

A CRITICAL REVIEW OF GREEN SYNTHESIS APPROACHES TO BIODIESEL PRODUCTION USING IONIC LIQUIDS

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ABSTRACT

The increment in the consumption of fossil fuel led researchers to focus on alternative sources like biodiesel. Biodiesel is acknowledged as encouraging substitute for petroleum, diesel because of its renewable, ecofriendly, better lubricating and sustainable properties. Ionic liquids, recognized for their unique properties, have emerged as promising catalysts in this process. They serve as effective catalysts by facilitating the transesterification of triglycerides derived from renewable feedstocks. Ionic liquids are called “new generation catalysts” because they increase the yield of process many times as compared to conventional catalysts, also ionic liquids can be recycled easily which leads to economic feasibility and reduced environmental impact of biodiesel process. The versatility of ionic liquids extends to their compatibility with different renewable feedstocks such as waste cooking oil, soyabean oil, jatropha oil, coconut oil etc. Their ability to dissolve both polar and non-polar compounds enhances reaction kinetics, resulting in higher conversion rates and increased biodiesel yield. The goal is to synthesize maximum yield of biodiesel using ionic liquids to contribute in an ecofriendly environment. This paper emphasizes their catalytic efficiency, recyclability, and selectivity. This review article explores the field of biodiesel production from waste cooking oil using ionic liquids as catalyst and focuses on recent studies and advancements on ionic liquids.

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LIST OF ABBREVIATIONS AND SYMBOLS

GHG - Greenhouse gases

NaOH – Sodium hydroxide

CH₃COOC₂H₅ – Ethyl ethanoate or ethyl acetate.

C₂H₅OH - Ethanol

FAME - fatty acid methyl esters

[BMIM][OH] - 1-butyl-3-methylimidazolium hydroxide

ILs - Ionic liquids

ChOMe - Choline methoxide

ChIm - choline imidazole

ChOH - choline hydroxide

FeCl₃ – Iron(III) chloride

[p-TSA]⁻ - p-Toluene sulfonic acid

[BMIM][HSO₄] – 1-Butyl-3-methylimidazolium hydrogen sulphate

CO₂ - Carbon dioxide

[BMIM] [PF₆] – 1-Butyl-3-methylimidazolium hexafluorophosphate

LCST - Lower critical solution temperature

[BMIM][OAc] – 1-Butyl-3-methylimidazolium acetate

[Hnmm]OH - 1-Butyl-3-methyl morpholine hydroxide

FFA – Free fatty acid

KOH – Potassium hydroxide

CHAPTER-1

INTRODUCTION

1. Introduction

The need to explore new energy resources has arisen due to the increase in energy consumption and dependence on fossil fuels. The excessive use of fossil fuels is the main cause of environmental problems, such as air pollution, the greenhouse effect, and global warming (Ong et al., 2021). Recent reports indicate that 87% of global CO₂ discharge result from the ignition of fossil fuels, with oil contributing 36%, coal accounting for 43%, and natural gas making up 20%. Reports also state that one third of global energy, with half related to oil, has been consumed by the transport system. The global population's continuous growth is projected to result in a 40% rise in fossil fuel consumption by 2040-2050 (Maliha et al., 2023).

According to the 2014 United Nations Environment Emission Gap reports, it is projected that the automotive industry will generate approximately 87 gigatons of greenhouse gases (GHG) by the year 2050 (Sharma et al.,2023). However, the introduction of biodiesel has resulted in a significant reduction in the emission of harmful greenhouse gases (Ullah et al et.,2015). As per the International Energy Agency, the global biofuel production hit a record of 171 billion liters in 2022, with biodiesel and bioethanol being the primary biofuels produced worldwide (Sharma et al., 2023). In 2010, the worldwide consumption was 11%, with an expectation that it would reach 15% in the industrial sector by 2030. The rise is attributed to the anticipated energy demand in global transport system reaching 130 EJ/year by 2030, which was lower in 2010 at 92 EJ/year. According to IRENA's 2016 Remap reports, the demand for liquid biofuel is projected to rise to 500 billion/year by 2030 (Maliha et al.,2023). This is operated by the transport sector, which reports for about 60% of the total oil demand and is reported to be the primary sector for fossil fuel consumption. Liquid biofuels are the best choice for reducing carbon emissions in the transportation sector (Canabarro et al.,2023; Wang et al , 2022; thangamani et al., 2021). This led researchers to shift their focus towards alternative, environmentally friendly renewable energy sources. In recent years, biodiesel has become widely recognized as a resource. Biodiesel comprises mono-alkyl esters of long-chain fatty acids derived from vegetable oil or animal fats (Hanna et al., 2005).

1.1 Biodiesel

Taking into account the historical context of biodiesel, the first industrial-scale biodiesel production plant began operations in 1991, initiated by Europe in Austria (w.k. rbitza et al., 2003). It is estimated that the ongoing oil and gas reserves will only last for a couple of decades. In response to the escalating demand for energy, biodiesel and bioethanol emerge as the forefront substitute technologies. Moreover, as a viable alternative for compression and ignition, biodiesel serves as a suitable substitute fuel (demirbas et al., 2007). Biofuel has the advantage of using renewable and natural raw materials (Marchetti et al., 2007). Biofuels can be divided into four generations according to their raw materials. From organic matter like Vegetable oil, potatoes or any edible substances, first generation biofuels are primarily extracted. Feedstocks such as cattle fodder, municipal waste, etc. are used to produce second generation biofuels. Third-generation biofuels are typically derived from algae biomass, while fourth-generation biofuels focus recombinant DNA technology (Ogunkunhle et al., 2019). The formation of biodiesel from used or waste cooking oil is relatively simpler and offers numerous environmental benefits. Kitchens and food industries globally give rise to 16.5 million tons of waste cooking oil, which is either disposed of in the sewage system or sent to landfills. As per the World Health Organization, 25% of wastewater pollution is attributed to waste cooking oil. Waste oil (approx. 1 liter) can pollute around one million liters of water, causing extensive damage to the aquatic ecosystem. In biodiesel synthesis, utilizing waste cooking oil ultimately addresses the issue of dumping or discharge (Rocho menses et al., 2023; Ulukardesler et al , 2023). Biodiesel contains a higher oxygen quantity compared to petroleum diesel, leading to a substantial decrease in the emissions of carbon monoxide, hydrocarbons, particulate matter, smoke, and noise when used in diesel engines (Gashaw et al, 2014; suzihaque et al., 2022, velmurugan et al., 2022, Aamir et al.,2022; B.Bankovic et al., 2012; Aghabarari et al., 2014).

1.2. Methods for Production of biodiesel

The method of biodiesel production rely on factors such as the raw materials used, the desired product quality, cost considerations, and environmental considerations. Every method possesses its own merits and drawbacks, and ongoing research is dedicated to enhancing the viability and efficiency of biodiesel production. The primary approaches for biodiesel production include:

1.2.1. Transesterification

The process of transesterification or alcoholysis, is similar to hydrolysis. The transesterification reaction requires three moles of alcohol and one mole of triglyceride to yield glycerin (1mole) and fatty acid methyl esters(3 moles). Various alcohols can be employed in this process, but methanol and ethanol are commonly preferred due to their physical and chemical properties as well as their cost-effectiveness. Triglycerides readily undergo reaction with methanol, and NaOH easily dissolves in the process. This process brings down the triglyceride's viscosity and therefore increases the physical properties of the fuel. Consequently, the fuel obtained through this transesterification process can be utilized with greater preference (Fukuda et al., 2001; Ma a et al., 1999). The transesterification process typically employs three categories of catalysts, namely alkali catalysts, enzyme catalysts, and acid catalysts. Each catalyst presents its own perks and limitations during the reaction.

