

## Desulfurization of Liquid Fuels by Selective Extraction Method: a Review



### Engineering

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### ABSTRACT

*Owing to the stringent environmental regulations in many countries for production of ultra low sulfur petroleum fractions intending to reduce sulfur emissions results in enormous interest in this area among the scientific community. Requirement of zero sulfur emissions enhances the prominence for more advanced techniques in desulfurization. Desulfurization by extraction is a promising approach having several advantages over conventional hydrodesulphurization. This paper is dealt with various new approaches for desulfurization of ultra clean gasoline, diesel and other liquid fuels by extraction with ionic liquids. Recent advancements for finding a long term solution for deep sulfur reduction by extraction method using various ionic liquids are reviewed. Very promising ILS are found to be pyridium and imidazolium based cations.*

### 1. INTRODUCTION:

Sulfur content in transportation fuel has become a serious concern throughout the world due to its severe environmental menace as well as its effects in catalysts and vehicle engines. Sulfur compounds are encountered in many areas in oil refinery. Common types of sulfur compounds in liquid fuels are listed in table 1. During combustion in the diesel engines, the sulfur compounds burn to form harmful sulfur oxides (SO<sub>x</sub>) and sulfate particulates. The nitrogen compounds are oxidized to nitrogen oxides (NO<sub>x</sub>). The exhaust emissions contain SO<sub>x</sub>, NO<sub>x</sub>, CO, CO<sub>2</sub>, PM and unburned hydrocarbons (HC). The particulate matter (PM) emitted from diesel engine consists of three main constituents: a carbonaceous core, a soluble organic fraction (SOF) and a mixture of SO<sub>x</sub> and water, and it has been found to be a human carcinogen. A few traces of sulfur present in the diesel fuels also poison the oxidation catalysts in the emission control system and reduce their effectiveness for the oxidation of harmful carbon monoxide, hydrocarbons and volatile organic matter [1,2]. The government imparts various environmental regulations and added stringent sulfur specification in fuels aiming at Zero sulfur emission. Table 2 lists diesel fuels standards in India. Bharat Stage IV diesel are cleaner fuels as they have lower sulfur content of 50 ppm compared to BS-III diesel which contain 350 ppmw sulfur respectively [3].

Many new concepts and technologies have been developed during the last 20 years to desulfurize the least reactive sulfur species from the diesel feed that make reaching the near zero sulfur requirements attainable cost effectively. The widely used method for desulfurization is catalytic hydrodesulphurization. In the recent years, refineries are facing higher amounts of sulfurous crude oil as feedstock due to the diminishing crude oil reserves and producing larger volume of products from high sulfur heavy oil fractions. This, along with the demand for ultra clean fuel, are increasing the desulfurization cost[4]. Severe operating conditions, i.e., very low space velocities, high temperatures, high hydrogen pressures, and the use of highly active catalysts, are inevitably required to produce ultra-clean fuel oils [5-7]. HDS show lesser satisfactory performance in removing Poly Aromatic Sulfur Heterocycles (PASHs) such as DBT due to steric hindrance[7]. Hence, it is necessary to develop an supplementary process to HDS for deep desulfurizing fuel. Hence, it is necessary to investigate new processes of fuel desulfurization as the existing HDS process supplement.

Some of the new promising methods for fuel desulfurization are adsorption, extraction, oxidative extraction, precipitation, membrane and biochemical processes. Extraction method is based on better solubility of sulfur compounds and aromatic hydrocarbons in relation to non aromatics in appropriate polar

solvent. This article reviews about various researches of selective extractive desulfurization process based on the type of solvent used for the extraction.

