of 44, 46, 38, and 23% were observed for the contaminants at concentrations of 100, 1000, 2000, and 3000 mg/L, respectively. The removal process for each of the concentrations continued over time, and the amount of contaminant declined. Moreover, the adsorption rate initially increased with increasing the concentration of the contaminant, but decreased from a level onwards, because of organoclay earlier saturated at high concentrations. The results indicated that the removal rate was much higher in the initial stage, and the slope of the graph decreased for long duration of time. After 6 hours, the removal efficiencies of contaminants in the concentrations of 100, 1000, 2000, and 3000 mg/L were 64, 78, 69, and 56%, respectively. These results were compared by many other reported data were desirable. For instance, Froehner et al. [25] were able to remove 30% of phenol in aqueous solution using organoclay made of bentonite and HDTMA surfactant.

Figure 4 shows the removal of phenol using organoclay with 200% CEC. After 2 minutes, the removal efficiencies for the contaminants with concentrations of 100, 1000, 2000, and 3000 mg/L were 58, 71, 68 and, 73%, respectively. The amount of contaminant for each concentration decreased with respect to long duration of time (Similar trend as shown in Figure 3), but the removal rate was much higher in the early

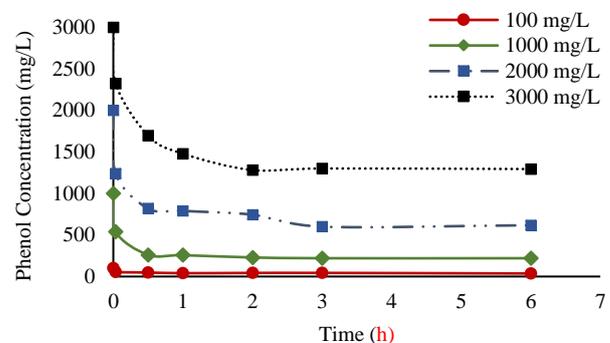


Figure 3. Removal of phenol at different concentrations using organoclay with 100% CEC

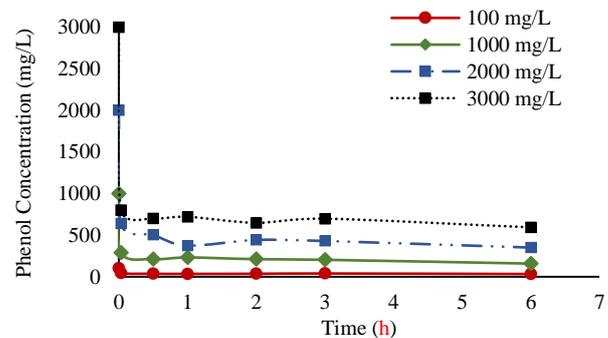


Figure 4. Removal of phenol at different concentrations using organoclay with 200% CEC

stage and the slope of the graph declined with respect to time. After 6 hours, the removal efficiency of the listed contaminants reached 68, 84, 82, and 80%, respectively. According to the results, the adsorption rate enhanced with an increase in surfactant concentration.

Finally, the ability of organoclay with 300% CEC for the removal of phenol is shown in Figure 5. At first 2 minutes, the removal efficiency of pollutants with concentrations of 100, 1000, 2000, and 3000 mg/L reached to 49.8, 55.3, 64.9, and 57.7%, respectively. After 6 hours, the removal efficiency of contaminants reached 69, 85.4, 81.5, and 76.6%, respectively. According to the results, there was no significant difference between the two organoclays with 200 and 300% CEC. Although the adsorption rate of the contaminants increased by increasing the surfactant from 100 to 200% CEC, after that, the adsorption rate inhibited by raising the CEC value to 300%, because of the surfactant desorbed during the experiment by clay and appears as foam in the experiment. The similar results were reported by Yapar et al. [26]. They had concluded that the adsorption of phenol increased with increasing the amount of adsorbent. However, this increase to a certain concentration was linear. With an increase adsorbent, the formation of coagulum was enhanced by cationic surfactant. Thus, increasing the resistance to particulate emissions and decreasing the more adsorption of phenol.

