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Adsorptive desulfurization of model oil using untreated, acid activated and magnetite nanoparticle loaded bentonite as adsorbent

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Abstract The present research work focuses on a novel ultraclean desulfurization process of model oil by the adsorption method using untreated, acid activated and magnetite nanoparticle loaded bentonite as adsorbent. The parameters investigated are effect of contact time, adsorbent dose, initial dibenzothiophene (DBT) concentration and temperature. Experimental tests were conducted in batch process. Pseudo first and second order kinetic equations were used to examine the experimental data. It was found that pseudo second order kinetic equation described the data of the DBT adsorption onto all types of adsorbents very well. The isotherm data were analyzed using Langmuir and Freundlich isotherm models. The Langmuir isotherm model fits the data very well for the adsorption of DBT onto all three forms of adsorbents. The adsorption of DBT was also investigated at different adsorbent doses and was found that the percentage adsorption of DBT was increased with increasing the adsorbent dose, while the adsorption in mg/g was decreased with increasing the adsorbent dose. The prepared adsorbents were analyzed by scanning electron microscopy (SEM), energy dispersive X-ray spectrometry (EDX) and X-ray diffraction (XRD).

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1. Introduction

Production of energy is one of the most imperative issues of modern era. Energy usage and economic activity are intimately associated, because useful goods production, services and more inclusive economic output need high energy usage. From 1980 to 2013 the annual energy usage in the world increased by an average of 404 quadrillion BTUs. Although over the same period, the percent energy attained from fossil fuels declined, still the share of energy from fossil fuel is

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about 82%, and about half of which emanates from petroleum. Thus in the world petroleum is considered one of the main sources of energy, but major problems from petroleum are due to its sulfur compounds. Sulfur compounds are present in petroleum in the form of free sulfur, sulfides, disulfides, mercaptan, thiophene, benzothiophene and dibenzothiophene [1]. During combustion of petroleum these sulfur compounds are converted into obnoxious Sulfur oxides (SO_x). SO_x resulting from combustion of sulfur rich fuels have become one of the serious environmental issues in the world as they are a major root of atmospheric pollution, global warming and acid rains.

Since last decade considerable attention has been paid to the desulfurization of diesel fuel oil and gasoline due to increasingly rigorous environmental protocols being imposed to decrease the sulfur content of fuel to minimum levels. Industrially the removal of sulfur compounds from liquid fuel is carried out by catalytic hydrodesulfurization process (HDS) [2]. Although, HDS is more effective for the removal of sulfur compounds such as: sulfide, disulfide, thiol, thiophene, benzothiophene, but dibenzothiophene most objectionable sulfur components present in the petroleum fractions are fewer reactive to this process [2]. Further enhancement of the catalytic HDS process for ultra-deep desulfurization is limited due to increasingly severe working conditions at high cost. Moreover, the deep HDS processes entailed substantial hydrogen consumptions, huge energy and significantly improved selectivity and reactivity of the required catalysts. Presently most of the catalysts used in the HDS process are very expensive and also cause unwanted sideways reactions, which lead to a decrease in the required octane number of gasoline [3]. Thus, from both economic and environmental considerations, recently other alternative ultra-deep desulfurization processes have been explored, such as: bio-desulfurization, oxidative desulfurization (ODS) using various catalysts, extractive desulfurization with different ionic liquids [4].

Presently new approach for the sulfur removal from liquid fuels is adsorptive desulfurization (ADS), which seems to be very promising and economical methods with regard to energy consumption, because the adsorption process can be achieved at ambient pressure, temperature and without use of any expensive hydrogen as well as catalyst. Moreover, some types of adsorbents have high adsorption capacity which can remove more refractory aromatic sulfur compounds very effectively [5,6]. In this regard, great deal of research on the desulfurization of liquid fuel has been diverted to adsorption process in order to develop new type of adsorbents with enhanced adsorption capacity, high regenerability and selectivity [7].

Sulfur compounds present in fuel affect its quality as well as the environment. Therefore, it is necessary to carry out more and more work in order to remove or decrease their concentration in petroleum fraction into formidable level by cheaper and easily operating method. In order to remove one of sulfur compound from petroleum cuts, the study was carried out on the removal of DBT from model oil by the adsorption method. In this study bentonite was used as an adsorbent. Bentonite; activated with acid and loaded with magnetite nanoparticles was also used as an adsorbent for the effective removal of DBT from model oil.

2. Experimental

2.1. Reagents and solutions

Dibenzothiophene (DBT) was purchased from Merck Company, and was used without further purification. Stock solutions of 1000 mg/L DBT were prepared in 1000 mL n-heptane and working solutions were then prepared from stock solutions by using the dilution formula.

2.2. Adsorbent preparation

2.2.1. Preparation of bentonite as adsorbent

Bentonite was obtained from soil sciences laboratory, Pakistan Council of Science and Industrial Research (PCSIR), Peshawar. The bentonite was grinded into powder form and was sieved by sieve size of 120 μm. The sieved bentonite was washed with distilled water for the removal of dust and other water soluble particles. The washed bentonite was dried in oven and was stored for further use.

2.2.2. Acid treatment of bentonite

25 g of cleaned and dried bentonite was taken in a round bottom flask and 150 mL of 0.1 N HNO₃ solutions was added to it. The bentonite and acid solutions in flask were refluxed with continuous stirring and heating (70 °C) for 3 h. After 3 h the bentonite slurry was filtered by Buchner funnel, then washed with distilled water, dried in oven and stored for further use.

2.2.3. Loading of magnetite nanoparticles on bentonite

Magnetite nanoparticles were loaded on bentonite by the following procedures: FeCl₂·4H₂O and FeCl₃·6H₂O were dissolved in 80 mL double distilled water in 1:2 ratio respectively; and were then shaken at 85 °C in a water bath shaker for 30 min. Then 30% ammonium hydroxide solutions of 20 mL were added to the mixture solutions and with ammonium addition the solutions color changed to black from orange immediately. Then 6 g of the pretreated bentonite was added to the mixture, and was then shaken for 2 h in a water bath shaker at 85 °C. After 2 h, the bentonite loaded with magnetite nanoparticles (MNLB) was filtered, then washed with water in order to remove extra chloride ions, and dried. The dried MNLB were checked through magnet. It was found that the magnet attracts all the MNLB because of its magnetic nature. The MNLB were then stored in a bottle for further experiments and characterization.

2.3. Batch adsorption program

Adsorption of dibenzothiophene (DBT) from model oil onto untreated bentonite (UB), acid activated bentonite (AAB) and magnetite impregnate bentonite (MNLB) was conducted at different adsorption parameters like: different contact time, adsorbent dose, initial DBT concentration, temperature. For each experimental set up, known volume, DBT concentration, and mass of the adsorbent were taken in a 100 mL Erlenmeyer flask, and were shaken for the respective time in temperature control water bath shaker. After the respective time, the Erlenmeyer flask was taken from the shaker and the mixture

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