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Synthesis pathway of biomass-derived 5-chloromethylfurfural for conversion and obtaining high concentration 2,5-dimethylfuran

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ABSTRACT

The rapid depletion of fossil-based resources, together with the increasing global energy demand, has stimulated intensive research into renewable alternatives. Among these, biomass-derived fuels have emerged as highly promising candidates, with particular attention given to furan derivatives for their versatile chemical reactivity and potential as liquid transportation fuels. While the production of 5-hydroxymethylfurfural (HMF) has been extensively studied, the synthesis of 5-chloromethylfurfural (CMF) has recently attracted growing interest as a novel platform molecule. CMF offers several advantages over HMF, including milder reaction conditions, lower polarity that facilitates separation in organic media, and the presence of chlorine as an excellent leaving group, enabling efficient downstream transformations. Notably, CMF can be converted into high-value compounds such as 2,5-dimethylfuran (DMF), a biofuel with favorable physicochemical properties for transportation applications. This review highlights recent advances in CMF production from lignocellulosic biomass, synthetic strategies for its valorization into DMF and other value-added chemicals, as well as the feasibility of scaling up these processes toward industrial implementation.

1. INTRODUCTION

The widespread use of fossil fuels such as coal, oil, and natural gas by humanity has led to their rapid depletion, making it impossible to meet human consumption demands. Additionally, the use of fossil fuels has severely impacted human health, significantly increasing the number of deaths due to emissions from engines burning fossil fuels, and has caused serious environmental issues, including the greenhouse effect (Bell & Gillingham, 2025). Alongside the collective efforts of countries worldwide to protect the human living environment, treaties have been signed to achieve net-zero emissions by 2050. Therefore, the top strategic objectives are to promote the development of renewable energy sources in an environmentally friendly manner. Effectively utilizing waste biomass can generate an efficient biomass energy source. To effectively address these issues, countries worldwide have implemented a series of policies on "carbon neutrality" (Wang & Zhang, 2020). Biomass energy refers to solar energy stored in plants as chemical energy (Gao et al., 2018). This is a new energy source that can replace petroleum fuels at large production scales. After being used, it can create a carbon cycle effect, aligning with the concept of "carbon neutrality".

Among the biofuels under active development, ethanol has emerged as a promising alternative to fossil fuels due to several advantageous properties. Ethanol can be produced from lignocellulosic biomass, making it a renewable fuel. The

combustion of ethanol results in lower net CO_2 emissions than conventional gasoline, helping mitigate greenhouse gas effects (Kumar & Sinha, 2025). Moreover, ethanol is free of sulfur compounds, thereby reducing SO_x emissions and improving air quality. Ethanol also has a high octane rating (~110), reducing the risk of engine knock and enabling higher compression ratios for improved engine efficiency (Tafesse & Nallamothu, 2025). Additionally, ethanol can be blended with gasoline at various proportions (e.g., E10, E85), thereby reducing dependence on traditional fossil fuels (Abel et al., 2021).

Ethanol, despite being a renewable fuel, presents several limitations. Its lower energy density compared to gasoline leads to higher fuel consumption and slightly reduced engine efficiency. Ethanol is also hygroscopic, which can cause phase separation, corrosion of fuel system components, and potential engine knocking (Khoa, 2025). Additionally, its production from crops like corn or sugarcane may compete with food supply, affecting food prices and raising environmental concerns such as soil erosion and water pollution (The Guardian, 2024).

2,5-dimethylfuran (DMF) is liquid at room temperature, with a boiling point 20 °C higher than ethanol and an octane number nearly 40% higher than ethanol. It is gaseous when burned in an internal combustion engine. It is insoluble in water and does not absorb moisture in the air. It is an ideal fuel and is known as the "second-generation biofuel." These properties make DMF a promising alternative in the near future (Zhang et al., 2013; Xiao et al., 2017a, 2017b; Wei et al., 2018). Table 1 presents the main physical and chemical properties of DMF fuel, comparing it with other fuels to illustrate its potential advantages for energy applications (Nguyen et al., 2021).

