

A Review on the Adsorptive Performance of Bentonite on Sulfur Compounds

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When sulfur is left in fuel, it has a tendency to be oxidized during combustion and cause problems to engines and harm when released into the atmosphere. One way to reduce sulfur content is by adsorptive desulfurization, which makes use of solid materials as adsorbents to adsorb sulfur from fuel selectively. This process is gaining interest from researchers as it can produce a high sulfur yield at mild temperatures and pressure, and the use of effective and regenerative adsorbents can lower the cost of the process. Clay adsorbents, like kaolinite, bentonite, and montmorillonite, are being studied as their availability and low cost make them good candidates. The objective of this paper is to review studies making use of bentonite as a desulfurization adsorbent and determine future research topics in this scope. It also aims to gain insight into the performance of bentonite and its mechanism in adsorbing different sulfur compounds. Several studies have made use of bentonite as an adsorbent for different compounds, specifically dibenzothiophene, dibenzothiophene sulfone, benzothiophene sulfone, dimethyl sulfide, propyl-mercaptan, and thiophene. Though these studies showed the wide range of effectiveness of bentonite to adsorb sulfur, modification to the adsorbent generally increases its effectiveness as it encourages chemisorption between the adsorbent and target molecule. It also aids in improving the physical properties of the adsorbent, like the surface area available for adsorption. The review showed that modifications to bentonite improved its performance in adsorbing sulfur, presenting the potential for industrial use with further research. For future studies, other modifications of bentonite can be investigated, such as activation and impregnation of other ions for adsorption of other sulfur compounds.

1. Introduction

Fuel for transportation is a non-negotiable commodity needed for industries to function. However, with the continuous consumption of liquid fuel, pollution becomes a problem. The challenge persists in lowering the sulfur content of transportation fuels to avoid irreversible damage to the environment and its inhabitants. Efforts to prevent the further worsening of air pollution levels around the world are being made by different constituents. Specifically, oil refineries have a large responsibility to take steps to reduce the sulfur content of their fuels to comply with stringent standards set by regulating bodies. When left in the fuel, sulfur is oxidized during combustion and can damage engines and exhausts. Sulfur can also be released into the atmosphere, which can result in the formation of acid rain, which can cause harm to humans, the environment, and infrastructure. Desulfurization is now necessary to reduce the sulfur in fuel. The most common method employed in the industry to reduce sulfur content is Hydrodesulfurization (HDS). The method of HDS treats sulfur in the fuel with the use of hydrogen gas to convert sulfur compounds to hydrogen sulfide (H₂S). Though popular and effective, issues arise with this process due to its expensive cost associated with the extreme operating conditions and limited ability to hydrogenate heterocyclic sulfur compounds (Srivastava, 2012). Oxidative desulfurization is also being studied as it can remove sulfur content through stable reactions and mild conditions (Choi, 2021). Another method for desulfurization is Adsorptive Desulfurization (ADS) which makes use of solid materials as adsorbents to selectively adsorb sulfur from fuel (Rezazakemi and Zhang, 2018). This process is gaining interest from

researchers as it can produce a high sulfur yield at mild temperatures and pressure (Yaseen et al., 2021). The use of effective and regenerative adsorbents can also contribute to a lower cost for the desulfurization process. Characteristics of a good adsorbent for ADS include its adsorption capacity, selectivity to sulfur relative to fuels, renewability, and durability (Rezakazemi and Zhang, 2018). Adsorbents rising in popularity are clay adsorbents, which are relatively new to the research scene for ADS. This section of materials is being studied for their adsorption capacities because of their abundance, low cost, stability, and environmentally benign characteristics (Tian et al., 2017). Some clay adsorbents are kaolinite, bentonite, and palygorskite. Several studies have been done to compare different types of clay minerals as adsorbents. One of which is the study of Shakirullah et al. (2012) made use of kaolinite, montmorillonite, palygorskite, and vermiculite to remove sulfur compounds from petroleum products. It revealed the differences between the clay minerals, wherein despite vermiculite having a slightly higher surface area, kaolinite resulted in a better performance compared to the other adsorbents due to differences in mineralogical composition. This highlights the effect of the differences of the types of clay adsorbents on their performance and interaction with sulfur compounds.

