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ISSN: 2488-9229
FEDERAL UNIVERSITY
GUSAU-NIGERIA**INTERNATIONAL JOURNAL OF SCIENCE FOR GLOBAL SUSTAINABILITY****A Review on Desulfurization of Kerosene using Ionic Liquids as Catalysts****Alhassan Yahaya and Mubarak Abubakar**

Department of Chemistry, Federal University Gusau, P.M.B. 1001, Gusau-Nigeria

Corresponding Author's Email & Phone: y.alhassan@fugusau.edu.ng, +2349061738187**Received on:** April, 2022**Revised and Accepted on:** June, 2022**Published on:** July 2022**ABSTRACT**

Kerosene, a very common household cooking fuel in Nigeria also has a number of secondary industry applications. It is a popular industrial and domestic energy source, due to its significant energy output. The major industrial applications of kerosene are in production of insecticides, pesticides and aviation fuels. However, issues associated with sulfur content limits its energy density and other industrial applications. Desulfurization of Kerosene is mainly targeting the reduction of sulfur related compounds. Effective, sufficient and develop sulfur removal technologies have been established. Acids and bases constitute the greater part of the catalysis in desulfurization, while, activated carbon is used in traditional desulfurization process, although, these technologies are not necessarily environmentally friendly. The use of Ionic Liquids (ILs) as green chemicals in sulfur removal has not been properly documented. Recently, the use of ILs in desulfurization is attracting the attention of research community. ILs are employed as catalysts and co-solvents due to their excellent acidity derived from inter-molecular bonding interactions. Therefore, ILs exhibit the tendency toward eliminating reactor corrosion; a major reaction engineering problem that has been posing serious challenges in desulfurization technologies. This research work, is aimed at reviewing desulfurization techniques and the use of ILs as green and environmentally friendly catalysts in desulfurization. Literature pool is provided with analyses of the potentials of ILs as emerging catalysts and their challenges discussed as well.

Keywords: Emission; extraction; ionic; liquids; kinetics and sulfur.**1.0 INTRODUCTION**

Kerosene, is an important lighting source in rural African and other parts of the World where electricity supply is inadequate. Because it is a carbon-rich fuel, and with high energy density, energy released from its complete combustion is enough to power many automotive engines, especially jet engines. Kerosene, is a distillation product in petroleum refining, obtainable within distillation temperatures between 150 °C and 275 °C, and largely consists of carbon chain ranging from six – to – sixteen atoms (Azam, 2019). The final Kerosene product from the distillate is formed after completing series of reactions proceeding either simultaneously or independently.

Catalytic reforming, alkylation, catalytic cracking and hydro-processing are significant reaction pathways in Kerosene production and refining (Opara, 2013). The resultant product constitute an array of compounds and their derivatives like straight alkanes, aromatics, alkyl benzene, and

naphthalene (Liqiong, 2019), with substantial concentration of sulfur and sulfur containing-compounds. Pyrolysis oil also contained significant amount of sulfur that is much higher than the recommended permissible limit for engine operation (Omidghane, 2020), suggesting sulfur removal is required during its upgrade to fuel. Traces of sulfur was detected in bye-product of drop-in fuels derived from waste fats, as well. Emission from sulfur containing fuels is a major source of environmental pollution. Sulfur oxides (Sulfur dioxide and sulfur trioxide), are important ingredients in the formation of acid rain, in the presence of air, smog and ozone. Not only as an environmental pollutant, sulfur, present in fuels, constitute both mechanical and, operational challenges in engines (Waqas, 2017). To attain low sulfur emission in fuels and, to improve the combustion properties; effective sulfur removing technologies are incorporated into petroleum refining process. Other than the aforementioned, additional forms of aromatic sulfur including

benzothiophene and its derivatives, alkyl and ring substituents, aromatic disulphide, thiolane, thiophene and its derivatives, and mercaptans. While traces of inorganic forms of sulfur like dissolved pyrites, elemental sulfur, and carbonyl sulphide among others, exist in Kerosene.

Traditional desulfurization techniques including; deep oxidative desulfurization (Azam, 2019), using low-cost adsorbent (Liqiong, 2019), extractive desulfurization (Swapnil, 2015), hydro-desulfurization, ultra deep desulfurization (Gaurav, 2015), and oxidative desulfurization (Mahdiah, 2017). These Sulfur removing (desulfurization) processes are significantly established in the pool of literature.

