

Kinetics Analysis of Manganese Dioxide Adsorbent on Desulphurization of Diesel Oil

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Abstract - Pollutant such as exhaust gases cause damage to human health and safety of other living organisms. Diesel, a derivative of petroleum is one of the major pollutants of air, water and soil. It is rapidly increasing every year causing grave and irreversible consequences. Adsorptive desulphurization of diesel oil has been investigated in a batch reactor using activated manganese dioxide as the adsorbent. The results obtained showed that desulphurization efficiency increased on increasing contact time. There was 53% reduction of diesel oil sulphur content at the end of the process. The respective square of correlation coefficient (R^2) values of pseudo-first order and pseudo-second order reaction models were estimated to be 0.8941 and 0.9987 respectively when the experimental data were fitted into them. Thus, pseudo-second order reaction model was found to better describe the desulphurization process with chemisorption being the rate limiting step. Also, the effects of desulphurization on other physico-chemical properties of diesel oil show insignificant changes after treatment. However, there is the need to improve on the methods and the adsorbents that will result in complete isolation of sulphur from diesel oil.

Keywords: Pollutants, desulphurization, diesel oil, kinetics, chemisorption.

1. INTRODUCTION

The quality of life on earth is linked directly to the overall quality of the environment. The natural ecosystem encompasses all living and non-living things existing on earth naturally. It is the environment that interacts with all living species. Petroleum hydrocarbons belong to the most widespread contaminants of soil, air and water (Trindade *et al.*, 2005). The largest and most widely used energy source in the world is crude oil (Adeyi *et al.*, 2014). However, it contains sulphur usually in the form of organic sulphur compounds. Sulphur compounds exist in various forms and can be classified into four main groups: mercaptans, sulphides, disulphides and thiophenes (Al-Malki, 2004). It is an undesirable component because it forms sulphur dioxide (SO_2) during fuel oil combustion (Shiraishi *et al.*, 2002). The combustion of sulphur compounds are not only causing corrosion but also contributing considerably to acid rains and air pollution (Song *et al.*, 2004), deforestation, smog, and global warming, as well as several human health concerns such as cardiovascular disease, cancer, creation of asthmatic symptoms and other respiratory diseases (Lin *et al.*, 2010). The simplest way to decrease the amount of SO_2 emitted into the air is to reduce the amount of sulphur in fuel (Sundaraman *et al.*, 2010). Hence, desulphurization of diesel oil is essential to overcome these challenges.

At present, oil desulphurization technology is broadly divided into two categories, which are hydrodesulphurization (HDS) and non-hydrogenation hydrodesulphurization (NHDS). HDS is used to remove sulphur from hydrocarbons in petroleum refineries that requires either increasing reactor residence time, or carrying out reactions under high temperature and pressure (Grossman, 2001). The catalysts used in HDS are not active in removing refractory sulphur compounds like thiophenes and its derivatives. These compounds require high hydrogen and energy consumption in

the HDS process. Non-HDS technology is a reverse case as it does not require hydrogen and energy consumption. Adsorption is the most common HDS alternative method currently used to achieve ultra-clean fuels (Tymchyshyn, 2008). It is often employed to remove trace impurities, such as the removal of trace amounts of aromatics from aliphatics (Takahashi *et al.*, 2002), and metal oxides are the adsorbents utilized in petroleum processing industries for the desulphurization of diesel oil (Hernandez-Yang *et al.*, 2004).

New Federal regulations require the removal of sulphur in both gasoline and diesel to very low levels, forcing existing technologies to be pushed into inefficient operating regimes (Nehlsen *et al.*, 2003). Today, most of the work is concentrated on the application of active metal oxides to form solid metal thiolates (Liu *et al.*, 2002). The process is based on thiophenes chemistry in which metal oxides react with thiophenes to form solid-metal-thiolates. The metal thiolates are insoluble in hydrocarbons and water at sufficiently low temperatures, permitting their removal by filtration (Zhang *et al.*, 2005). Many new combined technologies for desulphurization of crude oil include hydrogenation-bacterial catalysis method (Agarwal *et al.*, 2010), microwave-catalytic hydrogenation method (Leadbeater *et al.*, 2008), oxidative desulphurization in electrostatic fields (Guiling, 2004) and ultrasonic/microwave-catalytic oxidation method (Wen, 2009). In the past decade, researches have been concentrated on the desulphurization of diesel oil.

