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Performance of Surfactant-Type Polyoxometalate, $[(C_{16}H_{33})\ N(CH_3)_3]_6\ [PV_3W_9O_{40}]$ in Oxidative Desulfurization of Gas Condensate and Straight Run Gasoil

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ABSTRACT

A novel hybrid surfactant-type polyoxometalate, $[(C_{16}H_{33})\ N(CH_3)_3]_6\ [PV_3W_9O_{40}]$ was synthesized and characterized by FT-IR, SEM, and EDX analysis. Oxidation of sulfur compounds in gas condensate with H_2O_2 as oxidant using surfactant-type polytangestovanado phosphoric catalyst and acetonitrile as solvent was studied. The total sulfur content of the samples was determined by UV Fluorescence. The catalyst was evaluated for the oxidative desulfurization of gas condensate from Ilam Refinery Company and straight-run diesel from Kermanshah Oil Refining. The findings reveal that the catalysts preserve the Keggin structure. The sulfur level of a gas condensate can be lowered from a few hundred 1800 µg/g to 83 µg/g only at 5 min and the sulfur removal of 98.83% was obtained at 15 min. Also, the desulfurization rate of straight-run diesel can be up to 82% at 30 min. In addition, the reusability of the catalyst after five times showed that the catalytic activity had a decrease from 97.22 to 92.11 percent sulfur removal of gas condensate.

Introduction

Environmental concerns and the growing interest in fuel cell application have made it increasingly important to study the desulfurization of liquid transportation fuels. The combustion of fossil fuels containing sulfur generates sulfur oxides (SOx) and particulate matter (PM), leading to air pollution and cause to serious diseases of the human respiratory system [1]. Additionally, SOx molecules play a significant role in the formation of acid rain, which is dangerous for both the environment and man-made objects. [2]. Due to the extremely strict environmental rules, the sulphur content of diesel has been restricted to less than 15 g/g in the US since 2006, less than 15 g/g in Europe since 2009, and fewer than 50 g/g in Iran since 2008. [3, 4]. One of the most popular desulfurization techniques used in refinery processes since the 1950s is hydrodesulfurization (HDS) [5]. At high temperatures and partial pressures of hydrogen, the HDS process involves catalytic treatment with hydrogen to convert various sulphur compounds to H2S and sulphur free organic compounds [6]. It is not possible to achieve ultra-low sulphur levels due to the refractory S-containing aromatic compounds such as dibenzothiophene (DBT) and its derivatives, particularly the alkyldibenzothiophenes, unless

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extreme operating conditions of high temperature, high pressure, and high hydrogen consumption are met, as well as the use of a more active catalyst, which necessitates a significant capital investment. [7, 8]. As a result, alternative, more energy-efficient deep desulfurization methods, are desirable, such as extraction [9, 10], adsorption [11, 12], oxidation [13, 14], as well as bioprocess[15, 16].

One of the most promising approaches for removing sulphur at ambient temperature and atmospheric pressure is oxidative desulfurization (ODS). Heavy sulphides are oxidised in ODS by adding one or two oxygen atoms to the sulphur without breaking any carbon-sulfur bonds, generating sulfoxide and sulfone, respectively. Due to their enhanced relative polarity, the oxidised chemicals are then removed or adsorbed from the light oil. [17]. The different oxidants used in ODS process, such as organic hydroperoxides [18], molecular oxygen [19], peracids [20], hydrogen peroxide [21] and others. H2O2 is considered one of the greatest oxidising agents and is often utilised due to its perceived environmental friendliness, generating just water as a by-product. [22].

A range of heterogeneous and homogeneous catalysts have been researched for ODS process enhancement to reduce hydrogen peroxide consumption and reaction time, such as formic acid [23], acetic acid [20, 23], transition metals supported on porous solid [24], zeolites [25], hetropolyacides [26] and hetropolyoxometalate [27].

