زانكۆى سەڵاحەدین-هەولێر

Salahaddin university-Erbil

Desulfurization of fuels using Ionic Liquids (ILs)

Research project submitted to the council of the college of education at Salahaddin university in partial fulfillment of the requirements for the degree of B.Sc. at chemistry department

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Supervisor recommendation

I am the student's supervisor, Ibrahim Khaled Omer, I support that the student has completed all the requirements for submitting the research drawn entitled Desulfurization of fuels using Ionic Liquids (ILs), according to the numbered administrative order 3/1/5/1972 on 6th April 2024 in accordance with the instructions of Salahaddin university quality assurance and it is ready for discussion.

Supervisor: Dr. Essa Ismaeil Ahmed

Signature:

Date:

Research project lecturer

Dr. Dler D. Kurda

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Abstract

Here we are talking about the desulfurization of fuel by ionic liquid and summary of ionic liquid with desulfurization in their types in this way (Imidazolium-based ionic liquids, Pyridinium-based ionic liquids, Lewis and Brønsted acidic ionic liquids or redox ionic liquids, EDS performance of different ILs, Extraction temperature, Multiple extractions for model gasoline, Multiple extractions for model diesel, Extractive performance after regeneration, Regeneration, Effect of different ionic liquids on DBT removal, Effect of different catalytic systems containing $FeCl_3$ species on DBT removal).

List of contents

Introduction

Ionic Liquids (ILs)

compounds completely composed of bulky positively charged ions called cations and. negatively charged ions called anions. Ionic liquids use the boiling point of water as a point of reference: "Ionic liquids are ionic compounds which are liquid below 100 °C." More commonly, ionic liquids have melting points below room temperature; some of them even have melting points below 0 °C. (Kianfar. Mafi, 2020)

Generations of ionic liquids

There are three generations of ionic liquids which are briefly described below;

First generation

Ionic liquids are compounds that are widely used as solvents. These compounds have unique physical properties that can be enhanced by changing their cations or anions. In this Figure, these ionic liquids are introduced as the first generation. (Kianfar. Mafi, 2020)

