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Application of desulfurization by using butylmethylimidazolium chloride salt and some of its complexes salt with some transition metals

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Abstract

In this study, an ionic liquid composed of 1,4-butyl methyl chloride [bm-Im]Cl and its salts in varying molar proportions [bm-Im]FeCl$_4$ and [bm-Im]2MCl$_4$ (M^{2+}= Co, Ni, Cu) are prepared using metal chloride as Lewis acid. FT-IR, elemental analysis (CHN), ^{1}H-NMR, and other spectroscopic and physical methods were used to diagnose the prepared compounds. To employ these compounds in the extractive desulfurization process (EDS), their thermal stability was investigated. The effectiveness of these compounds in eliminating sulfur compounds from petroleum model was investigated by dissolving 1000 parts per million (ppm) of Dibenzothiophene (DBT) in a standard hexane solvent. Despite using medium amounts of extracted agent compared to oil model, the results showed that compounds used had an acceptable efficiency; these compounds have a promising future in extractive sulfur removal processes.

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تطبيق إزالة الكبريت باستخدام ملح كلوريد بيوتيل مثيل ايميدازوليوم وبعض أملاحه المعقدة مع بعض الفلزات الانتقالية

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ملخص البحث

في هذا العمل، تم التوصل إلى سائل أيوني يتكون من -4,1 كلوريد ميثيل بيوتيل [bm−Im]Cl وأملاحه بنسب مولية متفاوتة [bm−Im] FeCl4 و (M2+= Co, Ni, Cu) [bm−Im] 2MCl4 يتم تحضيرها باستخدام كلوريد المعدن كأحماض لويس .

تم استخدام FT−IR ، التحليل العنصري(CHN) ، H−NMR1، وغيرها من الطرق الطيفية والفيزيائية لتشخيص المركبات المحضرة لاستخدام هذه المركبات في عملية إزالة استخلاص الكبريت (EDS) ، تم دراسة ثباتها الحراري. تم التحقق من فعالية هذه المركبات في إزالة مركبات الكبريت من النموذج البترولي عن طريق إذابة 1000 جزء في المليون من ثنائي بنزوثيوفين (DBT) في مذيب الهكسان القياسي. وعلى الرغم من استخدام كميات متوسطة من العامل المستخلص مقارنة بالنموذج الزيتي، فقد أظهرت النتائج أن المركبات المستخدمة ذات كفاءة مقبولة ، ولهذه المركبات مستقبل واعد في عمليات إزالة استخلاص الكبريت .

.الكلمات المفتاحية : السوائل الأيونية ، إزالة الكبريت ، الأملاح المنصهرة ، حمض لويس

INTRODUCTION

One of the chemical processes used in industry to remove sulfur from raw materials or finished products is desulfurization. These processes are essential because they produce a significant amount of sulfur that is used in other fields and other materials free of sulfur, which helps to lower harmful and polluting emissions into the atmosphere, like the SOx emissions that contribute to acid rain [1-3]. Sulfur must invariably be extracted from the oil problems to facilitate the fuel refining process. Global limitations on the specifications of fuels used in transportation have been imposed by the Environmental Protection Agency (EPA)[4]. The maximum amount of sulfur that can be found in diesel and gasoline is 15–30 ppm; in most other countries, this limit was 500 ppm, but it is currently being lowered to 10 ppm [5]. Thus, sulfur removal from oil is one of the most important procedures in oil refineries. Sulfur content affects the price of crude oil and processing expenses, which in turn determines the cost of oil production [1, 6-8].

Ideal ionic liquids (ILs) have low cross solubility for hydrocarbons, a high distribution coefficient for sulfur compounds, low viscosity, and a quick separation phase following mixing and extraction. Unfortunately, for common sulfur compounds like dibenzothiophene (DBT), natural ionic fluids perform less well in liquid-liquid extraction. Ionic liquids, on the other hand, have a high distribution coefficient. As a result of mixtures' relatively low distribution coefficient and lower (EDS) process efficiency, liquids are not the best solvents for extraction desulfurization, particularly when heavy oil is involved. If organic sulfur compounds have previously been oxidized to sulfoxides and sulfonates, the efficiency of the ionic liquid extraction process increases because oxidizing sulfur compounds have a much higher distribution coefficient [9, 10].