In 2006, Rang and colleagues worked on advances in desulphurization research of liquid fuel in which it was stated that 4-methyldibenzothiophene, 4,6-dimethyldibenzothiophene and their alkyl-substituted derivatives were the key substances that needed to be separated from diesel fuel and fuel oil. They observed that these compounds required high hydrogen consumption in the hydrodesulphurization process, and common hydrogenation catalysts were not very effective for this hydrogenization. Zhang *et al.* (2009) recognized oxidative desulphurization (ODS) technology as one of the most effective methods for the removal of fuel oils, due to the very mild operation conditions it requires. The ODS methods was divided into H₂O₂ oxidation method, organic oxidant method, photochemical oxidation method, as well as those involving the use of plasma or ultrasound based on the oxidants involved.

Recently, oxidative desulphurization of jet and diesel fuels using hydroperoxide generated in-situ by catalytic air oxidation has been examined by Sundararaman *et al.* (2010). Lin and co-workers (2010) did theoretical review on progress in the technology for desulphurization of crude oil. Campos-Martin *et al.* (2011) reviewed the developments in selective removal of organosulphur compounds present in liquid fuels via oxidative desulphurization, including both the chemical oxidation and biodesulphurization. Adeyi and Aberuagba (2012) did comparative study of desulphurization potentials of two metal oxides-activated manganese dioxide and activated zinc oxide, including the kinetic analyses of the adsorption process. The ability of the immobilized spores of *Aspergillus flavus* cultured in increasing concentrations of sodium metabisulphite to remove sulphur from diesel oil was investigated by Adegunlola *et al.* (2012). Hammad and group (2012) demonstrated in-situ electrochemical desulphurization of crude oil and its fraction. Sundararaman *et al.* (2013) investigated oxidative desulphurization of crude oil incorporating sulphone decomposition by alkaline earth metal oxides. They explored a novel approach to desulphurization of whole crude oil where thiophenic sulphur compounds were oxidized to sulphones followed by sulphone decomposition into sulphur-free hydrocarbons.

In this research work, manganese dioxide was applied to isolate sulphur from diesel fuel in batch experiments. The work explored the influence of contact time of the adsorbent. X-Ray Fluorescence spectrophotometer was used to determine the remaining sulphur in each sample after adsorption. The kinetics analysis of sulphur uptake from diesel oil sample was investigated using pseudo-first-order and pseudo-second-order rate equations.

2. MATERIALS AND METHODS

2.1 Acquisition and Preparation of Adsorbent and Diesel Oil

Manganese dioxide (MnO_2) used was 98.95% pure, 1.7 mm particle size, 5.026 g/cm^3 density and products of M and B Laboratory Chemicals, England. Its adsorptive capacities was increased by oven-drying at 110°C for 4 hours. After that, it was stored in a desiccator.

The diesel oil sample used in this research was obtained from Kaduna Refining and Petrochemical Company (KRPC), Kaduna, Nigeria.

2.2 Desulphurization Experiment

Batch adsorptive desulphurization experiments were performed by contacting 2 g of the adsorbent-activated manganese dioxide (MnO_2) powder with 20 ml of diesel oil. The experiment was performed on magnetic stirrer (model A-034) for a period of 1 hour at 120 rpm using 250 ml flask containing 20 ml of diesel oil sample and 2 g of adsorbent at room temperature. Continuous mixing was provided during the experiment by agitating at constant speed (120 rpm) for better mass transfer with high interfacial area contact. The remaining sulphur in each sample after adsorption at different time intervals (60, 120, 180, 240, 300 and 360 minutes) was determined with the aid of X-Ray Fluorescence Spectrophotometer (SULPHUR-IN-OIL ANALYZER) using ASTM (D-2622) method. This was done by placing the filtered sample in an X-ray beam and intensity of the sulphur X-ray fluorescence was, thereby, measured.

