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Desulphurisation of Kerosene by Adsorption Process

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ABSTRACT: The secondary contaminant such as sulphur and other impurities represent one of the major threats to burning properties of kerosene and to environment. The sulphur contaminant produces a strong odor, which if not removed can cause some problems. Several method used to deodorized kerosene are extraction method by adding paraffin and solvent into kerosene, thereby removing the odor. In this study, activated carbon 10-50mm of particle size gotten from burnt woods was used to deodorize kerosene. The exercise was carried out using a specific quantity of untreated kerosene with different quantity of activated carbon at different contact time. Also iodometric method was used to test for sulphide level in the deodorized kerosene.

KEY WORDS: Deodorization, Activated carbon, Adsorption, Kerosene.

I. INTRODUCTION

Kerosene, one of the byproducts of petroleum was discovered in 1853 by Abraham Gesner. A British physician, Gesner developed a process to extract the inflammable liquid from asphalt. The term kerosene is derived from the Greek word keros. Kerosene is a combustible hydrocarbon liquid, widely used to power jet engines of aircraft (jet fuel) and some rocket engines. It is also commonly used as a cooking and lighting fuel for fire toys. Kerosene lamps are widely used for lighting in rural areas, where electrical distribution is not available or too costly for widespread use. Kerosene, a thin, clear liquid formed from hydrocarbons, with a density of $0.78-0.81 \text{ g/cm}^3$, is obtained from the fractional distillation of petroleum between 150 °C and 275 °C, resulting in a mixture of carbon chains that typically contain between six and 16 carbon atoms per molecule. Once the oil has been distilled into its fractions, further processing in a series of chemical reactors is necessary to create kerosene. Catalytic reforming, alkylation, catalytic cracking, and hydro processing are four of the major processing techniques used in the conversion of kerosene. These reactions are used to control the carbon chain distribution by adding or removing carbon atoms from the hydrocarbon backbone. These reaction processes involve transferring the crude oil fraction into a separate vessel where it is chemically converted to kerosene. Major constituents of Kerosene include n-Dodecane, alkyl benzenes, and naphthalene and its derivatives. The flash point of kerosene is between 37 and 65 °C (100 and 150 °F), and its auto ignition temperature is 220 °C (428 °F)[1-5]

II. TEST METHODS FOR SULPHUR

1. Copper Test - Free sulphur, hydrogen sulphide, and perhaps other sulphur compounds, in oils can be detected by the formation of a dark spot of copper sulphide when two or three drops of the oil are left on the polished metal for a sufficient length of time. The " copper dish " test for gasoline is based upon this reaction. It is generally believed that the degree of darkening depends upon the amount of sulphur present. This is not true. A topped Mexican crude, which contained 4.05 per cent of sulphur, and which gave off streams of hydrogen sulphide when heated in a test tube, did not perceptibly darken copper in 72 hours. Even when a strip of the



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metal was partly immersed in the oil and kept at 295 to 300° C for 2 hours, the lower portion was only slightly tarnished. The end of the strip which was not under the oil was, however, much blackened and covered with a varnish-like coating, which was probably formed by partial oxidation of the oil. Although the copper test can not be relied upon to show the presence of all sulphur compounds in oils, it is a delicate means of detecting free sulphur and hydrogen sulphide.

A series of solutions was made by dissolving recrystallized sulphur in a white, medicinal oil which did not darken copper and which was found to contain too little sulphur to detect by analysis. The strongest solution contained o. 100 per cent of free sulphur. By suitable dilution, samples containing down to 0.000 per cent, or i : z 000 000, of sulphur were prepared. On each piece of freshly sandpapered, washed and dried piece of copper was placed two or three drops of oil. A blank test was run to check the original oil as well as the air of the laboratory. With 0 to 0.050 per cent of sulphur the copper was blackened in a few minutes, with 0.033 to 0.07 per cent the copper was perceptibly darker in 30 minutes, and with 0.01 per cent inside of an hour. When left in contact with copper for 20 hours, oil containing as little as 0.0002 per cent of sulphur, or 2 : 1 caused a just perceptible darkening of the metal. At the final dilution no action could be detected. Similar tests were made with oil into which hydrogen sulphide had been passed for a few minutes. This solution, which instantly darkened copper, contained 0.037 per cent of sulphur, equivalent to 0.039 per cent of hydrogen sulphide. A portion of this was diluted to 100 volumes and then three or four times to 10 volumes each. It is evident that the copper test is much less delicate than the sense of smell. [9]