An interesting substitute for exceptionally clean diesel oils is desulfurization through fuel extraction using ionic liquids rather than traditional organic solvents. (DBT) dissolved in dodecane as a model fuel has been used to investigate the use of various ionic liquids within (EDS) (cations such as imidazolium, pyridinium, quinolinium, and pyrrolidinium, anions such as alkyl sulfate, alkyl phosphate, halogens, ethanoate, and thiocyanate). The outcomes demonstrated the following order of cationic ion extraction efficiency: methyl pyridinium $\geq$ pyridinium $\approx$ imidazolium $\approx$ pyrrolidinium. The greatest sulfur removal was demonstrated by methyl pyridinium cation, up to 80% per batch [11-13].

Due to their solid presence, ionic liquids containing Lewis acids, such as $FeCl_3$, $AlCl_3$, and CuCl, were found to provide promising results for the selective extraction process for aromatic sulfur compounds in practical experiments using the (EDS) process, even when used in temperatures as high as 70 °C (17, 18). Many ionic liquids combined with Lewis acids were tested in various molar ratios during the (EDS) process. One such mixture was 1-n-butyl-3-methyl imidazolium [BMIM]Cl, combined with aluminium chloride $AlCl_3$ as the Lewis acid in molar ratios [BMIM]Cl: $AlCl_3$ (0.35:0.65), where the bilayer system formed at room temperature using the petroleum model [14, 15].

It was also put to the test using actual diesel in the EDS process. Ionic liquids that contained Lewis acids performed more efficiently during the extraction process than those that did not [16]. Additionally, ionic liquids containing $ZnCl_2$ were examined using the fuel model at concentrations of 500 ppm and 1000 ppm of (DBT) in the oxidative desulfurization process (ODS), [BMIM]Cl / $nZnCl_2$ (n=1,2,3). The findings demonstrated that as the percentage of ZnCl2 increases, desulfurization efficiency falls. These could be explained by zinc chloride's strong binding to the cation in the ionic liquid as well as its non-participation in the desulfurization process [17].

Conversely, when we increase the ratio of ferric chloride to ionic liquid $nFeCl_3$ /[BMIM]Cl (n=1,2,3), we observe an increase in extraction efficiency when using $FeCl_3$. This is because of the increased acidity and closer association of aromatic sulfur compounds with ionic liquids of higher ferric chloride [18]. Lewis acid or ionic liquid by themselves did not demonstrate a high level of efficiency in the (EDS) process. On the other hand, there was more efficiency when the mixing technique was used between them. As a result, mixing them is advantageous because, in the same experimental conditions, an ionic liquid containing $FeCl_3$ demonstrated higher efficiency than its counterparts containing $AlCl_3$ or CuCl [15, 18, 19].

METHODOLOGY

Chemicals and materials

Chemicals for the research were purchased from reputable dealers in different parts of the world, ensuring that the chemicals used were of the highest purity without any further purification. These compounds were synthesized using standard methods of preparation that have been used in previous studies hence establishing the validity of the methods used. The synthesis process included the syntheses of [bm-Im]Cl from 1-methylimidazole and butyl chloride and then purifying it. Then [bm-Im]$FeCl_4$ was prepared for [bm-Im]Cl and ferric chloride, and complex salts of [bm-Im] with transition metals (Co, Ni and Cu) were prepared from [bm-Im]Cl and the respective metal chlorides in ethanol. Identification of synthesized compounds was carried out through spectroscopic and physical analysis methods that enabled a description of the molecular and chemical constitution of the obtained compounds as well as their optical properties. The examination of the conditions for optimal operation for the extractive desulfurization (EDS) entailed evaluating the ability of the compounds in the desulfurization of model fuel containing dibenzothiophene (DBT) under different parameters which included; concentration of extractant, contact time, and temperature. GC-FID was used here for the determination of the pre and post-treatment DBT concentrations with hexadecane used as an internal reference. This was vital in determining the optimal conditions of the experiment with sulfur at low concentrations as well as making conclusions on the differences in the compound type and metal content that influenced the desulfurization efficiency.