The quantity of sulphur retained in the adsorbent phase was calculated using Equation 1 (Demirbas *et al.*, 2004):

$$q_t = \frac{(C_o - C_t) V}{W} \quad (1)$$

where q_t = amount of adsorbed sulphur on adsorbent surface, mg g^{-1} ,

C_o = initial concentration of sulphur solution, mg kg^{-1} ,

C_t = final concentration of sulphur solution, mg kg^{-1} ,

V = volume, ml, and

W = mass of the adsorbent, g.

Also, the percentage sulphur removal (desulphurization) was estimated using Equation 2:

$$\% \text{Desulphurization} = \frac{(C_o - C_t)}{C_o} \times 100 \quad (2)$$

2.3 Kinetics Study

The kinetics of the adsorption was studied by analysing the adsorptive uptake of sulphur from the diesel oil sample at different time intervals. The pseudo-first order (Equation 3) and pseudo-second order (Equation 4) models were fitted to obtain the kinetics of the sulphur adsorption onto the activated manganese dioxide (AM). The linearity of each model, when plotted, indicated whether the model appropriately described the desulphurization process or not. The pseudo-first order model equation used is given as:

$$\ln(q_e - q_t) = \ln(q_e) - k_{s1} t \quad (3)$$

where q_e = equilibrium adsorption capacity, mg g^{-1} ,

q_t = quantity of adsorbed sulphur on adsorbent surface, mg g⁻¹,
 K_{s1} = adsorption rate constant of pseudo-first order kinetic model, min⁻¹,
 t = time, min.

The pseudo-second order model equation is expressed as:

$$\frac{t}{q_t} = \frac{1}{K_{s2} q_e^2} + \frac{1}{q_e} t \tag{4}$$

where K_{s2} is the adsorption rate constant of pseudo-second order, g mg⁻¹ min⁻¹. Other terms remain as stated in Equation 3.

The models described above were used with the premise that the adsorption of organic sulphur compounds from diesel oil, due to its low concentration, could be observed as the adsorption of one component represented by sulphur content in fuel with high selectivity of these adsorbents towards sulphur (Muzic *et al.*, 2008).

3. RESULTS AND DISCUSSION

3.1 Characterization of Diesel Oil Sample

The results obtained from the characterization of the diesel oil used in this study before and after desulphurization are given in Table 1.

Table 1: Characterization results of the diesel oil sample before and after desulphurization

Property	Sample before desulphurization	Sample after desulphurization	ASTM Standard
Sulphur content (wt%)	0.175	0.08225	0.05-6.0
Flash point (°C)	43.52	43.25	40 – 48
Viscosity (cSt)	4.32	4.33	4.0 - 6.0
Salt (wt%)	3.00	2.40	2.90 - 4.10
Base sediment & water (wt%)	0.10	0.10	0.1 - 0.3
API gravity	36.0	37.60	15 – 40
Specific gravity	0.8456	0.87	0.8 - 1.0058

From the results presented in Table 1, it was observed that there was reduction in the sulphur content of the examined diesel oil after desulphurization by 53% because the sulphur content reduced from 0.175 wt% (1750 ppm) to 0.08225 wt% (822.5 ppm). Both values, which were for before and after the experiment respectively, were found to fall within the range of the recommended ASTM standard of 0.05 – 6.0 wt%. This was an indication that the sampled diesel was ASTM standard type. Also, the adsorbent (activated manganese dioxide) used proved to be efficient in the desulphurization of the diesel oil. Previous studies such as those of Adeyi and Aberuagba (2012) and Lin *et al.* (2010) had shown that activated manganese dioxide was a better adsorbent than activated zinc oxide and some other metallic oxides for desulphurization of diesel oil. In addition, other properties (flash point, viscosity, salt content, base sediment and water content, API gravity and specific gravity) of the diesel oil were checked, and their values were discovered to fall within the ranges of ASTM standards when examined before and after the experiment (see Table 1).