2. Sodium test- If the oil is not too volatile, but can be heated to 250 to 300 ⊂ C without boiling out of the test tube, the sodium test can be applied. A test tube containing a few cubic centimeters of the oil is clamped in an inclined position over a flame which is regulated so that the oil is near its boiling point. A clean piece of sodium, 2 or 3 mm in diameter, is dropped in before the heating begins. After half an hour or longer, the oil is allowed to cool and is then poured off. The blackened lump of sodium is rinsed off with benzene, and dissolved in a few drops of water or 50 per cent alcohol. To the alkaline solution there are now added a few minute particles of sodium nitroprusside. If any sodium sulphide is present, the solution becomes pink to reddish purple, according to the amount. When only traces of sulphur are present, care must be taken not to mask the reaction by adding too much nitroprusside. Hydrogen sulphide does not produce a colored compound, so that the solution must not be acidified. Attempts to utilize the sodium method for the determination of sulphur were unsuccessful, because duplicate results varied too widely.[9]

III. METHODS OF DESULPHARIZATION SURVEYED

1. Adsorptive Desulfurization Of Kerosene And Diesel Oil By Zn Impregnated Montmorollonite Clay Desulfurization of kerosene and diesel oil has been carried out by selective adsorption through metals impregnated montmorollonite clay (MMT). Different metals were impregnated on MMT by wet impregnation method which included Fe, Cr, Ni, Co, Mn, Pb, Zn and Ag. The adsorption study was carried out in batch operation initially for 1 h time and at room temperature (25 ?C). The results show that high desulfurization was brought about by Zn-MMT. In the case of kerosene highest desulfurization of 76% and in the case of diesel maximum desulfurization process. Highest yield of desulfurization was obtained at 1-h stirring period, at room temperature (25 ?C) and using oil to adsorbent ratio of 20:1.5. Under optimized conditions the adsorbent was found to adsorb about 81% of DBT from the model oil containing 1000 ppm DBT dissolved in cyclohexane. EDX, Surface characterization and SEM analysis of the adsorbents used in the surface area, pore size and pore volume of the MMT has been found to be increased many fold with Zn impregnation. Also the surface morphology of the MMT has also been improved with Zn impregnation.[10]



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2. Oxidative Desulfurization Of Kerosene In The Presence Of Iron Chloride Ionic Liquid Catalyst And Ultra Sound- Oxidative Desulfurization of kerosene refinery in Tehran with sulfur content of 293% with iron chloride - hydrogen peroxide and ultra sonic liquid catalysts in the presence of acetic acid - formic acid and an oxidizing hydrogen peroxide has been studied. The effects of operating parameters such as temperature, reaction time, mole ratio of moles of sulfur oxidation (no/ ns), mole ratio of moles of acid per mol of sulfur (n acid / n s (on the desulfurization of kerosene checked (the molar ratio of oxidant to 15-40 and 20-80 mole ratio of sulfur to sulfur acid)

The results showed that the optimal conditions for the removal of sulfur from iron chloride catalyst system kerosene by 93% and sulfur content of residual 128 ppm is obtained. The effect of ultrasonic waves on system performance oxidation was studied, the results showed that the percentage of desulfurization systems, oxidation of acetic acid- hydrogen peroxide in combination with ultrasound 96% and without ultrasound was 93%, which indicates improved performance of oxidation in the presence of ultrasound [11].

