



From Principles to Practice: Recent Developments in Greener and Sustainable Organic Synthesis

Keshav Kumar Saini¹, Vandana Saini², Ravi Kant^{3,*}

¹Department of Chemistry, Government Post Graduate College, Noida, G.B. Nagar, UP 201301, India

²Department of Chemistry, Dyal Singh College, University of Delhi, New Delhi, India

³Department of Chemistry, Raj Rishi College, Alwar, Rajasthan

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ABSTRACT:

Green chemistry addresses synthesis processes in accordance with its traditional 12 principles, which help to make chemical processes more sustainable, save energy, reduce the toxic effects of reagents and end-products, lessen harm to humans and the environment, lower the risk of global warming and use renewable resources and waste from agriculture more wisely. Current developments in the environmentally friendly synthesis and production of inorganic, organic and coordination materials, compounds, nanomaterials, hybrids and nanocomposites are examined. Both widely used chemical compounds and whole novel materials have been synthesized using more environmentally friendly methods. This review includes a comprehensive discussion of the latest advances in the more environmentally friendly synthesis of inorganic, organic and coordination chemicals, materials, nanomaterials, hybrids and nanocomposites using green solvents like water and ionic liquids.

1. Introduction

In today's world, synthetic chemicals are used in everything from plastics to skincare items, electrical devices to toys, autos to construction materials, cosmetics, *etc.* [1,2]. Every industry is using a vast number of chemicals and leaving behind harmful products in the environment, which leads to serious environmental pollution [3]. The environment is being strained by the increasing use of synthetic chemicals in a wide range of industries that use the products, which is primarily accountable for climate change, potentially dangerous pollution and plastic waste issues that endanger the health of people and the ecosystem [4]. Since the 1950s, the use of synthetic chemicals has grown 60 times and by 2050, it is predicted to have quadrupled [5]. In the 1960s, these appalling figures ultimately sparked global alarm, which resulted in the 1990 implementation of the US Pollution Prevention Act [6]. The newly adopted rule prioritized pollution prevention by chemical industry researchers, more commonly known as "Green Chemistry," and compelled the business to develop ways that limit emissions and waste produced and released to the environment [7]. Reduced solvent use, switching to aqueous conditions, catalytic variations, microwave irradiation, ultrasound facilitation and photo-

chemical pathways are some of the techniques used in green chemistry, which refers to the design of chemical compounds and procedures that minimise or completely eradicate the production of potentially toxic chemicals [8,9]. The objective of green solvent research is to minimise environmental damage resulting from the use of hazardous solvents in organic chemical research. As a consequence, in recent decades, various solvent-free techniques and improved recycling protocols have been developed. However, these methods have their drawbacks. As a result, the writers examine several eco-friendly solvent alternatives. Reactions employing water alongside ionic liquids (ILs) in place of traditional organic solvents are highlighted in this review paper.

Green Chemistry framework

The following is an outline of the three primary points of the Green Chemistry framework:

Across the entire chemical life cycle, green chemistry strategies are implemented [10,11].

1. Throughout the entire chemical life cycle, green chemistry designs are implemented.



2. The practice of green chemistry strives for decreasing the inherent hazards associated with chemical processes and products by creating substitutes.

3. Green chemistry functions as a unified set of standards for design or principles [12,13].

Green Chemistry twelve principles

Paul Anastas coined the phrase "green chemistry" and outlined the twelve guidelines for implementing this seemingly novel approach in chemistry [14]. These guidelines mostly pertain to the investigation of synthetic chemistry [12-14].

1 Waste Prevention: Preventing waste is the very first of the 12 green chemistry principles. Cutting back on waste creation is better than cleaning it up afterwards it has been produced [18].

2. Atom economy: The Atom Economy principle and waste prevention are naturally related since it calls for all raw reagent utilized in synthesis or fabrication to be used as efficiently as possible or included in the finished product in order to reduce waste. Therefore, it is important to plan chemical synthesis so that the end product uses the rawest materials possible or creates synthetic products that use all of the elements needed for synthesis [19].

3. Low harmful Chemical Syntheses: Synthetic processes should, if possible, be developed to employ and produce materials with low or zero toxicity to the environment and human health [20].