Nevertheless, there has been increasing demand on the use of “greener solvents” such as CO₂ based solvents, bio-ionic liquids, liquid polymers and, bio deep eutectic solvents, as suitable replacement to harmful and, toxic inorganic solvents obtained from fossil fuels. This recent quest for green solvents and technologies for production of fuels and fuels related products is been intensively investigated in desulfurization science and engineering.

Evolving desulfurization techniques reported include; hydrodynamic cavity assisted desulfurization (Soroush, 2020) and, Bio-desulfurization (João, 2020). Also, process intensification desulfurization techniques proved to be highly effective, economically viable and environmentally friendly. This is achieved using combine oxidation by ILs as co-solvents (Shurong, 2018) and, the use of ILs as catalyst (Kulkarni, 2010).

The focus of this review, is to explore the versatility of ILs in desulfurization of Kerosene as catalyst. In so doing, literature trend in desulfurization is revisited with a view to understanding the mechanism of reaction, synthesis and application of this generation catalysts are critically examined. Literature on different desulfurization techniques and, applications of ILs are compiled.

2.0 KEROSENE DESULFURIZATION TECHNOLOGIES

2.1 Hydrodesulfurization (HDS)

Hydrodesulfurization (HDS) is one of the largely available and commercially viable desulfurization processes in many refineries. However, indications emerged that, HDS is inefficient in removing compounds like DBT and, its derivatives, owing to its aromatic sulfur-containing compound, but effective in non-aromatic sulfur-containing compounds like thiols, disulfides and sulphides (Mahdiah, 2017). High operation conditions like temperature and pressure are of the major disadvantages of HDS, as such, not all catalysts can withstand conditions in HDS, due to catalyst's instability, reduced efficiency and distorted selectivity. HDS operating temperature could be as high as 300 °C to 400 °C and pressure ranging from 3 – 6 MPa. Desulfurization under such severe reaction conditions, deactivates many catalysts and denatures its composition. These stringent reaction conditions add up to the production cost and risk of operation on a large scale (Swapnil, 2015).

In general, the most acceptable standard methods of calculating the efficiency of sulfur removal are by ASTM D-4294. In modern-day laboratories, sulfur analyzer is employed for direct evaluation. Most sulfur analyzers are built on the principle of energy dispersive X-ray fluorescence technique.

2.2 Oxidative Desulfurization (ODS)

Oxidative Desulfurization, is probably one of the most industrially viable desulfurization technologies. The mechanism is proposed to proceed by simple sulfur oxidation into corresponding sulfoxides and sulfone and the products are removed via extraction or adsorption mechanism (Mahdiah, 2017).

Typically, an oxidizing agent is required to facilitate the reaction. In this method, operation conditions are relatively mild, allowing effective catalytic activity, eliminate product destruction, and enhance recovery (Mahdiah, 2017). These bye-products (sulfones and sulfoxides) are further removed from the solvent matrix by simple liquid-liquid extraction, or by-adsorption. A preferential extraction is achieved by polar solvents. Prominent oxidants include K₂FeO₄,

NO_2 , H_2O_2 , O_2 and O_3 and organic peroxides are employed (Soroush, 2020).

An example of sulfone compounds in fuels belong to the S1 group, like alkyl derived benzothiophene or the benzo-homolog with two cyclic Sulphides (Jiyuan, 2021). The principle ODS reaction pathway is proposed by different research groups.

In this advance desulfurization process, a wide range of catalysis could be employed without compromises to the yield and quality of the product, including ILs as catalysts and co-solvents. The later, although showed promising desulfurization efficiency, but there are unresolved challenges associated with its high viscosity, which; lowers the mass transfer ratio, increases reaction time and consequently upturn the production economics (Soroush, 2020) (Pengyuan, 2020).

An advance form of ODS is the hydrodynamic cavitation ODS technology. This is an improved sulfur removal either by converting sulfur compounds into corresponding sulfoxides and sulfones radicals that are much suitable for attack by formyl and hydroperoxyl species, or by generating hydroperoxyl and hydroxyl species via water and hydrogen peroxide pyrolysis (Soroush, 2020).