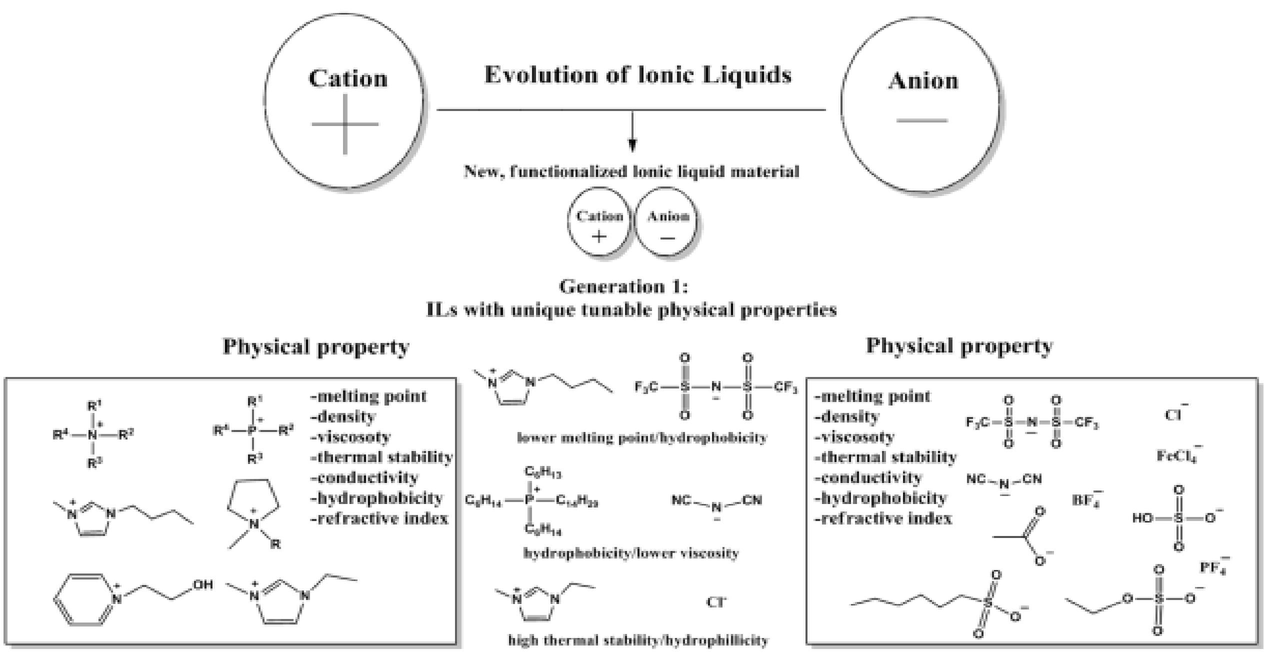

Figure 1. The first generation of ionic liquids with physical, chemical and biological properties (Kianfar. Mafi, 2020)

Second generation

With the increasing growth of these compounds, a group of ionic liquids was designed, which are known as ionic liquids with specific chemical use. These compounds have one or more specific functional groups on the cation that can interact and play a specific chemical role. For example, they are used as lubricants and complex ligands. In addition to the physical properties mentioned, these compounds also have chemical efficiencies, known as the second generation of ionic liquids. In Figure 2, these ionic liquids are introduced as the second generation. (Kianfar. Mafi, 2020)

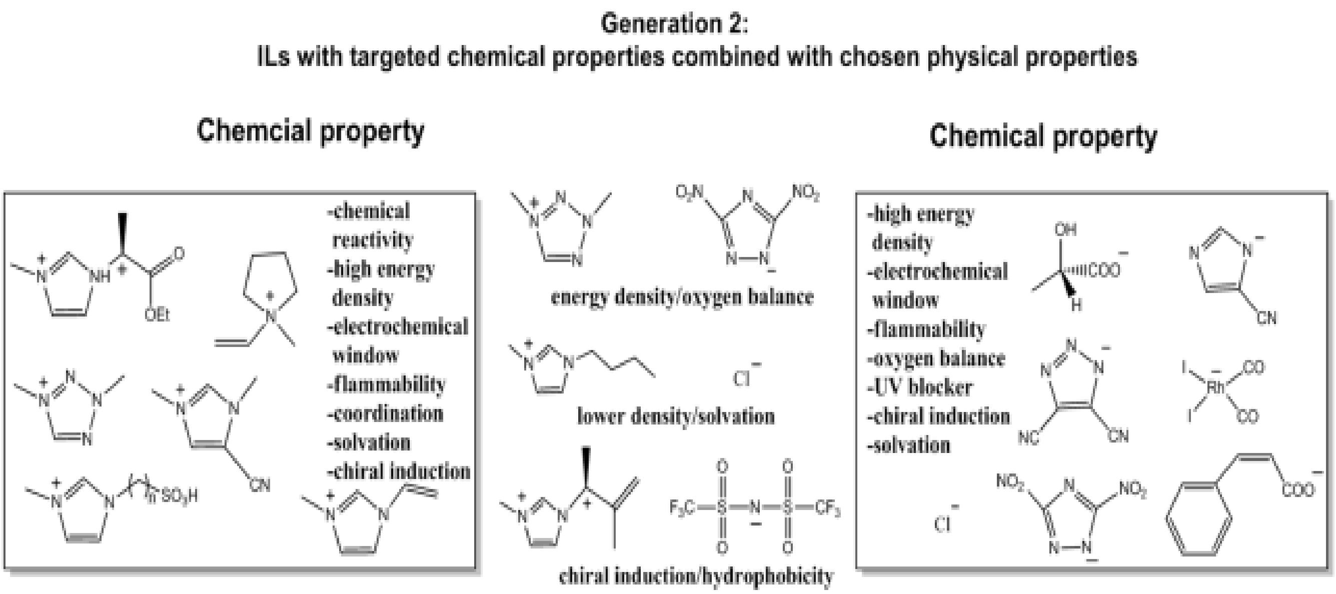

Figure 2. The first generation of ionic liquids with physical, chemical and biological properties. (Kianfar. Mafi, 2020)

