Advance of glucose conversion to 5-hydroxymethylfurfural using ionic liquid: mini review

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Abstract. Biomass has been used as a raw material to replace fossil fuel due to its abundance and renewability in recent years. The majority of biomass consists of carbohydrates such as glucose and fructose. The conversion of glucose into its derivative (i.e., 5-hydroxymethylfurfural (HMF)) opens several spaces to be studied. However, the yield of HMF is still low due to its ease of degradation into by-products and the difficulty of product separation. Therefore, ionic liquid (IL) is used as a solvent or media to avoid HMF degradation, which has high thermal stability, low toxicity, an un-flammable, wide range of solubility, physical and chemical stability. In addition, IL is recyclable, has good performance at dissolving biomass, and can act as a catalyst. Many important aspects of glucose conversion into HMF in IL as a media and catalyst can be deeply developed and investigated regarding conversion yield, which is affected by different ILs used, and conversion time. This review paper will discuss a detailed summary of the development of glucose conversion into HMF using IL.

1. Introduction
The depletion of fossil fuel reserves and their emissions give environmental impact, therefore require lawmakers to prioritize renewable green energy [1]–[3]. One of the material alternatives that can be used to produce energy is biomass. Biomass is thought to be suitable as an energy source as it is abundant, renewable, and considered to be carbon-neutral [4]. The main compounds of biomass are lignocellulose, hemicellulose, and lignin. As well known that carbohydrate derivatives of biomass are glucose and fructose. Glucose can be converted into many kinds of chemicals in organic solvent with or without catalyst and using a conventional method, one of the high potential chemicals is 5-hydroxymethylfurfural (HMF) [5]. HMF has many applications, i.e., fuels, flavors, fragrances, solvents, pharmaceuticals, agrochemicals, and polymers [6]. A few of HMF’s derivatives are levulinic acid (LA), ethyl levulinate (EL), 2-methyltetrahydrofuran (MTHF), 2,5-dimethyltetrahydrofuran (DMTHF), 2,5-dihydroxy-methylfuran (DHMF), and 2,5-furandicarboxylic acid (FDCA) [7], all of which are presented in figure 1.
LA can be processed further into acetyl acrylates or acetic-acrylic succinic acid used in copolymerization, diphenolic acid to replace bisphenol A in polycarbonate synthesis, or MTHF that can be used as fuel oxygenates or solvents [5]. EL can be used as a fuel additive in biodiesel [8]. MTHF can replace tetrahydrofuran (THF) as a solvent, substitute of gasoline, or gasoline blend [9]. DMTHF can be another choice of biofuel as it has similar properties to gasoline [10]. DHMF is utilized to produce many things such as polymers, drugs, resins, etc. [11]. 2,5-Bis(hydroxymethyl)furan can be used to produce polyurethane foams or polyesters [12]. FDCA is used to substitute terephthalic acid in polyesters synthesis [13]. Traditional solvents used in industrial processes are organic solvents. In a previous study, glucose is converted into HMF in DMF using H+T-Amberlyst-15 as a catalyst at 100 °C for 180 minutes with a yield of HMF around 42.3% [14]. Yang et al. converted mainly glucose and fructose in different types of organic solvents (i.e., DMF, DMSO, DMA, and n-butyl alcohol) and used various catalysts [15].

Nowadays, the technology used in biomass conversion product separation is categorized into extraction method, distillation method, adsorption method, and filtration method (membrane technology) [16]–[20]. The extraction method can be further categorized into traditional extraction, water extraction, in-situ extraction, supercritical CO2 (ScCO2) extraction, and deep eutectic solvent (DES) extraction [21]. With the extraction method, different reaction systems will need different extracting agents [21]. In traditional extraction in the aqueous system, MIBK with a high HMF transfer rate and MTHF with a high load of HMF is widely used in HMF extraction [22]. However, both of them are needed in large quantities, which means high cost. In-situ extraction is usually combined with a biphasic system. In a biphasic system, biomass conversion occurs first, and then the product is transferred into extracting agents. Its benefit is the suppression of HMF decomposition and polymerization while improving HMF yield and selectivity [23]. Through the distillation method, indirect distillation to separate HMF from its reaction system is feasible with some modifications [21]. Another study conducted by Hyung et al. uses nanofiltration and electrodialysis for separate HMF, Levulinic acid (LA) from hydrolysate agarose [24]. In that work, separation by nanofiltration can remove 62% LA and 91% HMF. Though filtration fails to give complete separation, it is still a greener method than liquid-liquid extraction. In addition, the performance of filtration can be enhanced by fabricating specialized membranes [17],[25],[26].