3.2 Desulphurization Process

The diesel oil sample used was found to contain 0.1750 wt% (1750 ppm) of sulphur; though this value was low compared to other diesel fuel from other sources, there was, however, the need to either remove it completely or reduce it to minimum acceptable standard of less than 500 ppm (0.05 wt%). To achieve this, activated manganese

dioxide (AM) was used as the adsorbent for the removal of the sulphur, and the results obtained are shown in Figure 1.

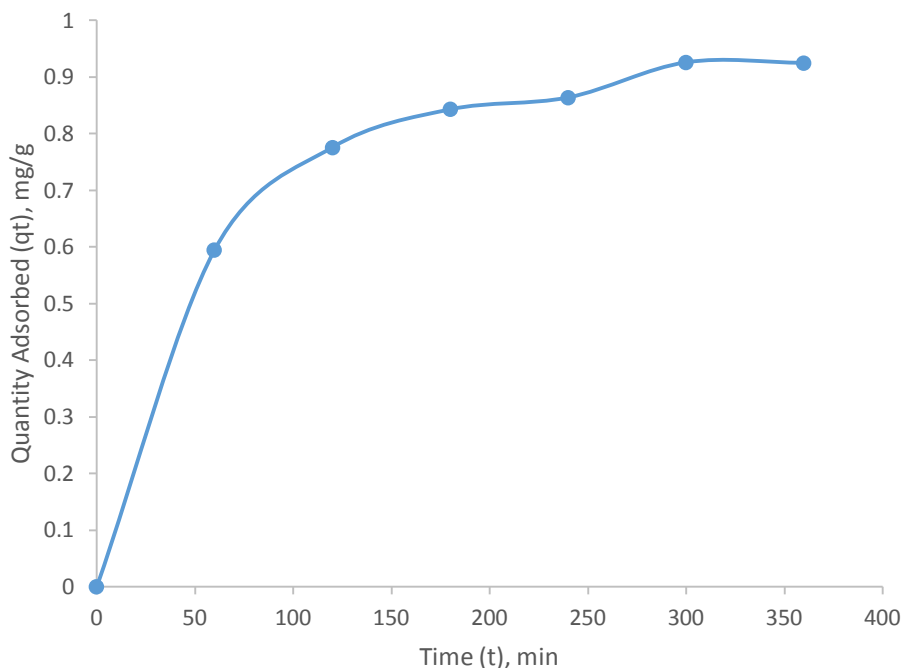


Figure 1: Dependence of adsorption capacity on time for the tested adsorbents at room temperature

According to the results given in Figure 1, the desulphurization with AM caused notable removal of sulphur at 60 minutes of about 34.00%. This was found to be due to the availability of large surface area of the adsorbent being available at the beginning for the adsorption of sulphur. The extension in contact time up to 300 minutes also caused significant increment in percentage desulphurization of the tested adsorbent from 34.00% to 52.86%. As can be seen, the increase in the contact time of the adsorbent has resulted in an increase in the sulphur uptake; similar phenomena was also observed by Onundi *et al.* (2010) and Okoye *et al.* (2010). The extension in time beyond 5 hours caused little changes in the percentage desulphurization. The maximum desulphurization of 52.89% brought about by the AM was observed to occur when the contact time was 6 hours.

Furthermore, it was discovered that the contact-time experimental results could be used to study the rate-limiting step of the adsorption process, as reported by Weber and Morris (Arivoli, 2007), and the outcomes of that have been obtained and presented as the kinetics of the process, given in the next subsection.

3.3 Adsorption Kinetics

Adsorption is a mass transfer process involving transfer of adsorbate from liquid phase into solid phase, and it includes 3 steps: (i) boundary layer mass transfer across the liquid film around the particle, (ii) internal diffusion/mass transport within the particle boundary as pore and/or solid diffusion, and (iii) adsorption within the particle and or on the external surface (Gueu *et al.*, 2007; Nounou *et al.*, 2010). In this work, the data obtained on dependence of adsorption capacity on time were used for kinetic analysis. Figures 4 and 5 respectively show the tests of pseudo-first order and pseudo-second order rate equations.