**Table 1**

Typical Sulfur Compounds and corresponding refinery streams for fuels

Sulfur compounds	Refinery streams	Corresponding fuels
Mercaptanes, RSH; sulfides, R <sub>2</sub> S; disulfides, RSSR; thiophene (T) and its alkylated derivatives, benzothiophene	SR-naphtha; FCC naphtha; coker naphtha	Gasoline (BP range: 25-225°C)
Mercaptanes, RSH; benzothiophene (BT), alkylated benzothiophenes	Kerosene; heavy naphtha; middle distillate	Jet fuel (BP range: 130-300°C)
Alkylated benzothiophenes; dibenzothiophene(DBT); alkylated dibenzothiophenes	Middle distillate; FCC LCO; coker gas oil	Diesel fuel (BP range: 160-380°C)
Greater than or equal to three-ring polycyclic sulfur compounds, including DBT, benzonaphthothiophene (BNT), phenanthro[4,5-b,c,d] thiophene (PT) and their alkylated derivatives and naphthothiophenes (NT)	Heavy gas oils; vacuum gas oil; distillation residues	Fuel oils (non-road fuel and heavy oils)

**Table: 2**

Diesel Fuels in India

Date	Particulars
1995	Cetane number: 45; Sulfur: 1%
1996	Sulfur: 0.5% (Delhi + selected cities)
1998	Sulfur: 0.25% (Delhi)
1999	Sulfur: 0.05% (Delhi, limited supply)
2000	Cetane number: 48; Sulfur: 0.25% (Nationwide)
2001	Sulfur: 0.05% (Delhi + selected cities)
2005	Sulfur: 350 ppm (BS III/Euro 3; selected areas)
2010	Sulfur: 350 ppm (BS III/Euro 3; nationwide)
2010	Sulfur: 50 ppm (BS IV/Euro 4; selected areas)

## 2. CURRENT DESULFURIZING TECHNOLOGIES:

### 2.1 Hydrodesulfurization:

Hydrotreating or hydroprocessing refers to a variety of hydrogenation processes which saturate unsaturated hydrocarbons and eliminate S [by hydrodesulfurization (HDS)], N [by hydrogenation (HDN)], O [by hydrodeoxygenation (HDO)] and metals [by hydrodemetallization (HDM)] from different petroleum streams in a refinery. Hydrodesulfurization or hydrotreating is one of the major standard method for desulfurization used in oil refinery. Hydrogenation reaction occurs due to cleavage of C-C Bond with net result of formation of C-H and H-S bonds. HDS is a high-pressure, high-temperature catalytic process that converts organic sulfur to hydrogen sulfide gas. It can also remove various types of sulfur compounds, some types of heterocyclic sulfur compounds existing in petroleum cannot be removed [8]

### 2.2 Adsorptive desulfurization:

Adsorptive desulfurizing units can provide low sulfur fuel for sulfur intolerant systems such as fuel cells and catalyst beds. Operability of a desulfurizer at ambient conditions without the requisite for hydrogen provides many advantages over conventional systems. When lower sulfur standards are enacted, the use of sorptive systems in combination with traditional HDS units would reduce the costs of retrofitting them [9].

### 2.3 Oxidative desulfurization:

Oxidative desulfurization (ODS) is considered a hopeful method for ultra-deep desulfurization of fuel oils. Under mild conditions, the organosulfur compounds are oxidized to their corresponding sulfoxides or sulfones. The process is carried out in the presence of a catalyst and an oxidising agent. The oxidized sulfur compounds are subsequently removed by extraction, adsorption, distillation, or decomposition [10-13]

### 2.4 Biodesulfurization:

Biodesulfurization is excellent desulfurizing technology due to its specificity towards aromatic sulfur containing compounds and mild operability[14].

## 3. EXTRACTIVE DESULFURIZATION:

Liquid Extraction, sometimes called solvent extraction, is the separation of the constituents of a liquid solution by contact with another insoluble liquid. Separation based on extraction is preferred over other separation methods for its advantage in less costlier easy disposal since it incurs no chemical consumption or by product formation unlike other chemical methods. In distillation depends on vapor pressure and the constituents are similar unlike extraction. In liquid Extraction, the major constituents of two phases are chemically different, and this makes separations according to chemical type possible.[15].