4. Developing materials that are safer: Chemical items ought to be made as non-toxic as possible while still accomplishing their intended purpose. Designing safer goods and processes can involve many challenges, one of which is minimising toxicity while preserving function and efficiency. To achieve this aim, one must comprehend not just chemistry but also environmental science and toxicological principles [21].

5. Safer solvent: If at all possible, solvents required for synthesis ought to be used sparingly and without harm. Environmental contamination makes chromatographic separations, which require significant amounts of solvents, challenging. The majority of traditional organic solvents are corrosive, poisonous and combustible. The creation of environmentally friendly solvents is required because their recycling is associated with energy-efficient distillation that results in significant losses [22].

6. Energy efficient design: Energy needs should be reduced because they have an influence on the economy and the environment. It is best to use synthetic procedures at room temperature and pressure [23].

7. Use of renewable materials-According to this green chemistry principle, where technically and financially possible, a feedstock or raw material ought to be reusable rather than diminishing [24].

8. Minimise derivatives: Avoid or reduce unnecessary derivatization (application of blocking groups, protection/deprotection and temporary changes to physical/chemical processes) as much as feasible because these procedures can produce waste and call for extra reagents.

9. Selection of catalysis: Stoichiometric reagents are inferior to catalytic materials (as selective as feasible). The catalysis principle encourages the adoption of biodegradable catalytic agents to safeguard the environment, which means using less energy, avoiding organochlorine compounds and using less water or wastewater.

10. Design for Degradation: Chemicals and other substances should be made to decompose into harmless degradation products at the end of their useful lives and not linger in the environment.

11. Analysis in Real Time to Prevent Pollution: To enable real-time, in-process monitoring and control before hazardous chemicals form, analytical techniques must be improved.

12. Safer Chemistry by Nature to Prevent Disasters: Chemical accidents, such as leaks, explosions and fires, can be reduced by selecting the right substances and their forms for the chemical process.

In the production and use of chemical products, green chemistry efficiently uses (ideally renewable) raw materials, gets rid of waste and stays away from using hazardous and/or poisonous reagents and solvents [25]. Using representative examples, the current review aims to demonstrate not only the wide range of reactions that can be carried out in water, without water, or using micro-waves but also how the reactions in water or without water can result in additional sustainability benefits that improve a process's overall environmental impact.

Green synthesis in water as a green solvent

Water has lots of distinctive chemical and physical characteristics, such as broad ranges of temperatures where it

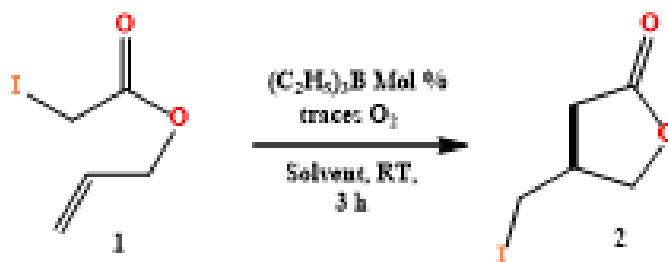


retains as liquid, a high heat capacity, highest dielectric constant, profound hydrogen bonding and an optimal oxygen solubility for aquatic life [26,27]. Water is able to influence the selectivity and accelerate rate of reactions several different kinds of chemical reactions [28].

The following are some recent developments in green synthesis using water as a solvent.

Oshima and colleagues [29] utilized the iodine transfer cyclization of α -iodoacetates for investigating the synthesis of metal-free bonds composed of carbon and carbon (Scheme-I). when triethylborane used as a free radicals

initiator at room temperature (r.t.), was unable to synthesis of the lactone in various organic solvents, including dichloromethane, dichloromethane, benzene and hexane [29]. Since poor yields were achieved in other solvents that are polar such alcohols, dimethylformamide (DMF), or dimethyl sulfoxide (DMSO), acetonitrile (ACN) the product had been produced under the same conditions with an excellent 78% yield in water. remarkably, large and medium-sized rings containing compounds (contain up to an 18-membered ring) had been synthesized successfully under these conditions.