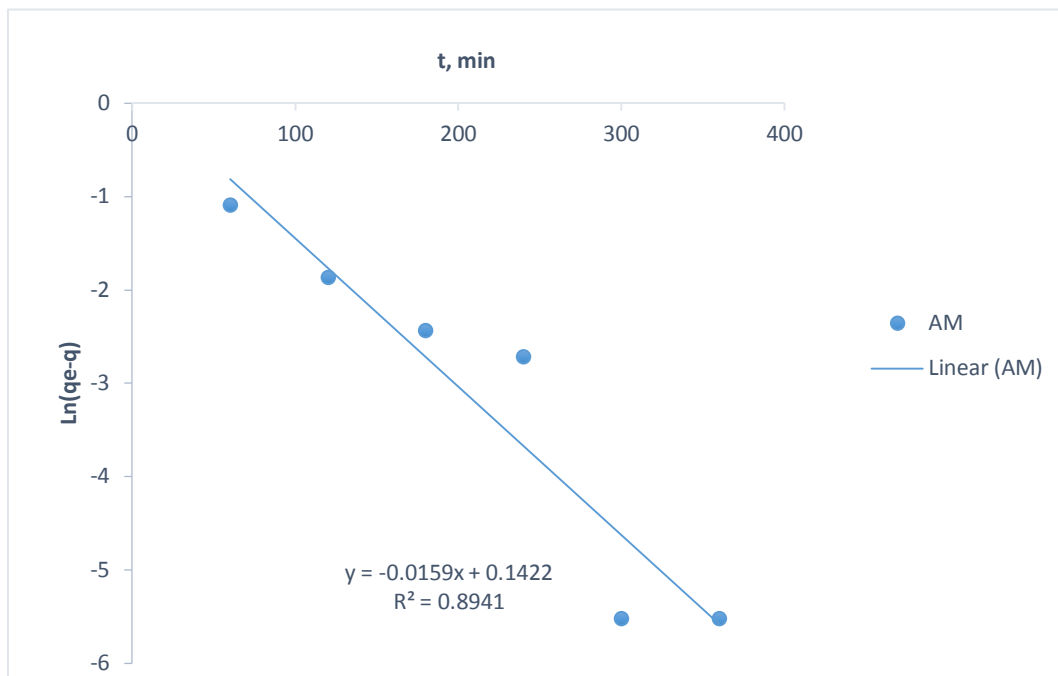


Figure 2: Pseudo-first order reaction model output

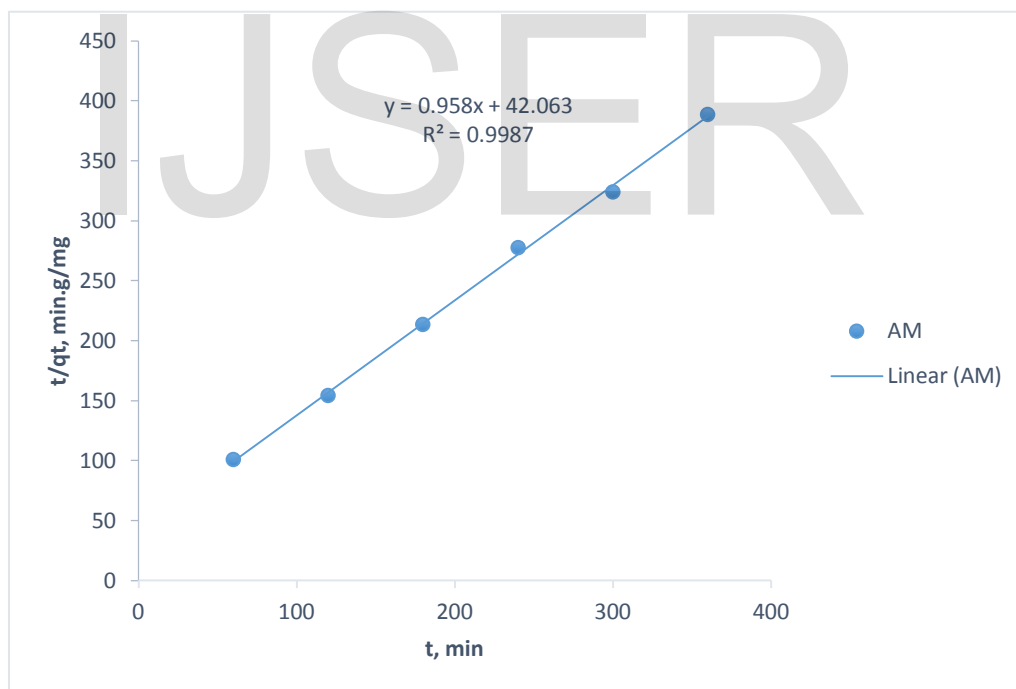


Figure 3: Pseudo-second order reaction model output

The kinetic analyses of both pseudo-first order model and pseudo-second order model were done in such a way that the equilibrium adsorption capacity, q_e , of the used adsorbents included the values measured after 400 min (0.9300 mg g^{-1}). It was assumed that the equilibrium of the system had been reached under this condition. From the results of fitting the data to the kinetic models, the respective square of correlation coefficients (R^2) value of pseudo-first order and pseudo-second order reaction model were estimated to be 0.8941 and 0.9987, respectively. Those results revealed that pseudo-second order reaction model was able to describe the data better due to the closeness of its correlation

coefficient value to 1 and the closeness of the values of its equilibrium adsorption capacity (q_e) to the estimated capacity values after 400 min. These also established that the adsorption of sulphur compounds by activated manganese oxide followed the pseudo-second order kinetic model, which was discovered to rely mostly on the assumption that chemisorption might be the rate-limiting step. In chemisorption, the sulphur sticks to the adsorbent surface by forming a chemical bond and tend to find sites that maximize coordination number with the surface. Similar hypotheses that the adsorption procedure in our study was primarily chemical adsorption were reported by Rozaini *et al.* (2010), Gupta *et al.* (2011) and Futralan *et al.* (2012). Thus, the sorption of sulphur was discovered to follow a pseudo-second order model, with chemical sorption as its rate limiting step.