POMs are anionic oxoclusters of early transition metals at their maximum oxidation state, such as Mo6+, W6+, V5+, and, less commonly, Nb5+ and Ta5+. POMs are classified into two groups based on their chemical composition: isopolyanions ([MmOy] p) and heteropolyanions ([XxMmOy] q), where M is the primary transition metal atom and O is the oxygen atom. The principal heteroatom X, which might be a non-metal, can also be integrated into the heteropolyanions (such as P, Si, As, and Sb) [28]. The use of hetropolyoxometalate in combination with surfactants makes use of advantages emulsion phase that reduces mass transfer limitation between two phases. Several researchers reported several kinds of surfactant-type polyoxometalate such as $Q_4W_{10}O_{32}$ (Q= quaternary ammonium cation with different lengths of the alkyl chain)[29], $[(C_{18}H_{37})_2N(CH_3)_2][PW_{12}O_{40}]$[30], and Phosphomolybdic acid (HPMo) modified with quaternary ammonium cation [31] as catalysts, leading to the formation emulsion droplet in ODS system with H_2O_2 as oxidant.

In this study, keggin tangestatvanadophsphoric acid was modified with hexadecyl ammonium [(CH3) N (C16H33)$_3$]$^+$ and used as a catalyst in an ODS system. The amphiphilic catalyst is made up of a lipophilic tail quaternary ammonium with a single long carbon chain that acts as a surfactant and a hydrophilic catalyst made up of hetropolyoxometalat. As an oxidant, hydrogen peroxide was employed, and gas condensate was chosen as the fuel model.

Experimental

Materials

Sodium tangestate ($NaWO_4.2H_2O$), phosphoric acid(85%), and acetic acid(99%) were purchased from Merck Co. (Germany) and hydrogen peroxide(30%) was purchased from B.P medical (Holland), and sodium metavanadate and acetonitrile from Sigma chemical co. Condensate with a sulfur content of 1800 µg/g and Straight-run diesel with a sulfur content of 5500 µg/g were obtained from a domestic gas and petroleum refinery respectively.

Characterization Methods

A Nicolet Nexus $_{470}$ FT-IR spectrometer was used to obtain the infrared spectra (IR) of the catalysts. The scanning limitations were in the 4000 to 400 cm-1 range. The SEM picture was created using the JEM-2100F instrument. EDAX (energy dispersive X-ray analysis) was used to account for the C, P, W, and V elements. Antek multitek vertical sulphur Model 734 UV Fluorescence was used to determine the total sulphur content of the samples.

Catalyst Preparation

First, 12 g of Na2WO42H2O was stirred into 15 ml of distilled water. After that, we progressively add 0.4 ml of 85% H3PO4 to this solution while vigorously mixing it. The pH of the solution was then adjusted to 7.5 by adding 2.2 mL of acetic acid, and the solution was maintained for one hour. After a while, the solution became cloudy and gradually a heavy white precipitate formed. The solid was filtered and first dried for 8 h at 80ºC, then, it was dried at 120 °C for about 3 h to get the solid of B-$Na_8H[PW_9O_{34}]\cdot 7H_2O$[32].

Then, 4 g of B-$Na_8H[PW_9O_{34}]\cdot 7H_2O$ and 0.61 g of $NaVO_3$ were dissolved in 20 ml of 1.0 M sodium acetate/acetic acid buffered at pH 4.8 and the solution stirred at 25 °C for 48h [33]. To this solution, 6 ml of hexadecyltrimethyl ammonium chloride was added slowly under mixing. The mixture was stirred vigorously for 1 h. A red solid was filtered off and then washed with an excess amount of water, and then it was left in air for 12 h and dried at 80 for 12 h.