1.2.1.1 Mechanism of transesterification by acid catalyst

In the process of transesterification of vegetable oil with acid catalyst, a carbocation is formed through the protonation of the carbonyl group in esters (Fig1). A tetrahedral intermediate is subsequently produced by the nucleophilic attack of alcohol on this carbocation. This intermediate then removes glycerol, forming a novel ester and regenerating the catalyst.

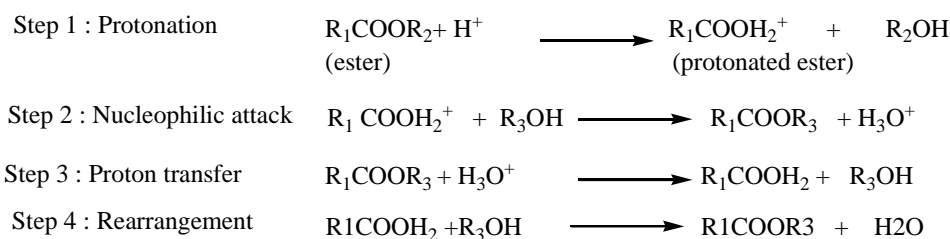


Figure 1: Transesterification by acid catalyst

1.2.1.2 Mechanism of transesterification by base catalyst

In this process, Initially base and alcohol reacts, producing a protonated catalyst and alkoxide. Subsequently, a nucleophilic attack is done by alkoxide on the carbonyl group of triglycerides, resulting in the production of a tetrahedral intermediate. The production of alkyl ester and the anion of Diglyceride occurs from this tetrahedral intermediate. Following this, deprotonation

of the catalyst takes place, regenerating the species which actively fuses with a second alcohol species to start a new catalytic cycle.

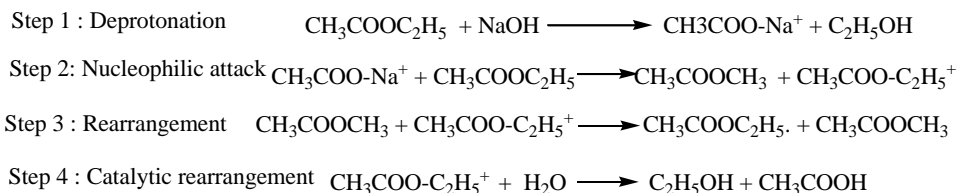


Figure 2: Transesterification by base catalyst

Utilizing carbonates, sodium, potassium, alkoxides as effective homogeneous catalysts, a significant portion of biodiesel can be generated from vegetable oil. Generally, the rate of the acid-catalysed process is slower than alkali catalysed process

1.2.2 Pyrolysis

The pyrolysis process involves heating a substance to change its form, with no air or oxygen present during the process (Ma a et al., 1999). In this procedure, prior to utilizing the oil, it undergoes an initial filtration to eliminate any remaining particles. Subsequently, the heating of oil up to 50°C eliminates its moisture content, ensuring the oil flows freely throughout the process. The pyrolysis process operates with a reaction time of approximately 3 to 6 hours with a temperature range of 200 to 400°C in the reactor where the pyrolysis process occurs, approximately 2 liters of oil are introduced each time. Upon completion of reaction, the vapor of the produced biodiesel from the reactor was condensed and accumulated in a receiving flask, displaying a yellowish color (M.A. Mohamed et al., 2017; demirbas et al l., 2003).

1.2.3 Supercritical technology

The transesterification reaction typically proceeds slowly at room temperature because of the two-phase characteristic of the alcohol-oil mixture. To address this issue, catalysts like heterogeneous and homogeneous catalysts are employed, with potassium hydroxide and sodium hydroxide being among the preferred choices. However, the use of catalysts introduces a challenge in separating the catalyst from the final product (S Saka et al., 2001). Several advancements have been made in this regard, revealing that the reaction rate can be heightened without the necessity of a catalyst. Achieving this involves heating methanol to its high temperature and pressure stage and initiating the reaction with the help of rapeseed oil. It has been observed that this method results in a single-phase methanol-oil mixture, in contrast to

the two-phase mixture obtained at room temperature. This occurs because, at the supercritical stage, there is a reduction in the dielectric constant of liquid methanol, leading to an increase in the proportion of oil in methanol (Gui et al., 2009; Zhu et al., 2016, martinez et al.,2020).

1.2.4 Reactive distillation

The reactive distillation process is utilized because of the various advantages of biodiesel and the increasing commercial demand. The simultaneous occurrence of reactions and separation is possible through this process. It is better than the traditional process, especially when it involves a reaction that can be reversed. The main reason for employing this approach is its high chemical conversion rate and cost-effectiveness. Furthermore, this method involves a reduction in the utilization of excess alcohol(D darnoko et al., 2000).

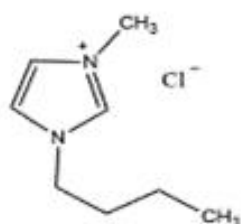
1.2.5. Catalytic distillation

The main focus in recent developments has been on minimizing pollution and adopting an eco-friendly process that does not have a adverse impact on the nature. An emerging approach called catalytic distillation is gaining prominence as a more environmentally friendly method for biodiesel production. Catalytic distillation is a procedure where production separation and chemical reactions occur simultaneously in a single operation. This results in reduced energy consumption and, consequently, lowers the cost of biodiesel production. Continuous separation of products from the reaction contributes to an increased yield of the final product (Gaurav et al., 2013; Bhate et al., 2004; kamath et al., 2006).

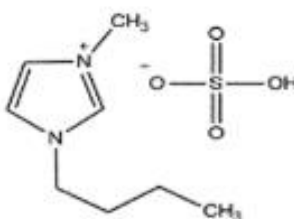
1.3 Ionic liquids

Ionic liquids (ILs) are acknowledged as the ‘next generation of catalysts’ within the industry of chemicals. The application of ionic liquids enhances the yield many times compared to conventional methods. Ionic liquids refer to organic salts which contains anions and cations, which remain liquid at room temperature (Fig 3). Ionic liquids consist of organic cations such as pyridinium, imidazolium, alkylated phosphonium, and sulfonium ions, as well as aliphatic ammonium ions, combined with either organic or inorganic anions (Yoo et al. 2017). Ionic liquids are characterized by their high catalytic activity, easy recycling, and low viscosity. They possess the ability to dissolve various kinds of substances, including polar and non-polar

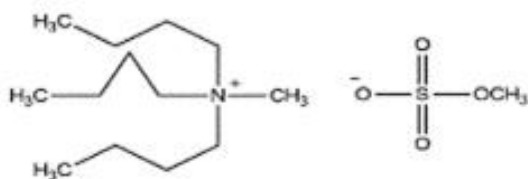
organic compounds, as well as polymeric and inorganic molecules, owing to their diverse structures (Andreani et al., 2012). Ionic liquid's exceptional thermal and chemical solubility can be determined with the help of cations and anions composition in use. Moreover, they possess the ability to adjust or modify the inherent characteristics of both the anion and cation components to achieve the desired outcome effectively. This is why ionic liquids are referred to as 'designer solvents' and are also known as 'Task-specific ionic liquids' (Ong et al., 2021; Muhammed et al., 2015). Ionic liquids are more expensive compared to conventional catalysts and solvents but they are ecofriendly or environmentally secure so these are highly preferred and are called as "green catalyst". Ionic liquids enhance the utility of glycerol by serving as an extraction solvent in the biodiesel production process (Zhang et al., 2023). Ionic liquids at room temperature, a new type of environmentally friendly chemical material, possess recyclable catalyst properties that have the potential for applications in biomass conversion (Liyang et al., 2022). Ionic liquids enhances the efficiency of transesterification for good biodiesel yield from waste cooking oil or vegetable oil. This is attributed to their heightened Lewis acidity, resulting in a substantial yield of biodiesel (Yaseen et al., 2017). Therefore, incorporating ionic liquids into the biodiesel synthesis pathway is advantageous for environmentally friendly production (Ong et al., 2021).