Numerous studies have demonstrated that phenol adsorption rises with increasing adsorbent dosage, because it was directly associated with an increase in active adsorption sites [26-28]. Nevertheless, increasing the amount of adsorbent in a system generally leads to the accumulation of clay minerals, which reduces the adsorption site for the adsorption of phenolic compounds. This accumulation of more particles desorbs the dissolved particles that loosely attach to the surface of the adsorbent [29].

The removal of phenol at a concentration of 100 mg/L using different organoclays is shown in Figure 6. According to the figure, there is not much differences between different organoclays in the removal efficiencies. Thus, it can be said that the increase of surfactant at low concentrations of phenol (such as 100 mg/L) has slightly affected on the adsorption capacity of organoclay and has no economic justification or benefit.

The rate of phenol removal at concentration of 3000 mg/L using different organoclays is illustrated in Figure 7. Accordingly, an increase in surfactant has a favorable and

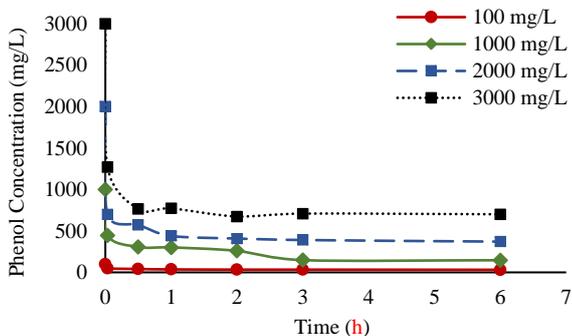


Figure 5. Removal of phenol at different concentrations using organoclay with 300% CEC

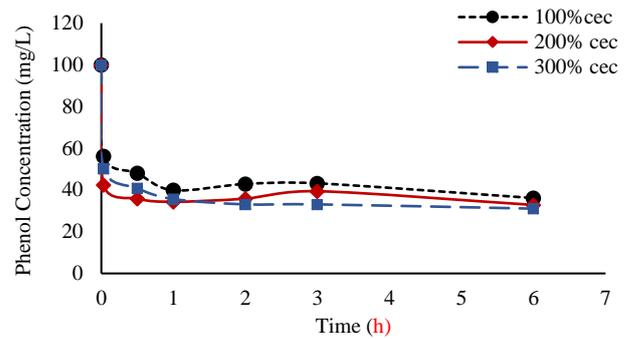


Figure 6. Impact of organoclay with different CECs on the removal percentage of phenol (Initial concentration of 100 mg/L)

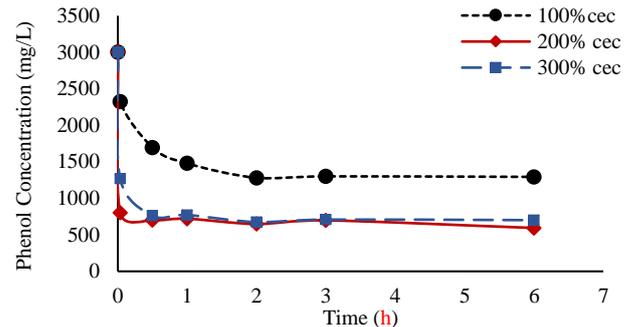


Figure 7. Impact of organoclay with different CECs on the removal percentage of phenol (Initial concentration of 3000 mg/L)

significant effect on organoclay adsorption capacity; especially in the first few minutes for removal of phenol at high concentrations such as 3000 mg/L and has great advantages and economic justifications. Of course, as pointed out, the adsorption capacity of organoclay with CEC 200% has no significant differences with that of CEC 300%, even organoclay with CEC 200% in some cases had performed slightly better than CEC 300%. Thus, increasing surfactant for high concentrations of phenol has caused a dramatic increase in the adsorption capacity; but, addition of surfactant may be useless from the above certain level and economically is not feasible to use excess amount of surfactant.

