New glycerol-based deep eutectic solvents as green extragents for diesel fuel

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The dearomatization, denitrification and oxidative desulfurization of commercial diesel fuels were investigated using glycerol-based deep eutectic solvents. The optimal conditions of liquid-liquid extraction processes were chosen as room and 90°C temperatures and 3 hours of mixing time. Ammonium chloride-glycerol, triethylammonium acetate-glycerol and choline chloride-glycerol were used as extractive solvents in the purification processes. Diesel-Deep eutectic solvents (Diesel-DESs) were taken as the volume ratios of 1:1 for the dearomatization and denitrification processes. The volume ratios of Diesel-DESs-H_2O_2 were 1:1:2 for the oxidative desulfurization. H_2O_2 was selected as the oxidative agent of oxidation desulfurization. Before and after separation processes, the exploitation properties were studied by ASTM standards and compared to commercial diesel. All separation processes were controlled by gas chromatography and ^{1}H NMR methods. Based on the NMR analysis, NH_4Cl/6Glycerol with H_2O_2 and ChCl/6Glycerol were the most effective extractive agents for purified diesel samples, from the alkyl aromatic, naphthenic, and heteroatomic compounds.

Keywords: deep eutectic solvents; glycerol; diesel; gas chromatography; ASTM standards; SDG 6; SDG 13.

Дизель отынын экстракциялауға арналған глицерин негізіндегі жаңа терең эвтектикалық еріткіштер

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Коммерциялық дизель отындарын деароматтандыру, денитрификациялау және тотығулы күкіртсіздендіру процестері глицерин негізіндегі терең эвтектикалық еріткіштерді (DES) қолдану арқылы зерттелді. Сұйық–сұйық экстракция процестерінің оңтайлы шарттары бөлме температурасы мен 90°C және араластыру уақыты 3 сағат ретінде таңдалды. Тазарту процестерінде экстрагенттер ретінде аммоний хлориді-глицерин, триэтиламмоний ацетаты–глицерин және холин хлориді-глицерин қолданылды. Деароматтандыру және денитрификация процестері үшін дизель-DES көлемдік қатынасы 1:1 болды. Тотығулы күкіртсіздендіру үшін дизель-DES-H_2O_2 көлемдік қатынастары 1:1:2 құрады. ODS процесінде тотығу агенті ретінде H_2O_2 таңдалды. Бөлу процестеріне дейін және кейін пайдалану қасиеттері ASTM стандарттарына сәйкес зерттеліп, коммерциялық дизель отынымен салыстырылды. Барлық бөлу процестері газ хроматографиясы және ^{1}H ЯМР әдістерімен бақыланды. ЯМР талдауының нәтижелері бойынша, NH_4Cl/6Glycerol + H_2O_2 және ChCl/6Glycerol тазартылған дизель үлгілерінен алкил ароматты, нафтендік және гетероатомды қосылыстарды жоюда ең тиімді экстрагенттер болып анықталды. Бөлу процесі GC әдісімен де бақыланды.

Түйін сөздер: терең эвтектикалық еріткіш; глицерин; дизель; газ хроматографиясы; ASTM стандарттары; SDG 6; SDG 13.

Новые глубокие эвтектические растворители на основе глицерина в качестве экологически чистых экстрагентов для дизельного топлива

