

Deep desulfurization of hydrotreated diesel fraction using oxidation process followed by extraction

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Abstract: The main goal of this research focuses on a desulfurization process for diesel fuel based on the chemical oxidation followed by liquid-liquid extraction. This technology was also applied on a model fuel (n-dodecan, o-xylene) contains sulfur compounds (dibenzothiophene and its derivatives 4-methyldibenzothiophene, 4,6-dimethyldibenzothiophene) simulating that exist in actual diesel fuel, by using hydrogen peroxide as oxidant and formic acid as catalyst at temperature 50°C with atmosphere pressure. Oxidation products (sulphoxides and sulphones) were extracted by acetonitrile. The results indicated that the present process can remove substituted dibenzothiophene from diesel fuel up to 99.4%, and rates conversion of dibenzothiophene, 4-methyldibenzothiophene, 4,6-dimethyldibenzothiophene were 95.4%, 97.4% and 99.3% respectively. This technique can be used as a subsequent process to the hydrodesulphurization, it considered as one of the most promising methods for removing sulfur compounds from oil fractions.

Keywords: Desulfurization, Oxidation, Extraction, Technology, Sulphoxides, Sulphones.

Introduction

The presence of sulfur compounds in petroleum fractions is the main cause of air pollution and acid rains. It can cause also several corrosion problems in vessels, pipelines, engines, refining equipment, and pumping^(1,2,3). The removal of sulfur compound from transportation fuels classified at the top of priority list of environment issues after the carrying out of worldwide strict emission regulations. In Europe, the sulfur level in max mass % for liquid fuels is presently limited at 0.015, 0.035 for gasoline, diesel fuel, respectively⁽⁴⁾. New sulfur limited of 0.003-0.005 mas% for gasoline and diesel fuel will be introduced in coming years^(5,6).

The most commonly used process for removing sulfur compounds from diesel fuel is hydrodesulfurization. This technique is highly efficient for removing simple sulfur compounds (thiols, sulfides, and disulfides), but it's limited in treating stubborn refractory sulfur compounds such as dibenzothiophene and its derivatives. More severe operating conditions are required to remove these compounds including higher hydrogen consumption and the use of special active catalysts. Moreover, use of additional infrastructure which needs higher pressure and temperature, and larger reactor volume with more active catalysts in the treatment facility. However, the operation at high conditions leads to a lot of problems, such as a one-time investment, high capital, higher manufacturing and operating cost and reduction of the length of the catalyst life cycle. All these conditions increase the price of the resulting fuel⁽⁷⁾. Therefore, several new processes are considered as alternative or complementary technologies to HDS for deep desulfurization. These include biodesulfurization, adsorptive desulfurization, extractive desulfurization, desulfurization via precipitation.

In biodesulfurization process (BDS) the removal of recalcitrant organic sulfur compounds from feedstock is depending on using a series of enzyme-catalyzed reactions, but two factors limit the application of this method.

Firstly, temperature, disinfection, stability and lifetime of biocatalyst extremely control the biodesulfurization processes. Secondly, the non-compatibility between the organic solvents and most bacterial kinds limits the application of BDS^(8, 9). While adsorptive desulfurization could be accomplished depending on physical adsorption of organic sulfur compounds on the solid sorbent surface and this process is called desulfurization by adsorption (ADS). ADS is based on the ability of solid sorbent to the selectively adsorb sulfur compounds from refinery streams. The sorbent properties such as capacity, selectivity towards sulfur compounds, attraction, and stability, in addition to quick overloading of the sorbent that required to treat high sulfur refinery streams limit this process⁽⁵⁾. Also, extractive desulfurization depends on the solubility difference between organic sulfur compounds and hydrocarbons in an appropriate solvent, but the resemblance of polarity between aromatic sulfide compounds and these without a sulfur atom make the process in effective⁽⁵⁾.

The essential step which the desulfurization via precipitation process based on is the formation of insoluble charge-transfer complexes then remove them in consequent stage. The emerging competition between organic sulfur compounds (DBT and its derivatives) and other non-sulfur aromatics in the formation of complex causes weak selectivity in removal of DBT and its derivatives, making the effectiveness of this process is very low⁽¹⁰⁾.