S. No.	Solvent	% Yield
1	Acetonitrile	13
2.	Benzene	<1
3.	Dichloromethane	<1
4.	Dimethyl formamide	13
5.	Dimethyl sulfoxide	37
6.	Ethanol	3
7.	Hexane	<1
8.	Methanol	6
9.	THF	<1
10.	Water	78

Scheme-I: Solvent assisted synthesis of lactones

Novartis *et. al.* [30] demonstrates that in certain situations, water can increase yield by decreasing or completely removing side reactions in addition to speeding up the chemical reaction. they utilized this approach for synthesis of 1-substituted-4-cyano-1,2,3-triazoles from 2-chloroacrylonitrile and organic azides at the industrial scale. The biggest challenge in this transformation lies in the fact 2-chloroacrylonitrile is known to polymerase in both basic and acidic solutions. The 1,3-dipolar cycloaddition proceeds by an aromatization, which results in formation of hydrogen chloride as a by-product in organic solvents

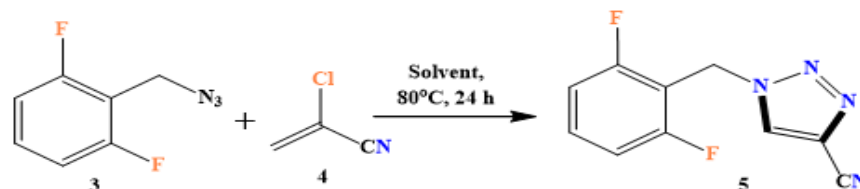
(Scheme-II). This reaction required use of an ample dilution or excess of this reagent to achieve good yield of the product because the hydrogen chloride that produces during the reaction increases the acidity of the reaction solution, which encouraging the polymerization of the olefin and reduced the yield of the desire product. Water proved to be a very practical and environmentally friendly substitute in this reaction. since it allowed the reaction to occur into the organic phase whereas the hydrogen chloride that generated was remains dissolved in the aqueous phase, preventing the alkene's polymerization.



Engberts *et al.* reported in their scientific study of the Diels–Alder reaction involving 3-aryl-1-(2-pyridyl)-2-propen-1-ones and cyclopentadiene (**Scheme-III**) [21]. They discovered that when compared to acetonitrile, the process conducted in water as a solvent was 287 times far more quickly. They also reported that, in comparison to the reaction in acetonitrile, the reaction in water, when Lewis acid and micellar catalysis were used, the rate of reaction was by a proportion of 1,800,000.

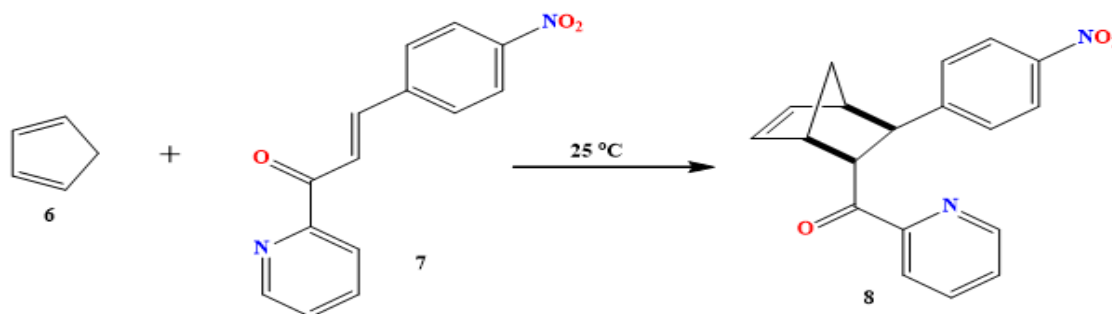
Sharpless *et al.* describe the in water reaction conditions are the reactions in which water is used as the solvent for the chemical reaction of reactants that are insoluble in water [31]. Furthermore, the research team reported a highly illustrative an example of an increase in the rate of the in water reaction involving the reaction of dimethyl azodicarboxylate and quadricyclane (**Scheme-**

IV). In number of different solvents, the time required for reaction completion was measured and it was observed that not only hydrogen bonding and dipolar effect of solvent play role in increasing the rate of reaction (18 h in C₂H₅OH as opposed to 36 h in DMSO, 72 h in DCM (dichloromethane), or more than 120 h in C₆H₅CH₃) [32]. Heterogeneity also contributed significantly to the more rate of acceleration with only a 10 min reaction time with in water as solvent. whereas, it takes 48 h for the reaction carried out in solventless condition. whereas, it takes 48 h for the reaction carried out in solventless condition. It is worthwhile to observe that the reaction completion time increases by 45 min when heavy water (D₂O) was taken as the solvent. This might have associated with the hydrophobic effects of solvent and the viscosity was higher, which obstructed the heterogeneous mixture from the mixing well.



