

Ultrasonic Intensification of Diesel Fuel Desulfurization: Review

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Abstract— The article shows that nowadays the strict requirements are being applied to the quality of diesel fuel (DF), especially to the content of organic sulfur compounds. These requirements must comply with European standards. Desulfurization of diesel fuels is carried out by different methods with the use of both classical technologies and non-traditional ones. One of the methods for the intensification of desulfurization is an ultrasonic treatment method, whose results used for desulfurization of diesel fuel are considered in this article.

Keywords— diesel fuel, desulfurization, ultrasonic exposure, non-traditional desulfurization methods, cavitating, sulfones

I. INTRODUCTION

Due to the fact that in recent years there has been a tendency to increase the consumption of diesel fuel (DF) throughout the world with the concurrent toughening of environmental requirements to its quality [1], the modern global market places high demands on the content of organic sulfur compounds in diesel fuels [2-4].

With the objective to reduce the harmful effects of sulfur on the environment and human health, most countries are more and more strictly regulating its content in diesel fuel every year.

The maximum sulfur content in automotive diesel fuel was reduced from 0.2-0.5% wt. in the 1980s to 15 ppm in the USA in 2006 and up to 10 ppm in 2010 in European countries [5].

Starting from January 1, 2011, the Russian Federation also limited the sulfur content, but to the level of 350 ppm, which corresponds to the Euro-3 standard.

Euro-4 standards allow the content of organic sulfur compounds to be not more than 50 ppm, and according to the Euro 5 standard it is 10ppm. [6]. In the most developed countries like the USA, Japan and the countries of the EU, the permissible sulfur content in fuel is currently 10 ppm [4].

In Russia, oil companies are doing a lot of work aimed at the massive adoption of domestic refineries in the production

of environmentally friendly diesel fuel with a sulfur content of no more than 0.035% and 0.005% and with the content of polycyclic aromatic hydrocarbons not more than 11% [1].

Since 2016 all diesel fuel in Russia have begun to be produced in accordance with the European standards, namely: more than 50% of diesel fuel produced in the country meet the requirements of Euro-3 standards (350 ppm sulfur content), Euro-4 (50 ppm sulfur content) and Euro -5 (10 ppm sulfur content) [7].

The classical method of removing sulfur from diesel fuel is the process of catalytic hydrodesulfurization performed at 350–450°C and at a hydrogen pressure of 3 MPa with Co-Mo or Ni-Mo catalysts supported on alumina [4].

Despite the fact that catalytic desulfurization hydroprocesses are widely used and are the main processes of desulfurization, they have a number of significant drawbacks being as follows: tough operating conditions, high operating costs, low utilization of raw materials and inability to satisfy all the tougher environmental standards for sulfur [14]. The reduction of sulfur in petroleum products up to 50 ppm and below by hydrofining processes requires large expenses.

Application of this technology enables to reduce sulfur content in the fuel to 300-500 ppm. It is necessary to apply another type of technologies being hydrogenless with the objective to gain the deeper purification to a degree of desulfurization equal to 97-99% (requirements of the EU and US standards). These technologies either individually or in combination with classical technologies enable to intensify desulfurization of motor fuels with regards to the requirements of European standards.

Therefore, the search for new unconventional methods for the removal of sulfur from motor fuels is highly relevant at present. The most intensive research in the sphere of non-traditional methods for diesel fuels desulphurization is carried out in the field of technologies based on the use of oxidative desulfurization, bio-desulfurization, ozonation, selective adsorption, ultrasonic treatment [8].

The most available and promising methods for isolating organic sulfur compounds are oxidative desulfurization ones, which apply various oxidizing agents, catalysts of extractants [4].

Oxidative desulfurization can be carried out at room temperature and atmospheric pressure, which can significantly reduce the cost of the process. During desulfurization sulfur compounds are oxidized to sulfones and sulphooxides, which are then easily removed by conventional separation methods, since their properties are significantly different from the properties of petroleum hydrocarbons that form the basis of fuels [9].

The extractants like methyl alcohol, ethylene glycol monoesters or aqueous acetone can be used for the removal of sulfoxides and sulfones formed during oxidative desulfurization.