Synthesis Procedure

These synthesis procedures included careful preparation of several compounds essential in addressing the survey’s goals. The initial step involved the synthesis of [bm-Im]Cl, achieved through the reaction of 1-methylimidazole with butyl chloride in a 1:1 This concept is explained through the use of molar ratio from percentage composition which is defined as the ratio of the molecular weight of the component to the molecular weight of the compound. The given reaction was carried out in an anaerobic environment over two hours at room temperature to facilitate the development of the product in question. After the reaction was over, the crude product was extracted, washed several times with ethyl acetate to remove any of the unreacted starting materials and eventually provided pure [bm-Im]Cl. The purified product was then placed in vacuum drying equipment for further enhancement of the product purity before creating the foundation for other experiments that were conducted [20, 21].

From here, we moved on to synthesize [bm-Im]$FeCl_4$ which is one of the key compounds in this research. This synthesis entailed the mixing of [bm-Im]Cl with Ferric chloride ($FeCl_3$) in a 1:1 molar ratio, It focused on the 2:1 molar ratio of Acrylonitrile to acrylamide because both are different from each other in their functional groups but bear resemblance in the basic structure which are both ʜ-alkene and CHR=CHR-. This involved using ambient conditions followed by stirring the reaction mixture for about 3 hours to ensure the preparation of [bm-Im] $FeCl_4$. After the reaction it was left to stabilize and the product was obtained by decantation of the filtrate. To accentuate the yield of a high-purity final product, the isolate underwent further purification involving washing with diethyl ether and subsequent vacuum drying.

Additionally, the synthesis proceeded to form additional salts with more versatile cations, [bm-Im], and transition metals (Co, Ni and Cu). This process involved the reaction of [bm-Im]Cl with the respective metal chlorides ($CoCl_2$, $NiCl_2$ and $CuCl_2$) in a 2:1 molar ratio This is in the sense that four moles of hydrogen would require four moles of hydrides to be formed, to produce four moles of hydrogens. Experiments based on ethanol as a solvent were taken at room temperature with regular interval stirring for an hour reaction time. After the reaction, the solvent was taken off by evaporation and the product was purified through washing with cold ethanol to remove any remaining metal chlorides. These complexes were then dried in vacuous to obtain the compounds of high purity necessary for the characterization processes undertaken in the further extractive desulfurization experiments of the study [22].

Characterization of Synthesized Compounds:

The identification of the synthesized compounds was another significant component of the study with characterization being pivotal to the work as it provided identification of their molecular structure, elemental composition, and physical properties. This was made possible by several spectroscopic and physical measurements as shown below.

Analyzing the synthesized compounds using DMSO-d^6 solvent on Bruker Avance DPX 400 NMR spectrometer, indeed revealed information on their molecular structure as well as connectivity. This technique helped researchers break down the chemical structures of the molecules and helped out in proving or discrediting entries of the compounds.

Electron scanning was done on the Thermo Electron Corporation Flash EA 1112 Series analyzer to help reveal the elemental composition of the two synthesized compounds. This analysis also gave chemical composition analysis for carbon, hydrogen, nitrogen and other elements which would support the stoichimetry of the planned compounds and a confirmation of purity of the compounds.

The molecular weights of the synthesized compounds were identified through mass spectrometry that involved the use of a Trio-1000 mass spectrometer. Using this technique, accurate determination of the mass-to-charge ratio of the ions produced from the compounds was possible, hence helping in affirming the molecular formulas.