Third generation

Some active pharmaceutical compounds have a structure that has classical ionic units and are biologically active and their toxicity has been investigated. Using these drugs, the new generation of ionic liquids of the third generation has been introduced. These compounds have very low toxicity and also have the physical properties of ionic liquids. This means ions can be used as drugs. In Figure 3, these ionic liquids are introduced as the third generation. (Kianfar. Mafi, 2020)

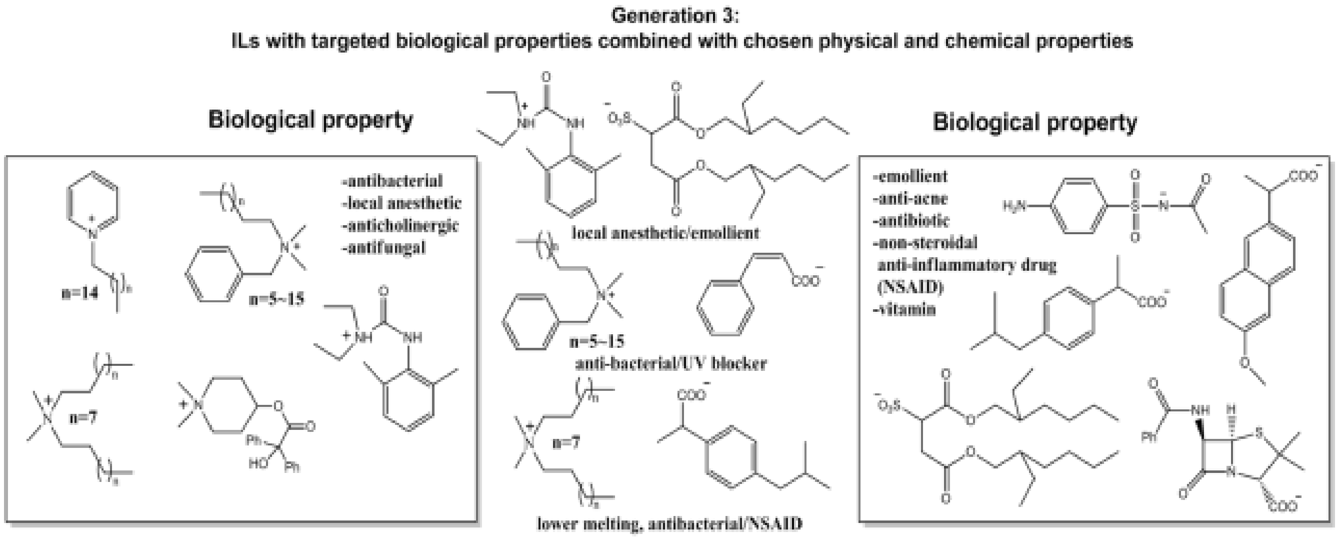

Figure 3. The first generation of ionic liquids with physical, chemical and biological properties. (Kianfar. Mafi, 2020)

Critical Properties of ionic liquid

Density: has been measured for the density as a function of the temperature for a range of imidazolium, pyridinium, ammonium, phosphonium and propidium based ionic liquids. For pure ILs.

1. **Viscosity**: Viscosity relates to the internal friction within the fluid which is caused by intermolecular interactions and is therefore important in all physical processes.
2. **Surface Tension**: The versatility of ILs has driven increasing interest in using them in extraction and multiphasic homogeneous catalytic reactions
3. **Specific Heat Capacity**: Heat capacity represents the relationship between energy and temperature for a specified quantity of material , this value relates to the kinetic energy.
4. **Thermal Conductivity** At present there are limited data available on the thermal conductivities of ionic liquids. (Valderrama and Robles 2007)

Applications of ionic liquids

Today, ionic liquids are widely used in various sciences and technologies. The most important use of ionic liquids is to act as a green solvent instead of volatile solvents. Today, ionic liquids have a wide range of other uses, some of which are briefly mentioned. (Kianfar. Mafi, 2020)

1) **Catalytic reactions:**

   Ionic liquids are used as a two-phase catalyst or substrate to stabilize other catalysts. In the presence of ionic liquids, it is possible to reuse the catalyst. The general state of reaction.