Catalysis in this area reported recently presented novel synthesis of carboxyl functionalized imidazolium Ionic Liquids (ILs) as a linker. In this task-specific catalytic design (Qi, 2022). This research group prepared a carboxyl functionalized ILs to form a composite with improved selectivity using phosphor-tungstic acid and Zirconium ion. Result indicated significant extractive and selective oxidative desulfurization of DBT from fuel.

A two-way reaction mechanism developed by both experimental and density functional theory computation proposed. A superoxide radical formation in the first step, and proceeded by formation of peroxotungstate, with both showing high oxidizability and stability as shown in **Fig. 1**.

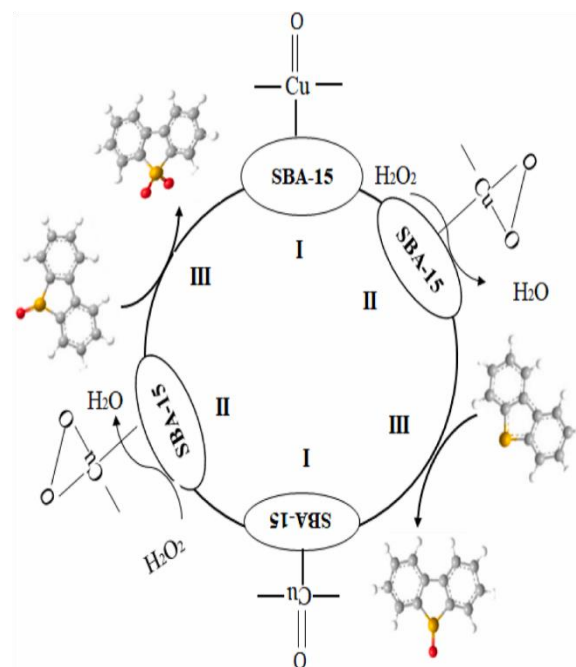


Fig. 1: Improved Ionic Liquids Linker Composite
(Adopted from Qi et al., 2022)

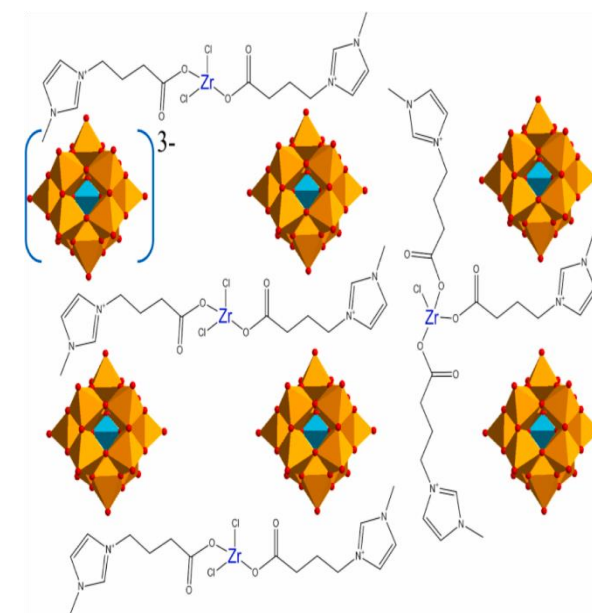


Fig. 2: Proposed Desulfurization Reaction Mechanism for Cu-based Catalyst
(Adopted from Wang et al., 2022)

Furthermore, to demonstrate the flexibility of catalysts design towards improving selectivity and to impact specific need ODS, a hierarchical

self-assembly 2D-channel silica catalysts was reported (Feng, 2022).

This research group explored the possibility of grafting amino group and successful immobilization of phosphotungstic acid. Authors attributed the performance to the improved molecular diffusion, with varying removal efficiency in the order; DBT > 4,6-DMDBT > BT > n-DDM (Moslem, 2022). This trend was supported by a similar research group reported by claiming sulfur removal decreased in the order: DBT > 4,6-DMDBT > BT (Ahmadian, 2022). In the later research, two mesoporous silica based materials were synthesized and characterized, then, employed in desulfurization using H₂O₂ as oxidant, while acetonitrile was extracted; achieving 99.9% DBT removal.