S. No.	Solvent	% Yield
1	Dimethylformamide	78
2	Ethanol	40
3	<i>n</i> -Heptane	46
4	Neat	72
5	Toluene	51
6	Water	98

Scheme-II: Beneficial use of water in the synthesis of triazole from azides and 2-chloroacrylonitrile



S. No.	Conditions	K _{rel}
1	Acetonitrile	1
2	Water	287
3	Cu(OSO ₃ C ₁₂ H ₂₅) ₂ 2.4 mM, water	1.8 × 10 ⁶

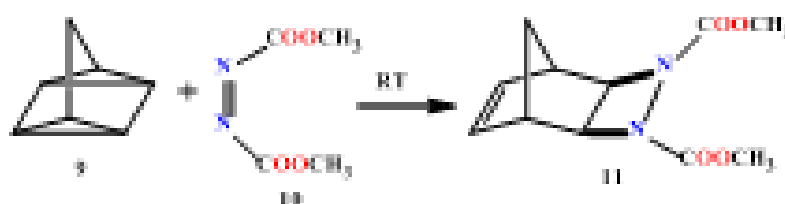
Scheme-III: Joint influence of water and catalysis in the rate increase of a Diels–Alder reaction



Kobayashi *et al.* [33] investigated the chiral scandium complex-catalyzed asymmetrical desymmetrization of meso-epoxides using amines. They reported that in contrast to dichloromethane or THF/water mixtures, they revealed that the chemical reaction of aromatic epoxy compounds with anilines derivatives resulted in an excellent yield of enantiomeric excess in water solvent (**Scheme-V**). These conditions were effectively used with a wide range of

reactants, although the reaction is restricted to aromatic amines. Furthermore, using scandium tris(dodecylsulfate) rather than scandium triflate afforded a greater yield.

Chaskar *et al.* [34] discovered that 3,4-dihydropyrimidine-2(1H)-ones (DHPMs) may be synthesized from an aldehyde, urea/thiourea and β -keto esters using iodoxy benzoic acid (IBX) (**Scheme-VI**). It was believed that IBX

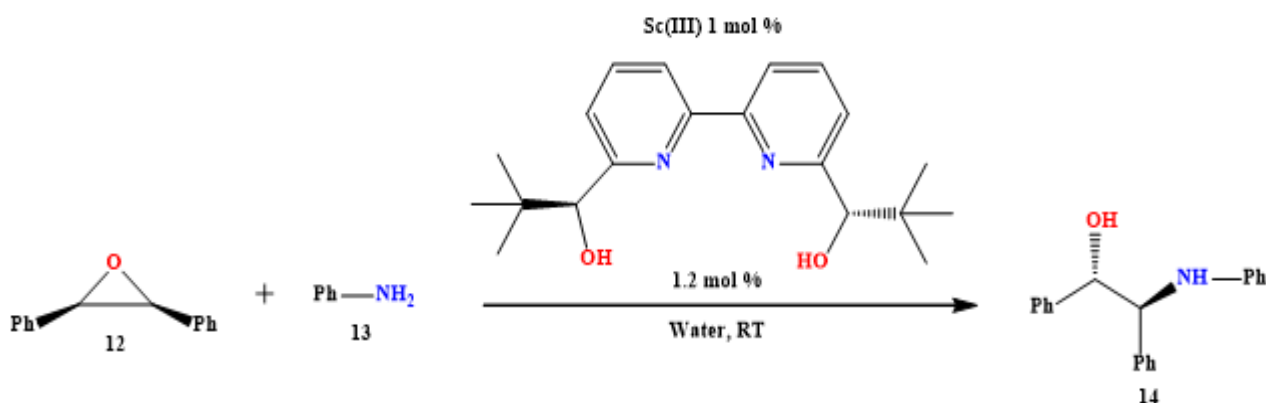


S. No.	Solvent used	Reaction time
1.	Acetonitrile	84 h
2.	Dichloromethane	72 h
3.	Dimethyl sulfoxide	36 h
4.	D ₂ O	45 min
5.	Ethyl acetate	120 h
6.	Methanol	18 h
7.	Neat	48 h
8.	Perfluoro hexane	36 h
9.	Toluene	120 h
10.	Water	10 min