In the present work we discussed oxidative desulfurization as a subsequent technology for hydrodesulfurization (HDS), it's generally consists of two processes; the first step is oxidation of organic sulfur compounds in fuels, and the next step is the removal of oxidized sulfur-containing compounds from the treated fuels. The reactivity of this technique was examined for two kind of diesel fuel and for a model fuel. Also the feasibility of using appropriate oxidation agent and catalyst at middle conditions was tested.

Experimental procedures:

The sulfur content in diesel fuel was determinate according to the method of Siddiqui et al.2006⁽¹¹⁾.

Materials:

All reagents and solvents in this work are available commercially and were used as received. The model compounds and chemicals, including: dibenzothiophene (DBT 98%), 4-methyldibenzothiophene (4-MDBT 96%) 4,6-dimethyldibenzothiophene (4,6-DMDBT 97%), n-dodecan, O-xylene 99%. Hydrogen peroxide 33%, Formic acid 98%, Hydrochloric acid 37%, Acetic acid 99.8%, Acetonitrile were obtained from Sigma Aldrich.

Fuels Samples:

The two samples used in this research are hydrotreated diesel and unhydrotreated diesel that obtained from Banaias Refinery Company. They undergo to experiments in central laboratory in Faculty of Science chemistry department of Damascus University.

The chemicals characterization for the oxidation products was accomplished by means of elemental analysis, infrared spectroscopy (IR), and nuclear magnetic resonance (¹H NMR and ¹³C NMR), which confirmed their structures.

Preparation of model fuel and mixture of oxidant with catalyst:

To prepare a solution which could act as an appropriate sample of real fuel, the model fuel was prepared by dissolving 0.1g of DBT in about 21 ml n-dodecan and 4 ml o-xylene. Hydrogen peroxide and formic acid mixtures for all experiments were freshly prepared each time; this mixture consists of 7 ml H₂O₂ 33% and 3ml HCOOH 98%. Oxidation of model fuel was studied at different temperatures (room temperature, 50°C, 80°C), during a period of time 2 hours with stirring, to study the effect of temperature on the oxidation process. Then, during the cooling process a white precipitate was formed. This product was taken, washed, dried and analyzed with different analytical techniques infrared spectroscopy (IR), elemental analysis, and nuclear magnetic resonance (¹H NMR and ¹³C NMR) to determine the structure.

Effect of acid selectivity on oxidation process:

Oxidation of model fuel was carried out in three experiments; each experiment uses a certain acid catalyst with the oxidant different from the acid that uses in the other experiment.

Three experiments using (7ml HCL 37%, 5ml CH₃COOH 99.8%, 3ml HCOOH 98%) with the same volume of the oxidant (7ml H₂O₂ 33%) in each experiment.

All these mixtures were using to oxidize the model fuel at 50°C for 2 hours. Then the products were collected and analyzed by (IR, ¹H NMR and ¹³C NMR) technique.

Oxidation other sulfur compounds.

In order to study the effect of this process on the substituted dibenzothiophene and so to obtain the real diversity of these compounds in the fuel, we studied the following compounds. These compounds are 4-MDBT and 4,6-DMDBT. So, the same previous model fuel was prepared twice. In the first we dissolved 0.1g of 4-MDBT instead of DBT in the model fuel, and in the second we dissolved about 0.1g of 4,6-DMDBT instead of DBT in the model fuel. Oxidation of two models fuel was carried out with a mixture of (H₂O₂ and HCOOH) at 50°C for 2 hours. The same steps were repeated as in the previous.

Oxidation of real diesel fuel and effect solvent selectivity.

For the oxidation of diesel oil an oxidizing medium was developed by mixing 1:1.5 ratio of (H₂O₂ 33% and HCOOH 98%) respectively. The mixture was added to three samples of 50ml of hydrotreated diesel and added also to three samples of 50ml of unhydrotreated diesel. The six samples were heated at 50°C for 2 hours. Aqueous peroxide/ acid phase from the diesel was completely separate out using separating funnel.