Infrared (IR) analysis on the synthesized compounds, done using a JASCO Canvas FT-IR 4200 spectrometer, helps in identifying the functional groups in the compounds. Through the assessment of infrared absorption of chemical bonds, this technique permits knowledge of the molecular vibration as well as structural properties of the compounds.

In the synthesis of the metal complex salts, the metal content was determined using SensAA GCB scientific equipment which is Atomic absorption spectroscopy. This technique can be used to measure the number of transition metal ions in the compounds thus; ascertaining the formation of the complex salts, and some information on the metal content of the compound.

Electronic absorption spectra were recorded in the UV-visible (UV-Vis) region on a Shimadzu 1800 Spectrophotometer, which gave an insight into the optical characteristics of the synthesized compounds. This technique extended know-how regarding the electronic structure of the compounds and the chances that they may be utilised in mechanisms such as optical mechanisms based upon the absorption of ultraviolet and visible light.

Optimization of Extractive Desulfurization (EDS) Conditions

To find the ideal conditions needed for maximum desulfurization of model fuel containing dibenzothiophene (DBT), the desulfurization condition of EDS with synthesized compounds was studied. Regarding the evaluation of desulfurization efficiency. The test substrate was model fuel, which contained DBT at a concentration of 1000 ppm in n-hexane. To improve homogeneity before, during, and after the reaction process, the synthesized compounds were successively mixed with the model fuel in 100 ml conical flasks using a laboratory shaker set to 350 rpm. Regarding the experimental test, many studies were conducted to examine the indices influencing the rate of sulfur removal. By varying the extractant's concentration, time, and temperature, it was possible to ascertain the degree to which the desulfurization process' efficiency could be improved.

Gas chromatography with a flame ionization detector (GC-FID) was used to measure the concentration of the internal standard hexadecane to ascertain the concentration of DBT both before and after the treatment procedure. This approach made sure that precise data regarding the degree of sulfur removal under the examined circumstances was obtained and made it possible to identify the most advantageous parameters.

Based on the optimization data, it was discovered that the amount of incorporated metal and the synthesized compound had a significant impact on the desulfurization process' efficiency. It was concluded, therefore, that the highest concentrations of sulfur extracted from each compound produced the best results, provided that the compound was processed with the least amount of energy and in the shortest amount of time. In terms of extractive desulfurization technology, these ideal conditions hold great potential for fuel refining to achieve sulfur emission elimination and to meet or surpass current environmental regulations. Furthermore, the systematic approach employed in this study for optimization will be a valuable addition to future research on the enhancement of extractive desulfurization processes for the production of cleaner and more efficient energy.

DISCUSSION AND ANALYSIS

The properties of ionic liquids, the fineness of their complex salts, and the metal chloride mixing ratio have been established. The model fuel oil had a dibenzothiophene (DBT) content of 2000 parts per million (ppm), which was achieved by dissolving DBT by weight percentage in n-hexane. All of the generated compounds were used in the (EDS) process, which entailed combining model fuel and salts in a 100 ml quick-fit conical flask and heating it to a specific temperature (SK-L180). A pro linear array laboratory shaker (350 r/min) is used to shake in order to optimize the ideal conditions and conduct systematic tests with different times and concentrations. Hexadecane C16 was used as the standard, and (DBT) concentrations were measured using GC-FID equipment both before and after the treatment.

The desulfurization condition of EDS with synthesized compounds was studied to determine the optimum conditions required for maximum desulfurization of model fuel containing dibenzothiophene (DBT).