The literature review shows that yield of HMF is relatively low under 50% used organic solvent as media. It can be reasoned that the reaction partly happened, and the separation is not optimal in the organic solvent. On the other hand, the organic solvent is many disadvantages if used in glucose conversion industrially, such as flammable, volatile, high volume usage, high operational cost, and high...
toxicity impact [27]–[30]. Moreover, they give low HMF yield while adding additional challenges at product separation, regenerating, and recycling organic solvent [27]. The difficulty regarding product separation is tied to low solubility in the aqueous phase, resulting in low HMF yield [27]. Based on this problem, an alternative is needed to substitute organic solvent in biomass conversion, and one of the potential solutions is by using ionic liquid (IL) [29],[31].

IL is an organic or inorganic salt that possesses some interesting properties such as its low melting point of under 100 °C, non-flammable, chemical stability, high thermal stability, low vapor pressure, and environmentally friendly as either catalyst or solvent [26],[29]. Moreover, ILs can be tuned to comply with a particular task by changing their cation and anion combination [32],[33]. Some applications of IL include the use as a catalyst for the reaction, as corrosion inhibitors, as supporting material in membrane separation, and as media in biomass conversion [27]. In this paper, a review focused on the advances of glucose conversion into HMF is provided. It contains detailed information on converting glucose into higher-value chemicals (HMF), reaction mechanism, conversion condition, and future prospects and challenges.

2. IL for biomass conversion

In 1983 the biomass conversion was conducted using pyridinium chloride to convert fructose and glucose into HMF with a yield of around 70% [34]. Moreover, Swatloski et al. examined the potential of 1-butyl-3-methylimidazolium [BMIM+] based IL coupled with various anions such as Cl-, Br-, SCN-, [BF4]-, and [PF6]- to dissolve cellulose. They showed that 1-butyl-3-methylimidazolium chloride ([BMIM]Cl) has the capability to dissolve cellulose with 25% solubility [34]. Their work is considered one of the pioneering studies that unveil the potential of IL as a solvent and the following application in biomass conversion [35].

Regarding cellulose dissolution using ILs, we need to know what makes an IL a good solvent for cellulose. Xu et al. investigated this matter by testing the solubilities of microcrystalline cellulose (MCC) in several [BMIM+] based IL with Brønsted basic anions [36]. They concluded that the solubility of cellulose increased almost linearly with increasing hydrogen bond accepting ability of anions in the IL. The role of cation was discussed by Zhao et al. [37]. They tested various imidazolium based-based chloride IL, 1-butyl-3-methyl pyridinium chloride ([BMPy]Cl), and 1-allyl-3methyl imidazolium cation ([AMIM]+). Their results indicated that the cations' heterocyclic structure and alkyl chain length affect the interactions between anion of the IL and cellulose. In addition, the longer alkyl chain the cation has, an IL’s ability to dissolve cellulose is decreased due to an increase in steric hindrance for the anion to bind with cellulose, and the addition of electron-drawing groups in the alkyl chains of an IL will increase improve its cellulose solvation capability [37],[38].