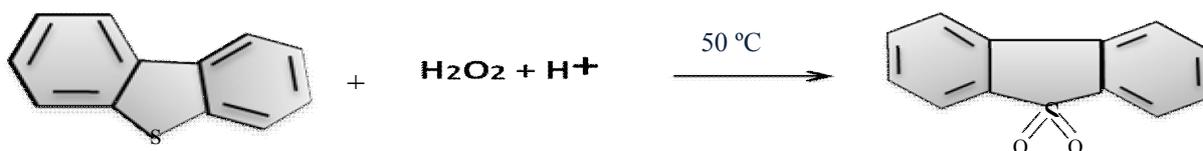
All samples washed thoroughly with 7ml of distilled water, the upper layers was submitted for total sulfur determination by using RX-360SH(Tanaka scientific), and the top layer of each sample was separated. In the oxidation process the typically bounded sulfur thiophenes get converted into sulfones, these sulfones from diesel were extracted using acetonitrile. So, all samples were washed again with 30ml of acetonitrile and the solvent layer was separated from the diesel, and diesel layer had been analyzed by RX-360SH to determine the total sulfur content.

The effect of direct extraction with acetonitrile on diesel quality.

About 30ml of acetonitrile were added for two 50ml samples of hydrotreated diesel and unhydrotreated diesel separately. All were mixed well and left at room temperature for settling down for overnight. The diesel layers were separated and submitted to total sulphur determination by using RX-360SH.

Results and discussion.

The experimental results indicate that: it is possible to convert thiophene, dibenzothiophene, 4-methyldibenzothiophene, 4, 6-dimethyldibenzothiophene into their corresponding sulfones. Where the following compounds were formed during the oxidation process, at first we get on the product "dibenzothiophenesulfone" dibenzo[b,d]thiophene 5,5-dioxide in the formula C₁₂H₈SO₂" as shown in the following reaction:



And then we get on 4-methyldibenzothiophene sulphone (4-MDBT₂) in the formula C₁₃H₁₀SO₂ and 4,6-dimethyldibenzothiophene sulphone (4,6-DMDBT₂) in the formula C₁₄H₁₂SO₂ in the same way. The rate of conversion 4-DBT into 4-DBT₂ was 97.4%, while the rate of conversion of 4,6-DMDBT into 4,6-DMDBT₂ was 99.4%.

The effect of temperature's and acids on the oxidative desulphurization of DBT is shown in tables (1),(2):

Table(1): DBT oxidation at different temperatures

Temperature (°C)	Rate of oxidation(DBT) %
Room temperature (22°C)	87%
50°C	95.40%
80°C	96.80%

The results in table (1)are shown that: reaction rate is increasing when reaction temperature increased from 50°C to 80°C.However oxidation at higher temperature than 80°Cwas unfavorable because it leads to oxidation of useful compounds present in fuel. Also at higher temperature more than 80°C hydrogen peroxide decomposes to undesirable side products other than hydroxyl radicals, which decrease the efficiency of diesel desulfurization process and effects on fuel quality⁽¹²⁾.

Table(2): Oxidation of DBT using different acids at 50°C

Acid catalyst	Rate of oxidation(DBT) %
HCl	87.2
HCOOH	96.2
CH ₃ COOH	84.6

From table (2): The results indicate that the formic acid has greater oxidation effect than in the case of both CH₃COOH and HCl, and the strong acid accelerate the decomposition of H₂O₂ to oxygen and water, resulting in a poor yield of oxidized DBT.

Results of elemental analysis

Table (3)shows theelemental analysis for isolated sulfone compounds (C₁₂H₈SO₂, C₁₃H₁₀SO₂, C₁₄H₁₂SO₂)

#	Name Compound	Formula	Testing values			Calculated values		
			C[%]	H[%]	S[%]	C[%]	H[%]	S[%]
1	Dibenzothiophenesulfone	C ₁₂ H ₈ SO ₂	66.72	3.659	14.327	66.65	3.70	14.81
2	4-methyldibenzothiophene sulfone	C ₁₃ H ₁₀ SO ₂	66.12	4.412	14.083	67.82	4.34	13.91
3	4,6-dimethyldibenzothiophene sulfone	C ₁₄ H ₁₂ SO ₂	68.96	5.415	13.333	68.85	4.92	13.11

The results indicate that the percentages of elementals for sulfone product C₁₂H₈SO₂ (calculated and testing) are convergent, the same matter for C₁₃H₁₀SO₂ and C₁₄H₁₂SO₂.