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Процессы деароматизации, денитрификации и окислительной десульфуризации коммерческого дизельного топлива с использованием глубоких эвтектических растворителей на основе глицерина были исследованы. В качестве оптимальных условий жидкостно-жидкостной экстракции были выбраны комнатная температура и температура 90°C, а также время перемешивания 3 часа. В процессах очистки в качестве экстракционных растворителей использовались системы хлорид аммония-глицерин, ацетат триэтиламмония-глицерин и хлорид холина-глицерин. Для процессов деароматизации и денитрификации дизельное топливо и глубокие эвтектические растворители использовались в объёмном соотношении 1:1. Для процесса окислительной десульфуризации объёмное соотношение дизельного топлива, глубокого эвтектического растворителя и H_2O_2 составляло 1:1:2. В качестве окислителя в процессе окислительной десульфуризации использовалась H_2O_2. До и после процессов разделения эксплуатационные свойства топлива изучались в соответствии со стандартами ASTM и сравнивались с характеристиками коммерческого дизельного топлива. Все процессы разделения контролировались методами газовой хроматографии и ^{1}H ЯМР-спектроскопии. На основании анализа ЯМР было установлено, что NH_4Cl/6Glycerol с H_2O_2 и ChCl/6Glycerol являются наиболее эффективными экстракционными агентами для очистки дизельного топлива, обеспечивая удаление алкилароматических, нафтеновых и гетероатомных соединений.

Ключевые слова: глубокий эвтектический растворитель; глицерин; дизельное топливо; газовая хроматография; стандарты ASTM; SDG 6; SDG 13.

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Article

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1. Introduction

Diesel engines are internal combustion engines, known for their lower fuel consumption than other analogs. They have numerous applications, including construction, underground mining, power generation and farming. Besides, they are used in various land and sea transport systems. However, controlling diesel motor emissions is important [1-5]. Petroleum diesels have nitrogen, sulfur, oxygen contents and aromatic hydrocarbons that are burned to produce hazardous pollutants such as SO_x and NO_x. On the other hand, aromatic hydrocarbons produce the output of soot and residues [6-9]. Reducing aromatic and heteroaromatic compounds in fuels is conventionally achieved through a process called hydrotreatment. The drawbacks of traditional hydrotreatment have contributed to the development of more "greener" purification methods [10,11]. A class of solvents known as ionic liquids was investigated for separating aromatic and heteroaromatic compounds from fuels [12-14]. The poor biodegradability, toxicity, complex synthesis, and high production cost are the shortcomings of their application in industry. Therefore, selective and eco-friendly solvents have been searched by scientists.

Deep eutectic solvents (DESs) are a new generation of solvents formed by a eutectic mixture of a hydrogen bond donor (HBD) and a hydrogen bond acceptor (HBA). They have good chemical and thermal stability. The raw materials of DESs are cheap, easily obtained, and environmentally friendly [15-18]. DESs have a wide range of applications in chemistry. Using DESs in chemical separation is interesting for producing clean motor fuels [19]. In many research papers, polymeric, metal complex, and acidic-based DESs were investigated as an extractive agent for the purification of organic sulfides [20-24]. Ammonium, phosphonium, and choline chloride-based deep eutectic solvents also showed sensitivity to the denitrification of fuels [25,26]. Moreover, several studies have also studied the liquid-liquid extraction (LLE) of aromatic compounds from fuels by DESs [27,28].

In the presented work, a new type of glycerol-based DESs [NH_4Cl/6Glycerol (DES1), $[TEAH]^+$ $[AcO]^-$/6Glycerol (DES2) and ChCl/6Glycerol (DES3)] were prepared and investigated as an extractive solvent for aromatic, sulfur and nitrogen compounds from the diesel fuel at room and 90°C temperatures. The optimal mixing time was chosen as 3 hours. The physical and chemical properties of diesel fuels were determined according to the ASTM standards. The separation efficiencies of each compound class were studied by ^{1}H NMR and GC analysis.

2. Experiment

All chemicals were obtained from Merck (Germany) and used as received. The tested diesel fuel was purchased at a fuel station in Azerbaijan.

2.1. Preparation of glycerol based DESs

DESs were prepared by mixing hydrogen bond donor (HBD) glycerol with hydrogen bond acceptors (HBAs) as choline chloride, ammonium chloride or triethylammonium acetate. The preparation of eutectic mixtures was carried out in a screw-capped bottle by magnetic stirring (800 rpm) at 20°C. According to our previous work, the optimal molar ratio of the HBA and HBD was selected as 1:6 [29]. The process was considered complete when the two components transformed into a homogeneous transparent liquid visually during the 2 hours.