Catalytic Reaction

In a beaker fitted with a magnetic stirrer, the catalytic oxidation of sulfur-containing organic molecules in gas condensate with hydrogen peroxide was performed. In a typical run, the solid catalyst (100 mg) was suspended at a constant temperature in a combination of fuel (20 mL) and H2O2 (at varied molar ratios O/S) (between 313 and 353 K). After the specific time, the catalyst was filtered and acetonitrile was added to the fuel under stirring for 5 min. Then the aqueous and hydrocarbon phases were decanted in a separatory funnel. Antek Instruments Model 734 Sulfur Analyzer was used to determine the sulphur content of the hydrocarbon phases. Finally, the catalyst was filtered and reused without further treatment. Using their starting concentration (C0) and concentration after t minutes of reaction (Ct), the removal or conversion (X) for a specific organic sulphur molecule was computed as X = (C0 - Ct)/C0Results and discussion

Characterization of Catalyst

Fig. 1 depicts the major elements of the catalyst's IR spectrum. Peaks in the 500-1100 cm1 range, corresponding to Keggin structural vibrations, were recognised. This indicates that $[(C_{16}H_{33})N(CH_3)_3]_6[PV_3W_9O_{40}]$ maintains the Keggin structure [34-36].

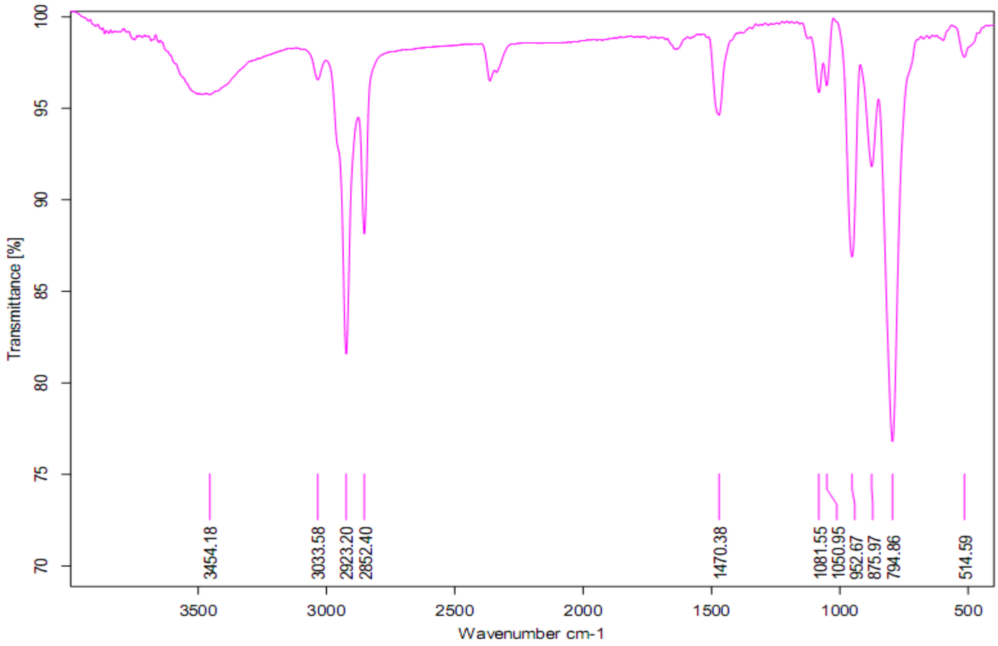

Fig. 1. The FT-IR spectra of $(C_{16}H_{33})N(CH_3)_3]_6[PV_3W_9O_{40}]$

Fig. 2 shows Scanning Electron Microscopy (SEM) of the synthesized catalyst to study the particle size and morphology. Filamentous structure with lengths less than 1µm was obtained. Fig. 3 and Table 1 exhibit the EDAX measurement findings, which revealed that the molar ratio of C:P:W:V in the catalyst was 118:1:8.8:3 in good agreement with the elemental analysis values.