There are two steps to transform biomass into fuels or chemicals, (1) pretreatment and (2) conversion [39]. Many kinds of catalysts and media have been used to test their performance in biomass conversion. Types of solvents used are water, organic solvents, mixed systems, and IL. The catalysts usually used in biomass conversion such as organic acid, salts, solid acid catalysts, ion-exchange resin, and zeolites [40],[41]. Additional methods can also be incorporated to increase the performance of biomass conversions, such as using microwave irradiation [42] and probe ultrasonication [43]. The conversion of mono/di/polysaccharides into furfural (FUR) or HMF in 1-ethyl-3-methylimidazolium hydrogen sulfate ([EMIM][HSO4]) was investigated. The results showed that furfural could be used to produce HMF with 88% HMF yield and 100% conversion rate at 100 °C for 0.5 hours. The same system is used to convert glucose into HMF, but the yield is very low at 3% even at the prolonged reaction time for 4 hours; the yield only goes up to 9% [44]. In [BMIM][Cl] at 100 °C for 4 hours, no glucose conversion was detected, but with the help of CrCl3, high HMF yield (91%) and conversion rate (91%) were achieved [44]. Cellulose was used in [BMIM]/CrCl3 system at 100 °C for 4 hours, the yield was 0%, but with the addition of H2SO4, the yield increased up to 9%. It was assumed that the addition of acid accelerates the hydrolysis reaction [44]. Using IL as reaction media, the conversion of biomass has been studied using many kinds of catalysts such as metal salts, mineral acids, solid acids, and zeolites. Metal salt and mineral acid are the most used catalyst as they have good catalytic properties in IL. Meanwhile, solid acid has better performance in water [45]. Several IL types usually used in biomass conversion are summarized in table 1.
3. The general condition of biomass conversion using IL and catalyst

In general, the temperature of HMF production is 100-300 °C [58]. That range needs to be lower to reduce energy consumption and avoid the degradation of biomass. For example, glucose conversion cannot be conducted at high temperatures as glucose will degrade into glycolaldehyde, pyruvaldehyde, glyceraldehyde, etc. [59]. To achieve lower reaction temperature, ILs can be used to make the conversion reaction more effective and efficient [27],[50].

Stahlberg et al. [2] investigated the catalytic performance of lanthanide catalyst in glucose conversion into HMF in ILs. In comparison, YbCl₃ and Yb(OTf)₃ performed better than the other lanthanide catalysts in both [EMIM]Cl and [BMIM]Cl. Furthermore, the effect of temperature and reaction time is discussed during glucose dehydration in [BMIM]Cl using YBCl₃ as a catalyst at 160 °C. The maximum HMF selectivity is reached after 10 minutes, and the maximum HMF yield after 30 minutes. The selectivity and yield both decreased, showing HMF degradation to humins that occurs at high temperatures. The same substrate, IL, and catalyst are used but with a higher temperature of 200 °C. It gave a quicker and higher HMF yield at 26% after less than 10 minutes. The downside of using the higher temperature is that HMF degradation happens way faster [2]. Several groups have accomplished lower reaction temperatures (<50 °C). Zhang et al. tested several metal chlorides as a catalyst and [BMIM]Cl as solvent at 80 °C, 50 °C, and lower temperature. It resulted in WCl₆ as the most promising catalyst with 53% and 42% HMF yield at 30 °C and 22 °C, respectively, for 4 hours [61]. Qi et al. also did similar work. They were able to convert fructose at 25 °C using [BMIM]Cl as the solvent and Amberlyst-15 sulfonic ion-exchange resin as a catalyst with 80% HMF yield [58]. The reaction was possible by pre-dissolving biomass in IL at elevated temperatures. When the mixture has cooled down, a high viscosity mixture is formed. With the help of a co-solvent, the mixture’s viscosity is reduced, and stirring can be done easier [58].

With provided data in table 2, it can be seen that most experiments regarding conversion of biomass using IL are done in temperatures between 80 to 140 °C. However, a lower reaction temperature below 50 °C is made possible by pre-dissolving biomass in IL and with the help of a co-solvent. The different reaction time is also shown by the used biomass as a feedstock.