2.2 LLE experiments

The dearomatization and denitrification of diesel fuels were carried out at room temperature for 3 hours. The volume

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ratios of DESs and diesel fuel were 1:1. The oxidation desulfurization (ODS) of real fuels was done at 90°C in 3 hours. The oxidative agent of ODS was 30% H_2O_2. The optimal conditions of this process were at the volume ratio of DESs/ Fuel/H_2O_2 = 1:1:2 [30].

2.3 Analytical methods

The amounts of sulfuric, nitrogen and aromatic compounds after LLE were determined using an NMR spectrometer (UltraShield Magnet) AVANCE 300 MHz (300 MHz for the ^{1}H) and a GC Agilent chromatography. The following formula calculates the extraction efficiencies:

$$Y\,(\%) = C_0 - C \,/\, C_0 \times 100\%$$

Y (%) – removal efficiencies of aromatic, nitrogen and sulfur compounds; C_0 – the initial concentration, C – the concentration after liquid-liquid extraction of each compound.

The exploitation properties of diesel and cleaned diesel samples were studied according to the ASTM standards. The cetane number and cetane index were calculated according to the literature [31].

The kinematic viscosity (by Ostwald-Pinkevitch viscometer), density (by pycnometer), corrosion properties (by copper corrosion, at 3 hours and 50°C), flash point (by Pensky-Marten's apparatus) of fuel samples were studied according to the ASTM D445, ASTM D2320, ASTM G31 and ASTM D93.

3. Results and Discussion

The three types of glycerol-based DESs were used for the separation of aromatic and heteroatomic hydrocarbons from commercial diesel fuels: NH_4Cl/6Glycerol (DES1), $[TEAH]^+$ $[AcO]^-$ /6Glycerol (DES2) and ChCl/6Glycerol (DES3). The dearomatization, denitrification processes of diesel fuel were carried out in the room, but ODS processes at 90°C temperatures. DESs/Diesel fuels or DESs/Diesel fuels/ H_2O_2 were taken as 1:1 and 1:1:2 molar ratios.

3.1 The results of liquid-liquid extraction

First, the content of commercial diesel fuel, as well as diesel fuels purified using DESs, was studied. Amount of paraffinic, naphthenic, aromatic hydrocarbons in commercial diesel and diesel samples after LLE by DESs 1-3 were calculated by ^{1}H NMR [32]. In Table 1, the percentage content of hydrocarbons in diesel samples was given.

As can be seen from Table 1, the purification of diesel with DES1(H_2O_2) and DES3 is more effective for the separation of the aromatic hydrocarbons. The NMR results showed that the percentage amount of aromatic hydrocarbons reduced from 8.87 to 4.58 with DES1 (H_2O_2) and from 8.87 to 4.84 with DES3. Based on the GC results for sample 1 (LLE by DES3) and sample 2 (LLE by DES1), the removed individual naphthenic, aromatic and heteroaromatic hydrocarbons were given in Tables 2 and 3.

We observed a negative influence (or opposite direction of extraction) on phase equilibrium after 3 hours for volume ratios of 1:1 and 1:1:2.

Table 1 – The percentage content of hydrocarbons in diesel samples

Diesel samples	The percentage content of hydrocarbons, %		
	The aromatic compounds	The naphthenic compounds	The paraffinic compounds
Diesel	8.87	38.10	53.03
Diesel (DES1)	8.61	34.45	56.94
Diesel (DES2)	8.68	39.12	52.20
Diesel (DES3)	4.84	32.31	62.85
Diesel (DES1+H_2O_2)	4.58	35.11	60.31
Diesel (DES2+H_2O_2)	7.93	36.71	55.36
Diesel (DES3+H_2O_2)	5.00	35.00	60.00

As can be seen from Table 2, mainly alkyl naphthenic, alkyl aromatic and alkyl heteroatomic compounds were purified from sample 1 and n-paraffins, which are a very important component for diesel fuels, were not affected by DESs. The presence of n-paraffins in diesel fuel after LLE, can be explained by the lack of centers in this class of hydrocarbons, which could interact with DESs.