Extractive desulfurization (EDS) is more striking since extraction is a well established process technology carried out at or around ambient temperature and pressure and without the need for hydrogen, and does not modify the chemical structure of the fuel components. However, it is required that the extractant be sufficiently selective for the sulfur compounds (S-compounds) without affecting the octane number of the fuels[16]. The exclusive part of the extractive distillation unit is that it segregates the sulfur and aromatic components prior to the hydrotreater[17]

This paper proposes the use of Ionic Liquids (ILs) as extracting agent in liquid-liquid extraction as an alternative or supplemental technology for the desulfurization of refinery streams.

## 4. IONIC LIQUIDS:

An ideal extracting agent should have the following properties: (1) The partition coefficient for S-compounds (ratio of S-concentration in the extracting agent to the one in the oil), above all for dibenzothiophene-derivatives, should be high (2) The regeneration should be easy, e.g. by distillation, or at least the extraction should be reversible. (3) The agent should be absolutely insoluble in oil. (4) The (S-free) hydrocarbons of the oil matrix should not, or only to a small extent, be soluble in the extracting agent. (5) The agent should feature a high thermal and

chemical stability, be non-toxic and environmentally benign.

Ionic liquids will be of best choice for such ideal behavior. Ionic liquids are purely ionic, salt-like materials, which are liquid below 100°C. Commonly, they have melting points below room temperature, with some even below 0°C. Solidification of the Ionic Liquid will take place at lower temperatures. The strong ionic interaction within Ionic Liquids results in a negligible vapor pressure, unless decomposition occurs. It makes the material non-flammable and highly stable thermally, mechanically and electrochemically. Furthermore, it imparts very appealing solvent properties and immiscibility with water or organic solvents that results in biphasic systems.

They are useful as solvents and catalysts in alkylation [18], polymerization[19], Diels-Alder[20], electrochemistry[21]. Ionic liquids can be used as extractants for the separation of aromatic hydrocarbons from aliphatic hydrocarbons. Extraction of aromatics from mixed aromatic/aliphatic streams with ionic liquids is expected to require less process steps and less energy consumption than extraction with conventional solvents because ionic liquids have a negligible vapor pressure.

The application of making use of ionic liquids for extraction processes is promising because of their non-volatile nature.[22]. Some of the ionic liquids is shown in figure 1.

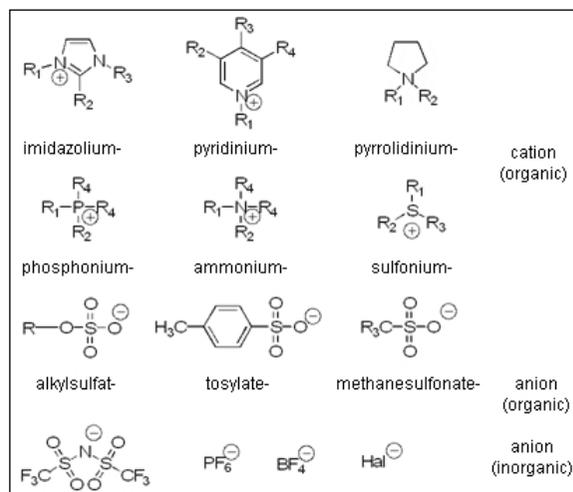


Fig. : 1 Typical structure that combines with organic cations with inorganic or organic anions.