The most recent classification of adsorbents being studied is Metal Organic Framework-based adsorbents (MOF). It has caught the eye of researchers because of its characteristics such as high pore volume, designable crystalline structure, and adjustable pore size. By varying the metal and organic linker of the MOF, the pore size can be modifiable (Saha et al., 2021). With these strong capabilities, using MOF also has drawbacks. Though it can adsorb the larger compounds that HDS might not be able to treat, sulfur selectivity must be established for it to be a viable adsorbent for desulfurization. It also has poor thermal and mechanical stability.

The search for an effective and efficient adsorbent for desulfurization is constantly in progress. Since clay adsorbents are stable, uncostly, and show great potential for sulfur adsorption, this study focuses on bentonite, which has been subject to study by different authors. This review paper differs from previous desulfurization reviews as it focuses on bentonite as an adsorbent of different sulfur compounds. Bentonite has the potential to be an effective desulfurization agent because of its stability, something that is yet to be acquired with MOF. This review is aimed to arrive at an overview of how bentonite interacts with different sulfur compounds, consolidate past studies on bentonite for desulfurization, and identify future research topics in this area.

2. Methodology

A scope was first established to denote the limitations of this review paper properly. This paper includes studies on the use of bentonite as a desulfurization adsorbent from liquid fuel. The keywords “bentonite,” “adsorbent,” and “desulfurization” was used to search for studies in Google Scholar and Science Direct. Studies making use of raw or modified bentonite for the removal of any sulfur compound were selected. The target sulfur compound and modification, if any, were then determined. Interactions between bentonite and sulfur were identified in the study being reviewed.

3. Desulfurization techniques

The relevant processes to reduce sulfur content in fuel are discussed in this section.

3.1 Hydrodesulfurization

In petroleum refining, hydrotreating is done to remove impurities from feedstock, prepare the feed for downstream plant units, and meet product specifications. This converts undesired aromatics, olefins, and organosulfur compounds to more stable products (Ortega, 2021). The most common process for sulfur removal used in oil refineries is HDS, wherein hydrotreating is applied to reduce the sulfur content of the feed stream selectively. The chemical reaction is generally characterized by the contact of hydrogen gas with the sulfur-containing organic compound, which will yield H_2S and the desulfurized organic (Mohammadi, 2021).

Hydrotreating is performed with the use of a catalyst that dictates the selectivity of the reaction. For desulfurization, a combination of Co and Mo oxides on an alumina matrix is used, but this could also vary with the use of Ni and Pt (Fahim et al., 2010). Though HDS has been widely used for several decades, research on catalysts for deep desulfurization of fuels is still being performed because of the difficulty of fully hydrogenating bulky sulfur compounds such as Dimethyldibenzothiophene (DMDBT) and Dibenzothiophene (DBT) (Huirache-Acuña et al., 2021). However, in the summary of studies that Shafi and Hutchings (2000) provided, it is shown that the reaction temperature and pressure are all high, with average temperatures and pressures around 300 °C and 3.5 MPa. This presents a downside in the economic aspect of HDS as a higher cost is necessary to reach an elevated temperature and pressure. Its difficulty in removing sterically hindered sulfur compounds still presents a challenge. Because of this, other desulfurization techniques are studied.