It can be seen from Table 3, in sample 2, alkyl naphthene, alkyl aromatic and alkyl heteroatomic compounds were purified. In Figures 1-3, chromatograms of commercial diesel and diesel samples 1 and 2 after LLE extractions were given.

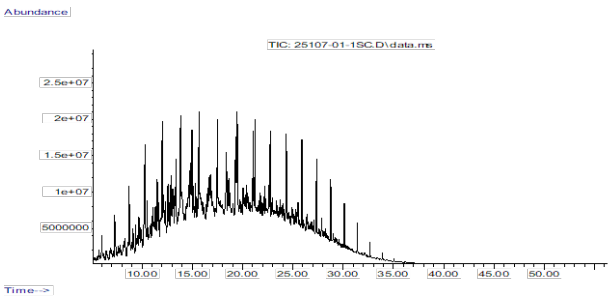

Figure 1 – The chromatogram of diesel fuel

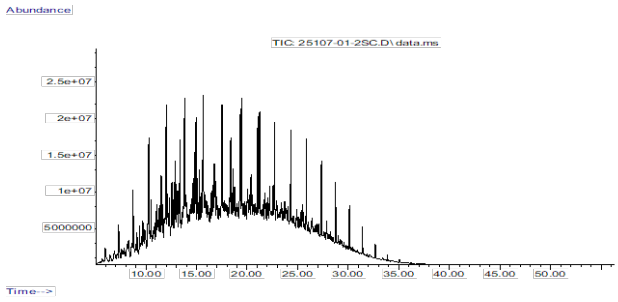

Figure 2 – The chromatogram of diesel after LLE by DES3 at room temperature

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Table 2 – The separated naphthenic, aromatic and heteroaromatic hydrocarbons from Sample 1

Naphtenic hydrocarbons	Retention time	Area	Alkyl aromatic hydrocarbons	Retention time	Area	Heteroaromatic hydrocarbons	Retention time	Area
Decaline	8.255	0.0488	1-Ethyl-3-methylbenzene	6.8642	0.0417	1-Acetyl-1,2,3,4-tetrahydropyridine	23.2909	0.3027
2-Methyldecaline	9.0981	0.2238	1,3-Dimethylbenzene	7.6804	0.0419	1-(2-Methylphenyl)-isoquinoline	37.8328	0.0041
1-Cyclohexyl-3-methylbenzene	14.3982	0.559	1,4-Dimethyl-2-ethylbenzene	8.1906	0.1149	2-Heptadecylthiophene	28.9348	0.0707
1,2,3-Trimethylcyclohexane	5.758	0.0409	1-Methyl-4-propylbenzene	8.3463	0.1143			
1-Ethyl-4-methylcyclohexane	6.1499	0.0404	1,2,4,5-Tertamethylbenzene	9.1625	0.1475			
2-Ethylbicyclo [4,4,0] decane	10.9561	0.3199	Decahydro-2,3-dimethylnaphthalene	9.9519	0.2319			
n-Heptylcyclohexane	12.8785	0.8235	1,2,3,4-Tertahydro-5-methylnaphthalene	11.6273	0.5707			
n-Nonylcyclohexane	16.7127	1.1107	1,2,3,4-Tertahydro-2,7-dimethylnaphthalene	12.5886	1.0116			
1-Methyl-2-propylcyclohexane	21.5027	0.4655	1,2,3,4-Tertahydro-1,8-dimethylnaphthalene	12.7174	0.3995			
n-Tetradecylcyclohexane	25.5248	0.5853	1-(1-Methylethenyl)-3-(1-methylethyl)benzene	13.0557	0.6429			
1,2-Diethylcyclohexadecane	25.1006	0.3323	1,2,3,4-Tetrahydro-6,7-dimethylnaphthalene	14.5325	0.4036			
Cycloeicosane	26.0833	0.2634	1,2,3,4-Tetramethylbenzene	9.1625	0.1475			
n-Pentadecylcyclohexane	27.0821	0.5111	2-(2-butenyl)-1,3,5-trimethylbenzene	15.3648	0.5012			
Heneicosylcyclopentane	28.4515	0.2134	3-Methyl-9*H*-flourene	19.7575	0.5081			
n-Decylcyclopentane	29.853	0.2416	1,2,3,4,5,6,7,8-Octahydro-1-methylanthracene	20.9013	0.5667			
Cyclooctaeicosane	32.4736	0.0813	1,2,3,4,5,6,7,8-Octahydro-2-methylanthracene	20.7939	0.4744			
Cholestane	35.8029	0.0151	Cymene	8.6202	0.1019			