### 4.1 Imidazolium based ionic liquids:

Major research of studies of desulfurization by extraction is by using Imidazolium based ionic liquids. Likhanova et al. [23] studied 17 ionic liquids and studied their efficiency in sulfur removal. The results show that ILs containing imidazolium cations are the best counterpart for increasing the efficiency of sulfur removing. The efficiency to remove sulfur compounds are not affected due to the presence of methyl substituents in position 2. Chunxi Li et al.[24] studied EDS performance of N-butylimidazole derived dialkylphosphate ILs, viz. [BMIM][DMP] and [BEIM][DEP]. [BMIM][DMP] seems to be promising for extractive removal of aromatic sulfur compounds (S-compounds) from fuel oils, and show strong preferential extraction for aromatic S-compound versus toluene. The results show that the sulfur removal selectivity for a specific IL is dependent on the molecular structure of the S-compounds. It follows the order dibenzothiophene > benzothiophene > thiophene > 3-methyl thiophene, The EDS performance is attributed to the formation of complex between  $\pi$ -electrons of aromatic S-compound and the charged imidazolium ring of the IL, which is largely affected by the size of the alkyl substitutes via steric effect and dispersion interaction involved. The extraction of sulfur containing aromatic compounds such as 2,5-dimethylthiophene, thiophene, benzothiophene and dibenzothiophene (1 wt %) present in n-octane follows the similar trend observed by

benzene derivatives. The interaction of these sulfur compounds with the ionic liquid is through CH $\cdots$  $\pi$  bonds and the quantity of sulfur compounds extraction increases with the increase of the  $\pi$ -density and decreases with the degree of alkyl substituents as already observed in the extraction procedures by 1-n-butyl-3-methylimidazolium n-octylsulfate and 1-ethyl-3-methylimidazolium ethylsulfate.

The extraction of benzothiophene from n-hexane was studied using 1-ethyl-3-methylimidazolium ethyl sulphate ([EMIM][EtSO<sub>4</sub>]) and 1-ethyl-3-methylimidazolium acetate ([EMIM][CH<sub>3</sub>COO]) at 308.15 K to analyze the performance of ionic liquids in the extractive desulphurization of aromatic sulphur compounds from petroleum fuels. Very high selectivity values were achieved for the ternary experiments done on the systems, 1-ethyl-3-methylimidazolium ethyl sulphate-benzothiophene-n-hexane and 1-ethyl-3-methylimidazolium acetate-benzothiophene-n-hexane. It was found that while the selectivity was higher for the ethyl sulphate-based ionic liquids, the distribution coefficient was higher for acetate-based ionic liquids[25].

Hu Yufeng and his co-workers[26] investigated extractive removal of sulfur compounds from Dongying and Liaohe diesel fuels with [BF<sub>4</sub>]<sup>-</sup> based ionic liquids. [C<sub>8</sub>Py][BF<sub>4</sub>] < [C<sub>8</sub>mim][BF<sub>4</sub>], remains unchanged as the mass ratio decreases from 1:5 to 1:2 or 1:1, implying that the used mass ratio has a little influence on the relative absorption capacities of the examined ionic liquids. Imidazolium-based ionic liquids show higher extraction efficiencies than pyridinium-based ionic liquids, presumably owing to the fact that the rings of the S-compounds are similar to the imidazolium head ring. With the 1:1 mass ratio of ionic liquid/diesel fuel, the rates of the first desulfurization of Dongying and Liaohe diesel fuels using [C<sub>8</sub>mim][BF<sub>4</sub>] amount to 29.96% and 39.76%, suggesting that [C<sub>8</sub>mim][BF<sub>4</sub>] is a promising extractant for desulfurization of these diesel fuels.

Eleven Lewis acid ionic liquids, [Bmim]Cl/FeCl<sub>3</sub> (Bmim: 1-butyl-3-methylimidazolium chloride), [Omim]Cl/FeCl<sub>3</sub> (Omim: 1-Octyl-3-methylimidazolium chloride), T<sub>8</sub>Cl/FeCl<sub>3</sub> (T<sub>8</sub>Cl: Tri-octyl methyl ammonium chloride), T<sub>4</sub>Cl/FeCl<sub>3</sub> (T<sub>4</sub>Cl: Tributyl methyl chloride), D<sub>10</sub>Cl/FeCl<sub>3</sub> (D<sub>10</sub>Cl: didecyl dimethyl ammonium chloride), and [Emim]Cl/AlCl<sub>3</sub> (Emim: 1-ethyl-3-methylimidazolium), have been synthesized and investigated for desulfurization of liquid fuels by Swapnil A. Dharaskar et al.[27]. [Bmim]Cl/FeCl<sub>3</sub> was the most promising ionic liquid and performed the finest among studied ionic liquids exhibiting better extractive performance for dibenzothiophene with the maximum desulfurization efficiency of 75.6% and they may be reused without regeneration with considerable extraction efficiency of 47.3%.