2) **Stability of nano catalysts in an ionic liquid medium:**

   Metal nano catalysts such as gold, platinum, palladium, rhodium, and ruthenium are widely used in organic reactions. The problem with nano catalysts is that they bind together in reaction environments and become clumpy, greatly reducing their activity.

3) **Solvent:** As mentioned, the main use of UV fluids is as a solvent. One of the most important benefits of using ionic liquids is increasing the speed of reactions and improving orientation relative to other solvents.

4) **Electrochemistry:** Some ionic fluids were the best examples for electrochemical devices such as power storage, fuel cells, photovoltaic cells, and electric hydration. This is due to the very high electrochemical stability, high conductivity, and wide temperature performance range.

5) **Liquid-liquid extraction:** One of the methods used for separation is liquid-liquid extraction. This method is used in industry because it is very energy efficient.

Desulfurization

Desulfurization or desulphurization is a chemical process for the removal of sulfur from a material. or the removal of sulfur compounds from a mixture such as oil refinery streams processes are of great industrial and environmental importance as they provide the bulk of sulfur used in industry. (Kulkarni, M. Afonso,2010)

Type of desulfurization: (Saha, Vidyacharan, Dalai,2020)

1. Hydrodesulfurization (HDS).
2. Oxidative Desulfurization (ODS).
3. Bio-Desulfurization (BDS).
4. Extractive Desulfurization (EDS).
5. Adsorptive Desulfurization (ADS).

Advantages And Disadvantage of Desulfurization methods

Advantages and disadvantage of desulfurization methods are shown in Table 1:

Table 1: Advantages and Disadvantage of Desulfurization methods. (Saha, Vidyacharan, Dalai,2020)

Type	**Advantages**	**Disadvantages**
HDS	1)Well-known mechanism and conventional technology 2)In light fractions, hydrogen gas is feasibly applied for desulfurization. 3)Removal of sulfides, thiols, and thiophenes successfully	1)Needs high pressure and temperature. 2)Employs expensive catalyst. 3)Decreases octane grade of gasoline.
BDS	1)Modest operating conditions i.e. lower pressure and temperature 2) Environmentally friendly with fewer greenhouse emissions. 3)Produces less acid rain gases. 4)High specificity of the enzyme.	1)Reduces fuel value. 2)Very slow rate. 3)Deep desulfurization cannot attain (10–100 ppm sulfur). 4)Sensitive
ADS	1)Operates at a lower temperature 2)No need of H2 3)No release of H2S 4)Capability to eliminate refractory S-compounds.	1) High amount of sorbent is required for surface reactions. 2)Poor selectivity of S-compounds. 3)Less adsorption capability, hence demanding multiple large adsorbent beds.

ODS	1)non-catalytic process. 2)Low-cost raw materials. 3)For light fractions, the mechanism is well studied. 4)The rate of reaction is larger as compared to hydrodesulfurization. 5)No use of costly H2 gas. ✓Minor temperature and pressure.	1)Robust oxidizing agent. g·H_2O_2 is costly for larger-scale applications. 2)Catalyst is required for deep desulfurization. 3)More reactions on oxidation may result in the production of non-essential
EDS	1)Easy to integrate with a refinery method. 2)Solvents such as DESs can be recycled. 3)Does not require hydrogen. ✓No use of catalyst. 4)Does not react with desired fuel oils.	1)Solubility of sulfur in solvents is restraint therefore appropriate choice of solvent is necessary. 2)Higher efficiency can be achieved. 3)The task to eliminate

Desulfurization by Ionic Liquids

can be applied for the desulfurization of liquid fuel owing to their very low vapor pressure. ionic liquids as class of green solvents can play a major role in the deep desulfurization of diesel fuel. For this reason, focuses on the current status in application of ionic liquids for achieving ultra-low-sulfur diesel (ULSD). To get a comprehensive perspective about the topic, other techniques of desulfurization are also discussed in brief in the introduction. Here we propose that the appropriate removal method should be selected according to different systems. To achieve deep desulfurization using ionic liquids, a better understanding regarding the regeneration of ionic liquids is vitally important. (Kulkarni. M. Afonso,2010)