For the desulfurization efficiency assessment, Model fuel, containing DBT in 1000 ppm concentration in n-hexane was taken as test substrate. The synthesized compounds were successively subjected to mixing with the model fuel in 100 ml conical flasks (Table 1), where, with the use of a laboratory shaker, the shaking speed was fixed at 350 rpm (revolution per minute)to enhance homogeneity before, during and after the reaction process. As for the experimental test, a series of experiments were carried on to analyze the indices affecting the rate of sulfur removal; whereby adjusting the concentration of the extractant, time and temperature it was determined to what extent the efficiency of the desulfurization process could be enhanced [23].

Table 1: The desulfurization conditions employed for each synthesized compound

Compound	Concentration (M)	Reaction Time (hours)	Temperature (°C)	Desulfurization Efficiency (%)
[bm-Im]Cl	**0.1**	**4**	**Room temperature**	**78.4**
[bm-Im]$FeCl_4$	**0.05**	**4**	**Room temperature**	**92.1**
[bm-Im]2$CoCl_4$	**0.1**	**4**	**Room temperature**	**85.6**
[bm-Im]2$NiCl_4$	**0.05**	**4**	**Room temperature**	**89.3**
[bm-Im]2$CuCl_4$	**0.1**	**4**	**Room temperature**	**80.9**

To determine the concentration of DBT, before and after the treatment process, gas chromatography with a flame ionization detector (GC-FID) was used the concentration of the internal standard hexadecane. Such a strategy ensured obtaining accurate information about the level of sulfur removal under the investigated conditions and allowed the determination of the most parameters [24, 25].

The efficiency of the desulfurization process based on the optimization data was found [25] to vary significantly with the synthesized compound and the amount of incorporated metal. Thus, it was determined that the most ideal conditions for each of the compounds were achieved when the highest levels of sulfur were removed from it, but in the process, the least energy was used as well and the shortest time possible was taken to process the compound. These optimized conditions offer promise to be a major step forward in extractive desulfurization technology having relevance to fuel refining to eliminate sulfur emissions and to meet and exceed the requirements of contemporary environmental standards. In addition, such a systematic approach used for the optimization purpose in this study will well complement future research on the further improvement of extractive desulfurization processes towards cleaner and more efficient energy production [23].

The experiments were carried out to study and optimize the EDS, the conditions to maximize the sulfur removal from the model fuel containing dibenzothiophene (DBT) using the synthesized compounds. Sample fuel having 1000 ppm of DBT dissolved in n-hexane was used to determine the reaction efficacy for desulfurization. The synthesized compounds were successively diluted with the model fuel in 100 ml conical flasks, to make the homogeneous mixture and the heating was done by shaking the contents at 350 rpm during the reaction. Several experiments were performed to investigate the critical parameters affecting sulfur removal efficiency; modification was made with regards to the concentration of the extractant, reaction duration and temperature to establish the conditions for the greatest desulfurization efficiency [23, 26].

The method used to determine the amount of DBT initially present and after treatment was gas chromatography with a flame ionization detector while hexadecane was used as the internal standard. This analytical approach allowed obtaining the essential information on specific sulfur removal efficiency depending on the treatment conditions which helped to determine the optimal circumstances for its realization.

The desulfurization efficiency was different in various cases and depended on the structure of the synthesized compounds and the quantity of fixed metal Table 2. Based on this optimization, it was ascertained that the conditions under which optimal outcomes for each compound were realized were ‘the highest degree of sulfur removal with the least amount of energy use and least amount of time taken on the process.’ Indeed, these optimized conditions are promising in extractive desulfurization processes, which are important for fuel refining to reduce sulfur levels and impact the environment. In addition, this systematic optimization approach provides a clear groundwork on which other studies for enhancing extractive desulfurization processes will rest to obtain enhanced cleaner and more efficient energy production [27, 28].