Table 3 – The separated naphthenic, aromatic and heteroaromatic hydrocarbons from Sample 2

Naphtenic hydrocarbons	Retention time	Area	Alkyl aromatic hydrocarbons	Retention time	Area	Heteroaromatic hydrocarbons	Retention time	Area
Decaline	8.255	0.0488	Ethylbenzene	5.6024	0.0067	1-Ethanamine-1H-pyrazole	28.9242	0.0721
2-Methyldecaline	9.0981	0.2238	*o*-Xylene	5.6936	0.0225	1-Acetyl-1,2,3,4-tetrahydropyridine	39.6963	0.0001
Ethylcyclohexane	5.248	0.0054	1-Ethyl-2-methylbenzene	6.8697	0.0391	1-(2-Methylphenyl)isoquinoline	37.8328	0.0041
1,1,3-Trimethylcyclohexane	32.8442	0.0526	1,2,4-Trimethylbenzene	7.3154	0.3053	2-Heptadecylthiophene	28.9348	0.0707
1,2,3-Trimethylcyclohexane,	7.7343	0.0426	1,2,3-Trimethylbenzene	7.7343	0.0426			
1-Ethyl-4-methylcyclohexane	5.4037	0.0064	1,3-Dimethylbenzene	5.6936	0.0225			
2-Ethylbicyclo [4,4,0] decane	10.950	0.3272	1,2,3,4-Tertahydro-5-methylnaphthalene	12.1323	0.82			
n-Heptylcyclohexane	12.8733	0.6545	1,2,3,4-tetrahydro-6-methyl naphthalene	11.6275	0.5779			
n-Nonylcyclohexane	28.4355	0.2186	1-(1-Methylethenyl)-3-(1-methylethyl)benzene	7.6859	0.0465			
1-Methyl-2-propylcyclohexane	27.9253	0.1172	1,2,3,4-Tetrahydro-2-methylnaphthalene	10.7737	0.2293			
n-Tetradecylcyclohexane	25.5088	0.5473	1,2,3,4-Tertahydro-2,7-dimethylnaphthalene	12.5726	0.756			
1,2-Diethylcyclohexadecane	32.9946	0.0367	1,2,3,4-Tertahydro-2,8-dimethylnaphthalene	12.7122	0.4065			
Heneicosane	26.7815	0.3739	2-Methyl-9*H*-flourene	19.7361	0.5144			
n-Decylcyclopentane	28.5482	0.2926	1,2,3,4,5,6,7,8-Octahydro-2-methylanthracene	20.8853	0.5175			

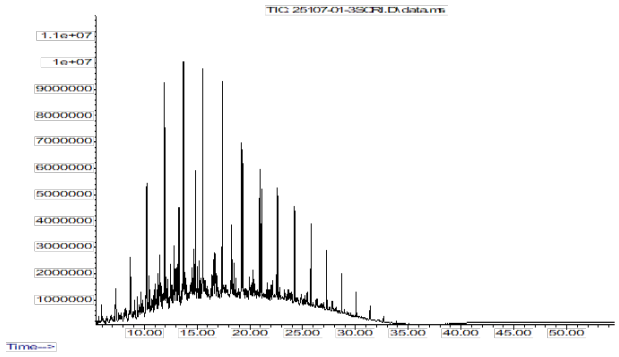


Figure 3 – The chromatogram of diesel after LLE by DES1 at 90° C temperature

3.2 The studying of exploitation properties of diesel fuels

The exploitation properties of diesel samples after purification at room and 90°C were investigated and compared according to the ASTM. The relative density (g/cm^3) at 20°C of diesel samples after LLE is shown in Figure 4.