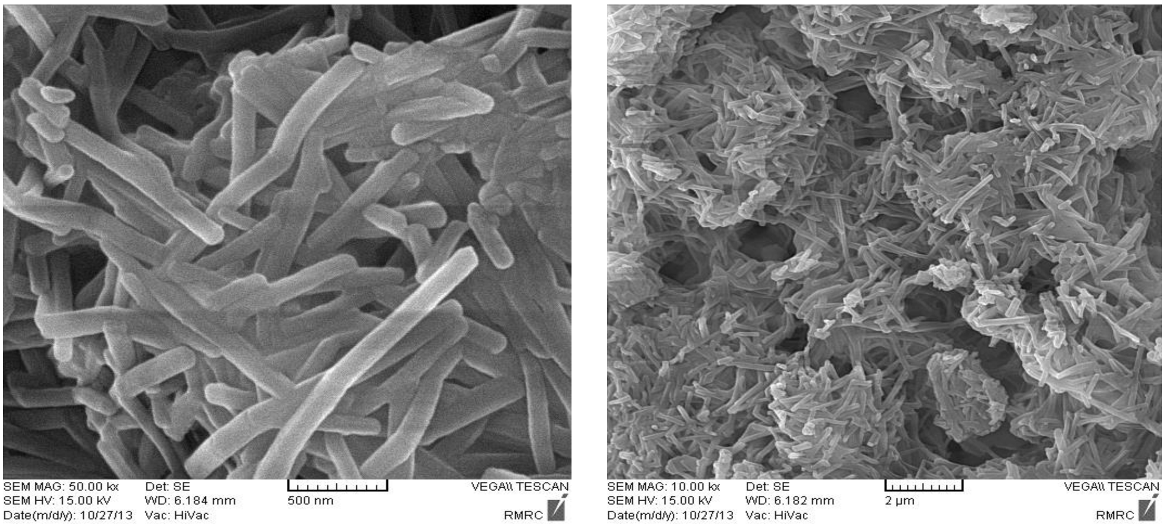

Fig. 2. The SEM images of $(C_{16}H_{33})N(CH_3)_3]_6[PV_3W_9O_{40}]$: (a) scale of 500 nm, (b) scale of 2µm