Imidazolium-based ionic liquids

Table 2: Imidazolium-based ionic liquids:(Alonso, Arce et al. 2008)						
	Type of IL used	Fuel type	Ratio	Conditions	Extraction	Reference
1	1-ethyl-3-methylimidazolium tetrafluoroborate ($EMIM^+BF_4^-$),1-butyl-3 methylimidazolium hexafluorophosphate ($BMIM^+PF_6^-$) and1-butyl-3-methylimidazolium tetrafluoroborate ($BMIM^+BF_4^-$)	Transportation fuel	1:5	Room temperature	30%	(Zhang and Zhang 2002)
2	l-butyl-3-ethylimidazolium ethyl sulfate ($BEIMEt^+SO_4^-$), l-ethyl-3-ethylimidazolium ethyl sulfate, ($EEIMEt^+SO_4^-$), l-ethyl-3-methylimidazolium ethyl sulfate ($EMIMET^+SO_4^-$), l-ethyl-3-methylimidazolium methyl sulfate ($EMIMMe^+SO_4^-$), l-butyl-3-methylimidazolium methyl sulfate ($BMIMMe^+SO_4^-$), and 1,3-dimethylimidazolium methyl sulfate ($MMIMMe^+SO_4^-$)	diesel	1.1	room temperature	70%	(Mochizuki and Sugawara 2008)
3	1-methyl-3- octyl imidazolium tetrafluoroborate ($C_8MIM^+BF_4$)	model fuel	NA	room temperature	80%	(Alonso, Arce et al. 2008) (Alonso, Arce et al. 2007)

1) It has been found that the structure and size of the cation and anion of an IL can affect the absorption process. For most model aromatic compounds selected, the ILs $BMIM^+PF_6^-$ and $BMIM^+BF_4^-$ have shown higher absorption capacities than $EMIM^+BF_4^-$. Further observation reveals that absorption is favored for molecules with a higher density of aromatic

2) p electrons. For example, thiophene with a five-membered ring had a stronger interaction with the ILs than the nonaromatic isobutyl thiol. They also treated ILs with actual fuel and found about 30 wt.% of sulfur was preferentially removed with little change in the content of the aromatics.

3) It has been found with an increase in the length of alkyl chains, i.e. IL $BEIMEt^+SO_4^-$ having the longest alkyl chain, showed the highest extraction yield. reported a good selectivity for DBT over diphenyl sulfide and diphenyl disulfide

Pyridinium-based ionic liquids

Table 3: Pyridinium-based ionic liquids:(Holbrey, López-Martin et al. 2008)						
	Type of IL used	Fuel type	ratio	conditions	extraction	reference
1	pyridinium ILs for the extraction of sulfur compounds	diesel	NA	room temperature	0.5%	(Gao, Luo et al. 2008)
2	DBT from dodecane	diesel	NA	room temperature	81–83%	(Holbrey, López-Martin et al. 2008)

1. It has been found that pyridinium-based ILs, viz. N-butyl pyridinium tetrafluoroborate ($BPy^+BF_4^-$), N- It has been found that pyridinium-based ILs, viz. N-butyl pyridinium tetrafluoroborate ($BPy^+BF_4^-$), N-hexyl pyridinium tetrafluoroborate ($HPy^+BF_4^-$), and N-octyl pyridinium tetrafluoroborate ($OPy^+BF_4^-$) have neg gable solubility in the fuel
2. It has been found that ILs with ethanoate and thiocyanate anions gave the best extraction performance with each cation. These two anions with the pyridinium cations showed highest extraction performance with 81–83% of the DBT removed in one contact.

Lewis and Brønsted acidic ionic liquids or redox ionic liquids

Table 4: Lewis and Brønsted acidic ionic liquids or redox ionic liquids: (Bösmann, Datsevich et al. 2001)						
	Type of IL used	Fuel type	ratio	conditions	extraction	reference
1	1-n-butyl-3-methylimidazolium (BMIM $+Cl^-$) with $AlCl_3$	diesel	1: 1	Room temperature	NA	(Bösmann, Datsevich et al. 2001)
2	DBT	diesel	2	Room temperature	NA	(Ko, Lee et al. 2008)

1. it has been found the Lewis-acidic IL, showed much higher efficiency in sulfur extraction from real diesel in comparison to Brønsted-acidic and neutral IL. it is reported that more extraction steps are necessary in the case of 'real' diesel oil to reach future technical sulfur content specifications.
2. It has been found decrease in sulfur. It was suggested that Lewis's acid–base interaction and Fe^{3+} can form p-complexation bonding with aromatic sulfur compound and thus enhances the extraction of sulfur species.