3.2 Adsorptive desulfurization

At ambient temperature and pressure, ADS can selectively remove sulfur from fuel. This is an advantage as opposed to the use of HDS. Since adsorbents are regenerable to some extent through desorption and regeneration, this helps utilize the adsorbents to their maximum capacity. Desirable traits of an adsorbent for desulfurization include porosity, physical and chemical stability, being environmentally benign, and amenability to regeneration (Omar and Verma, 2022). One of the oldest and most common types of adsorbents used is carbon-based adsorbents, specifically Activated Carbon (AC). AC is utilized in various industries, such as wastewater treatment, water purification, the food and beverage industry, and the pharmaceutical industry (Muttil et al., 2023). In ADS, AC is desirable because of its low cost, high surface area, and high porosity (Saha et al., 2021). Materials with a high surface area and porosity show the potential of becoming good adsorbents, as these pores serve as sites for adsorption. Modification of raw AC by modifying the functional groups on its surface helps in increasing its sulfur yield. This is due to the better interaction with the sulfur in the fuel, according to Yaseen et al. (2021), who used Zn and Mn-loaded AC to reach 95 % desulfurization. Another commonly used adsorbent is zeolite, which has pores and chambers that contribute to the breaking down or exclusion of other molecules. This results in a high selectivity for sulfur compounds (Rezakazemi and Zhang, 2018). Its metal-based active sites make it possible to perform cation exchange, making it a commendable adsorbent for sulfur removal (Saha et al., 2021). The zeolite that was ion-exchanged with metal ions, specifically copper and silver, resulted in high sulfur removal rates and also showed good regeneration capability (Cerutti et al., 2019). MOFs also show good performance as adsorbents as they have a high surface area, porosity, and number of active sites. They are composed of an inorganic center, which could be comprised of metal ions, and are linked together by di- or poly-dentate chelating organic bridges called linkers (Kampouraki et al., 2019). In the study of Mguni et al. (2021), thiophene was adsorbed by MOF at 64 % desulfurization due to the high affinity of thiophene to the immobilized nickel metal ion. Desulfurization of 92 % was achieved by Sundararaman et al. (2010), who used beta zeolite to adsorb sulfones in jet fuel. Another choice for adsorbents for ADS is clay adsorbents, which is the focus of this study. Since clay adsorbents are abundant and stable, it has become an attractive subject for desulfurization studies. Naturally occurring in nature, clay adsorbents are made up of alumina, silica, and water. Since the surface of the clay minerals contains exchangeable ions, studies have been done to exchange these with other ions and check their adsorption capabilities (Tonk et al., 2022). Comparisons of the different types of clay minerals as adsorbents have been performed, and modifications have been tested to achieve higher desulfurization rates. With the multiple studies done on several adsorbents, the focus will be put on bentonite, which will be reviewed in this paper.

4. Theory of adsorption

Adsorption is the adherence of atoms, molecules, or ions from a bulk fluid to the surface of a solid adsorbent (Bakhtyari et al., 2020). This results in the increase of solute concentration near the liquid-solid interface. Adsorption can happen through physical or chemical mechanisms. As discussed by Rezakazemi and Zhang (2018), physisorption is when Van der Waals forces become present on electric dipoles of the sulfur molecule and adsorbent. This results in the adherence of the molecule without changing its composition. This is depicted in Figure 1a, where the blue particles represent the solute molecules adhering to the surface of the adsorbent. Chemisorption is when chemical bonding occurs between the adsorbate and the adsorbent. The species are bonded by the exchange or sharing of electrons between the adsorbate and the species on the surface of the adsorbent (Bakhtyari et al., 2020). A diagram of this mechanism is shown in Figure 2, where the red particles represent the different species found on the surface of the adsorbent. The solute molecules attach to the chemical entities on the adsorbent through chemical bonds, as opposed to bonding through physical attraction.

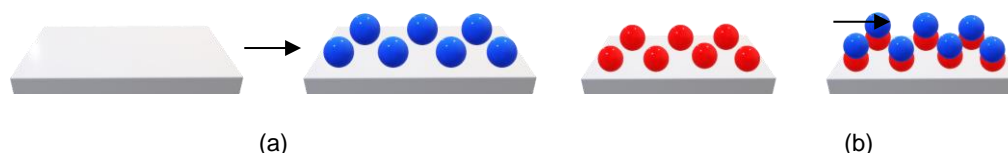


Figure 1: (a) Diagram of physisorption mechanism. (b) Diagram of chemisorption mechanism. Blue circles represent solute molecules. Red circles represent species on the surface of the adsorbent

5. Bentonite as adsorbent

Bentonite, with or without modification, can be used to adsorb Propyl Mercaptan (PM). Yi et al. (2013) loaded CuCl_2 on bentonite, which increased the adsorbed sulfur. Their characterization showed that the total number of Lewis acid sites on the surface of bentonite increased with metal insertion, creating more active sites and

promoting sulfur adherence. Tang et al. (2011b) studied multiple modifications on bentonite to adsorb PM, loading it with Cu^{2+} , Cu^+ , Fe^{3+} , and MnO_4^- . The metal with the best performance is KMnO_4 , followed by Fe^{3+} because the metals affect the dissociation of PM to $\text{C}_3\text{H}_7\text{S}^-$ and H^+ and the oxidation of $\text{C}_3\text{H}_7\text{S}^-$ to disulfides. The two other metals, Cu^{2+} and Cu^+ , are nucleophilic and react with the thiols through π -complexation.