Scheme-IV: Time to completion for the cycloaddition of quadricyclane with azodicarboxylate

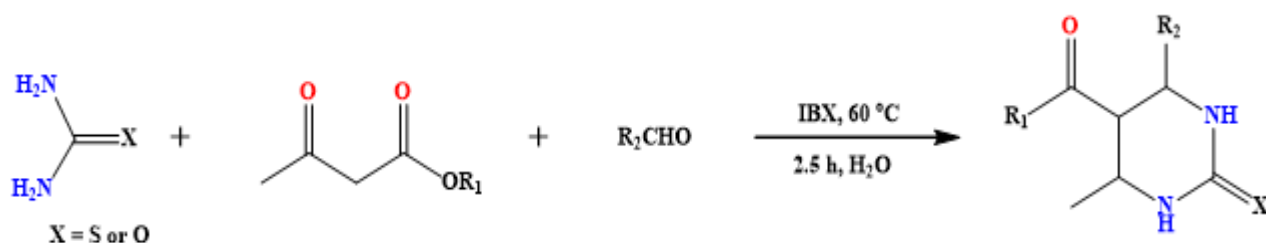
would expedite the production of iminium by activating both ketones and aldehydes. Results were not as good when using different basic catalysts like such as 1,4-diazabicyclo[2,2,2]octane (DABCO). Water was found

to be the most efficient solvent. Additionally, approximately sixty percent of the total IBX could be retrieved to be utilized again without losing its activity for the subsequent reaction cycles.



S. No.	Solvent	Sc(III)	% Yield	ee (%)
1	Dichloromethane	Sc(OTf) ₃	85	74
2.	THF/Water (9:1)	Sc(OTf) ₃	<5	71
3.	Water	Sc(OTf) ₃	15	85
4.	Water	Sc(OSO ₃ C ₁₂ H ₂₅) ₃	89	91

Scheme-V: Effect of the solvent on the scandium-catalyzed asymmetric desymmetrization of epoxides



Scheme-VI: 3,4-Dihydro-2-(1H)-one Synthesis Catalyzed by IBX in water

Green synthesis in Ionic liquids as a green solvent

Ionic liquids were previously known to only a small number of academics and researchers over the past 20 years [35]. At the present time, ionic liquids have been recognized as highly promising, potent and environmentally friendly green solvents [36]. Due to their comparatively low viscosity and vapour pressure, elevated thermal and chemical robustness, high electrical conductivity, wide electrochemical window and high solubility of organic, inorganic and polymeric materials and gases, ionic liquids have garnered growing attention and been effectively employed in a variety of reactions, such as environmentally friendly solvents and catalysts, over the past few years [37]. One appealing aspect of ionic liquids is their easily adjustable solubilities, which allow them to phase separate from both organic and aqueous environments based on the selection of cations and anions [38]. One can also adjust substrate solubility. Ionic liquids are

made up of larger organic cations and smaller anions, which determine their solvating characteristics [39]. Anions include things like formate, hexafluorophosphate, acetate, halides and benzoate. Anions of tetrafluoroborate, nitrate, methanesulfonate, sulphuric acid, tosylate, phosphate and trifluoromethanesulfonate [40].