In this case, oxidative desulfurization is more economical while the cheapest oxidizers being air oxygen, hydrogen peroxide, organic peroxides and others are used [10].

The outcomes of a significant amount of research devoted to the problem of DF desulphurization are partially reflected in review articles and thesis researches having been published since the 2000s [1, 2, 4, 11-24].

One of the main lines of research, which is currently of great priority, is the intensification of diesel fuels desulfurization.

Numerous studies show that the intensification of diesel fuels desulfurization is achieved by using unconventional methods, and ultrasonic treatment is the most effective of them [4, 23-61].

II. ULTRASONIC INTENSIFICATION OF DIESEL FUEL DESULFURIZATION

The study of processes and the experimental application of ultrasonic processing in various technological processes of oil refining began in the 1980s [25].

The interest in the ultrasonic treatment of hydrocarbons is primarily due to the potential of various refining processes at relatively low average temperatures and pressures compared to similar parameters of traditional refining technologies [28, 29], as well as a significant improvement in the properties of petroleum under the influence of ultrasound [5, 21, 22].

Currently, ultrasound is widely used in various processes of oil refining and oil production. Thus, attempts are being made to experimentally and practically apply high-intensity ultrasonic vibrations in thermomechanical action on oil (to initiate low-temperature cracking under conditions of cavitation and ultrasonic vibrations without using catalysts [26-29]), decontamination of sludge deposits [30], separation of water from oil in preparation of commercial products [31], reduction of the oil viscosity during transportation [32,33], intensification of numerous operations in oil production [25], etc.

The uniqueness and effectiveness of ultrasonic exposure is caused by the phenomenon of cavitation, which is the effective means of concentrating the energy of a low-density ultrasonic wave into a high energy density associated with pulsations and collapse of cavitation bubbles in the liquid phase [34].

Analysis of the use of ultrasonic treatment with the objective to intensify the desulfurization of petroleum products by means of various methods enables to draw conclusions about the prospects of using ultrasound to clean oil and petroleum products from organic sulfur compounds.

Studies [35] consider the effect of complex ultrasonic treatment of raw materials and catalysts in the hydrotreatment of the diesel fraction for the production of environmentally friendly diesel fuel. For this purpose, an ultrasonic activation unit for raw materials and an industrial catalyst was created and tested as part of a hydrotreatment unit for desulfurization of a straight-run diesel fraction.

According to the results of research [35], industrial tests were conducted on the basis of the pilot plant of All-Russian Scientific Research Institute for Oil Refining and proposed

technical solutions for upgrading the catalytic hydrotreating technology of the diesel fraction by means of its preliminary ultrasonic activation.

The pretreatment of a straight-run diesel fraction by ultrasound exposure, carried out on a designed pilot plant, showed an activating effect of ultrasound on organic sulfur compounds contained in it, which contributed to an increase in the depth of sulfur removal during subsequent catalytic hydrodesulfurization of the diesel fraction.

At the industrial hydrotreating unit of the diesel fraction under the conditions of temperature equal to 360° C, a pressure of 3 MPa, a volumetric feed rate of 3 h⁻¹ with preliminary ultrasonic activation, the desulfurization reached 97.0%, and when being hydrotreated without activation, it reached 96.4%.

The results of ultrasonic intensification of the catalytic oxidative desulfurization of straight-run diesel fraction are given and the process and technological parameters of the process are determined in [36].

The studies [35, 36] show that ultrasonic exposure increases the degree of cleaning from organic sulfur compounds by about 2 times, thus eliminating the need for additional heating and increasing the pressure in the reaction chamber.

The outcomes of experiments on the desulfurization of diesel fractions from organic sulfur compounds using various methods indicated the effectiveness of ultrasonic treatment, the most efficient of which was the least energy-intensive method of preliminary ultrasonic activation of the catalyst during 30 sec.

In recent years, especially foreign countries pay considerable attention to the oxidative desulfurization of diesel fuels with the application of hydrogen peroxide and various catalysts as an oxidative system.

According to the author [21, 22], ultrasonic exposure is rather promising in the process of selective oxidation of DF organic compounds by hydrogen peroxide due to intensive heat and mass transfer and minimizing the residence time of the raw material in the reaction zone.