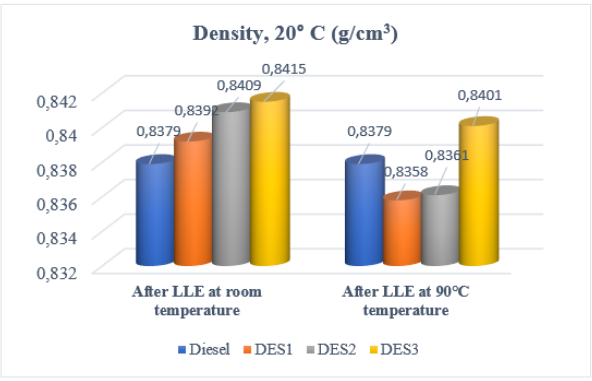


Figure 4 – The relative densities of commercial diesel and treated diesel samples by DESs

As shown in Figure 4, the density of purified fuel samples using DESs at room and 90°C temperatures increased slightly (0.003 and 0.002 accordingly) compared to diesel.

Figures 5 and 6 demonstrate the dependence of viscosity (mm^2/s) at 20°C and 40°C after the LLE. As can be seen from Figure 6, the viscosity at 40°C for the diesel sample after LLE by DES3 and DES2 showed the best results. So, the viscosity decreased from 5.1651 to 4.8042 at room temperature and from 3.1781 to 3.091 mm^2/s at 40°C (for room temperature extraction). This can be explained by the release of relatively high-viscosity alkyl naphthene, alkyl aromatic and alkyl heteroatomic compounds from the content of diesel fuels.

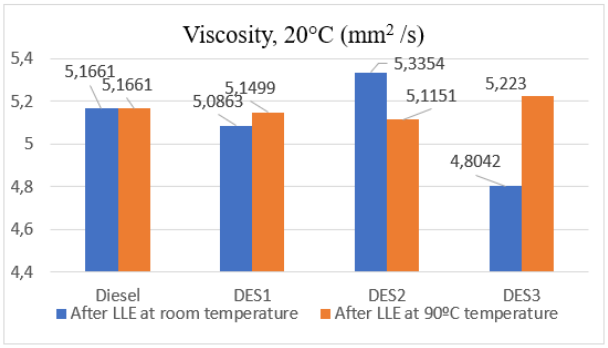


Figure 5 – Viscosity of commercial diesel and diesel samples at 20°C

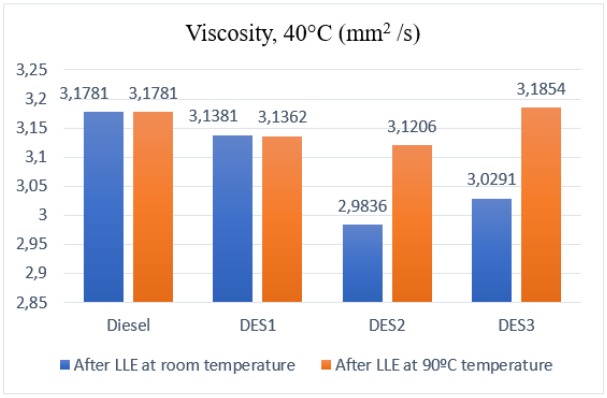


Figure 6 – Viscosity of commercial diesel and diesel samples at 40°C

Sulfur content in fuels was determined according to the ASTM D1266 after purifications at room and 90°C temperatures. Based on the obtained results, we can note that sulfur content (ppm) decreased in both purification conditions. The higher result was observed at room temperature by DES3 (amount of sulfur decreased from 50 up to zero ppm, Figure 7).