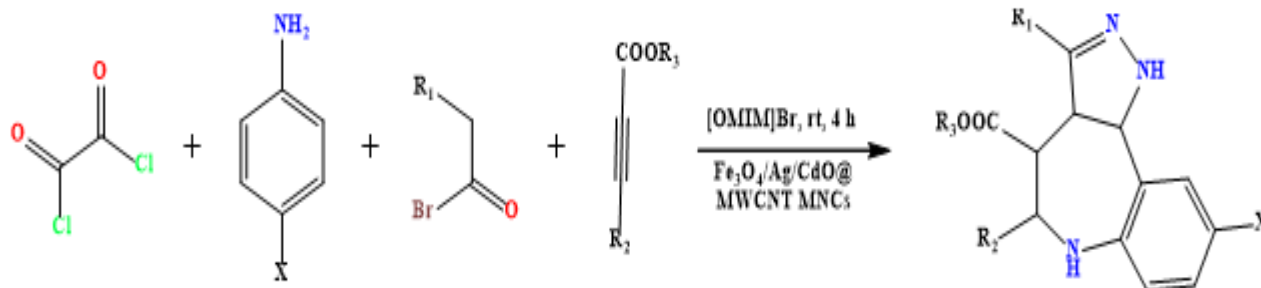
The following are some recent developments in green synthesis using water as a solvent.

Kadhim and *et. al.* [41] used ionic liquids [OMIM]Br as solvent and magnetic nanocomposite Ag/Fe₃O₄/CdO@MWCNTs as catalyst in the one pot multicomponent reaction between oxalyl chloride, aniline, alkyl bromides, hydrazine and activated acetylene to prepare produce benzoazepine derivatives at r.t. (room temperature). With a yield of up to 97%, this catalyst speeds up the reaction quickly. Ag/Fe₃O₄/CdO@MWCNTs were chosen for organic reactions because of the significant characteristics of Fe₃O₄ and CdO-NPs. Fe₃O₄ magnetized nanopar-

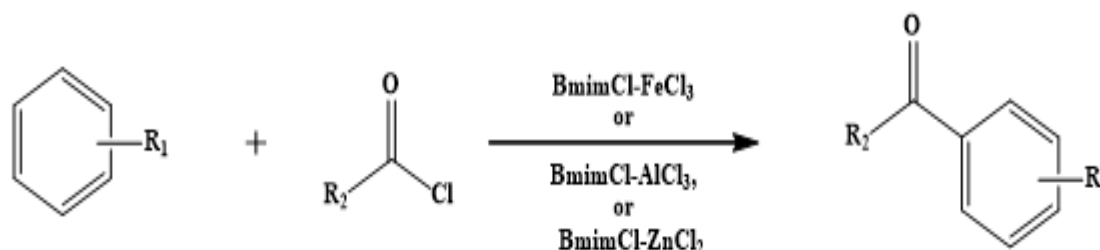


ticles (MNPs) have garnered a lot of attention due to their easy recovery from the resulting reaction mixture. While

CdO-NPs absorb visible light, they do not recombine as many electron-hole pairs as CuO NPs do (**Scheme-VII**).



Scheme-VII: Azepine derivative synthesis using multiple component reaction



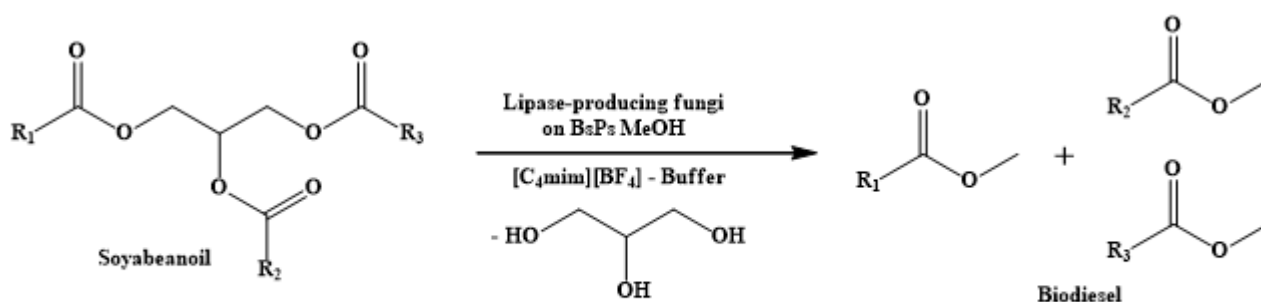
Scheme-VIII: Synthesis of benzophenone and its derivatives by Friedel-Crafts acylation using BmimCl-FeCl₃, BmimCl-AlCl₃, and BmimCl-ZnCl₂ ionic liquid