Highest selectivity was observed for Cu impregnated Mordenite, since it had high acidic sites, chemical and thermal stability and, Si/Al ratio among other stated advantages (Moslem, 2022).

Similarly, Wang et al. (2022), reported the synthesis of hierarchical mesoporous-silica Mo oxides catalyst through impregnation. The authors emphasized the outstanding superior performance of the treated catalysts in effective desulfurization even at room temperature and showed excellent turnover frequency. Other advantages of the mesoporous material were its increased activity, low cost, and selectivity.

2.3 Extractive Desulfurization (EDS)

Deep Extractive Desulfurization (EDS) is gaining scientific attention due to its ambient operating temperature, pressure and need no catalyst like hydrogen without prejudice to the fuel quality, stability, non-toxicity and reduced environmental impact (Swapnil, 2015).

In extractive desulfurization, solubility play significant roles in dissolving the sulfur compounds into solvent matrix. Important solvent properties were predicted using Conductor-like Screening Model for Real Solvents (COSMO-RS) using experimental data. Such evaluation from quantum-chemical calculation allows visualizing thermodynamic

properties and solvent behavior especially intermolecular contribution of enthalpy.

Extractive desulfurization conducted in a micro-channel contactor was conducted using deep eutectic solvents (DESs) (Ahmad, 2022). In this research, eight difference DES were synthesized from polyethylene glycol and propionic acid yielded remarkable sulfur removal of between 62.16% and 78%. Properties of the reaction studied including volumetric mass transfer, interfacial area and slug length showed promising advantages in intensification of extractive desulfurization process.

In this reported intensification, using micro-channel, solvent volume is reduced significantly. It was highlighted that there was the need to investigate the geometric parameters of micro-channel on the efficiency of desulfurization. There has been reported correlation between DES mixing ratio and its corresponding sulfur removal.

Interestingly, DES performance in desulfurization is aided by the technique employed. Three different desulfurization techniques (ODS, EDS and ultrasound) were used in heavy oil desulfurization using Choline chloride based DES. Report showed highest sulfur removal for Ultrasound (62%), in contrast to EDS (26.78%) and ODS (37.28%) respectively (Mohammed, 2022).

Choline based DES are termed as one of the promising bio-ILs with interestingly unique properties with knee interest in their bio-degradability, bio-compatibility, and limited toxicity as compared to other ILs (Rubén, 2022). According to Gomes (2019), Choline based materials are more bio-degradable, bio-compactable, and showed reduced toxicity, either in their natural and, synthetic forms, even when they exist in ILs and eutectic solvents.

This is unarguably true, since both the anions and cations use in the synthesis of bio-ILs are obtained from biocompatible materials like protein-derived amino acids, glycosides-derived and carboxylic acid-derived among others natural sources of bio-based chemicals.

Recently, Mofidi *et al.* (2022), proposed the mechanism of DES extraction in ultrasound assisted desulfurization. Accordingly, similarity-compatibility theory explanations, reaction starts with extraction of oil into DES matrix, which upon addition of the oxidant, forces the sulfur components dissolve into the acidic component, thus, reducing the oil, making all the remaining sulfur compounds dissolved in the aqueous phase. The properties have either physical or chemical dimension.

Generally, choosing suitable solvent is key in development of EDS, as much as its price, volatility, safety and impact on the environment.

2.4 Adsorptive Desulfurization (ADS)

This technique is based on availability of adsorbing surface where residual sulfur it bounded. Activated carbon provides this needed surface adsorption. Excellent properties of activated carbon like, oxygen-rich specific surface area and, advanced porosity are suitable features allowing selective catalysts impregnation and, adsorption via carbon fibres (Syed, 2016). In the case of reactive adsorption, a stable chemical complex is formed from the reaction of π -bond present in the aromatic sulfur compound with adsorbent, stronger than van der Waals attraction holding sulfur (Waqas, 2017).