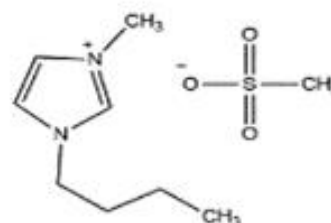
1-Butyl-3-methylimidazolium chloride



1-Butyl-3-methylimidazolium hydrogen sulfate



Tributylmethylammonium methyl sulfate



1-Butyl-3-methylimidazolium methanesulfonate

Figure 3: Structure of ionic liquids

1.3.1 Classification of ionic liquids

Ionic liquids stand out as a group of liquid salts, marked by their distinctive properties that set them apart from conventional liquids. ILs are widely used in fields such as synthetic chemistry, catalysis, and electrochemistry due to their flexibility. The classification of ionic liquids can be affected by various factors, like type of anions and cations, and their specific uses.

1.3.1.1 Cationic ionic liquids

Cationic ionic liquids are ionic liquids that have cations with a positive charge. Typically, these cations are organic or inorganic in nature and do not have hydrogen atoms attached directly to the cationic center. The positive charge of the cation is a key factor in these applications, which can result in specific interactions or reactions, like those in ion exchange processes or solvents for polar compounds. Cationic ionic liquids possess stability and have applications in various fields, including electrochemistry, catalysis, and separation processes. Cationic ionic liquids can be categorized depending upon their cations, which may include imidazolium, pyridinium, ammonium, and phosphonium (Fig 4). Cationic ionic liquids have the capacity to catalyze the transesterification reaction, which transforms triglycerides from oils or fats into FAME(fatty acid methyl esters) and these are important ingredients of biodiesel. There are many benefits of using cationic ionic liquids which includes increased catalytic efficiency, reduced waste production, and an environmentally sustainable process compared to traditional approaches.

1.3.1.2 Anionic ionic liquids

Anionic ionic liquids are a category of ionic liquids distinguished by presence of negatively charged anions . These liquids have been studied for their potential as catalysts or reaction media because of their unique properties. Anionic ionic liquids can be categorized on the basis of their anions, which may include halide, hexafluorophosphate, sulfonate and Tetrafluoroborate. They can be applicable in various processes, including electrochemistry,

separation processes, catalysis, gas capture, sensor and fuel cells. They explored for their promising application in producing biodiesel, especially for transesterification. The hydroxide anion can be found in [BMIM][OH] i.e 1-butyl-3-methylimidazolium hydroxide, and has been analysed in biodiesel production. The hydroxide anion in [BMIM] [OH] has the potential for facilitating the transesterification process.