3. Desulphurization By Adsorption-Sulphur causes environmental pollution if present in the liquid fuels. It is therefore needed to develop efficient and affordable processes for desulphurization of liquid fuels. Present review paper is aimed and studying the research carried out in order to investigate the mechanism of removal of sulphur from liquid fuel by adsorption using different adsorbent. Adsorption is a low cost and important physical process for the removal of sulphur from liquid fuel. Adsorption is a process of accumulation of one substance over another. It occurs between liquid-liquid, gas-liquid, gas-solid, or liquid solid interface. Typical liquid separation includes the removal moisture dissolve din gasoline decolourization of petroleum products and fractionations of mixtures of aromatic and paraffinic hydrocarbons .Adsorption may be physical or chemical. In physical adsorptions or activated adsorption is an interaction between the solid and adsorbed substance [6-8].

IV. MATERIALS AND METHODS

- Method: Adsorption by activated carbon
 - 1. Powdered activated carbon (PAC)
 - 2. Granular activated carbon (GAC)

Materials: Raw kerosene, Activated carbon (PAC & GAC)

Equipment: Filtering paper, measuring cylinder, glass sample bottle, conical flask, glass funnel beaker, burner, tripod stand and holders for funnels. Procedure:

- 1) Desulphurization of kerosene using adsorption process simply means removal of sulphur compounds like sulphide as in this case from product.
- 2) The method used is a Batch adsorption, consists of contacting finely activated carbon with the raw kerosene for a given period of time in a beaker. The activated carbon used in this process is typically supplied in a powdered form to maximize surface area for mass transfer and minimize agitation for suspension.
- 3) A specific amount of activated carbon is weighed in a triple weighing balance to determine the weight, and poured into a beaker. 20ml of raw kerosene is poured into the beaker containing the weighed activated carbon and allowed for a given time to enable adsorption to take place. The adsorbent, been the activated carbon, adsorbed the adsorbate (impurities) in the raw kerosene, which leaves it partly or wholly deodorized.
- 4) After the process completion, (typical contact time: 15 75 minutes) the spent carbon is separated from



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the kerosene (e.g., by filtration) and regenerated or disposed of.

- 5) A filter paper with funnel was used to separate the purified kerosene from activated carbon.
- 6) Iodometric method was used to test for sulphide in kerosene after the deodorization process. A clean conical flask was used to measure 5ml of deodorized kerosene.
- 7) 0.75ml of 0.025N of iodine solution was added to kerosene and allowed for 10 minutes observational time, the sample was titrated with 0.025N sodium thiosulphate, using starch as indicator.
- 8) The change from blue black to colorless indicates the end point.

V. OBSERVATIONS

Table 1: For Powdered activated carbon (PAC)

Sr.no.	Mass of PAC used	Volume of untreated kerosene used	Contact Time of raw kerosene and carbon	End point Results (titration with sodium thiosulphate)	End point Results In S ² (mg/1)
1.	1 gm	20 ml	15 min	6.8 ml	484
2.	1 gm	20 ml	30 min	5.4 ml	372
3.	1 gm	20 ml	45 min	2.0 ml	100
4.	1 gm	20 ml	60 min	1.1 ml	28
5.	1 gm	20 ml	75 min	0.7 ml	4

Table 2: For Granular activated carbon (GAC)

Sr.no.	Mass of PAC used	Volume of untreated kerosene used	Contact Time of raw kerosene and carbon	End point Results (titration with sodium thiosulphate)	End point Results In S ² (mg/1)
1.	4gm	20 ml	15 min	9.0ml	660
2.	4gm	20 ml	30 min	4.0ml	260
3.	4gm	20 ml	45 min	2.7ml	156
4.	4gm	20 ml	60 min	2.2ml	116
5.	4gm	20 ml	75 min	1.6ml	68