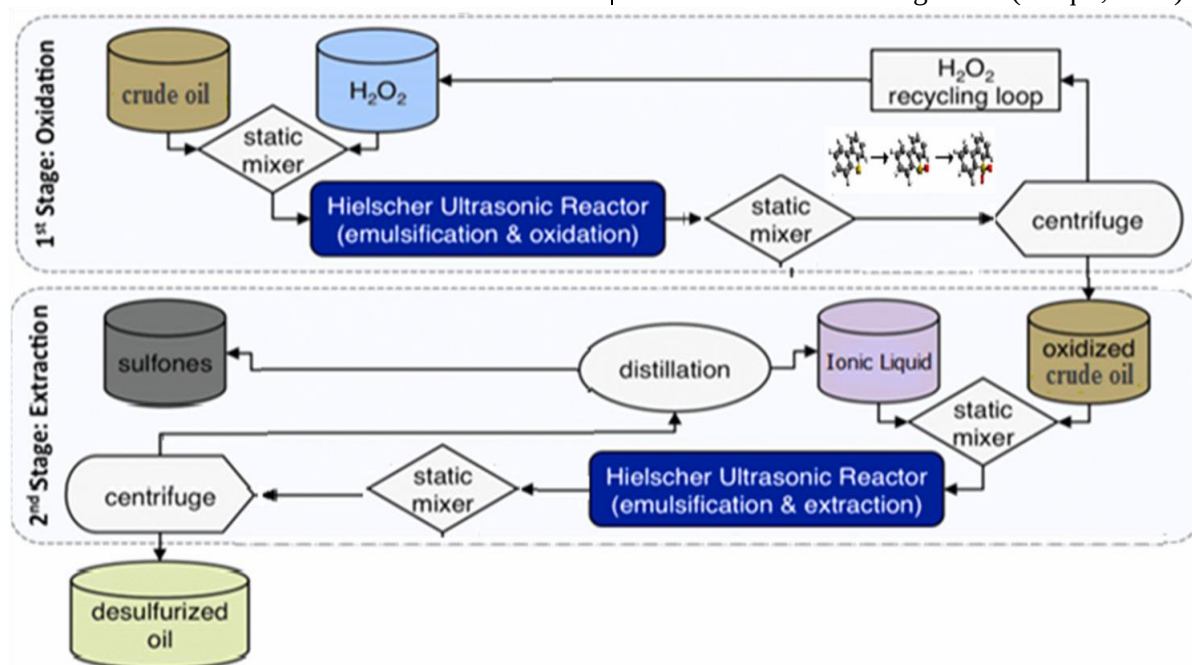


Fig. 3: Two-step Ultrasound Assisted Oxidative Desulfurization flow chart (Adopted from (Mohammed, 2022)).

Excellent refractive index, high stability, low toxicity, and optical as well as electronic properties are some of the unique features of these catalysts, making them suitable in application and synthesis. This is in addition to their already established particle associated properties, specifically surface area, porosity, crystal lattice and morphology (Ismail, 2016). The mechanism of adsorptive desulfurization is believed to be either via a physical interaction or reactive adsorption between the removing sulfur and the adsorbent, where sulfur is bonded on the surface of the adsorbent (Waqas, 2017).

2.5. Photo-catalytic Desulfurization (PCDS)

Photo-catalytic desulfurization is no doubt one of the emerging techniques gaining research attention. Mohamed and Ismail (2022), conducted the thiopene photocatalytic oxidative desulfurization in a photoreactor with a constant supply of O_2 . In their report, authors synthesized a mesoporous D-Ag₂O/ZrO₂ hetero-structure by a facile sol-gel under visible-light at room temperature. Findings revealed a promoted activity of photo-catalysts, which is attributed to the light scattering, synergistic effect, and rapid radical formation as factors responsible for such pronounced reactivity.

This new and promising desulfurization technique is found suitable for removal of ultra-low sulfur present in fuels.

Advantages of this technique including the use of visible light source as a cheap and eco-friendly technique. Other advantages include high selectivity, reusability of photo-catalysts, absence of air, and flexible catalyst design (Yingying, 2022). The authors concluded that, using Cu and Ni promoted electron transfer, resulting in improved surface permeability, reaction area, and low density.

2.6. Bio-sulfur desulfurization (BDS)

It was earlier highlighted in this paper, not all sulfur compounds are suitable for HDS and ODS, Dibenzothiophene (an organo-sulfur compound) and its corresponding derivatives, for example, are highly resistant to many desulfurization techniques. Thus, the selective desulfurization of this class of sulfur containing compound requires careful removal. Bio-sulfur desulfurization, employs enzymatic degradation pathways.