EDS performance of different ILs

Table 5: EDS performance of different ILs: (Nie, Li et al. 2007)						
	Type of IL used	Fuel type	ratio	conditions	Extracted yield	reference
1	[BMI][N(CN)2]	diesel	1: 1	Room temperature	68.9%	(Nie, Li et al. 2007)
2	[EMI][$N(CN)_2$]	diesel	1: 1	Room temperature	55.6%	(Nie, Li et al. 2006)
3	[S_2] [$N(CN)_2$]	diesel	1: 1	Room temperature	51.2%	(Ko, Lee et al. 2008)
4	[$EtMe_2S$] [$N(CN)_2$]	diesel	1: 1	Room temperature	45.5%	(Gao, Luo et al. 2008)

- in other IL molecules with a highly polarizable p-electron density tend to insert into the molecular structure of ILs. [BMI][N(CN)$_2$] exhibits the highest S-extraction efficiency for both TS and DBT.

Parameters affecting desulfurization

Temperature

Table 6: Extraction temperature: (Huang, Chen et al. 2004)

	Type of IL used	Fuel type	ratio	conditions	extraction	reference
1	[BMI][N(CN$_)2$] for TS	diesel	1: 1	Room temperature	40.6%	(Huang, Chen et al. 2004)
2	[BMI][N(CN)$_2$] for DBT	diesel	1: 1	Room temperature	7.7%	(Huang, Chen et al. 2004)

- The S-extraction efficiency for TS slightly decreases to 40.6% at 55 ◦C, a difference of only 6%; for DBT, the extraction is 7.7%. Such insensitivity to temperature can be understood due to the low viscosity of the ILs used in this study. Therefore, S-extraction can be performed at or below room temperature.

Table 7 shows Multiple extractions for model gasoline: (Huang, Chen et al. 2004)

Table 7: Multiple extractions for model diesel:(Huang, Chen et al. 2004)

	Type of IL used	Fuel type	ratio	conditions	extraction	reference
1	[BMI][N(CN)$_2$] for TS	gasoline	1: 1	Room temperature	100%	(Huang, Chen et al. 2004)
2	[BMI][N(CN)$_2$] for DBT	gasoline	1: 1	Room temperature	100%	(Huang, Chen et al. 2004)
3	[HMMPY][NTf$_2$]	gasoline	1: 1	Room temperature	26.7%	(Asumana, Haque et al. 2013)

- for TS in model oil, after 5 cycles, the Scantent in the gasoline remarkably dropped from 599 to 4 ppm (less than the current S-limit in fuel oils), an upsurge of almost 100% S-removal; for DBT in diesel fuel, after only 4 cycles.

Table 8: Multiple extractions for model diesel:(Huang, Chen et al. 2004)

	Type of IL used	Fuel type	ratio	conditions	extraction	reference
1	[BMI][N(CN)$_2$] for TS	diesel	1: 1	Room temperature	100%	(Huang, Chen et al. 2004)
2	[BMI][N(CN)$_2$] for DBT	diesel	1: 1	Room temperature	100%	(Huang, Chen et al. 2004)
3	[HMMPY][NTf$_2$]	diesel	1: 1	Room temperature	98%	(Asumana, Haque et al. 2013)

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Table 9 :Extractive performance after regeneration: (Huang, Chen et al. 2004)

	Type of IL used	Fuel type	Ratio	conditions	extraction	reference
1	[BMI][N(CN)$_2$] for TS	diesel	1: 1	Room temperature	45.2%	(Huang, Chen et al. 2004)
2	[BMI][N(CN)$_2$] for DBT	diesel	1: 1	Room temperature	65.3%	(Huang, Chen et al. 2004)

- After regeneration, its original structure was unchanged. The S-extraction performance of the regenerated IL was investigated indicating an inconspicuous decrease in the S-extraction efficiency after 6 regeneration cycles a clear indication that [BMI][N(CN)$_2$] is favorable for industrial applications because of its cost-effective regeneration, good reusability and simple operating conditions.