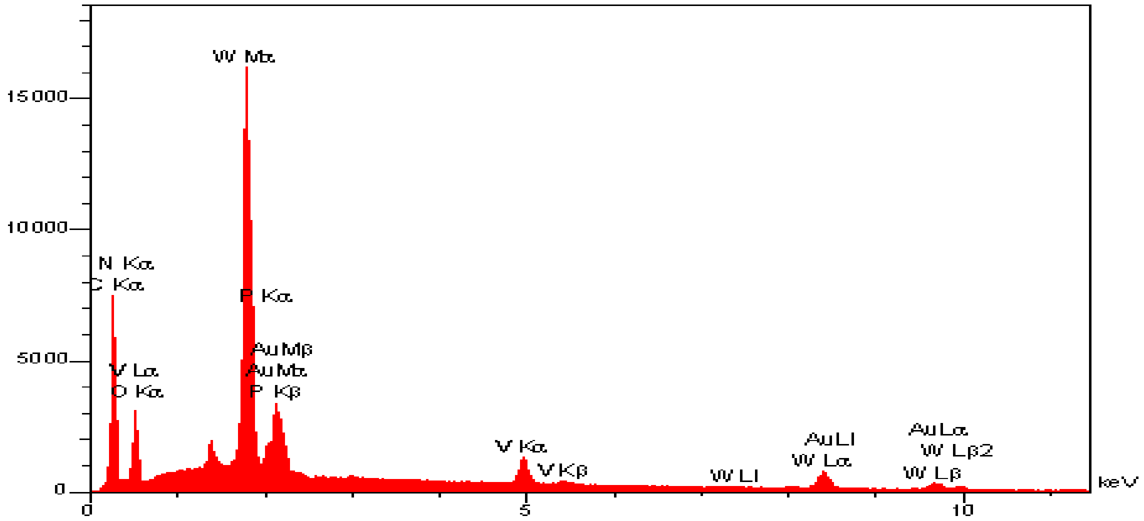

Fig. 3. The EDAX analyze of $(C_{16}H_{33})\ N(CH_3)_3]_6[PV_3W_9O_{40}]$

Table 1. EDX result of $(C_{16}H_{33})\ N(CH_3)_3]_6[PV_3W_9O_{40}]$

Element	Weight (%wt)	Atomic %
C	31.13	35.41
P	0.68	0.36
W	35.54	42.83

Catalytic Activity

Many types of oxidative desulfurization systems were based on the use of H_2O_2 as an oxidant. These systems as a liquid biphasic reaction system are limited by the transfer of reagents between organic and aqueous phases, which results in an excessive consumption of hydrogen peroxide to accomplish ultra-desulfurization. Phase transfer catalysts are an attractive choice for minimising mass transfer limitations by promoting reagent transfer between phases and resulting in good mixing between two phases via the creation emulsion droplet. The amphiphilic catalyst composes of the hydrophilic active catalyst and the lipophile surfactant (as was shown in Fig.4) that causes the catalyst be neither soluble in the oil phase nor aqueous phase, but with stirring continuously, by forming an emulsion droplet, reaction between two phases is facilitated and consumption of hydrogen peroxide is reduced to stoichiometric [37].

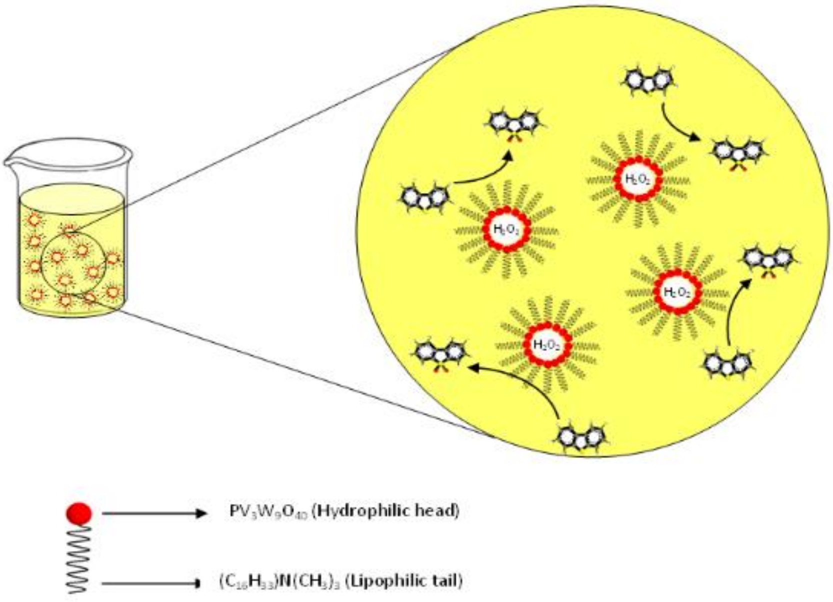

Fig. 4. The oxidation of benzothiophene in emulsion droplets

The influence of the molar ratio O/S on the sulfur removal rate is shown in Fig. 5. Stoichiometrically, 2 moles of H_2O_2 is consumed by 1 mol of sulfur compounds. Sulfur removal of gas condensate increased with an increasing O/S molar ratio at a reaction temperature of 40 °C. It can be seen from Fig. 5 that when the O/S molar ratio was increased from 2.5 to 5.5, the sulfur removal increased from 93.6% to 96.4%. Sulfur removal of 61.3% was obtained with no hydrogen peroxide only in the presence of catalyst and extraction at the next step which is referred to as catalyst activity and molecular oxygen diffused to the system as oxidant and extraction by acetonitrile.