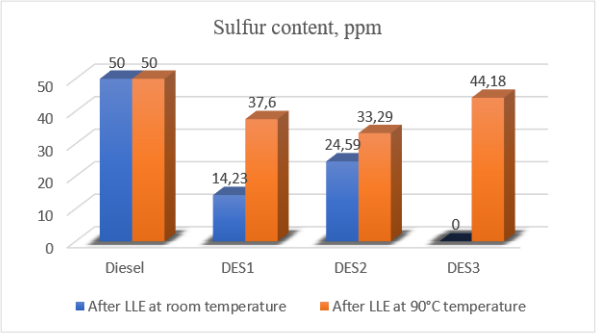


Figure 7 – The sulfur content of commercial diesel and diesel samples

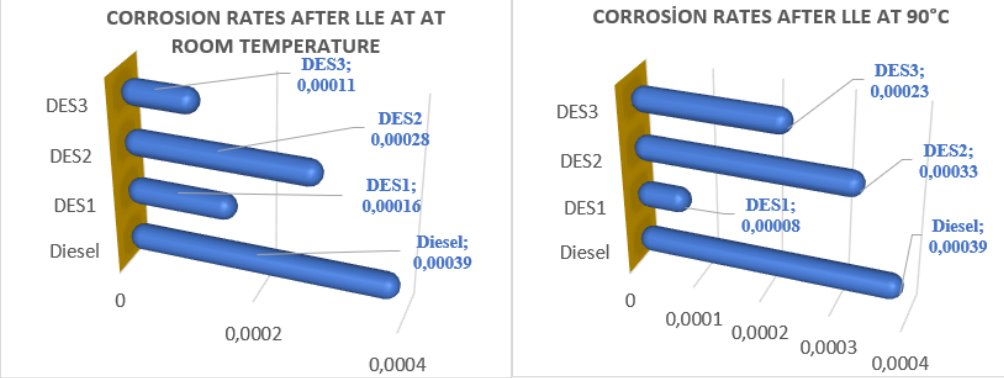


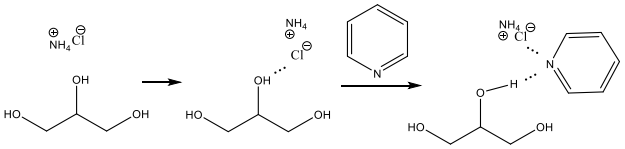
Figure 8 – The corrosion activities of diesel samples before and after LLE

The corrosion activities of diesel samples before and after LLE are given in Figure 8. As seen from the figure, there is a significant decrease in corrosion rate in fuel samples after LLE compared with commercial diesel. It is obvious that DES3 exhibits a more effective result for the anticorrosion ability of cleaned fuel at room temperature. After LLE at 90°C by DES1, the lowest corrosion rate was observed in both purification conditions. The removal of corrosive sulfur, nitrogen and alkylaromatic compounds from the content of commercial diesel fuel can explain the indicated results.

In the Table 4 was showed flash, cloud, pour points, cetane number and cetane index of investigated fuel samples.

According to Table 4, the flashpoint is increased after each extraction process. The cloud point and pour point of cleaned diesel samples by DESs decreased slightly in each room temperature extraction condition. The fuels treated by DES3 at room temperature exhibited the lowest cloud (-4°C) and pour (-17°C) temperatures. The diesel fuel cleaning with DES2 at 90°C showed +2°C cloud point and -19°C pour points compared to commercial diesel (accordingly +10 and -12°C). As seen, the pour point decreased by 3-5 units, despite an increase of the amount of paraffinic hydrocarbons. It may be connected to the extraction of high molecular mass alkyl heteroatomic, alkyl naphthenic and alkyl aromatic compounds. The higher diesel index of fuel was observed at room temperature by DES1. The obtained results can be explained by the removal of short as well as long alkyl chain naphthenes and aromatic hydrocarbons from the composition of commercial diesel fuel.