Table 1. Physical and chemical properties of DMF compared to other fuels

Property	Gasoline	Diesel	n-Butanol	Ethanol	DMF
Molecular formula	C_4-C_{12}	C_{10}-C_{15}	$C_4H_{10}O$	C_2H_6O	C_6H_8O
Energy density (MJ/L, 20°C)	32-34.8	40.3	29.2	18.4-21.2	30
Liquid density (kg/m^3, 20°C)	745	820	810	791	890
Auto-ignition temperature (°C)	420	246	385	434	286
Stoichiometric air/fuel ratio	14.7	14.3	11.2	8.9	10.7
Boiling point (°C)	27-225	282-338	117	78	93
Latent heat of vaporization (kJ/kg)	351	270-301	708	920	389
Oxygen content (%)	0	0	21.6	34.78	16.67
Surface tension (mN/m)	20	28	24.6	22.3	26
Kinematic viscosity (cSt)	0.37-0.44	2-4.5	3.6	1.5	0.57
Cetane number	10-15	40-45	25	8	9
Research octane number	90-100	-	98	110	119

2. METHOD

The research method chosen by the author for this paper is the synthesis and analysis of the theoretical basis of various documents and texts from previous research by different authors on biofuels. Selecting important information necessary for the research topic. Then, proceeding to analyze, evaluate, and systematically compile statistics on the pathways of conversion from biomass through the hydrolysis process to produce CMF, from the abundant CMF raw material obtained through different processes to catalyze the conversion into DMF fuel, which is a liquid biofuel and an important fuel source for transportation vehicles.

3. RESULTS AND DISCUSSION

3.1. The production pathway of DMF from biomass sources

After harvesting, crops that provide food for humans will produce a large amount of biomass waste, such as straw, corn stalks, corn cobs, peanut shells, and oat husks. It takes a lot of time and incurs significant costs to collect and process these biomass wastes. Previously, producing DMF from biomass was very difficult and inefficient, which did not attract many scientists to research it. In recent years, due to advancements in science and technology, the production of DMF has become simpler and faster, creating a biofuel source applicable to transportation.

These fuels can be produced by efficiently converting five-carbon sugars, such as xylose and arabinose, into furfural, and six-carbon sugars, such as fructose and glucose, into 5-hydroxymethylfurfural (HMF). Furfural and HMF can be further processed into furan fuel (Westbrook et al., 2021). Currently, there are two main conversion pathways to produce DMF from biomass, as shown in Figure 1. The first is that after the initial pretreatment, biomass is broken down into simple carbohydrates or non-hydrolyzed monosaccharides such as glucose and fructose, which are then selectively dehydrated to remove three oxygen atoms to produce HMF, and then HMF continues to remove two oxygen atoms through hydrolysis to produce DMF. The second pathway involves cellulose or glucose first producing 5-chloromethylfurfural (CMF), which then hydrolyses to DMF (Tong et al., 2020).

DMF is produced from biomass waste, thus it does not compete with the human food supply chain but transforms waste into a source of energy. Due to the readily available, low-cost biomass feedstock, biomass-based DMF production will become the main method in the future.

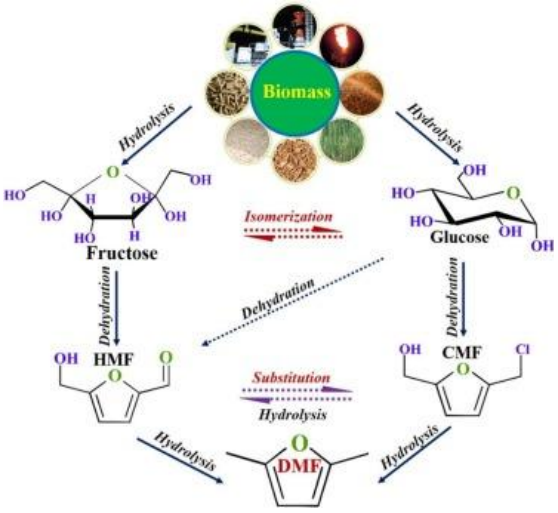

Figure 1. The production pathway of DMF from biomass

(Source: Hoang et al., 2021)

CMF is a chlorinated derivative of HMF. Its structure and molecular capacity are similar to HMF, but CMF is more stable under acidic conditions, and its good hydrophobicity makes it easier to extract into organic solvents. As one of the downstream products of cellulose hydrolysis, CMF is considered an important biomass platform compound due to its numerous functional groups. Various reactions can occur, such as hydrogenation, oxidation, and etherification (Shaoyu et al., 2025). Among them, DMF produced by hydrogenation selected from CMF is considered a high-quality liquid fuel due to its unique physical properties.