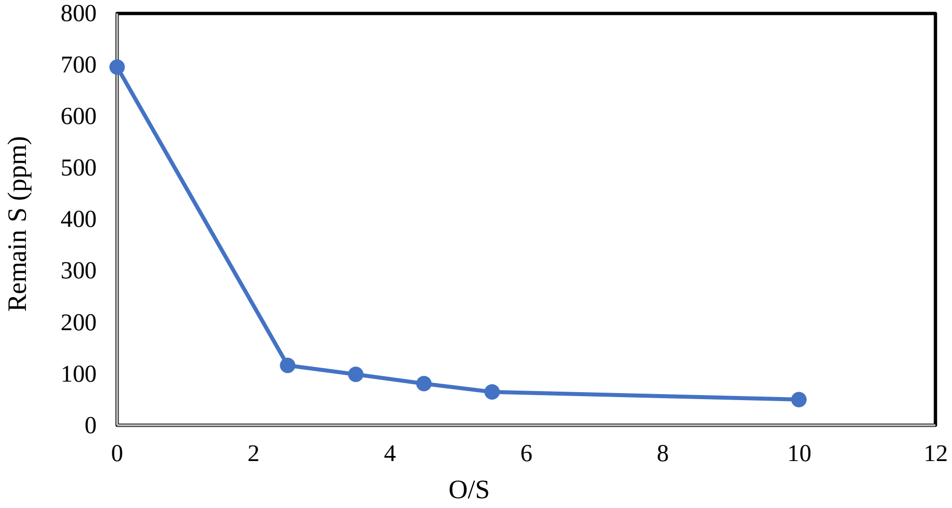

Fig. 5. The influence of the H_2O_2/sulfur molar ratio (O/S) on reaction rate. (T = 40 ˚C, gas condensate (S: 1800 ppm), Catalyst: 0.1 g)

Fig. 6 shows the sulfur removal with different amounts of catalysts. As the number of catalysts rose, the conversion increased. When the amount of catalyst was 0.1 g, sulfur in the gas condensate can be removed from 1800 to 99 ppm at the O/S=3.5 and to 67 ppm with increasing the amount of catalyst to 0.15 g. The experiment was also carried out in the absent catalyst at the molar O/S=4.5 the sulfur removal of 55.91% was obtained and this amount was increased to 97.17 % in the presence of 0.1 g of catalyst.

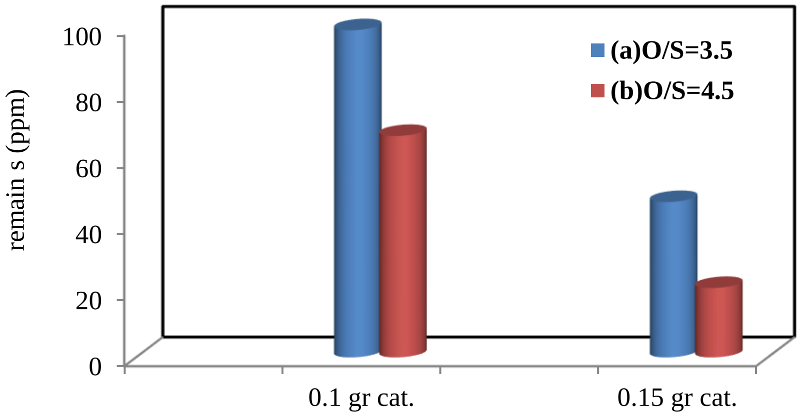

Fig. 6. The influence of catalyst amount on the reaction rate At conditions: T = 40 ◦C, gas condensate (S: 1800 ppm)

Fig. 7 shows the sulphur removal at different reaction times. The results demonstrated that, at 40 °C the sulfur in gas condensate can be removed from 1800 to 83 ppm only at 5 min. when reaction time rose from 5 to 15 min, the sulfur remain reached 62 ppm.