Table 1. The general IL that is usually used in biomass conversion [46],[47]

| IL       | Structure | Melting Point (K) | Liquid Density (kg/m³) | Viscosity (Pa.s) |
|----------|-----------|-------------------|------------------------|-----------------|
| [EMIM] [Cl] | ![Structure] | 350.42 [48] | 1110 at 358.14 K, 101.325 kPa [49] | 0.065 at 353.15 K, 101.325 kPa [50] |
| [EMIM] [Br] | ![Structure] | 350.2 [51] | 1378.39 at 352.77 K, 101.325 kPa [52] | 0.0012 at 101.325 K, 101 kPa [53] |
| [BMIM] [Cl] | ![Structure] | 340.1 [54] | 1057.70 at 343.14 K, 101.325 kPa [49] | 0.257 at 343.15, 102 kPa [55] |
| [BMIM] [Br] | ![Structure] | 350.8 [56] | 1268.23 at 353.08 K, 101.325 kPa [52] | 0.057 at 373 K, 101.325 kPa [57] |
merize and dehydrate glucose to HMF at action temperatures and long reaction times until it achieves its optimal value, whereas at low temperatures and short reaction durations, it will decrease and the required reaction time will continue to grow at high reaction temperatures.

Typically, an acidic catalyst is utilized to catalyze the dehydration of glucose or fructose to improve conversion rate. Hu et al. have conducted experiments comparing the conversion of glucose to HMF in Tetraethylammonium chloride (TEAC) at 120 °C for 30 minutes on various catalysts. In this experiment, they discovered that by using a CrCl$_3$·6H$_2$O catalyst, they could produce an HMF conversion yield of 70.3%, which was hypothetically attributed to the stronger Lewis acidity Cr$^{3+}$ [68]. In addition, time and temperature are also factors that play a role in conversion maximization. Based on research conducted by Hu et al., increasing the reaction temperature will increase the conversion of glucose into HMF and reduce the required reaction time. In this experiment, when the reaction temperature occurred at 100 °C, the yield of HMF reached 64.4% in 120 minutes, at 120 °C, the yield of HMF reached 70.3% in 30 minutes, and at 130 °C it reached 71.3% in 10 minutes [68]; Xin et al. also explained in their experimental results, the conversion of glucose to HMF will continue to grow at high reaction temperatures and long reaction times until it achieves its optimal value, whereas at low temperatures and short reaction durations, it will decrease and the effect of time is not very clear [69].

| Table 2. The results of various biomass conversion |
|-----------------------------------------------|
| **Biomass** | **Solvent** | **Catalyst** | **T (°C)** | **t (min)** | **The yield of HMF (%)** | **Ref.** |
| Fructose   | [HMIM]Cl    | [HMIM]Cl    | 90         | 45          | 92                       | [62]    |
| Fructose   | DMSO        | [NMP][CH$_3$SO$_3$] | 85         | 120         | 87.8                     | [12]    |
| Glucose    | [HMIM]Cl    | NHC/Cr      | 100        | 360         | 81                       | [63]    |
| Glucose    | [EMIM]Cl    | Yb(OTf)$_3$ | 140        | 360         | 10                       | [2]     |
| Glucose    | [EMIM]Cl    | CrCl$_2$    | 100        | 180         | 68                       | [64]    |

4. The advance of glucose conversion into HMF in IL media

The yield of HMF is highly dependent on the reaction conditions of its production process. With the correct reaction conditions, the maximum yield of HMF can be achieved. The key to converting glucose to HMF is to create a catalyst system that can both isomerize and dehydrate glucose to HMF [65]. Typically, an acidic catalyst is utilized to catalyze the dehydration of glucose or fructose to improve conversion rate [66],[67]. Hu et al. have conducted experiments comparing the conversion of glucose to HMF in IL media [70]. As in the experiment conducted by Yin et al., the yield of HMF increased from 17% to 91%. This may be achieved when using the organic solvent, but it goes a high boiling point can hinder its application [75]. In general, as shown in table 3, the optimum temperature that can be used to convert glucose to HMF is in the range of 100-130 °C. To compare the performance of IL in converting glucose into HMF, we can see the difference in the yield of HMF achieved and the reaction time for various ILs at a temperature of 100 °C with CrCl$_3$·6H$_2$O as a catalyst in figure 2.