Results of IR analysis for sulphone products " C₁₂H₈SO₂, C₁₃H₁₀SO₂, C₁₄H₁₂SO₂"

Table (4) shows the spectroscopies data (IR) for isolated sulfone compounds C₁₂H₈SO₂,C₁₃H₁₀SO₂, C₁₄H₁₂SO₂

Compound	IR
1	$\nu[(S)]=1286.29, 1163.38 \text{ cm}^{-1}$ (at T=50°C, HCOOH as catalyst) 
2	$\nu[(S)]=1285.32, 1158.04 \text{ cm}^{-1}$ (at T=50°C, HCOOH as catalyst) 
3	$\nu[(S)]=1281.47, 1154.19 \text{ cm}^{-1}$ (at T=50°C, HCOOH as catalyst) 

We had the same result when we used different acids at different temperatures in DBT case.

Results of nuclear magnetic resonance (NMR) for'' C₁₂H₈SO₂, C₁₃H₁₀SO₂, C₁₄H₁₂SO₂''

Table (5) shows Spectroscopy data (¹³C NMR, ¹H NMR) for isolated sulfone compounds C₁₂H₈SO₂, C₁₃H₁₀SO₂, C₁₄H₁₂SO₂.

Compound	¹³ C NMR		¹ H NMR
1	121.58 ppm 122.2 ppm 130.4 ppm 131.6 ppm 133.8 ppm 137.7 ppm		7.28 ppm (2H) 7.57 ppm (2H) 7.66 ppm (2H) 7.84 ppm (2H)
2	121.58 ppm	131.86 ppm	7.27 ppm (2H) 7.50 ppm (2H) 7.63 ppm (2H) 2.64 ppm (CH ₃ group)
	122.2 ppm	133.85 ppm	
	130.4 ppm	137.85 ppm	
	16.98 ppm (CH ₃ group)		
3	118.77 ppm	131.91 ppm	7.27 ppm (2H) 7.50 ppm (2H) 7.63 ppm (2H) 2.73 ppm (CH ₃ group)
	119.46 ppm	133.4 ppm	
	130.91 ppm	137.59 ppm	
	16.87 ppm (CH ₃ group)		

The results indicate that the compound C₁₂H₈SO₂ has 12 carbon atoms; the previous chemical shifts are the shifts attributable to six carbon atoms in the first half of the compound, and the fact that it's a symmetric compound. The six carbon atoms in the second half of the compound have the same chemical shifts that belong to the first half of the compound. The same matter for the substituted dibenzothiophene C₁₃H₁₀SO₂ (4-MDBT₂SO₂) and C₁₄H₁₂SO₂ (4,6-DMDBT₂SO₂) and there is an additional absorption peaks due to the methyl group (CH₃) as it explained in the previous table.

¹H-NMR spectrum of the compound C₁₂H₈SO₂ (DBT₂SO₂) shows four proton groups their shifts explained in the previous table, the compound DBT₂SO₂ is symmetric, and protons in every site are exposed to split. So, every site will has two protons (doublet doublet), while in ¹H-NMR spectrum of the compound 4-MDBT₂SO₂ the appearance of additional absorption peak due to (CH₃) group protons at 2.64 ppm, also ¹H-NMR spectrum of the compound 4,6-DMDBT₂SO₂ shows hiding for absorption peak due to proton group which has the shift 7.66 ppm and appearance of sharp absorption peak has shift equal 2.73 ppm due to two methyl group protons.

Oxidative desulfurization (ODS) of diesel fuel.

Some of general properties of unhydrotreated diesel and hydrotreated diesel are given in the table (6).

Table (6): general properties of unhydrotreated diesel and hydrotreated diesel.

CHARACTERISTICS	Un hydrotreated diesel		Hydrotreated diesel	
	METHOD	VALUE	METHOD	VALUE
Sp. GR. At 60/60 F°	D-1298	0.8696	D-1298	0.8374
Density at 15 C°	By Table/23B/	0.8692	By Table/23B/	0.8370
Water by dis % vol	D-2709	NIL	D-2709	NIL
Flash point pro A C°(P.m)	D-93	70	D-93	70
C.F.P.P C	IP-309	+4	IP-309	-4
Pour Point C	D-97	-10	D-97	-10
Distillation IBP C	D-86	180	D-86	180
5% VOL at C		204		197

10% VOL at °C		219		208
50% VOL at °C		284		264
90% VOL at °C		360		336
95% VOL at °C		=		255
At 200 °C %VOL		4		7
At 250 °C %VOL		27		40
At 300 °C %VOL		62		63
At 350 °C %VOL		88		74
At 360 °C %VOL		90		96
Kin viscosity at 37.8 °C cst	D-445	3.25	D-445	2.7
Aniline Point °C	D-611	59	D-611	67
Diesel. Index	BY CAL	43	BY CAL	58
Corrosion test 3h@50 °C	D-130	1a	D-130	1a
Sulfur % wt	D-1266	0.3262	D-1266	0.0176
Acids @ Alkalis mg KOH/g	D-974	0	D-974	0

The results of the oxidative desulfurization of diesel are given in the following tables:

Table (7): Results of oxidative desulfurization of hydrotreated diesel.