3.2. Methods for synthesizing 5-chloromethylfurfural from biomass

At present, DMF is mainly prepared from HMF from biomass-based platform molecule (Hu et al., 2020; Karimi et al., 2021; Li et al., 2021; Pisal et al., 2021). Although higher selectivity and yield have been achieved, there are problems, including higher reaction temperatures (120-220 °C) and longer reaction times (2-24h). In addition, HMF is mainly prepared from fructose at a higher cost, and the active chemical properties and hydrophilicity of HMF lead to the difficulty of subsequent separation and purification, which further limits the process of large-scale preparation of DMF using HMF as a raw material.

There are a few reports on the synthesis of DMF from CMF. Although CMF is a new biomass-based platform molecule that can be directly prepared from raw materials such as cellulose and biomass in high yield under mild conditions, its stability and hydrophobicity make it easier to separate and purify in the future (Mascal, 2019).

Compared with HMF, CMF exhibits not only higher stability under acidic conditions but also distinct kinetic and thermodynamic advantages (Karimi et al., 2025; Soukup-Carne et al., 2024). While HMF readily undergoes rehydration to levulinic acid (LA) and polymerization into humins under acidic media, CMF's chloromethyl substituent withdraws electron density from the furan ring, thereby enhancing stability and facilitating more selective hydrogenolysis to DMF under milder conditions. This combination of improved selectivity, lower reaction temperature, and shorter reaction time underscores the kinetic benefits and thermodynamic favorability of CMF as a superior platform for DMF production.

In the early 1900s, Fenton and Gostling (1901) obtained CMF by treating ketohexoses and cellulose with HCl in ether. Haworth and Jones (1944) reported the use of a two-phase solvent system to synthesize CMF. The yield of synthesized CMF from sucrose was 21 mol%. However, when using

glucose, cellulose, and biomass as raw materials, the yield of CMF obtained was relatively low.

Table 2 summarizes the studies on the synthesis of CMF from various biomass feedstocks, arranged according to the extraction solvent system. The results indicate that the CMF yield is strongly influenced by the type of carbohydrate precursor and by reaction parameters, including time and temperature.

Table 2. Reported studies on the synthesis of CMF from biomass

Feedstock	Solvent	Catalyst	T/°C	t/h	CMF Yield/mol%	Ref.
Glucose (5mmol)	$CHCl_3$	$ZnCl_2$/HCl (1:3)	45	10	11.2	(Wu et al., 2015)
Fructose (5mmol)	$CHCl_3$	$ZnCl_2$/HCl (1:3)	45	10	52.5	(Wu et al., 2015)
Sucrose (5mmol)	$CHCl_3$	$ZnCl_2$/HCl (1:3)	45	10	51.5	(Wu et al., 2015)
Cellulose (5mmol)	$CHCl_3$	$ZnCl_2$/HCl (1:3)	45	10	4.4	(Wu et al., 2015)
Glucose (5mmol)	$CHCl_3$	$CrCl_3$/HCl (1:3)	45	10	13.9	(Wu et al., 2015)
Fructose (5mmol)	$CHCl_3$	$CrCl_3$/HCl (1:3)	45	10	61.6	(Wu et al., 2015)
Sucrose (5mmol)	$CHCl_3$	$CrCl_3$/HCl (1:3)	45	10	71.7	(Wu et al., 2015)
Cellulose (5mmol)	$CHCl_3$	$CrCl_3$/HCl (1:3)	45	10	5.5	(Wu et al., 2015)
D-Fructose (5mmol)	$CHCl_3$	HCl(37%)/H_3PO_4(85%) (1:1)	45	20	35.8	(Gao et al., 2013)
D-Fructose (5mmol)	$CHCl_3$	HCl(37%)/H_3PO_4(85%) (4:1)	45	20	46.8	(Gao et al., 2013)
D-Fructose (5mmol)	DCE	HCl(37%)/H_3PO_4(85%) (4:1)	45	20	28.8	(Gao et al., 2013)
D-Fructose (5mmol)	1-Chlorobutane	HCl(37%)/H_3PO_4(85%) (4:1)	45	20	22.6	(Gao et al., 2013)
Palmarosa (1.0g)	$CHCl_3$	HCl(35%)+NaCl(2.5wt%)	100	1	76.5	(Singh et al., 2020)
Lemon grass (1.0g)	$CHCl_3$	HCl(35%)+NaCl(2.5wt%)	100	1	72.4	(Singh et al., 2020)
Citronella grass (1.0g)	$CHCl_3$	HCl(35%)+NaCl(2.5wt%)	100	1	65.8	(Singh et al., 2020)
Rice bran (4wt%)	$CHCl_3$	HCl (32%)	100	2	22.7	(Lakmini et al., 2022)
Rice hull (4wt%)	$CHCl_3$	HCl (32%)	100	2	29.6	(Lakmini et al., 2022)
Broken white rice (4wt%)	$CHCl_3$	HCl (32%)	100	2	52.2	(Lakmini et al., 2022)
Immature brown rice (4wt%)	$CHCl_3$	HCl (32%)	100	2	48.0	(Lakmini et al., 2022)
Sucrose (0.12M)	CH_2Cl_2	HCl (32%)	100	0.04	51.0	(Brasholz et al., 2011)
D-Fructose (0.11M)	CH_2Cl_2	HCl (32%)	100	0.04	80.0	(Brasholz et al., 2011)
D-Glucose (0.11M)	CH_2Cl_2	HCl (32%)	100	0.04	15.0	(Brasholz et al., 2011)
D-Glucose (0.11M)	DCE	HCl (32%)	120	0.083	58.0	(Brasholz et al., 2011)
HFCS (37wt%)	CH_2Cl_2	HCl (37%)	100	0.317	85.0	(Kohl et al., 2017)
Cellulose (1.4wt%)	DCE	LiCL/HCl	65	30	71.0	(Mascal & Nikitin, 2008)
Glucose (1.0wt%)	DCE	HCl	100	3	81.2	(Mascal & Nikitin, 2009)