The proposed separation mechanism of pyridine by the choline chloride:glycerol based DES is illustrated below.



3.3. Regeneration and recycling of solvent

Considering the future industrial importance, DESs must be regenerated for separation processes. After the purification of diesel fuel samples, diethyl ether was used for the recycling of DESs. The volume ratios of DESs/diethyl ether were taken as 1:1. The regeneration times for DESs are 3 hours at room temperature. We would like to note that, after the fifth regeneration occurs, there is a decrease in volume and deterioration of solvent purity. The purification of DESs was controlled by NMR ^{1}H analysis. Below is a schematic illustration of the liquid-liquid extraction process.

Table 4 – Some physical properties of commercial diesel and diesel samples

Parameters	ASTM Methods	Experimental data						
		Diesel	LLE at room temperature			LLE at 90° C temperature		
			DES1	DES2	DES3	DES1	DES2	DES3
Flash point, °C, min.	D93	79	85	86	95	83	95	97
Cloud point, (°C)	D2500	10	0	-2	-4	4	2	6
Pour point, (^{0}C)	D2500	-12	-15	-17	-17	-12	-19	-18
Cetane number, min.	D975-14	47.11	50.12	46.55	55.69	53.87	49.14	53.50
Diesel Index	-	53.705	58.582	51.358	61.954	57.170	54.512	58.779

Scheme 1 – A basic illustration of LLE by DESs

3.4. An environmental or economic assessment of the DESs

It should be noted that glycerin is a by-product of biodiesel production; its disposal is one of the actual problems. Ammonium chloride is one of the cheapest salts produced by thousands of tons per year. The choline chloride is also industrially produced as a supplement (typically 60-70%) for compound feed, preventing fatty infiltration of the liver in animals and birds, improving their growth and productivity. All DES components are environmentally safe and biodegradable.

4. Conclusions

The combined processes of dearomatization, denitrification and oxidative desulfurization of commercial diesel fuel studied by a new type of glycerol-based DESs. Ammonium chloride, triethylammonium acetate ($[TEAH]^+[AcO]^-$) and choline chloride were selected as hydrogen bond acceptors of DESs. The molar ratios of HBA/HBD were chosen as 1:6. The liquid-liquid extraction was carried out at room and 90°C temperatures in 3 hours of mixing time. The dearomatization and denitrification of diesel were carried out at the volume ratios of DESs/diesel fuels as 1:1 at room temperature. The oxidative desulfurization process was conducted at the volume ratios of DESs/diesel/H_2O_2 as 1:1:2 at 90°C.

NMR analysis of diesel, purified diesel samples demonstrated that DES1 with H_2O_2 and DES3 were more extractive agents for the separation of alkyl aromatic, naphthenic, heteroatomic compounds. In these cases, after room temperature extraction, viscosity was decreased from 5.1651 to 4.8042 and from 3.1781 to 3.091 mm²/s at 40°C. Besides, the best result was observed at room temperature by DES3; the amount of sulfur decreased from 50 down to zero ppm. DES3 exhibits a more effective result for the anticorrosion ability of cleaned fuel at room temperature (weight loss decreased from 0.00039 to 0.00011 g).

The obtained results showed that sulfuric and nitrogen compounds were separated in both extraction conditions. The separation process was controlled by NMR and GC methods. Based on the GC chromatograms, the individual content of diesel samples before and after LLE processes was demonstrated.

CRediT authorship contribution statement

Ibrahim Mamedov: conceptualization, supervision, validation; Sayad Niftullayeva: investigation, writing – original draft; Yegana Mamedova: investigation, writing – original draft; All authors have discussed the results, read the manuscript and agreed with its content.

Disclosure statement

No potential conflict of interest was reported by the author(s).

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