#### 4.2 Pyridium based ionic liquids :

N-butylpyridinium tetrafluoroborate ([BPy]BF<sub>4</sub>) is found to have best effect for selective removal of sulfur-containing compounds from gasoline at room temperature among other pyridium based ionic liquids with extraction rate of 45.5%. The extraction desulfurization ability of ILs connects with their chemical properties, such as the cation or anion structure, the size of ILs. Compared to N-ethylpyridinium-based ILs, N-butylpyridinium-based ILs with the substitution of a longer alkyl group to the pyridinium ring, have higher desulfurization ability

. Regenerated of used ILs are carried out by rotary evaporation or re-extraction using tetrachloro-methane[28]

#### 4.3 Aqueous ionic liquids:

Desulphurization of jet fuel, diesel oil, heavy residue and commercial furnace oil is carried out through extraction with aqueous solutions of sodium chloride, barium chloride, sodium hydroxide, mercury chloride, arsenic trioxide, potassium iodide, lead acetate, calcium hydroxide, zinc chloride, aluminum chloride, hydrochloric acid and sulphuric acid among which HgCl<sub>2</sub> and Ca(OH)<sub>2</sub> are most efficient in sulfur removal. The total sulphur depletion of 60 % and 58 % has been achieved in case of jet fuel oil, 71 % and 62 % in case of diesel oil, 68 % and 67 % in case of heavy residue and 67 % and 69 % in case of commercial furnace oil with 10% HgCl<sub>2</sub> and 5% Ca(OH)<sub>2</sub> aqueous solutions, respectively.[29]

#### 4.4 Anion composition in IL's:

Anion composition play an important role than cation in improving the efficiency of ILs for sulfur compound extraction. For desulfurisation of fuels ILs containing chloroaluminate and chloroferrate as anion are good extracting anions. The efficiency of sulfur removal decreased significantly when anions containing other chlorometalates (i.e. Sn, Zn, Cu) are used. Though acetate anion have lesser efficiency than chloroaluminates, it showed remarkable desulfurising ability than other anions since it could be recycled and reused without lost their performance significantly in three cycles. Furthermore this organic anion is halogen-free, moisture insensitive, and thermally stable[23]. However chlorometallate ILs are not suitable for large scale applications due to their limited stability and toxicity. Further researches shows that tetrafluoroborate or hexafluorophosphate ILs are also appropriate for the extraction of model-sulfur compounds. In spite of its higher hydrolysis stability their use is not optimal because of its higher price of starting material. To avoid these stability and corrosion problems, completely halogen-free ILs are used for the extraction of S- and N-compounds[30].

#### 5. EXTRACTION BY OTHER SOLVENTS:

Extractive desulfurization of FCC gasoline with two solvents (sulfolane and furfural) was studied. Obtained results show that lower sulfur content in samples after liquid extraction with sulfolane was achieved at temperatures of 50 °C and sulfolane / FCC ratio above 2 at process duration of 30 minutes. Maximum desulfurization efficiency was obtained at high levels of temperature and sulfolane / FCC gasoline ratio; time in researched range of values had small impact[31]. Various other solvents are also used for desulfurization.

#### 6. CONCLUSION :

Desulfurization of fuels by extraction using ILs proves to be promising approach eliminating the disadvantages of hydrodesulphurization because of its milder operating conditions and be able to remove those sulfur compounds that are difficult to remove by hydrodesulphurization. Ionic liquids comprised of number of solvents having remarkable properties proves to have higher desulfurizing efficiency without H<sub>2</sub> consumption. Cations, Anions, Chemical Structure, temperature are important parameters which affects extraction. Further experiments for further understanding of properties of ILs and its regeneration ability are needed.

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