The adsorption of Dimethyl Sulfide (DMS) using bentonite was also investigated. The study of Huang et al. (2013) showed that DMS favors interaction with Ag^+ on the surface more than the surface acid sites found on the surface of bentonite. This strong interaction caused DMS molecules to hardly reach the interlayer of the Ag^+ -modified bentonite. The interaction was characterized by physical intermolecular forces and a sigma bond between DMS and Ag^+ . Yi et al. (2013) used CuCl_2 -loaded bentonite to adsorb DMS. The Cu^{2+} forms bonds with the sulfur atom in the thiol to form copper (II) mercaptide. Since the sulfur atom has a low electronegativity, this allows it to provide lone pair electrons to the copper atom and form a sigma bond at the weak Lewis acid sites on the adsorbent. The disulfide equivalent of DMS, Dimethyl Disulfide (DMDS), was used in the study of Mikhail et al. (2002). Acid-activated bentonite was superior to the other adsorbents because of the dissociation of the water molecule in its silicate-silicate structure, resulting in Brønsted acid sites. The acid treatment also resulted in hydrogen sites on the surface of the adsorbent, creating a disruption in the charge equilibrium of the bentonite lattice and creating more active sites for the adsorption of DMDS. A similar study also used acid activation to create H^+ -bentonite. Al-Bidry and Azeez (2020) compared H^+ -bentonite with Fe^{3+} -bentonite and showed that Fe^{3+} impregnated on the bentonite showed better performance in adsorbing organic sulfur compounds from crude oil than the other. The treatment of bentonite with the metal enhanced the surface area and decreased the particle size, resulting in an improved desulfurization performance. The 37.28 % sulfur removal of H^+ -bentonite increased to 81.44 % sulfur removal of Fe^{3+} -bentonite at the same conditions.

Bulkier sulfur atoms are harder to remove from fuel using traditional desulfurization processes. As such, adsorbents have also been tested to remove cyclic sulfur compounds. Ali (2018) removed thiophene from model oil using raw and Na^+ -impregnated bentonite. Results showed that the latter outperformed the former with 87 % desulfurization by Na^+ -bentonite against the 62 % desulfurization of the raw bentonite. This increase in sulfur removal is due to the higher occurrence of activated groups on the surface of the adsorbent, leading to acid-base interactions and π -complexation between Na^+ and thiophene molecules. Another study used Ag^+ -loaded bentonite to remove alkyl DBT from liquid fuel. Tang et al. (2011a) discovered that at optimum conditions, Ag^+ -loaded bentonite performed better than raw bentonite because the nucleophilic activity between the Ag^+ ions and alkyl dibenzothiophenes enabled them to have complex reactions, as well as the presence of weak Lewis acid sites on the adsorbent. Ullah et al. (2020) also studied the adsorption of DBT with the use of bentonite and AC composites. Their study showed that the adsorbent with 50 % bentonite and 50 % AC performed better than the other ratios of adsorbent. This indicates that a synergistic effect between the two adsorbents allowed for better sulfur removal, resulting in better surface acidity and mesoporous structure. A 97.22 % desulfurization was achieved at the optimal parameters when this ratio of bentonite and AC was used. In the study of Saeed et al. (2022), bentonite was made more hydrophobic with the use of different modification reagents. Benzyl Tri-n-Butyl Ammonium Bromide (BTB), Dioctyl Sodium Sulfosuccinate (DSS), Benzethonium Chloride (BTC), and Bis (2-ethylhexyl) Amine (BEHA) were used to enhance hydrophobicity and improve interactions with the organic matter. Increased carbon content after reagent treatment indicated successful modification. At the conditions of 45 °C, 60 min, 0.5 g of bentonite-BTC, and 30 mL of fuel oil, 96.76 % and 95.83 % sulfur removal were reached for kerosene and diesel oil. On top of the increased hydrophobicity, the good performance of the organoclay was attributed to the increase of interlayer spacing and increased porosity upon modification.