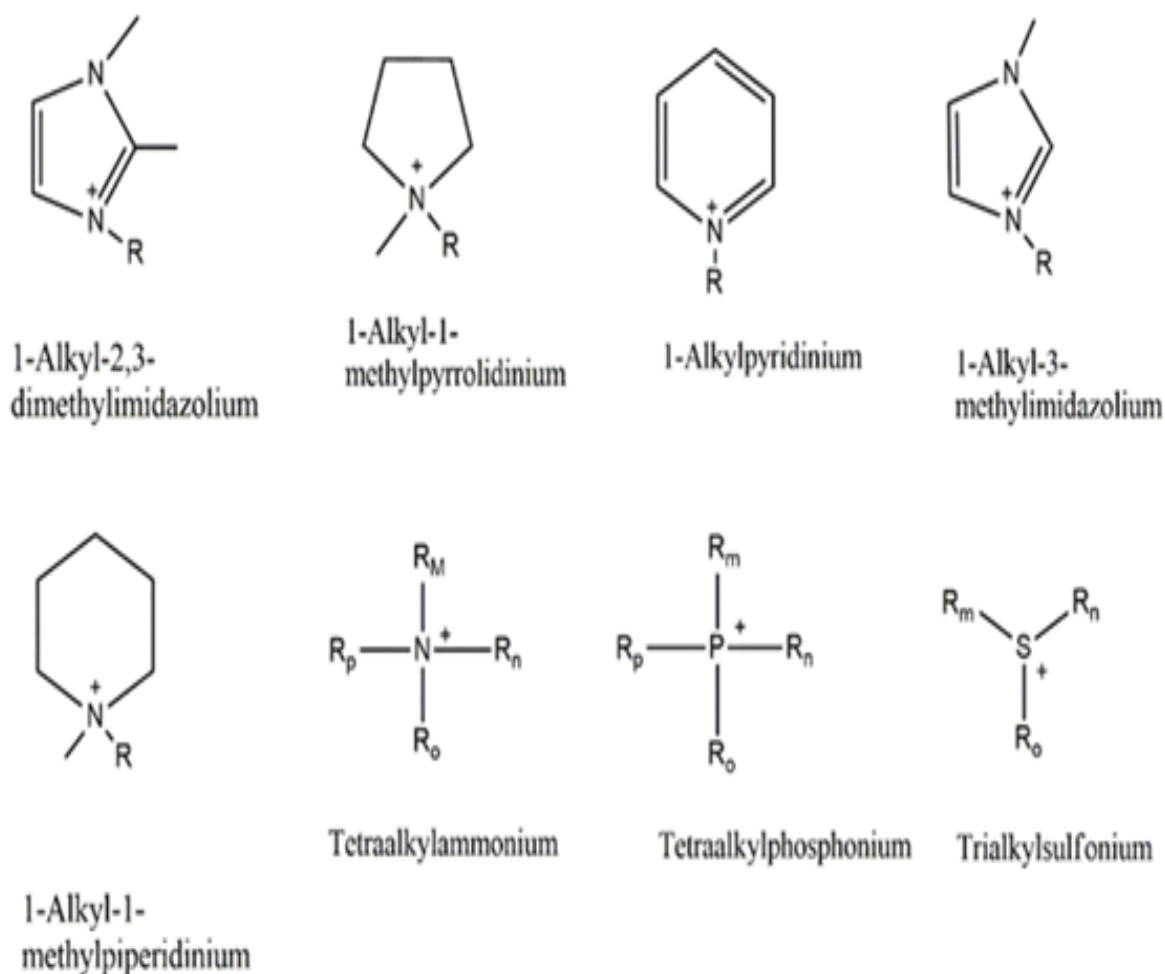


Figure 4: Cationic ionic liquids

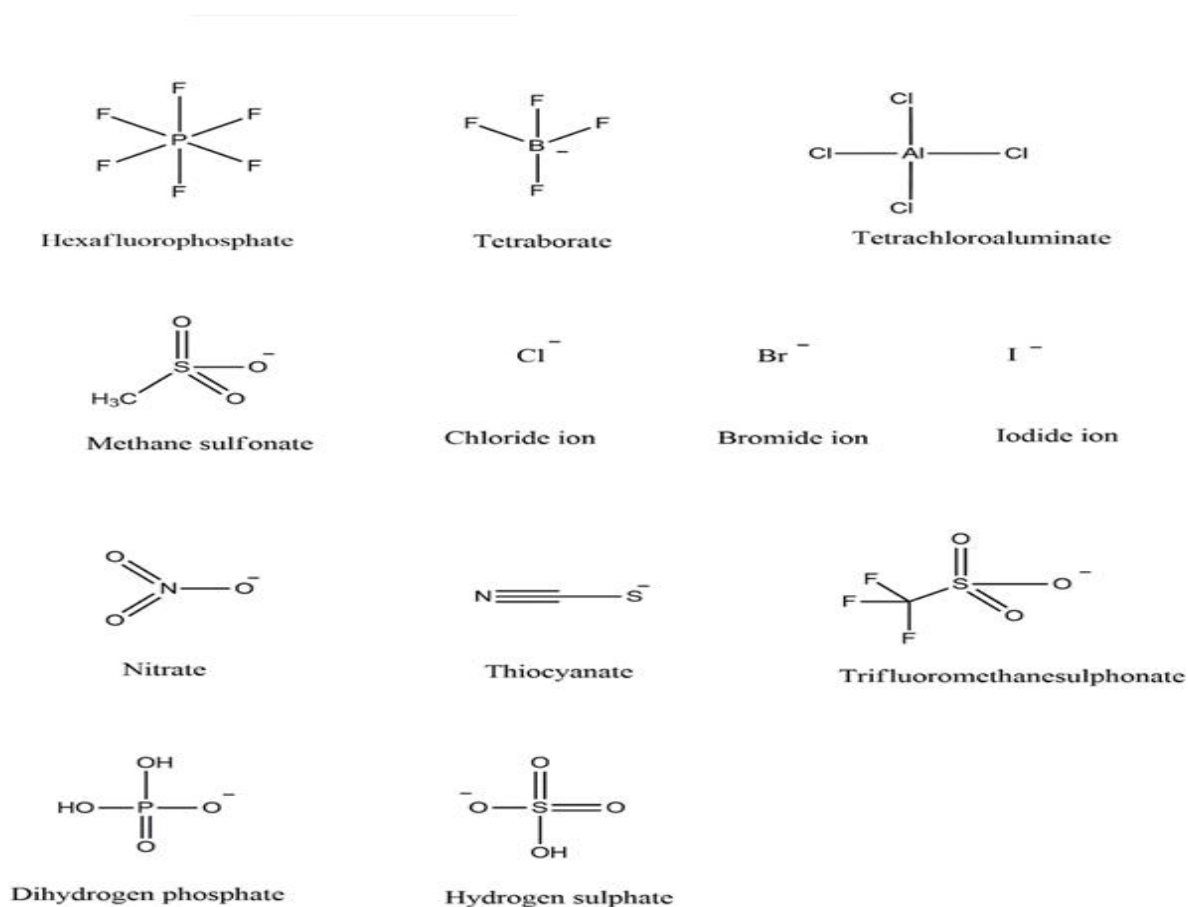


Figure 5 : Anionic ionic liquids

1.3.2 Applications of ionic liquids

The distinctive properties of ionic liquids (ILs) lead to broad range of applications in different fields.

1.3.2.1 Ionic liquids as lubricants additives

Use of Ionic liquids (ILs) as lubricant additives has gained attention because of their distinct properties and potential benefits in enhancing lubrication performance. properties like low flammability, thermal stability, volatility, enable them to act as perfect lubricants. ILs can withstand higher pressures and temperatures, making them superior lubricants compared to conventional synthetic oils. IL lubricants protect surfaces by establishing a structured layer that keeps moving parts separated (Somers et al., 2013).

1.3.2.2 Ionic liquid as electrolytes

ILs are often used as electrolytes in various electrochemical devices due to their high ionic conductivity, stability, versatility and safety. ILs are used as electrolytes in sodium-ion and lithium-ion batteries. The lithium-ion batteries contains lithium salts which can be dissolved in ionic liquids forming an electrolyte. This electrolyte allows lithium ions (Li^+) to participate in the intercalation reaction within these batteries. Another factor contributing to their effectiveness as electrolytes is their low vapor pressure, resulting from strong electrostatic interactions. This property is why most ionic liquids are challenging to ignite, making them compatible with Li-ion batteries (Jonsson et al., 2020).

1.3.2.3 Ionic liquids in biphasic system

Ionic liquids are frequently employed in biphasic systems, which encompass two liquid phases that are immiscible with each other. In biphasic systems, they are utilized as solvent in one phase and remain immiscible with the other phase. Ionic liquids typically function as the non-aqueous phase in biphasic systems (Imam et al., 2021). The properties of ionic liquids, such as their high thermal stability, low volatility and tunable solvation capabilities are distinctive which lead to their utilization. Ionic liquids in biphasic systems are commonly used for separation processes, liquid-liquid extraction, and biocatalysis.

1.3.2.4 Ionic liquid as biocides

Ionic liquids (ILs) are developing as popular biocides because of their antimicrobial properties. The use of ILs as biocides can be used to manage microbial populations effectively in different applications, while taking into account environmental and safety concerns through careful design and implementation. ILs possess the capability to stop the growth and reproduction of microorganisms like bacteria, fungi, and algae. The chemical composition and interactions between ILs and biological systems lead to these characteristics. ILs are used as biocides in various field, such as disinfectants, antiseptics, crop protection, disinfecting drinking water, and antimicrobial coatings. Ionic liquids' capacity to dissolve specific compounds enables them to dissolve targeted contaminants, aiding in cleaning processes and ultimately leading to reduced waste disposal expenses (Kohli et al , 2019).

1.3.2.5 Ionic liquids in biodiesel synthesis

Ionic liquids (ILs) garnered interest in biodiesel production due to their potential as catalysts, solvents, or additives. Certain ILs can catalyze the transesterification reaction and enhance reaction rates, selectivity, and yield. Biodiesel synthesis can be assisted by ILs as solvents by

providing a medium for alcohol and triglycerides so that it can react in catalyst's presence. ILs have the potential to provide advantages such as improved solubility, decreased waste generation, and product separation that is easier. The properties of biodiesel can be modified by adding ILs, which can improve its stability, lubricity, or cold flow properties. The overall quality and performance of biodiesel as a fuel can be enhanced by this.

1.4. Biodiesel production through the use of diverse ionic liquids and various raw materials

Biodiesel synthesis with diversity of ionic liquids and various raw materials entails considering several important factors, including the choice of ionic liquids, raw materials, transesterification reaction, separation and purification of biodiesel. Ionic liquid's utilization with different oils for biodiesel synthesis is shown in Table 1.

1.4.1. Castor oil with basic ionic liquid

Castor oil can be used in biodiesel production with basic ionic liquids. The [BTBD] [OH] ionic liquid can be reused and recycled after the procedure due to its thermostability. It exhibits partial solubility in polar solvents and is also water soluble, however in non-polar solvents it is insoluble. The ionic liquid can be used again a minimum of six times, indicating that [BTBD][OH] remains stable even at high temperatures. The recyclability and stability of this catalyst make it a suitable choice for this procedure, particularly when compared to [BMIM][OH] and [TBA][OH]. Under optimal reaction conditions, this basic ionic liquid demonstrates a high yield of 96% (fang et al., 2013; long et al., 2011).