Sulfur % wwt ASTM -D-4294	Diesel Index BY CAL	Aniline point °C ASTM-D-611	Sample Number	Sample Kind
0.0176	58	67	1	Hydrotreated diesel before oxidation
0.0166	-	-	1	Oxidized hydrotreated diesel and washed with distillate water (9 ml HCOOH -13.9 ml H ₂ O ₂)
0.0167	-	-	2	
0.0163	61	72	3	
0.0008	60	71.2	1	Hydrotreated diesel treated with oxidation and extracted with acetonitrile
0.0007	60	71	2	
0.0003	59	70.4	3	
0.0166	-	-	1	Oxidized hydrotreated diesel and washed with distillate water (9 ml HCOOH -17.25 ml H ₂ O ₂)
0.0165	-	-	2	
0.016	60	71	3	
0.0006	60	71.6	1	Hydrotreated diesel treated with oxidation and extraction with acetonitrile
0.0003	59	70.4	2	
0.0001	60	71.8	3	

Table (8): Results of oxidative desulfurization of unhydrotreated diesel.

Sulfur % wt ASTM - D-4294	Diesel Index BY CAL	Aniline point C° ASTM-D-611	Sample Number	Sample Kind
0.3262	43	59	1	Unhydrotreated diesel before oxidation
0.2854	44	61	1	Oxidized un-hydrotreated diesel and washed with distillate water (9 ml HCOOH -13.9 ml H2O2)
0.2844	-	-	2	
0.2818	-	-	3	
0.1416	48	67.2	1	Unhydrotreated diesel treated with oxidation and extracted with acetonitrile
0.0834	47	65.8	2	
0.0553	48	68	3	
0.3158	49	68.6	1	Oxidized unhydrotreated diesel and washed with distillate water (9 ml HCOOH -17.25 ml H2O2)
0.3137	47	66	2	
0.2969	48	68	3	
0.2865	46	64.4	4	
0.0488	46	64.2	1	Unhydrotreated diesel treated with oxidation and extracted with acetonitrile
0.0487	47	65.8	2	
0.0486	47	65.4	3	

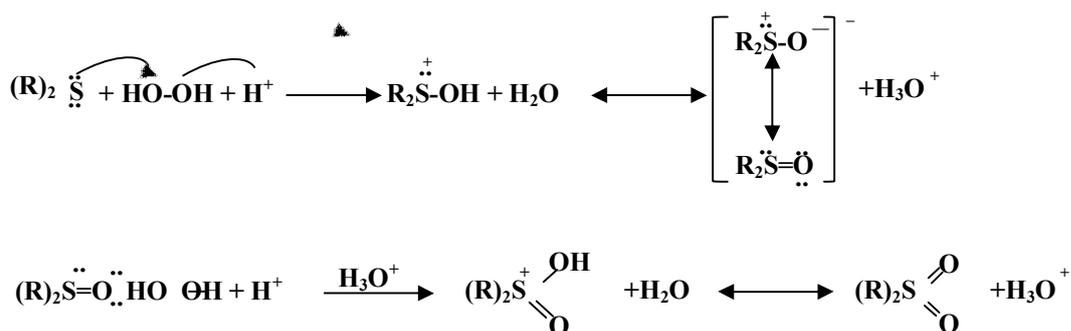
According to the obtained data, the total sulfur content in hydrotreated diesel was lower after the oxidation/extraction process (from 0.0176 wt % to 0.0003 wt %) and in unhydrotreated diesel was lower after oxidation /extraction process (from 0.3262 wt % to 0.0553 wt %), while aniline point it's obvious that it raised after oxidation/extraction on diesel fuel (hydrotreated,unhydrotreated) and that indicate to decrease in percentage of aromatic sulfur compounds in it.