Basing on laboratory experiments it was shown in [6] that this method can be significantly intensified due to ultrasonic treatment. A straight-run diesel fraction selected in the production of Gazprom neftekhim Salavat (with viscosity equal to 2.50 cSt, density 0.88 g/cm³, sulfur content 0.94% by weight) was used for the study of ultrasonic treatment. A 35% hydrogen peroxide (H₂O₂) was used as an oxidizing agent, the industrial catalyst KT-40 and iron chloride FeCl₃, ammonium tungstate 5(NH₄)₂O: 12WO₃H₂O, and distilled water effective in such cases were applied as catalysts.

A series of experiments were carried out with the objective to select rational modes of oxidation for the organic sulfur compounds of the diesel fraction in [6]. These experiments varied the types of catalyst, the amount of oxidizer, the intensity and time of ultrasonic exposure varied.

The studies devoted to determining the activity of catalysts with the application of 35% hydrogen peroxide indicated the

highest activity of ammonium tungstate. The degree of purification from organic sulfur compounds using this catalyst was equal to 29.1%.

The efficiency of desulfurizing straight-run diesel fraction with the use of ammonium tungstate as a catalyst significantly increased in accordance with the amount of hydrogen peroxide (2-7%) when using ultrasonic treatment. The degree of purification from sulfur compounds increased by 50%.

The study of the intensity of ultrasound exposure within the range of 10-25 W/cm², and the processing time equal to 2-8 minutes enabled to establish a rational oxidation mode achieved at an intensity of ultrasound exposure equal to 20 W/cm² during 4 minutes.

The energy consumption for ultrasonic treatment is very high under the conditions of desulfurizing diesel fuel in an industrial environment. In this case, it is advisable to pre-activate the catalyst in an ultrasonic field and then feed it into the reaction chamber with a mixer [6]. The outcomes of a comparative experiment on diesel fuel desulfurization from organic sulfur compounds (implemented with the application of various methods for 4 minutes) showed the effectiveness of this solution. The degree of cleaning from sulfur-containing compounds was 19.2% without ultrasonic treatment, mixing of reaction mixture with catalyst pre-activation during 1 min was 25.3% and the ultrasonic treatment of the reaction mixture without stirring was 28.9%.

Sulphco has developed the process of oxidizing water-fuel emulsion with hydrogen peroxide under ultrasonic exposure over a catalyst at 70-80°C and atmospheric pressure in an ultrasonic reactor with the objective to reduce the sulfur content in diesel fuel [37-38].

In one minute the sulfur content in diesel fuel was reduced from 550 to 10 ppm. According to company estimates, the process was cheaper than hydrotreating. The disadvantage of this method was the need for the subsequent extraction of organic sulfur compounds from the purified organic phase, which in its turn led to additional expenditure of reagents and budget.

This is confirmed by the study [35], which shows that ultrasound exposure is rather promising in selective oxidation of diesel fuel organic compounds by hydrogen peroxide due to the intensive heat and mass transfer, minimizing the residence time of the raw material in the reaction zone.

Studies [39-50] show that the use of ultrasound in the liquid-phase oxidation of organic compounds increases the rate of initiation and oxidation.

It was shown in [45] that the oxidation rate of organic compounds of middle distillate oil fractions increases when using hydrogen peroxide and acetone under the influence of an ultrasonic field. The efficiency of hydrotreatment increases under the influence of ultrasound and the sulfur content drops from 1.78 to 1.34%.

The studies [4, 46-51] found that organic sulfur compounds can be removed from the fuels of various origins (including those of oil origin) by the method in which oxidative desulfurization is combined with ultrasound.

Ultrasonic treatment allows reducing the reaction time of sulfur components with an oxidizing agent up to the level of several minutes [52].

The work [53] describes the continuous method of oxidative desulfurization of petroleum products under ultrasonic action.

Diesel fuel is mixed with water, hydroperoxide and aliphatic hydrocarbons C_{15} - C_{20} and the resulting multiphase reaction medium is continuously treated by ultrasound in a flow-through mode. This method enables significant reduction of the amount of organic sulfur compounds in the final product.

Ultrasonic treatment of the diesel fraction in the presence of hydrogen peroxide aqueous solution and a quaternary ammonium salt as an interfacial catalyst reduces the sulfur content in the fuel to less than 0.25% [54, 55].