Table 1: Utilization of ionic liquids in various oils for biodiesel synthesis.

Oils	Ionic liquids
Waste cooking oil	[BMIM][HSO ₄] (Ullah et al., 2015). [BSMBIM][CF ₃ SO ₃] (Ullah et al., 2017). [BMIM][OH]/Mg-Al-La (Sun et al., 2016). [SO ₃ H(CH ₂) ₃ Vim][HSO ₄] with PDVB (Liang et al., 2013).
Bitter apple oil	[SBP][HSO ₄] [SPP][HSO ₄] [MSPPHSO ₄] [MSBPHSO ₄] (A. Elsheikh et al., 2014).

Castor oil	[BTBD][OH] [BMIM][OH] (fang et al., 2013). [TBA][OH]
Tung oil	N-methyl-imidazole based ILs [Ps-MIM][p-TSA] [ps-N-Ch(Me) ₂][HSO ₄] [Ps-MIM][HSO ₄] (yang et al., 2016). [ps-N-Ch(Me) ₂][p-TSA]
Soyabean oil	[Hnmm]OH (Ren Q. et al., 2014). [OmPy][BF ₄] (liu et al., 2011). [Bmim][Im] - (imidazolide based IL) (luo et al. 2013). [Ch][OH] [Ch][Ome] [Ch][Im] (fan et al., 2013). 1,2-bis(3-methylimidazolium-1-yl) ethylene imidazolide 1,2-bis(3-methylimidazolium-1-yl) ethylene imidazolide 1,4-bis{3-methylimidazolium-1-yl} butylene imidazolide 1,6-bis(3-methylimidazolium-1-yl) hexylene imidazolide (fan M et al., 2013). [C ₄ SO ₃ Hpy][HSO ₄] [C ₃ SO ₃ HEt ₃ N][HSO ₄] [C ₄ SO ₃ HMIM][HSO ₄] [C ₃ SO ₃ HPy][HSO ₄] [C ₃ SO ₃ HMIM][HSO ₄] (Fan MM et al., 2012). [NMPH][CH ₃ SO ₃] [Et ₃ NH][Cl]- AlCl ₃ (Fauzi et al., 2022).
Coconut oil	[PhCH ₂ N _{1,1} PrSO ₃ H][HSO ₄] [PhCH ₂ N _{1,1} PrSO ₃ H][Tos] [CyN _{1,1} prSO ₃ H][HSO ₄] (Qiu et al., 2016). [Ps-MIM][Tos] [Ps-MIM][HSO ₄]
Jatropha oil	[BMIM][CH ₃ SO ₃]-AlCl ₃ [BMIM][CH ₃ SO ₃]- FeCl ₃ (Gyo et al., 2011). [BSMIM]Cl (khiratkar et al., 2018)
Palm oil	[ChOH] (phromphithak et al., 2020). [HSO ₃ -BMIM][HSO ₄] (ding et al., 2018). [Taz-prSO ₃ H][HSO ₄] [Taz-prSO ₃ H][CH ₃ SO ₃] [Taz-prSO ₃ H][Tos] [Taz-prSO ₃ H][CF ₃ SO ₃] (li et al., 2018).
Rapeseed oil	[BMIM][IM] (luo et al., 2013). [BSO ₃ HMIM][HSO ₄] (fan et al., 2017).

Sunflower oil	[BMIM][IM] [TMA][His] [TBA][His] [Ch][Leu] [Ch][Arg] [Ch][Ala] [Ch][Ser] [Ch][Phe] [Ch][His] [Ch][Met] [TBA][Arg] [TMA][Arg] [Ch][Gly]	(Ong et al., 2021).
Cottonseed oil	IMC ₂ OH IMC ₃ OH IMC ₄ OH IMC ₅ OH IMC ₆ OH	(Liang et al., 2010).
Castor oil	[BTBD][OH] [BMIM][OH] [TBA][OH]	(ong et al., 2021).
vegetable oil	[BMIM][AlCl ₄] [BMIM][CuCl ₄] [BMIM][CoCl ₄] [BMIM][FeCl ₄] [PSMIM][HSO ₄] [BMIM][NiCl ₄]	(Schuchardt et al., 1998)
Olive oil	[C ₁₆ MIM][NTf ₂]	(ong et al., 2021).

1.4.2 Palm oil with choline hydroxide and triazolium-based ionic liquid

Previous research has explored biodiesel production through the transesterification process utilizing choline ionic liquid compounds, which includes choline methoxide (ChOMe), choline imidazole (ChIm), and choline hydroxide (ChOH). Choline hydroxide (ChOH) ionic liquid, known for its environmentally friendly properties, was utilized in biodiesel synthesis as catalyst from palm oil. This choice ChOH as the ionic liquid resulted in the highest biodiesel yield, with a remarkable 95% achieved. Biodiesel production has been implemented on a large scale, but the microwave transesterification process is currently only available in laboratory scale. Microwave irradiation compared to conventional heating has been investigated in terms of its impact on oil conversion rates, and it has been found that microwave heating leads to a higher

conversion rate. Previous studies have shown that microwave-heated methods lead to faster reactions and less pollution (Hsiao et al., 2011, Lin et al., 2015). Choline hydroxide (ChOH) ionic liquid was employed in the biodiesel synthesis from palm oil, and the experimental design followed the Box-Behnken method. This choice was made due to its lower percentage error, typically around 2.5-3.4%, aiming to maximize the yield of biodiesel. The ionic liquid was synthesized by microwave heating a continuous flow reactor in methanol, with potassium hydroxide and choline chloride in methanol being used in equimolar amounts. To achieve the highest biodiesel yield, the optimal oil to methanol molar ratio was set at 1:13:24, with a flow rate of 20 ml/min and a microwave power of 800W (Phromphithak et al., 2020).

Previously, from palm oil synthesis of biodiesel involves using choline hydroxide as the ionic liquid. Now, the focus has shifted to utilizing triazolium-based ionic liquids for biodiesel production. To avoid saponification and corrosion, avoid alkali catalysts and acidic ionic liquids (Aziz et al., 2013; Jeong et al., 2010). N-methyl-imidazolium-based ionic liquids have been utilized; however, they exhibit low hydrothermal stability (Jeong et al., 2010). Therefore, 1, 2, 4-triazolium-based ionic liquids turned out to be employed, as it is believed that they possess superior hydrothermal stability owing to their chemical inertness. Several 1,2,4-triazolium-based ionic liquids were synthesized, including [Taz-prSO₃H][Tos], [Taz-prSO₃H][CH₃SO₃], and [Taz-prSO₃H][HSO₄], [Taz-prSO₃H][CF₃SO₃]. The transesterification process was carried out using these four ionic liquids, and their performance was evaluated. It was observed that [Taz-prSO₃H][CF₃SO₃] exhibited the highest catalytic activity among the four ionic liquids (Jain et al., 2005; Brushett et al., 2010; Li et al., 2018).