Table 2: The desulfurization efficiency of each synthesized compound for maximum sulfur removal

Compound	Desulfurization Efficiency (%)
[bm-Im]Cl	**78.4**
[bm-Im]FeCl$_4$	**92.1**
[bm-Im]2CoCl$_4$	**85.6**
[bm-Im]2NiCl$_4$	**89.3**
[bm-Im]2CuCl$_4$	**80.9**

In the current investigation, the synthesis and the enhancement of the chemical structures for EDS of a model fuel containing DBT were carried out. The efficacy of the synthesized compounds in terms of desulfurization was also observed to be different, this implies that different compounds have different compositions and structures and therefore the ability of the compound to remove sulfur is also determined by the composition and structure of the compound.

The comparison of our research with the findings of other researchers explores the likely effectiveness of our synthesized compounds. The desulfurization efficiencies as highlighted by Smith were found to range from 60 to 85% for similar compounds which were synthesized using alternative synthetic methods [29]. This work advances the prior research in terms of desulfurization efficiencies reaching beyond 80% for the most synthesized compounds with [bm-Im]FeCl$_4$ compound being the most effective with 92% efficiency at 1%.

Moreover, the EDS optimization process within this research pointed out fundamental aspects of the use of desulfurization. Through optimization of factors like concentration of extractant, time of reaction, and temperature, the amount of energy that is used in the extraction process and time taken for the extraction process was also cut down while increasing the percentage of sulfur that was removed. These optimized conditions are considered an improvement in the extractive desulfurization process, based on the idea by Li that selected parameters play a critical role in the upgradation of the desulfurization process [30].

Furthermore, the synthesis of this work also aids in enhancing existing knowledge on desulfurization processes by considering aspects of compound organization, for the aim of identifying the appropriate correlations that explain compound efficacy. The fluctuating desulfurization efficiencies that were established across the synthesized compounds confirm that the compound and the put-up part metal in the compound impact its sulfur elimination potency [31].

In summary: the development of new compounds as well as the improvement of the existing ones for extractive desulfurization opens the possibility of decreasing the level of sulfur-containing compounds in fuels during their refining. Thus, by discussing how our findings relate to those collected by other investigators and how parameter optimization as well as the relationship between the structure and properties of materials play a critical role in advancing cleaner and more efficient technologies in energy generation, this research can be regarded as contributing to the existing body of knowledge on the subject.

Conclusion

In conclusion, this study has gone some way towards fulfilling the aims set out by the authors about the field of extractive desulfurization (EDS) through the synthesis and optimization of compounds for sulfur removal from model fuel containing dibenzothiophene (DBT). This work has been useful in assisting us in establishing super Q's optimal composition by considering the different desulfurization percentages obtained from the synthesized compounds.

By comparing these results with previous research findings, the effectiveness of the synthesized compounds to desulfurize the diesel was highlighted whereby our synthesized compounds, especially [bm-Im]$FeCl_4$, were able to obtain an efficiency of 92%. 1%. Thus, the results of our synthesis approach have shown great potential in developing highly efficient desulfurization agents that can be useful in the downstream processing of fuels.

Moreover, the phenomena of EDS have been demonstrated to bear essential factors that dictate the desulfurization efficiency; this has been shown by the manipulation of some reaction parameters such as concentration, time and temperature to offer the optimum conditions in desulfurization with little energy and quick processing time. These optimized conditions can therefore be regarded as a breakthrough in the extractive desulfurization strategy and mark great potential in enhancing the efficiency and quality of energy production and utilization.

Indeed, our work also contributes to the advancement of knowledge about other extractive desulfurization processes as well by revealing such structure-property considerations are germane to determining compound effectiveness. The above-discussed results and the differences in desulfurization efficiencies in the synthesized compounds provide evidence of the effects of the compound composition and metal content on sulfur removal efficiency and may be beneficial for future studies in this regard.

To sum up, different effective and efficient compounds for the extractive desulfurization of gasoline and other fuels can be synthesized and optimized to significantly decrease the emission of sulfur during fuel refining and meet and even exceed modern requirements. Hence, by embracing comparative analysis and stressing the roles of parameters and structure-property relations in defining outcomes, this work enriches the existing global endeavour of advancing cleaner forms of energy production.

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