The side formation of 2- and 3-benzothiophenesulfones can be avoided using a fluorine-containing phase transfer catalyst [54].

The following three layers: oxidized diesel fuel (upper layer), residue (lower layer) and aqueous layer (middle) are formed under the conditions of ultrasonic processing of diesel fuel in the presence of an aqueous solution of hydrogen peroxide. The residue is less than 1% of the total fuel and it contains about 2% sulfur [55].

The review article [4, 56, 57] states that sulfur recovery efficiency does not exceed 66% per 2.5 hours under the conditions of oxidative desulfurization of the diesel fraction. The combination of ultrasonic treatment with oxidative desulfurization enables to obtain the sulfur removal degree equal to 99% during a few minutes with the 87% yield of refined fuel [55].

The effect of the Fenton agent and ultrasound on the degree of desulfurization of diesel fuel was investigated in [58].

The authors found that adding Fenton-agent to the hydrogen peroxide or acetic acid system increases the efficiency of desulfurization, and ultrasonic treatment in the process of desulfurization gives a synergistic effect [23, 58].

Under the most favorable conditions being as follows: temperature equal to 400° C, ultrasound with the frequency of 28 kHz, Fe^{+2}/H_2O_2 0.05 mol/mol, pH 2.1 in an aqueous medium and the reaction time of 15 min the sulfur content in diesel fuel decreased from 568.75 $\mu\text{g/g}$ to 9.5 $\mu\text{g/g}$.

A new method for producing diesel fuel with ultra-low sulfur by oxidative desulfurization by means of ultrasound was proposed by the authors in [55].

The example of benzothiophene being the model sulfur-containing compound of fuel demonstrates the possibility of quantitative catalytic desulfurization with suitable oxidizing agents during several minutes. Catalytic oxidation and sonication followed by solvent extraction at normal temperature and pressure were used for diesel fuels with different sulfur content. The degree of removal of sulfur-

containing compounds reached more than 99% under the conditions of short contact time.

The effect of the frequency and intensity of ultrasound in the process of oxidative desulfurization of diesel fuel in the presence of H_2O_2 and organic acid at 20°C, a catalyst/fuel ratio of 0.05, a stirring speed of 300 rpm, a reaction time of 15 min, an ultrasound frequency of 28 kHz, and the radiation intensity 0.408 W/cm² followed by 2-time solvent extraction (DMF) were studied.

The degree of removal of sulfur-containing compounds during extraction is 94.8 and 67.2% for ultrasonicated fuel and non-sonicated fuel, respectively [55].

It was shown in [20–24] that the process of catalytic oxidation of sulfur-containing organic compounds in hydrocarbon media is intensified under the action of ultrasound.

The use of ultrasonic treatment enables to increase the degree of desulfurization by 2–4 times in comparison with catalytic desulfurization without ultrasound, which implies a deeper desulfurization of hydrocarbons. The oxidized sulfur-containing organic compounds resulting from such a sonocatalytic reaction are capable of irreversible transition into droplets of a reverse aqueous hydrocarbon emulsion formed in the ultrasound field.

An important advantage of technological processes with the use of ultrasound is the possibility of their implementation in devices of any structural materials, shape and volume, under the conditions of carrying out the processes in continuous mode at elevated temperature and pressure [60–62].

The use of ultrasound favours the intensification of mass transfer in a desulfurization reactor [23]. Cavitation, which occurs with processing the liquid phase by ultrasound, contributes to breaking the liquid droplets by making up the oxidizer/hydrocarbon system into submicron and nano-sized droplets. Such a decrease in the diameter of the droplets contributes to the reaction that takes place at the interface, and enables to avoid the application of a phase transfer catalyst.

Table 1 shows some methods for the desulfurization of diesel fuel with and without ultrasonic treatment and shows the efficiency of ultrasonic treatment [23]. As it is seen from Table 1, the ultrasonic processing of the diesel fraction intensifies the desulfurization in all cases of ultrasound application. Thus, the degree of sulfur extraction does not exceed 66% per 2.5 hours with the oxidative desulfurization of the diesel fraction. The use of ultrasonic treatment with oxidative desulfurization of diesel fuel can increase the degree of sulfur removal up to 99% in a few minutes with the yield of purified fuel equal to 87%.