1.4.3 Coconut oil with imidazole based ionic liquid

Biodiesel can be produced by utilizing a novel Bronsted acid as an ionic liquid catalyst from coconut oil. The use of H₂SO₄ or HNO₃ as acid catalysts in the past was based on their high catalytic activity. However, they posed limitations such as corrosiveness, challenging recovery, and environmental pollution, which restricted their usage (Sun et al., 2010). N-methylimidazole-based Bronsted acidic ionic liquids are more water stable and effective in reactions, but their high cost makes them unsuitable for large-scale biodiesel production (Yang et al., 2015). Four novel Bronsted acids such as [CyN1,1PrSO₃H][HSO₄], [CyN1,1PrSO₃H][Tos], [PhCH₂N1,1PrSO₃H][HSO₄] and [PhCH₂N1,1PrSO₃H][Tos]; were developed for biodiesel

production from coconut oil, which offer a cost-effective alternative to imidazole-based ionic liquids. A solubility test was conducted on the ionic liquids before starting the experiment, which revealed that they were soluble only in methanol. Several experiments were carried out, altering various parameters including the time of reaction (ranging between 20 - 180 minutes), the type of ionic liquid used, the molar ratio of methanol to oil (ranging from 6:1 to 18:1), and the reaction temperature (353-393K). Biodiesel obtained after the reaction, with the findings showing that the ionic liquid containing tosylate provided superior results compared to the one containing bisulfate. The ionic liquid based on [CyN1,1PrSO₃H]⁺ stands out among all others due to several advantages. It exhibits superior catalytic activity, the cost of its cation source, N,N-Di-methyl cyclohexylamine, is more economical compared to N-methyl-imidazole, and the formulation of the ionic liquid derived from [CyN1,1PrSO₃H]⁺ is simple. Although sulfuric acid was not environmentally friendly, this ionic liquid made from it is eco-friendly (shen et al., 2014; yang et al., 2016).

1.4.4 Jatropha oil with choline hydroxide and bronsted acidic ionic liquid

Biodiesel production utilizes the transesterification process, which is catalyzed by using either a basic or acidic catalyst. Potassium hydroxide (KOH) and sodium hydroxide (NaOH) catalysts are avoided because of their tendency to undergo saponification reactions and extended reaction times. Acidic catalysts like sulfuric acid and p-toluene sulfonic acid are avoided due to their long reaction times, challenging recovery process and harmful nature (anh N.phan et al., 2008; C.C. Enweremadu et al., 2009). Several ionic liquids have been utilized to transesterify *Jatropha curcas* oil, but they require high temperatures and produce suboptimal yields. The mixture of Jatropha oil and methanol was mixed with CHOH as the catalyst, and the temperature was adjusted accordingly. Upon completion of reaction, the mixture was left to cool down and then separated to obtain biodiesel. It was found that different concentrations of the ionic liquid resulted in varying yields of biodiesel (A.V.Bornade et al., 2017). As per the findings, [BMIM][CH₃SO₃], a Bronsted acidic ionic liquid, exhibits the maximum activity of catalyst for oleic acid with a 93% esterification rate. However, the biodiesel yield is only 12% at 120 degrees Celsius. It was further reported that by introducing FeCl₃ or any MCl₃ to [BMIM][CH₃SO₃], the biodiesel yield increased significantly to 99.7% at 120 degrees Celsius. The metal ions supplied the Lewis acid sites in ionic liquids, with trivalent metallic ions providing more sites than bivalent ones, which led to this phenomenon. FeCl₃, CuCl₂, and AlCl₃ exhibited the highest level of activity (Guo et al., 2011; Alexander A.M. Lapis et al., 2008).

1.4.5 Tung oil with Bronsted acid ionic liquid

Tung oil, derived from a woody plant, is a inedible vegetable oil with high oil ratio. Therefore, it is a preferable alternative for biodiesel production (wang et al., 2015). Anhydrous conditions are necessary because alkali catalysts undergo saponification, while acidic catalysts lead to corrosion (luu et al., 2014). Ionic liquids have been employed due to their environmentally friendly nature. The four Bronsted acid ionic liquids utilized for biodiesel production from Tung oil are; N,N-dimethyl-N-(3-sulfopropyl) cyclo-hexyl ammonium hydrogen sulphate, 1-(3-sulfopropyl)-3-methylimidazolium tosylate, N,N- dimethyl- N-(3-sulfopropyl) cyclo-hexyl ammonium tosylate and 1-(3-sulfopropyl)-3-methylimidazolium hydrogen sulphate. Previous research indicated the high catalytic activity of N-methyl-imidazole based ionic liquids. Hence, ionic liquids of this type were selected for biodiesel production. Ammonium-based ionic liquids exhibit superior catalytic activity. The Bronsted acidity of the [p-TSA]⁻ anion is higher than that of the [HSO₄]⁻ anion, resulting in a higher yield with [p-TSA]⁻ (wang et al., 2014; yang et al., 2017).

1.4.6 Cottonseed oil with di-cationic functional ionic liquid

Functional ionic liquids were engaged as catalysts due to the drawbacks of alkali and acidic catalysts, as they are environmentally friendly and provide a good yield. For instance, alkane sulfonic acid with Bronsted acidic ionic liquid was produced, which exhibited good catalytic activity. Specifically, 1-(4-sulfonic acid) Butyl pyridinium hydrogen sulfate ionic liquid showed activity comparable to concentrated sulfuric acid (wang et al., 2007; Han et al., 2009). Synthesis of a di-cationic ionic liquid resulted in improved stability, while an acidic di-cationic ionic liquid having an alkane sulfonic acid group performed better in the reaction. The basic di-cationic ionic liquids such as Bis -(3-methyl-1-imidazole)-ethylene dibromo salt, Bis-(3-methyl-1-imidazole)-butylene di-hydroxide, Bis-(3-methyl-1-imidazole)-pentylene di-hydroxide, Bis-(3-methyl-1-imidazole)-propylene Dihydroxide, and Bis-(3-methyl-1-imidazole)-hexylene di-hydroxide; were synthesized using cottonseed oil. The catalytic activity of various ionic liquids was assessed. It was observed that the activity depended on the carbon chain length, with bis-(3-methyl-1-imidazolium)-ethylene di-hydroxide showing the best performance, resulting in a biodiesel product exceeding 98% (ling et al., 2010).

1.4.7 Soyabean oil with [Hnmm]OH ionic liquids

[Hnmm]OH (1-butyl-3-methyl morpholine hydroxide), a basic ionic liquid derived from morpholine alkaline, is employed in biodiesel synthesis using soybean oil. According to reports, the use of [Hnmm]OH as a catalyst, with an increase in its dosage from 1% to 4%, results in an increment in biodiesel yield from 61.2% to 96.6%. The ionic liquid [Hnmm]OH exhibited excellent basic properties, thermal stability, and can be readily separated from the reaction mixture without any impurities (Ren Q. et al., 2014; J.D. Rocha et al., 2012).

1.4.8 Waste cooking oil with [BMIM][HSO₄] ionic liquid

Oil is a vital food component consumed globally, and its disposal into the environment leads to environmental pollution. The handling of waste cooking oil can be best done by converting it into biodiesel. Base catalysis cannot be utilized for converting waste cooking oil into biodiesel because of the potential formation of soap. Consequently, homogeneous catalysts were employed, but, they come with their own drawbacks such as potential corrosion and challenging separation from the reaction medium. Heterogeneous catalysts were employed; however, they also come with drawbacks. The green catalysts were developed to address these challenges. As part of this effort, [BMIM][HSO₄] was created, which can be separated easily and do not cause corrosion (Dogan et al., 2016; liu et al., 2009; Man et al., 2015; kavlak et al., 2022).