Several studies have tested the performance of bentonite to adsorb oxidized cyclic sulfur compounds, specifically Benzothiophene Sulfone (BTO) and Dibenzothiophene Sulfone (DBTO). Choi et al. (2017b) compared the performance of bentonite with two other adsorbents, activated clay and kaolinite, in adsorbing BTO. Bentonite was second to kaolinite in terms of adsorption capacity. Their study revealed that the adsorption systems follow the trend of a fast adsorption rate during the first 2 h of contact, then slow down until equilibrium. Choi et al. (2017a) also tested the adsorption performance of the three adsorbents on DBTO. Bentonite was second to activated clay in terms of adsorption capacity, and it was determined that chemisorption is the rate-limiting step for DBTO adsorption from 25 °C to 55 °C. The different functional groups on the surface of the clay minerals also play a role in their adsorption performance. Bentonite was also used in the study of Chen et al. (2016) to remove sulfones from oxidized diesel oil. The use of bentonite for sulfones was proved to be successful as it reduced sulfur content from 1,109.3 ppm to 6 ppm. The effectivity of the system was achieved by increasing the adsorbent dosage to increase adsorption sites, increasing temperature to promote collision between solute and adsorbent, and increasing the agitation rate to allow effective transfer of solutes from the bulk solution to the surface of the adsorbent, also to limit the thickness of the liquid boundary layer. Their study also resulted in the same finding as Choi et al. (2017a), where chemisorption limited the rate of adsorption. A comparison of the desulfurization performance of bentonite by different studies is shown in Table 1. Though several studies have desulfurization percentages higher than 70 %, others can be potentially investigated further to identify ways to

improve sulfur adsorption. The review shows the scarcity of recent studies with bentonite as its focus. A large gap can also be observed in the period between studies. Though done with different modifications, the table shows the good performance of bentonite for adsorbing the bulkier, heterocyclic sulfur compounds. This offers the idea of bentonite being used as a supplementary process of HDS to be able to remove the sulfur compounds that HDS does not fully treat.

Table 1: Comparison of previous studies utilizing bentonite as adsorbent

Modification	Sulfur Adsorbed	Desulfurization (%)	Reference
Cu ²⁺ -loaded	DMS and PM	58.75	Yi et al. (2013)
Cu ²⁺ -loaded	PM	58.75	Tang et al. (2011b)
Cu ⁺ -loaded	PM	67.75	Tang et al. (2011b)
Fe ³⁺ -loaded	PM	48.30	Tang et al. (2011b)
MnO ₄ ⁻ -loaded	PM	85.10	Tang et al. (2011b)
Fe ³⁺ -loaded	Organic sulfur compounds	81.44	Al-Bidry and Azeez (2020)
H-loaded	Organic sulfur compounds	37.28	Al-Bidry and Azeez (2020)
Na-loaded	Thiophene	91.00	Ali (2018)
bentonite and AC composite	DBT	97.22	Ullah et al. (2020)
bentonite-BTC	Total sulfur	96.76	Saeed et al. (2022)
N/A	BTO	72.88	Choi et al. (2017b)
N/A	DBTO	47.20	Choi et al. (2017a)
N/A	Sulfones	99.00	Chen et al. (2016)

6. Conclusions and recommendations

The use of bentonite as an adsorbent was found to be effective in removing sulfur compounds in fuel oil. Studies reviewed showed that the modifications on bentonite performed better than raw bentonite. Modifications, such as acid activation and metal impregnation, increased sulfur adsorption due to the increased active sites and the creation of Lewis acid sites on the surface of bentonite. This promotes an interaction between the adsorbent with the basic sulfur compounds. The knowledge generated from this review can lead to the possible upscaling of alternative desulfurization processes and adaptation by fuel refineries. The review showed that bentonite can be at par with some of the more established adsorbents. From the reviewed studies, AC, zeolite, and MOF reached 95 %, 92 %, and 64 % sulfur removal. At the right conditions, bentonite can reach 99 % desulfurization of model oil. The affordability of bentonite also pushes its attractiveness as a sulfur adsorbent and can be considered an advantage compared to other types of adsorbents. Future studies can tackle other modifications, such as the acid used for activation, the ion impregnated on the adsorbent, and testing composites of bentonite with other types of adsorbents. Testing with high-sulfur fuel oil will also help in the push for commercializing adsorptive desulfurization. Experimentation with this can display the performance of bentonite in uncontrolled samples and non-ideal conditions. Computational studies on the cost and performance trade-off of bentonite with other adsorbents can also be explored to drive the use of bentonite as an agent for desulfurization.

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