A proposed reaction mechanism suggested that, reaction proceeds by protonation, electrophilic aromatic substitution, thiolate formation and, sulfur released in the form of HSO_3^- (João, 2020). But predominant reaction mechanism for sulfur removal by these enzymes was proposed to be an electrophilic aromatic substitution (Geronimo, 2017). Although this had proven to be effective at laboratory scale, the fear of enzyme contamination, slow reaction rate and cost are challenges needed to be addressed.

Some of the excellent desulfurization enzyme that demonstrated efficiency are 2'-hydroxybiphenyl-2-sulfinate desulfinase (Geronimo, 2017) and, *Rhodococcus erythropolis* strain (Nakayama, 2002) (Davoodi-Dehaghani, 2010).

2.7. Ultrasound Assisted Oxidative Desulfurization (UAOD)

Recently, ultrasound assisted oxidative desulfurization of Arabian extra light oil showed sulfur removal of 55.1% was achieved (Jiyuan, 2021). According to this research group, sulfur compounds identification conducted using molecular characterization highlighted the presence of S_1 , S_2 , O_4S_2 and O_2S_1 species, with

the later showing great abundance and characterized by the presence of carbon number 11-20 and greater than 20 but less than 40 were observed. Major advantages of Ultrasound assisted desulfurization is that, it improves catalytic performance, promotes mass transfer and produces more radical ion formation.

The investigation into reaction kinetics suggested UAODS proceeds by pseudo-first order reaction pathway, thus, increasing reaction temperature has a consequential increase in rate of reaction (Jiyuan, 2021) (Moslem, 2022). For example, because EDS is exothermic, increasing temperature deters the extraction efficiency.

Similarly, a mixture of activated charcoal and wood of *Prosopis juliflora* tree was modified by ILs (troctylmethylammonium chloride) and used in ultrasound assisted adsorptive desulfurization of model fuel in the presence of n-hexane as solvent (Christina, 2022). Catalysts modification formed mesoporous material with improved specific area, resulted in up to 99% efficiency but reaction mechanism obeyed pseudo-second order kinetics in contrast to the usual first order. This claim was also supported by Luo *et al.* (2022), proposing a complete chemisorption or chemisorption via valence forces sharing or exchange electrons between removing sulfur and the adsorbent, at the rate determining step of the desulfurization reaction.

One of the major advantages of ultrasound assisted oxidative desulfurization is its ability to accommodate different solvents. In achieving effective mass transfer rates during ultrasonic desulfurization, there is a collapse of cavitation bubble and repeated oscillations resulting from intensive micro-mixing. Another advantage is the system is reducing reaction time. Reaction time is low when employing ultrasound system since there is improvement in the interfacial mass transfer between the participating phase components (Mohammed, 2022).

3.0 CATALYSIS IN KEROSENE DESULFURIZATION

Like any other chemical syntheses and processes, Kerosene desulfurization process requires using catalysts at different stages of sulfur removal. Different methods are employed in using

catalysts in desulfurization. For examples, in the case of physical adsorption (impregnation method), desired properties of liquid or ILs are impacted on solid surface serving as binder.

On one hand, the advantages of this method include; ease of separation, reusability, and improved reaction surface area. On the other hand, the major disadvantage is leaching of active catalyst constituents during the impregnation process (Mahdieh, 2017).

Immobilization is another promising method different from impregnation (Pengyuan, 2020). This process involves supporting desired property on a binder that is a porous solid material. For example, an interesting POM immobilization on solid material induced an encapsulated-POM properties in the final matrix.

Interestingly, ILs are not only promising desulfurization catalysts, but equally effective, versatile and tunable. One of its versatility is in its ability to be used as supported on heterogeneous catalysts, by sol-gel method (Mahdieh, 2017), silica gel adsorption (Liqiong, 2019) and so on.

Results of ILs incorporation into silica-gel matrix, suggested increasing surface area, producing effective sulfur desulfurization up to 99.1% (Mahdieh, 2017).

Suitable oxidation reaction catalysts in ODS included Polyoxometalates (POM). Attractive features of these catalysts consisted of their excellent catalytic ability, abridged size, super-selectivity to oxidation of organic sulfur compound like of S_1 , S_2 , O_4S_2 and O_2S_1 species, present as bonded compounds, and cost as major advantages. Their potentials are restricted by poor recyclability and separation difficulty during and aftermath of reaction (Pengyuan, 2020).