Additionally, Table 1 shows that ultrasonic treatment of the diesel fraction in the presence of an aqueous solution of hydrogen peroxide and a quaternary ammonium salt as an interfacial catalyst reduces the sulfur content in the fuel to less than 0.25%, i.e. desulfurization efficiency of the above examples is the highest and is equal to 99.75%

In all cases, the impact of ultrasound on diesel fuel or its mixture with oxidizing agents, catalysts, organic acids or aqueous medium leads to an increase in the particles reactivity in the mixture [53, 60, 61].

TABLE I. COMPARISON OF THE EFFICIENCY OF DF DESULFURIZATION WITH AND WITHOUT ULTRASOUND TREATMENT

Raw material	Oxydizing agent	Catalyst, other conditions	Sulfur removal rate
Diesel fuel	Without oxydizing agent	Hydrotreating (temperature 360°C, pressure 3 MPa, feed space velocity -3 h ⁻¹ , without activation by ultrasound	96.4 %
		Hydrotreating (temperature 360°C, pressure 3 MPa, feed space velocity -3 h ⁻¹), with pre-activation of raw materials by ultrasound	97.0 %
	H ₂ O ₂	-	66% (2.5 hours)
		Acetic acid/Ultrasound	97.4%
		Acetic acid/Na ₂ WO ₄	> 90 %
		Organic acid / without ultrasound	67.2 %
		Organic acid / ultrasound	94.5 %
		Organic acid / Activated carbon	>95%
		Quaternary ammonium salt / ultrasound	up to 0.25 %
		Ammonium tungstate / without ultrasound	29.1%
		Ammonium tungstate / with ultrasound	79.1 %
		Mo/Al ₂ O ₃	97.8
	Fe ²⁺ /ultrasound	from 568.75 to 9.5 mg/g	
	H ₂ O ₂ /acetone	ultrasound	75.28%
	H ₂ O ₂ /alcohol oxide	MnO ₂ /Al ₂ O ₃	from 500 to 1 ppm
Tert-butylhydroperoxide	Fe- MoO ₃ / Al ₂ O ₃	from 440 to 88 ppm	
ozone	Submicron bubbles	>69%	

Evidence of unusually high efficiency of ultrasonic treatment is the fact that dibenzothiophene and other sulfur-containing organic sulphides, which are the most persistent organic sulfur compounds in diesel fuels, are easily converted to the corresponding sulfones under relatively mild conditions of temperature and pressure and under ultrasonic treatment.

The increased polarity of sulfones compared to sulfides ensures their high degree of susceptibility to removal using traditional separation processes based on polarity. The advantage of ultrasonic treatment in the DF oxidative desulfurization is that the oxidation proceeds selectively with respect to the conversion of compounds containing sulfur. Notably, there are no changes in the components of diesel fuel that do not contain sulfur. Another advantage of the use of ultrasound in the DF desulfurization is that the transformation occurs in a very short period of time, that is, during the period significantly less than one hour, preferably within 10-20 minutes on average.

III. CONCLUSION

1. Intensification of diesel fuel desulfurization is achieved by using unconventional methods and ultrasonic treatment is the most effective of them.
2. Ultrasonic treatment of the diesel fraction in all cases of ultrasound application leads to the increase in the degree of sulfur removal from diesel fuel. Thus, the oxidative desulfurization of the diesel fraction with hydrogen peroxide leads to the situation when the degree of sulfur recovery does not exceed 66% during 2.5 hours. The use of ultrasonic treatment allows increasing the degree of sulfur removal to over 99% in a few minutes with a yield of purified fuel equal to 87%.
3. The highest degree of sulfur removal to less than 0.25% of the mass is achieved by using ultrasonic treatment of the diesel fraction in the presence of an aqueous solution of hydrogen peroxide and a quaternary ammonium salt as a phase transfer catalyst. The efficiency of desulfurization is 99.75% wt.
4. Ultrasonic treatment of diesel fuels can serve as a logical addition to the large-capacity hydrotreating process, and can be used as an independent method of deep desulfurization.

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