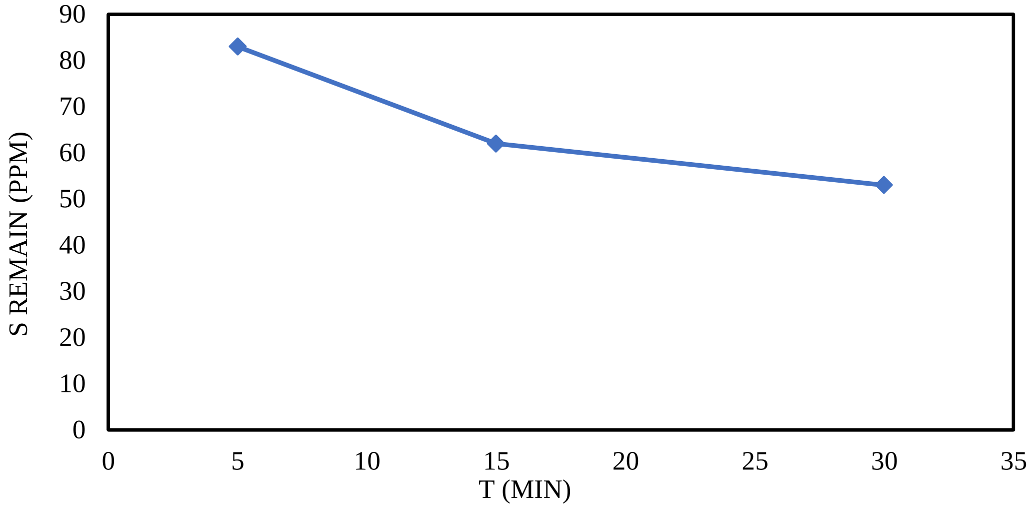

Fig. 7. The influence of time on reaction rate, Conditions: T = 40 °C, gas condensate (S: 1800 ppm), O/S=4.5

The Reusability of Catalyst

The reusability of the catalyst was investigated in the ODS process of sulfur compounds in the gas condensate. After the reaction, the catalyst was recovered by filtration and reused in the next experiment run without any regeneration treatment of the catalyst. Results showed that the catalytic activity had a few decreases after the first run and in the next runs changes were not significant as was shown in Table 2.

Table 2. The results of reusability of catalyst $(C_{16}H_{33})\,N(CH_3)_3]_6[PV_3W_9O_{40}]$

Recycle Number	Remain Sulfur(ppm)	Sulfur Removal (%)
1	50	97.22
2	95	94.72
3	122	93.22
4	123	93.16
5	142	92.11

Catalyst Performance for Straight-Run Diesel Fuel

The ods process of sulfur compounds of straight run diesel from Kermanshah Oil Refining and Distribution Company with initial sulfur content of 5500 ppm. was carried out and showed in Fig. 8 with the molar ratio O/S=4 and amount of catalyst 0.2 g, the sulfur removal 78.69% was obtained at reaction time 60 min after two extraction step by acetonitrile. Also in the molar ratio O/S=8, ODS of diesel was carried out that sulfur removal of 81% was obtained at 30 min.

Ghorbani et al. [38] applied MoO3/ SBA-15 catalyst for desulfurization of the same diesel samples from Kermanshah oil refinery (under operating conditions of T=67 °C, time=42 min, H2O2/S=8 and catalyst dosage=0.004 g/ml) and sulfur removal efficiency were reported 46.29%. As can be shown, the (C16H33)N(CH3)3]6[PV3W9O40] catalyst has a greater efficiency in removing sulphur from diesel, indicating its superiority over comparable samples.