In the case of fats, sulfur compounds are converted into hydrosoluble sulfone that are suitable for removal in polar solvents. Selectivity and distribution ratio are two important parameters in liquid-liquid extraction, and both are related to activity coefficients. The former, is found to be inversely proportional to partition coefficient, such that, the higher the partition

coefficient, the lower the selectivity and vice-versa (Canales, 2016).

Precious metals oxide catalysts provide the needed selectivity and specialty towards sulfur removal. Prominent metal oxides of PbO_2 and MnO_2 and metal halide got significant literature recognition.

Effective sulfur removal by these metal oxide and metal halides catalysts is achieved using reactive adsorbent desulfurization. It employs the chemical impregnation of desired special metal on an adsorbent material to ensure effective separation of catalyst, its regeneration, recycle and reuse during refining process.

Nanoporous carbon adsorptive desulfurization of dibenzothiophene was conducted and the selectivity of active sites was identified and investigated (Seredych, 2010). Result highlighting the correlation between effective adsorption and number of active sites, while selectivity is attributed to the concentration of the adsorbent material.

Choosing a suitable adsorbent is critical to this process, as different adsorbents show varying performances. Depending on the adsorbent selectivity and adsorption capacity, Copper-based adsorbents, for example, exhibited excellent selectivity owing to the formation of a coporous-thiophenic π -complexes in a fuel mixture.

A similar observation was reported by Fazle *et al.*, (2022) using ZSM-5 mesoporous silica Cu constructed catalyst for batch and continuous adsorptive desulfurization of thiophene. The research indicated the MMZ materials were highly selective when compared to ZSM-5, resulting from improved diffusion pathway and enlarged surface area (Swapnil, 2015).

Using ILs catalysts in EDS, especially imidazolium based ILs had higher Nerst partition coefficient, a key industrial parameter in determining extraction efficiency of catalysts. A major drawback in the potentials of ILs in desulfurization is their high prize, although recent discovery of non-halogenated ILs proved to be

inexpensive but equally effective in reaction catalysis (Katarzyna, 2021).

The ionic size and structure of cations and anions involved in complex compound formation and are strong indicator of extractive desulfurization performance. It is generally agreed that smaller anions showed excellent adsorption behavior, since upon increasing non-polar alkyl chain of carboxylic anions lowers its Henry's law constant value, resulting in distinctively lower solubility, much higher than corresponding amino acids or phosphate based anions (Moreno, 2018).

Increasing operating temperature not below 45 °C lower the ILs viscosity thereby improving its flexibility, and subsequently increase sulfur removal. Presence of some hetero-atoms, and steric effect of alkyl group in the aromatic ring, and S-containing compounds with varying S-atom electron density in fuels like diesel and kerosene are factors limiting sulfur removal of ILs (Soroush, 2020).

Although sulfur removal during petroleum refining is achieved, traces are reported in its products like kerosene, and diesel. Effective desulfurization of Kerosene and Diesel was achieved via selective reactive adsorption using montmorillonite clay impregnated with Cr, Ni, Co, Mn, Pb, Zn, Ag and Fe. It was concluded that the locally available clay was suitable for adsorptive sulfur desulfurization with increases adsorption owing to metals impregnation, and subsequent improvement in catalyst properties including surface area, pore size and volume, especially with Zn (Waqas, 2017).

4.0 CONCLUSION

Industrial application of Kerosene is limited by the presence organic and inorganic sulfur compounds and its derivatives. Effective removal of these sulfur compounds by desulfurization techniques is achieved when using suitable catalysts. Literature surveyed showed improved catalysis engineering and use of green catalysts. Ionic liquids are effective, suitable and efficient in desulfurization. Review of the reaction kinetics indicated a pseudo-first order. It is concluded that

careful selection of anions and cations could ensure task specific ILs are formed.

ACKNOWLEDGEMENT

Authors acknowledged the funding of this research through the Institutional Based Research (IBR) of Federal University Gusau, Nigeria, funded by Tertiary Education Trust Fund TETF/DR&D/CE?UNIV/GUSAU/IBR/2020/VOL.1

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