Table 10 : Regeneration: (Asumana, Yu et al. 2010)

	Type of IL used	Fuel type	Ratio	conditions	extraction	reference
1	[BMTH] [N(CN)$_2$] for TS	model oil	1: 1	Room temperature	49.6%	(Asumana, Yu et al. 2010)
2	[BMTH] [N(CN)$_2$] for DBT	model oil	1: 1	Room temperature	69.5%	(Asumana, Yu et al. 2010)
3	$Et_3NHCl-AlCl_3$ for TS	model oil	1: 1	Room temperature	25%	(Wytze Meindersma, Podt et al. 2005)
4	$Et_3NHCl-AlCl_3$ for DBT	model oil	1: 1	Room temperature	40.2%	(Wytze Meindersma, Podt et al. 2005)

- S-compounds in the vicinity of solvent molecules are repelled; after regeneration, ILs are reused without a noticeable decline in efficiency for model oils as demonstrated in the literatures.

Type of ionic liquids

Table 11 :Effect of different ionic liquids on DBT removal: (Nie, Li et al. 2006)						
	Type of IL used	Fuel type	ratio	conditions	extraction	reference
1	[bmim]Cl/$FeCl_3$	gasoline	1: 3	Room temperature	99.2%	(Nie, Li et al. 2006)
2	[omim]Cl/$FeCl_3$	gasoline	1: 3	Room temperature	87.0%	(Nie, Li et al. 2006)
3	$Et_3NHCl/FeCl_3$	gasoline	1: 3	Room temperature	68.3%	(Nie, Li et al. 2006)

- ionic liquids showed higher extractive ability than $Et_3NHCl/FeCl_3$, which could be attributed to the principle of similitude-compatibility for aromatic cycle-containing ILs.

Effect of catalysts

Table 12: Effect of different catalytic systems containing FeCl3 species on DBT removal: (Nie, Li et al. 2006)

	Type of IL used	Fuel type	ratio	conditions	extraction	reference
1	[bmim]Cl/$FeCl_3$	gasoline	1: 3	Room temperature	66.0%	(Nie, Li et al. 2006)
2	Anhydrous $FeCl_3$+ H_2O_2	gasoline	1: 3	Room temperature	99.2%	(Nie, Li et al. 2006)
3	[bmim]Cl/$FeCl_3$+ H_2O_2	gasoline	1: 3	Room temperature	57.4%	(Nie, Li et al. 2006)

- It is clearly demonstrated that a combination of extraction and catalytic oxidation in [bmim]Cl/$FeCl_3$.The remarkable advantage of this process in the desulfurization of model oil(gasoline) by mere solvent extraction with IL can also be seen.

Table 13: Two desulfurization systems with different substrates: (Zhu, Li et al. 2008)

	Type of IL used	Fuel type	ratio	conditions	extraction	reference
1	DBT	model oil	1: 3	Room temperature	99.2%	(Zhu, Li et al. 2008)
2	4,6-DMDBT	model oil	1: 3	Room temperature	90.3%	(Zhu, Li et al. 2008)
3	BT	model oil	1: 3	Room temperature	75.9%	(Zhu, Li et al. 2008)

- Therefore, reactivity was mainly affected by the steric hindrance of the methyl groups, which became an obstacle for the approach of the sulfur atom to the catalytically active species.

Conclusion

Here we talked about desulfurization fuel by ionic liquid, summary about ionic liquid and desulfurization and its types and we selected the best types of desulfurization, that's how it was (Multiple extraction for model oil of gasoline and diesel) because for TS in model oil, after 5 cycles, the Scantent in the gasoline remarkably dropped from 599 to 4 ppm (less than the current S-limit in fuel oils), an upsurge of almost 100% S-removal; for DBT in diesel fuel, after only 4 cycles. And (Effect of different ILs on DBT removal) because ionic liquids showed higher extractive ability than $Et_3NHCl/FeCl_3$, which could be attributed to the principle of similitude-compatibility for aromatic cycle-containing ILs.

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