1.5 The potential for ionic liquid catalysts to be recycled

The utilization of ionic liquid catalysts in biodiesel production becomes promising as they possess unique features such as tunable solubility, high thermal stability, and recyclability. The probability of recycling ionic liquid catalysts is a major advantage for sustainable biodiesel production. Ionic liquid catalysts are attractive options for researchers and industries seeking greener catalytic solutions due to the fact that they can be recyclable and help in contributing sustainability and efficiency of biodiesel synthesis processes. Recyclability of ionic liquids is more advantageous than conventional homogeneous solvents. The most common method for recycling or recovering ionic liquids is distillation due to their low vapor pressure (felizaedo et al., 2006).

1.5.1 Processes for the recovery of ionic liquids

The recovery methods can be customized based on their specific properties and the desired purity level for recycling. The sustainability and cost-effectiveness of using ionic liquids in various applications is greatly influenced by their role.

1.5.1.1 Distillation

Ionic liquids can be recovered by distillation (most common method). The process involves heating the mixture to evaporate the ionic liquid, which is then condensed back into liquid form. This method allows for the recovery of ionic liquids through distillation, where methyl butyrate and methanol are separated because of their maximum volatility and minimum boiling points in comparison with ionic liquids. Steam and vacuum distillation techniques can be advantageous in an ionic liquid catalyst system for product separation, but there has been a decrease in catalytic activity following the distillation process. The Friedel-Crafts benzoylation reaction employed these techniques to separate the reaction products by using ionic liquids as catalysts.

1.5.1.2 Extraction

The high thermal energy needed for the evaporation method makes it somewhat unsustainable for recovering ionic liquids, rendering it an impractical option. The extraction of ionic liquid using carbon dioxide is an alternative to evaporation, which does not require high thermal energy. This method is more effective than the evaporation method because carbon dioxide (CO₂) and ionic liquids are insoluble, while organic compounds are soluble in CO₂. This helps in the separation of organic compounds from ionic liquids (fauzi et al., 2012).

1.5.1.3 Crystallization

Crystallization methods can be employed to recover certain types of ionic liquids that form crystals under specific conditions. Temperature and other parameters can be used to precipitate and separate the ionic liquid from the solution.

1.5.1.4 Adsorption

Activated carbon or resins are used in adsorption techniques to selectively adsorb ionic liquid from a solution. The ionic liquid that has been adsorbed can be removed using a suitable solvent or heat. The recovery of [BMIM] [PF₆] from wastewater has been investigated by researchers who found that hydrophilic ionic liquids are less absorbed than hydrophobic ones. Hydrophilic

ionic liquid's adsorption can be increased by incorporating hydroxyl groups on activated carbon. The adsorption of hydrophilic ionic liquids can be enhanced by incorporating hydroxyl groups on activated carbon (Sklavounos et al., 2016).

1.5.1.5 Membrane separation

Ionic liquids can be separated on the basis of their molecular size and charge using membrane separation techniques, such as reverse osmosis or nanofiltration. Ionic liquids, because of their diverse sizes, are retained within the system, while components other than the ionic liquid diffuse through the membrane (Fauzi et al., 2012). Nanofiltration is applied in the process of separating ionic liquids from solvents. This method is especially used when the ratio of ionic liquid is minimum in the mixture and for systems that are not suitable for distillation. Nanofiltration membranes can separate monovalent and divalent ions, as well as charged and neutral compounds. During this process, non-volatile products and ionic liquids were initially dissolved in water. Subsequently, the product was rejected, while the ionic liquid permeated through the membrane (Haerens et al., 2010).

1.5.1.6 Induced phase separation

Induced phase separation is a technique that involves separating phases in a mixture by changing conditions, such as temperature or solvent composition. The salting-out is a simple technique used to create biphasic systems by combining electrolytes or salts with an aqueous solution of ionic liquid. The utilization of inorganic salts which is utilized in this method to produce a water-rich phase and an ionic liquid-rich phase, while minimizing the cost of inorganic salts. Carbon dioxide-induced phase separation is an environmentally friendly process in which CO₂ is introduced to trigger a phase separation of the solute, ionic liquid, and CO₂-rich phase. Temperature-induced phase separation can only occur in certain types of ionic liquids. It requires minimal energy consumption, making it highly efficient. Some aqueous solutions of ionic liquids exhibit lower critical solution temperature (LCST) phase behavior, allowing them to be separated into multiple phases by adjusting the temperature (Lan Mai et al., 2014).

Research gap

- The catalysts widely used are hazardous in nature so there is need to focus on environmentally friendly catalyst for biodiesel production.
- The low yield of biodiesel production is the bottleneck in the scalability of the current technologies.

To address these gaps, following objectives are proposed in the present work:

Objective 1: Use of Ionic Liquid as more efficient and cost-effective catalyst.

Objective 2: Characterization of the waste cooking oil.

Objective 3: Use of the catalyst for esterification/ transesterification of waste cooking oil.

Objective 4: Comparison of properties of diesel and biodiesel produced from the waste cooking oil

CHAPTER:-2

MATERIALS AND METHODS

2.1 Materials

Waste cooking oil was purchased from the local eatery joint and commercially available [BMIM][OAc] ionic liquid and other chemicals such as Isopropyl alcohol, phenolphthalein, Sodium Hydroxide (NaOH), Potassium Hydroxide (KOH), methanol were purchased from local vendors.

2.2 Characterization of waste cooking oil

Waste cooking oil is the left out oil after frying food. It is a glycerol ester which contains different types of important fatty acids. Since waste cooking oil contains waste in the form of grease and fats it is not good for the consumer as well to the environment.

Taking the waste cooking oil, we can use five parameters on which we can conclude the characteristics of waste cooking oil to be used for biodiesel production and the five parameters are : kinematic viscosity, saponification , flash point , moisture content and free fatty acid value.

2.2.1 Kinematic viscosity

The value of kinematic viscosity of waste cooking oil was found to be in the range of 39.56-45.45, which is around 40-50 times more than that of kinematic viscosity required for synthesis of biodiesel according to ASTM D6751 which is around 1.9-6.0 mm²/s, because of the high value of biodiesel it will cause incomplete combustion in the combustion chamber that will effect the engine's operation.

2.2.2 Saponification

The saponification value for waste cooking oil was found to be in between 199-201mgKOH/g. The presence of free fatty acid in the oil is the reason for the occurrence of saponification. The

higher the value of free fatty acid the more the value for saponification and this high value of saponification will affect the production of biodiesel.

2.2.3 Flash point

It is the minimum value at which enough vapours are produced from the fuel and this leads to the ignition i.e. flame generation. Flash point value is useful in predicting if there is any fire hazard during storage, use and transport. The value of flash point of sample of waste cooking oil was 199-205°C. According to the ASTM D6751 the value for that for biodiesel is 130°C. The high flash point value in case of waste cooking oil is because of the exposure with high heat during the food preparation process.

2.2.4 Moisture content

0.050% is the maximum percentage according to the ASTM D6751 for the production of biodiesel but the moisture content was found to be 0.15% for the sample of waste cooking oil. The presence of water in the oil has the bad effect on the production of biodiesel as it will cause hydrolysis and also low yield of biodiesel.