Changzhi Li *et al.* [42] reported that Ionic liquids (ILs) comprising BmimCl-FeCl₃, BmimCl-AlCl₃ and BmimCl-ZnCl₂ were used as dual catalyst-solvents in Friedel-Crafts acylation processes to produce benzophenone and its derivatives. Compared to the other two ILs and standard organic solvents, BmimCl-FeCl₃ exhibited much greater catalytic activity. Good to exceptional yields of acylation products (up to 97%) were achieved using these reaction systems in a brief reaction time. Higher yields were obtained in a shorter reaction time when using solid catalysts such GaCl₃/kaolin, as opposed to solventless reaction of benzoyl chloride with benzene. Moreover, hydrolysis of the product and neutralization of the reaction mixture are necessary for solution-based processes employing Lewis acids such as AlCl₃ and FeCl₃; in the IL, the product was only extracted with cyclohexane, greatly simplifying the work-up (**Scheme-VIII**).

Shogo Arai *et al.* [43] used fungal entire-cell bio catalysts in ionic liquid to show the methane degradation of soybean oil. Due to lipase's innate positional specificity,

even though w-ROL had the maximum activity, a single application of w-ROL did not produce all of the ME. The combination of w-ROL and r-mdIB produced a high ME concentration. Despite w-ROL's deactivation in ILs (ionic liquid) [Bmim][BF₄]. They reported that the combination of two whole-cell biocatalyst types, w-ROL and r-mdIB, resulted in high ME conversion. After being incubated in [Bmim][BF₄] after a period of 72 h, the function of w-ROL decreased to a third of its initial value in a stability test (**Scheme-IX**).

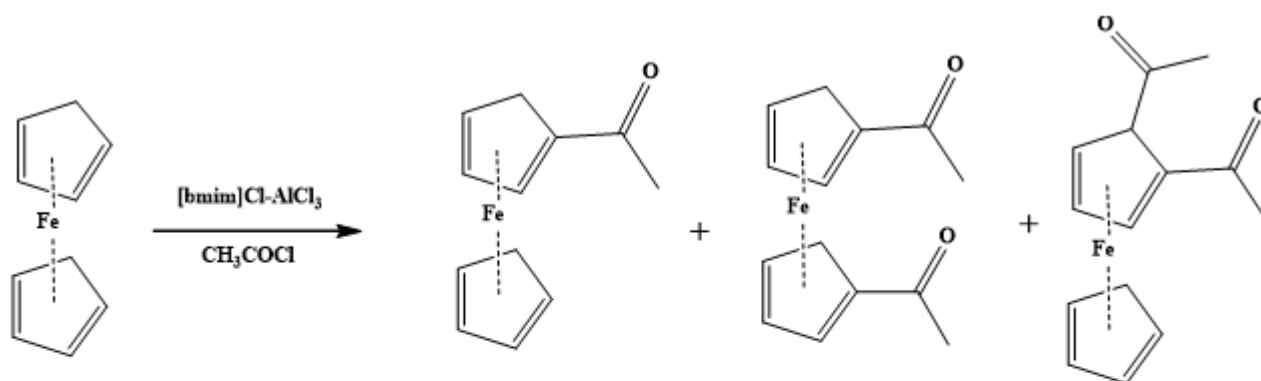
Arwa Sultan Alqahtani *et al.* [44] reported that acidic chloroaluminate ionic liquids have also been used to acylate ferrocene. Ferrocene combines with acylating reagents (RCO)₂O and RCOCl (where R = Me, Ph, Pr, *n*-Bu and *t*-Bu) in ionic liquid [emim]I±AlCl₃ to produce mono-substituted and 1,1'-bis-substituted compounds, A further product, 1,2-diethanoylferrocene, is isolated under somewhat different circumstances employing acidic [bmim]Cl±AlCl₃ and ethanoyl chloride as the acylating reagent (**Scheme-X**).