| Table 3. Glucose conversion to HMF in various ILs |
|-----------------------------------------------|
| **IL** | **Catalyst** | **Temp (°C)** | **Time (min)** | **Reactor** | **Yield (%)** | **Ref.** |
| [BMIM]Cl | CrCl$_3$ | ML,400W | 1 | Microwave | 91 | [70] |
| [DBDIm]I | H$_2$SO$_4$ | 100 | 120 | Oven | 82.2 | [27] |
| TEAC | CrCl$_3$·6H$_2$O | 130 | 10 | Oven | 71 | [71] |
| [Emim]BF$_4$ | SnCl$_4$ | 100 | 180 | Oven | 62 | [72] |
| [BMIM]Cl | CrCl$_3$ | ML,400W | 2 | Microwave | 55.4 | [73] |
| [DBDIm]I | H$_2$SO$_4$ | 100 | 120 | Oven | 52.1 | [27] |

Note: MI: Microwave Irradiation

From table 3, the largest HMF yield is 91% using the microwave irradiation process. Microwave irradiation can effectively accelerate and increase the yield of HMF [74],[75]. The heating process by applying MI is heating to the molecular level, compared to conventional heating such as oil heating [74]. As in the experiment conducted by Yin et al., the yield of HMF increased significantly, and the reaction time was faster as in the process with [BMIM]Cl and catalyst CrCl$_3$ and assisted by MI, the yield of HMF increased from 17% to 91%. This may be achieved when using the organic solvent, but it goes a high boiling point can hinder its application [75]. In general, as shown in table 3, the optimum temperature that can be used to convert glucose to HMF is in the range of 100-130 °C. To compare the performance of IL in converting glucose into HMF, we can see the difference in the yield of HMF achieved and the reaction time for various ILs at a temperature of 100 °C with CrCl$_3$·6H$_2$O as a catalyst in figure 2.
Figure 2. HMF yield (%) and time in [BMIM]Cl [71], [EMIM]Cl [76], [C4Cl1im][HSO4] [77], TEAC (tetraethylammonium chloride) [68], Bu-DBUCI [78], DMSO [79]

Figure 2 shows that in a range time of 120-180 minutes, the first HMF conversion that reached its maximum was using TEAC, which was within 120 minutes, while the highest conversion was produced by [BMIM]Cl with an HMF yield of around 71%. The best type of IL to convert glucose into HMF more effectively than the other four ILs, as shown in figure 2 is [BMIM]Cl. In addition, temperature also played a crucial role in optimizing the production of HMF from glucose. As shown in Wang et al.’s report, the experiment of glucose conversion into HMF with the addition of TBAC or tetrabutylammonium chloride and CrCl3.6H2O as a catalyst, the higher the temperature, the higher the formation of by-products [80]. The HMF yield was at 29.6% at 130 °C, 34.8% at 140 °C, and it grew over time until it reached its peak at 43.7% after 90 minutes.

Meanwhile, at higher temperatures, the HMF yield will initially peak and then decrease continuously. Based in the investigation shows that the HMF yield will be optimal at a specific temperature. Naturally, the ideal temperature for each IL will be different.

5. Conclusion and future outlook
The HMF is well known as a potential compound to produce other valuable platform chemicals. There have been many studies showing high HMF yield. However, future efforts are still necessary to prevent the formation of by-products and the loss of HMF. Currently, the utilization of IL for HMF production from glucose conversion is massively conducted and being developed further. The selection of IL type and the catalyst is one strategy to optimize the production of HMF from glucose conversion. The reaction conditions such as reaction time and temperature vary depending on the used IL and the catalyst. IL possesses a threshold temperature to achieve optimum HMF recovery. The increasing temperature can promote the production of by-products. Therefore, the appropriate temperature is the crucial factor to explore. In addition, suitable ILs for glucose conversion are needed to make recycling and reuse processes easy, even saving on production costs and still paying attention to the environmental impact. In the future, there will be more research on the optimization of glucose conversion into HMF using an IL-based process and the implementation of HMF production on a large scale (i.e., industrial scale).
6. References

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