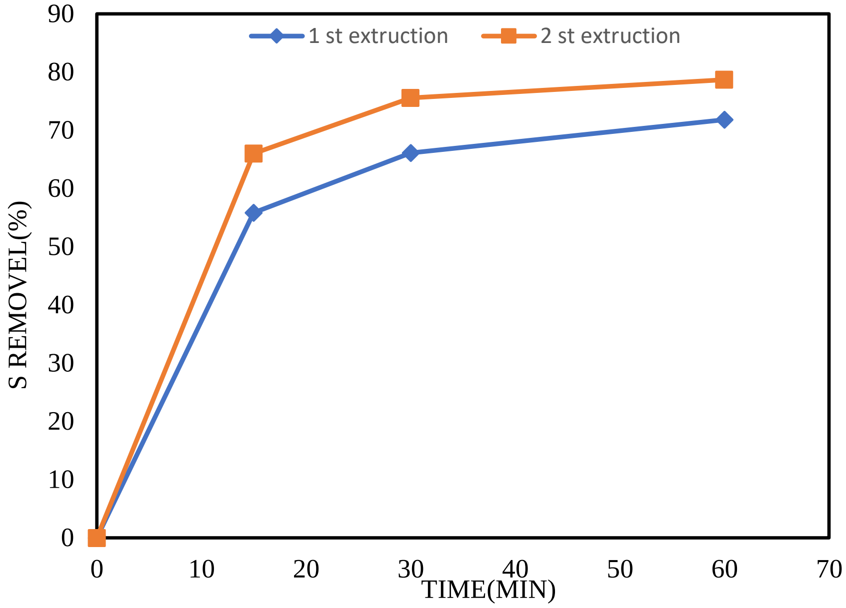

Fig. 8. The oxidation of sulfur compounds in diesel fuel versus time of reaction, Conditions: T = 40 ˚C, diesel (S: 5500 ppm),O/S=4,cat. Amount=0.2gr

Mechanism

Fig. 9 illustrates a plausible mechanism for the biphasic oxidation of DBT. By reacting the Keggin heteropolyanion with hydrogen peroxide, an active hydroperoxo polyoxometalate is generated, and the hydroperoxo species then loses a water molecule to produce the active peroxo species. The oxygen atom transfer from peroxo polyoxometalate to the sulfur-organic substrate occurs in the emulsion phase to produce sulfoxide. The peroxo polyoxometalate is then regenerated at the interface by interaction with H2O2. The sulfoxide is then further oxidised by another peroxo group to generate sulfone [39, 40].

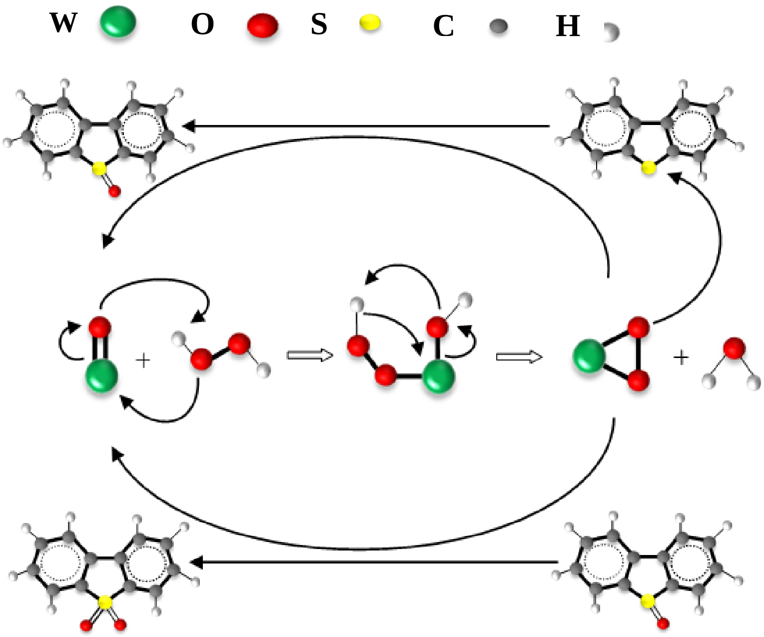

Fig. 9. The mechanism of the ODS process in the presence of (C16H33)N(CH3)3]6[PV3W9O40]

Conclusions

$(C_{16}H_{33})N(CH_3)_3]_6[PV_3W_9O_{40}]$ catalyst was successfully prepared and used in the ODS process of sulfur compounds in gas condensate and straight-run diesel. This catalyst was solved neither in the oil phase nor in aqueous phase. But stirring by the formation of emulsion droplets caused better mixing of two phases and decrease mass transport limitation that resulted enhancement performance of the ODS process. Results showed that catalyst is very active in the oxidation sulfur compounds in gas condensate and diesel fuel. Only in five min, the sulfur removal of 96.38% of gas condensate was obtained. The reusability of the catalyst also was good and had no significant decrease in activity after five-run without any regeneration of the catalyst. Also, the sulfur removal of 81% was obtained at 30 min after two times extraction by acetonitrile for straight-run diesel.

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