2.2.5 Free fatty acid value

We can find the presence of free fatty acid (chemical molecule present in waste cooking oil) by the colour change during titration. The value for free fatty acid in sample oil was 4.051% while the maximum value for biodiesel is 0.05 mg KOH.

High free fatty acid value will cause low biodiesel yield because of saponification, it will also leads to high viscosity.

Parameters	Waste cooking oil	Biodiesel
Kinematic viscosity	39.56- 45.45,	1.9-6.0 mm ² /s
Saponification	199-201mgKOH/g	179-190mgKOH/g
Flash point	199-205°C	130°C
Moisture content	0.15%	0.050%

Free fatty acid	4.051%	0.05 mg KOH
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Table 2 - parameters defining waste cooking oil and biodiesel

Since from above characterisation we can see that the value of 5 parameters kinematic viscosity, saponification, flash point, moisture content and free fatty acids are higher than that one need for the biodiesel according to the ASTM D6751, so to reduce these values we need to do transesterification which will convert the waste cooking oil sample into biodiesel.

2.3 Acid value :-

It is the number of milligrams of potassium hydroxide which is required to neutralize the free fatty acids present in one gram of fat.

2.3.1 Significance of acid value calculation :-

- Acid value calculation provides information about the quality of oil used.
- It measures the FFAs content (free fatty acid's content) in the oil. FFA value will be high if the oil is too old or it is of poor quality (it starts degrading).
- From Acid value, we can determine whether pretreatment (esterification) is needed or not.
- It is important to calculate because high content of FFAs can disturb the process of transesterification process, so it is necessary to reduce the FFAs content.
- It ensures high yield of biodiesel and efficient production.

2.3.2 Procedure :-

1. we took 1g of oil in the beaker and added 10ml of isopropyl alcohol into it.
2. Then we added 1 drop of phenolphthalein to it.
3. We mixed the solution with glass rod and the solution turned out colourless
4. For titration , we filled the burette with N/10 NaOH solution.

5. Then we titrated the solution by adding drops of NaOH solution filled in the burette.
6. After the completion of titration, the solution became pinkish in colour.
7. Burette reading was noted down.

2.3.3 Calculated acid value for waste cooking oil :-

<u>Initial value</u>	<u>Final value</u>	<u>Value used</u>
20	20.5	0.5
20.5	21	0.5
21	21.3	0.3

Table -3 : Acid value calculation

Calculation-

Acid value = $56.1 \times 1/10 \times \text{volume of NaOH value used} / \text{weight of oil used}$

Volume of NaOH = 0.5ml (burette reading)

Weight of waste cooking oil used =1gm

Acid value = $56.1 \times 1/10 \times 0.5/1$

Acid value of waste cooking oil = 2.805

So the **FFA value** = Acid value / 2

= $2.805/2 = 1.4025$ FFA in waste cooking oil.

FFA content should be within 2 for any oil which is used for making biodiesel. If this value comes greater than 2 then it should be brought down to less than or equal to 2 with the help of esterification process. The calculated value came out to be 1.4025 (less than 2), so there was no need for esterification process. Direct transesterification process was done.

2.4 Procedure used for transesterification

For transesterification we used waste cooking oil, which we collected from the food shop and [BMIM][OAc] Ionic liquid as catalyst and followed the following procedure :-

1. 100ml of waste cooking oil was taken in the beaker.
2. we kept it at 60° C on hot plate.
3. Measured 0.5gm of KOH was dissolved in 20ml of methanol in separate beaker. We mixed alcohol with catalyst and then we mixed it with waste cooking oil.
4. The solution was Kept stirring at constant temperature for 1- 2 hours (60° C).
5. Then we checked whether the reaction proceeded or not.
6. After the transesterification reaction, we waited for glycerol to settle down to bottom. This happens because glycerol is heavier than biodiesel and lower solubility of glycerol in ester.
7. The setting began immediately, but the mixture was left for minimum eight hours (preferably 12 hrs) to make sure all of the glycerol was settled out. Glycerol was very dark and highly viscous while biodiesel looks yellow and low viscous. The glycerol volume should be approximately 20% of the original oil volume.
8. Glycerol was removed by separating funnel and biodiesel was washed with distilled water (2-3 times).
9. we dried biodiesel at 500 degree Celsius for 3-4 hr to remove water molecules from it.
10. we then stored the biodiesel in glass bottle or container.
11. Finally, the methyl ester layer (biodiesel) was kept for purification and assessment.



For transesterification we used both the catalysts i.e. acidic as well as basic catalyst but the yield was too low. This made us to use the ionic liquid and we therefore used the commercial ionic liquid for the production of biodiesel following the process given above.

2.5 Comparison of properties of diesel and biodiesel produced from the waste cooking oil :-

Properties	Units	diesel	biodiesel
Density	g/ ml	0.84	0.87
Kinematic viscosity at 40°C	mm ² /s	3.5	5.2
Calorific value	KJ/ Kg	43472	39127
Acid value	mg KOH/ g oil	<0.8	0.44
Flash point	°C	110	188.5
Ash content	%	0.01	0.01

Table-4 : comparison of properties of biodiesel and diesel

CHAPTER -3

CONCLUSION AND FUTURE SCOPES

3.1. Conclusion:-

This review involves into the current and prospective outlook of biodiesel. Furthermore, various ionic liquids and renewable feedstocks engaged in biodiesel production have been explored. Utilization of diverse ionic liquids in catalytic systems leads to a significant increase in biodiesel yield. Ionic liquids are exceptional in terms of their recyclability, dissolving capacity, high chemical stability, selectivity, and non-flammability, making them more efficient than conventional heterogeneous and homogeneous catalysts. This is why ionic liquids are referred to as "new generation catalysts." Ionic liquids find diverse applications, including use as lubricants, additives, electrolytes, solvents, catalysts, and more. Various raw materials such as rapeseed oil, waste cooking oil, soybean oil, jatropha oil, castor oil, tung oil, etc., are employed in the biodiesel preparation process using different ionic liquids. Numerous imidazolium-based ionic liquids have been reported to be highly efficient. Additionally, ionic liquids functionalized with Brønsted and Lewis acid sites have been reported to have numerous applications. Various methods for producing biodiesel have been investigated, such as transesterification, pyrolysis, intensification techniques, reactive distillation, and others. Ionic liquids possess the characteristic of recyclability or reusability. Therefore, after the reaction is finished, they can be easily recovered using various separation techniques such as distillation, crystallization, etc., and subsequently reused.

3.2 Future scopes :-

We can analyse from the literature survey that there is increase of about 75% in efficiency of the process by using ionic liquid. Ionic liquid could help to twice the yield of biodiesel rather than the traditional catalysts that were used. Researchers are continuously trying to explore new catalysts and methods or techniques to improve the process of production. Advancements in biodiesel production aim to enhance the efficiency and reduce the impact on environment.

Using waste cooking oil helps in reducing waste disposal issues and also provides feedstock of low cost.

Also to optimize the reaction conditions, synthesis pathways, there is need for continuous researches so that the process of production of biodiesel becomes commercially viable. Many policies are introduced by many countries to encourage people about the use of biofuels. Researchers are more focussed on enhancing conversion , improving the fuel quality, alternations in development of catalysts and importantly in the collection of waste cooking oil. These will take the biofuel industries to an another level and will help in solving many fuel related problems. Jobs creation can also be done by biofuel industries in different fields like waste oil collection , in production and distribution of biodiesel. So these provide help in job opportunities.

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