In all experiments, the adsorption of phenol reached the equilibrium in about 5 to 6 hours that the result is in line with the results of investigations carried out by Djebbar et al. [30] and Sarkar et al. [31]. The equilibrium data (hours reported) for adsorption of phenol in these two studies were 5 and 6 hours, respectively.

Investigating the adsorption capacity of organoclay

The adsorption capacity of organoclay with 100% CEC was examined by 0.4 g organoclay at a concentration of 1000 mg phenol /L. After completing the adsorption process, the slurry solution was centrifuged to separate the clay. The clay was cold dried and used again for the removal of phenol at a new concentration of 1000 mg/L. This experiment was repeated several times (for fixed duration of time 6 hours), and the results are represented in Figure 8. The removal rates of phenol in the first and second times were achieved to be 88 and 70%, respectively. For long duration of time, the adsorption rate declined after each use and even dropped to 16.73% at the eighth time use.

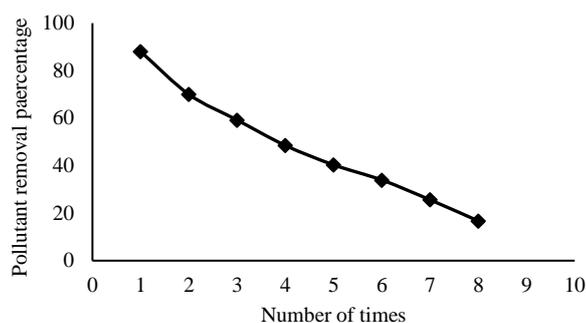


Figure 8. Adsorption capacity of organoclay

CONCLUSIONS

The results of this study suggest that organoclay was modified as a good adsorbent for the removal of phenol, especially for high concentrations in a short period of time. An increase in the surfactant at low concentrations of phenol has slightly affected on the adsorption capacity of organoclay and had no economic justification and advantages. However, to remove its high concentrations, increasing surfactant has a favorable and remarkable effect on the adsorption capacity of organoclay, especially in the initial stage of adsorption process (for the first few minutes) and enjoys an economical benefit, but addition of surfactant concentration was useless from certain level, and it is not economically reasonable to apply high surfactant concentration. Actually, adsorption of phenol increased with an increase in the adsorbent dosage, because it is directly associated with increased adsorption sites. However, increasing the amount of adsorbent in a system generally leads to the accumulation of clay minerals, which reduces the adsorption site for the adsorption of phenolic compounds. This accumulation of more particles desorbs the dissolved particles that loosely attach to the surface of the adsorbent. Based on the obtained results in this study, organoclay can be an alternative in compare to complex and expensive systems for the removal of phenol due to its simplicity, high performance, and low cost.

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Persian Abstract

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چکیده

فنل و متابولیت‌های آن به دلیل سرطان‌زایی، سمیت، ماندگاری طولانی مدت در محیط، از جمله ترکیبات آلی خطرناک به شمار می‌روند. هدف از این مطالعه سنتز organoclay به عنوان جاذب با پایداری فیزیکی و شیمیایی بالا، سازگاری محیطی و کاربرد آن برای تصفیه محلول‌های آبی آلوده به فنل است. در این پژوهش، organoclay با استفاده از ترکیبی از بنتونیت سدیم و سورفکتانت ستیل تری‌متیل آمونیوم برمید (CTAB) با سه ظرفیت مختلف تبادل کاتیون (CECs) ساخته شد. همچنین، ظرفیت جذب organoclay سنتز شده در استفاده مجدد تا زمان اشباع تعیین شد. نتایج نشان داد که راندمان حذف فنل در غلظت ۱۰۰، ۱۰۰۰، ۲۰۰۰ و ۳۰۰۰ میلی‌گرم در لیتر به ترتیب ۶۸، ۸۴، ۸۲ و ۸۰ درصد بود. به طور کلی، استفاده از organoclay با توجه به سادگی فرایند، کارایی بالا و مقرون به صرفه بودن آن می‌تواند جایگزینی برای سیستم‌های پیچیده و گران قیمت برای از بین بردن فنل از محلول‌های آبی باشد.