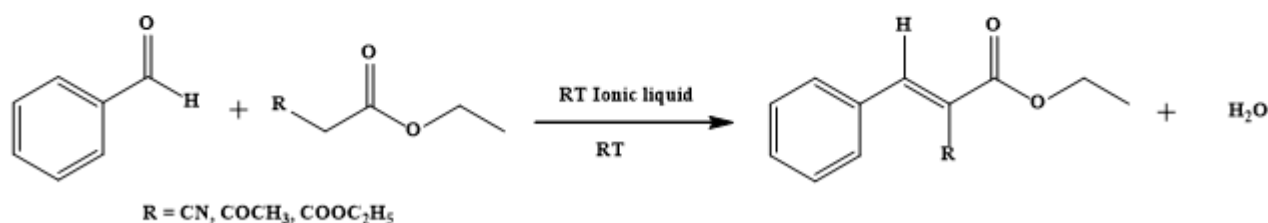
Scheme-IX: The methane degradation of soybean oil using [Bmim][BF₄] ionic liquid

E.J. García-Suárez *et al.* [45] reported a cheap, environmentally friendly method for synthesis of new IL (ionic liquid) at room temperature from readily available, natural feedstocks (amino acids and choline hydroxide), with the only byproduct being water. They produced α,β -unsaturated carbonyl compounds with good transforma-

tions and high selectivities when used as catalysts in the Knoevenagel testing reaction among benzaldehyde and several active methylene compounds at room temperature and without the need for a solvent. Cholinium and aminoacetate ions' catalytic function in the Knoevenagel condensation is examined (**Scheme-XI**).



Scheme-X: Acylate ferrocene using ionic liquid as solvent



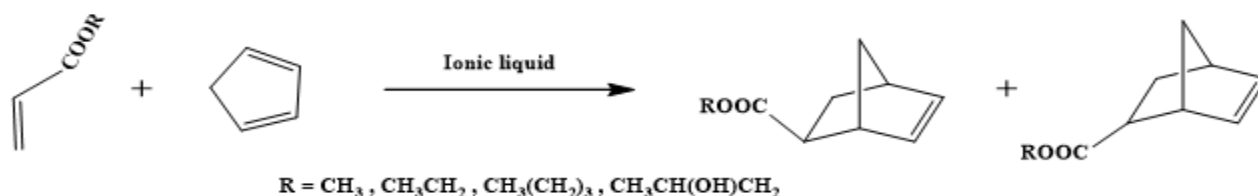
Scheme-XI: Condensation between benzaldehyde and different active methylene compounds using ionic liquids by Knoevenagel condensation reaction

Waldemar Stefaniak *et al.* [46] investigated that the Diels-Alder reaction between cyclopentadiene and alkyl esters of acrylic acid has been mediated by ionic liquids made of 1-(n-butyl)-1-methylpyrrolidinium cation and anions like bis(trifluoromethylsulfonyl)imide [NTf₂], trifluoro-

methanesulfonate [OTf] and tris(pentafluoroethyl)trifluorophosphate [FAP]. In the absence of the catalyst and with ionic liquids present, the reactions take place at ambient temperature.



They also demonstrated that the type of dienophile and the characteristics associated with the counter-anion in the ionic liquid determine the transformation and stereo-selectivity of the reaction. The reactivity of the acrylates utilized for these syntheses was comparable in the liquids containing [NTf₂] and [FAP] anions, according to a comparison (**Scheme-XII**).



Scheme-XII: Diels-Alder reaction between cyclopentadiene and alkyl esters of acrylic acid in the presence of ionic liquids

Conclusion

The utilization of sustainable chemical-based methods has grown substantially over the last ten years. However, there are still plenty of challenges to overcome, particularly in the widespread adoption of sustainable technology in the chemical industry. One prospective approach that has surfaced is the application of Engineered surfactants to assist the chemistry of water. In a chemical reaction's process mixture, the solvent may be the most underappreciated element. Its primary role in the past has been to fill the response space, enabling chemical engineering processes such as heating, mixing and separation. Solubility is a key objective, as a solvent must dissolve reactants, often at high or peak concentrations. Solvents must be inexpensive and usually widely available. The ability of solvents to be recycled has grown in significance as environmental consciousness has increased. The use of "green solvents", including acetone, esters (ethyl acetate), alcohols and water as solvents, has become more popular over the past ten years.

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