4. CONCLUSION

From the results obtained in this research work, desulphurization efficiency was found to increase with increase in contact time. Also, there was reduction in the sulphur content of the examined diesel oil by 53% after desulphurization. However, the need to improve on the methods and the adsorbents that will result in ultra-clean sulphur free diesel fuel was found necessary. Furthermore, the kinetics analysis of manganese dioxide adsorbent on desulphurization of diesel oil proved that the activated manganese dioxide was efficient during the adsorption of sulphur compounds from diesel oil. Pseudo-second order kinetics model was observed to fit better into the experimental data obtained than pseudo-first-order model. Thus, the sorption of sulphur has been discovered to follow a pseudo-second order model, with chemical sorption being its rate limiting step.

NOMENCLATURE

C_e – balance sulphur content in diesel oil, mg kg⁻¹,
 C_0 – initial sulphur concentration, mg kg⁻¹,
 C_t – sulphur content in diesel oil at specified time, mg kg⁻¹,
 K_{s1} --adsorption rate constant of pseudo first-order kinetic model, min⁻¹,
 K_{s2} - adsorption rate constant of pseudo-second order, g mg⁻¹ min⁻¹,
 W – Mass of adsorbent, g,
 q_m – maximum adsorption capacity, mg g⁻¹,
 q_t –quantity of adsorbed sulphur on adsorbent surface, mg g⁻¹,
 q_e –equilibrium adsorption capacity, mg g⁻¹,
 t – time, min.

ACKNOWLEDGEMENT

Authors are indebted to Mallam Idris of Fuel and Gas Laboratory, Kaduna Refinery and Petrochemical Company, KRPC, Kaduna Nigeria, for providing the necessary facilities during the course of this research work.

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