Changes of the reaction results are depending on the amount of (H₂O₂). However, the diesel conversion (removal sulfur) increased markedly when the amount of H₂O₂ increased from 13.9ml to 17.25ml. Total sulfur content in hydrotreated diesel lower to 0.0001 wt % and lower in unhydrotreated diesel to 0.0486 wt %.

We can explain oxidation of hindered sulfur compounds as the following:

Sulfur compounds can oxidized to their corresponding sulfones products through sulfoxides stage in the presence of acid / H₂O₂. The oxidation of organic sulfur compounds are depending on position of alkyl group and electron density. The reactivity is increased with the increase of electron density on sulfur atom. The reactivity of DBT derivatives are influenced by the electron donation of substituted methyl group and increased as the flowing: 4,6-DMDBT > 4-MDBT > DBT

The following proposed mechanism has helped to explain the reaction requirements for oxidation of hindered compounds that exist in diesel⁽¹⁴⁾.



The first step is electrophilic attack of the H₂O₂ by the electron of sulfur atom. The increasing of electron density at sulfur atom plays a major factor on the rate of oxidation reaction. Beside that: the position of alkyl group would have effect on electron density of DBT_s. The attached alkyl groups to the aromatic rings will increase the electron density at sulfur atom. So, the reactivity of molecules such as (4,6-DMDBT) is substantially higher than of (DBT)⁽¹³⁾.

The effect of acetonitrile extraction on diesel quality

The hydrotreated and unhydrotreated diesel samples were submitted to direct acetonitrile extraction without any oxidation. The results are shown in these tables.

Table (9).Results of direct extraction of unhydrotreated diesel.

Sulfur % wt ASTM-D- 4294	Diesel Index By CAL	Aniline point °C ASTM-D-611	Sample Number	Sample Kind
0.3262	43	59	1	Un hydrotreated diesel without any treatment process
0.25	48	68	1	Unhydrotreated diesel before oxidation but extracted directly with acetonitrile

Table (10).Results of direct extraction of hydrotreated diesel.

Sulfur % wt ASTM - D-4294	Diesel Index BY CAL	Aniline point C° ASTM-D-611	Sample Number	Sample Kind
0.0176	58	67	1	Hydrotreated diesel without any oxidation process
0.011	62	73.8	1	Hydrotreated diesel extracted directly with acetonitrile.

Using acetonitrile as a solvent to extract sulfur compounds from diesel samples without any pretreatment for these samples cause a markable effect, that the total sulfur content was reduced from 0.3262 wt % to 0.25 wt %.

For the hydrotreated diesel sample without any oxidation treatment, the total sulfur content was reduced from 0.0176 wt % to 0.011 wt % after some hours and extracted with 30ml of acetonitrile.

The efficiency of extracting process depends on the volume ratio of solvent to solute. So, the smaller ratio of acetonitrile to the diesel lead to more difficulty and long time to get on good separation, but the greater volume of acetonitrile cause loss of diesel layer and cost higher. In the experiments 30ml of acetonitrile to 30ml diesel requires overnight to produce a better separation.

The results indicate that the direct extraction didn't give promising results. Because, it causes removal some hydrocarbons with sulfur compounds removal, that due to small difference of polarity between aromatic hydrocarbons and sulfur compounds and this effect on the product, and didn't reduce the sulfur content to the required level. While if sulfur containing compounds that are present in diesel fuel oxidized to their corresponding sulfones their solubility in polar solvents would increase with an increase in their polarity. The dipole moment of the oxidized sulfur containing compounds increases and they are more soluble in polar solvents.

Conclusion:

The oxidative desulfurization sulfur(ODS) technology has been found a very active way for the oxidation of model fuel. The conversion of DBT was nearly more than 95% at 50C° and the reaction time was 2 hours.

The ODS of diesel fuel has been successfully applied at the previous conditions and the result showed decreasing sulfur content from 0.0176 wt % to 0.0001 wt % for hydrotreated diesel and from 0.3262 wt % to 0.0486 wt % for unhydrotreated diesel, by oxidation with H₂O₂ / HCOOH and liquid-liquid extraction with acetonitrile.

Almost 99% of sulfur compounds in the original hydrotreated diesel and about 85 wt % of sulfur compounds in the original unhydrotreated diesel were removed by oxidation reaction followed by extraction process.

The direct extraction of diesel fuel without any oxidation has resulted in about 37.5 wt % sulfur removal from hydrotreated diesel and 23.36 wt % sulfur removal from unhydrotreated diesel.

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