Case B: For constant time period

Table 3: For Powdered	activated carbon	(PAC)
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Sr.no.	Mass of PAC used	Volume of untreated kerosene used	Contact Time of raw kerosene and carbon	End point Results (titration with sodium thiosulphate)	End point Results In S2(mg/1)
1.	0.25gm	20 ml	45 min	5 ml	340
2.	0.50 gm	20 ml	45min	2.1ml	108
3.	0.75gm	20 ml	45 min	1.4 ml	52
4.	1 gm	20 ml	45 min	0.9 ml	12
5.	1 .25gm	20 ml	45 min	0.5 ml	20

CASE A



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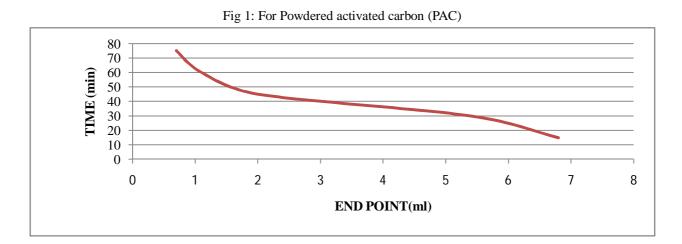
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Table 4: For Powdered activated carbon (PAC)

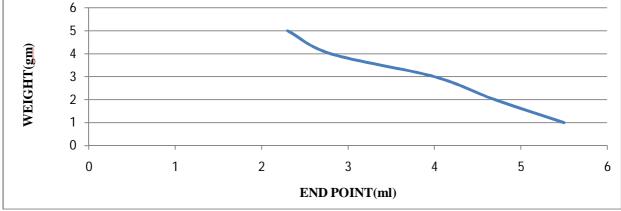
Sr.no.	Mass of	Volume of	Contact Time	End point Results	End point
	PAC used	untreated	of raw	(titration with sodium	Results In
		kerosene used	kerosene and	thiosulphate)	S2(mg/1)
			carbon		
1.	1 gm	20 ml	45 min	5.5 ml	380
2.	2 gm	20 ml	45 min	4.7 ml	316
3.	3 gm	20 ml	45 min	4.0 ml	260
4.	4 gm	20 ml	45 min	2.8 ml	164
5.	5 gm	20 ml	45 min	2.3 ml	126

VI. RESULTS AND DISCUSSION

From above calculation Case A: For constant weight of activated carbon









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Case B: For constant time period

Fig 3: For Powdered activated carbon (PAC)

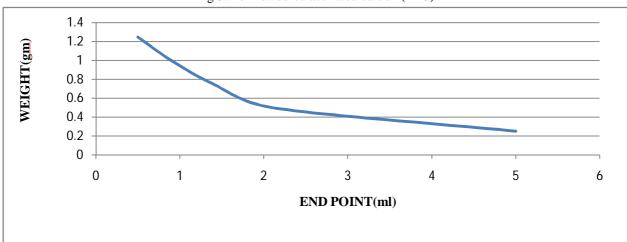
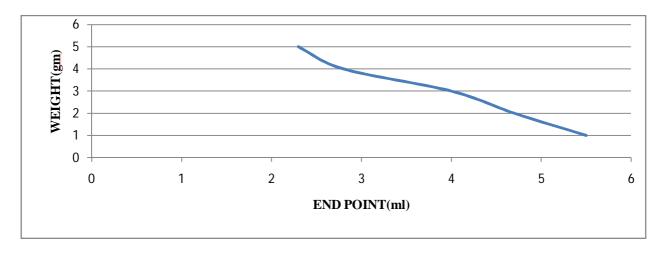


Fig 4: For Powdered activated carbon (PAC)



VII. CONCLUSIONS

The desulphurization of kerosene was studied varying adsorbent, time and quantity of adsorbent. It was found that powdered activated carbon is a more effective method. Granular activated carbon was found to be less effective. Hybrid methods like oxidative desulphurization with ultrasound maybe more effective with respect to time consumed for adsorption.

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