Feedstock	Solvent	Catalyst	T/°C	t/h	CMF Yield/mol%	Ref.
Sucrose (1.0wt%)	DCE	HCl	100	3	89.7	(Mascal & Nikitin, 2009)
Cellulose (1.0wt%)	DCE	HCl	100	3	83.5	(Mascal & Nikitin, 2009)
Corn stover (1.0wt%)	DCE	HCl	100	3	80.2	(Mascal & Nikitin, 2009)
Glucose (250mg)	DCE	HCl (5ml)	80	0.25	71.0	(Breeden et al., 2013)
Glucose (500mg)	DCE	HCl (5ml)	70	0.167	85.0	(Breeden et al., 2013)
Cellulose (1.7wt%)	DCE	$ZnCl_2$ (3.4wt%)	80	2	72.0	(Bhat et al., 2022)
Glucose (2.1wt%)	DCE	BTBAC (0.2wt%)	90	3	64.0	(Onkarappa & Dutta, 2019)
Glucose (0.05M)	DCE	HCL (37.2 mmol)	80	2	38.0	(Antonyraj et al., 2021)
Fructose (0.005M)	MIBK	$AlCl_3 \cdot 6H_2O$ (0.005M)	120	5	50.3	(Zuo et al., 2016)
Fructose (0.002M)	DCE	$AlCl_3 \cdot 6H_2O$ /HCl (0.015M)	120	0.5	86.0	(Chen et al., 2020)
Sucrose (0.002M)	DCE	$AlCl_3 \cdot 6H_2O$ /HCl (0.015M)	120	0.5	80.0	(Chen et al., 2020)
Cellulose (0.002M)	DCE	$AlCl_3 \cdot 6H_2O$ /HCl (0.015M)	120	0.5	30.0	(Chen et al., 2020)
Bamboo (0.002M)	DCE	$AlCl_3 \cdot 6H_2O$ /HCl (0.015M)	120	0.5	29.0	(Chen et al., 2020)
Bamboo powder (0.002M)	DCE	$AlCl_3 \cdot 6H_2O$ /HCl (0.015M)	120	0.5	35.0	(Chen et al., 2020)
Fructose (1.0wt%)	Toluene	HCl	100	1	51.0	(Smith et al., 2021)
Fructose (1.0wt%)	CPME	HCl	100	1	46.3	(Smith et al., 2021)
Fructose (1.0wt%)	Toluene	HCl	80	4	79.0	(Smith et al., 2021)
Fructose (10g/L)	Anisole	HCl (26%)	90	0.07	79.0	(Rojahn et al., 2022)
HFCS (0.1mol)	C_6H_5Cl	HCl (35%)	75	1	90.0	(Szmant & Chundury, 1981)
D-Fructose (0.1mol)	C_6H_5Cl	HCl (35%)	75	1	95.0	(Szmant & Chundury, 1981)
D-Glucose (0.1mol)	C_6H_5Cl	HCl (35%)	75	2	30.1	(Szmant & Chundury, 1981)
Corn starch (0.1mol)	C_6H_5Cl	HCl (35%)	75	2	14.2	(Szmant & Chundury, 1981)
Glucose (3.03 wt.%), mannose (0.07 wt.%), xylose (0.090 wt.%)	C_6H_5Cl	HCl	90	2	96.7	(Bueno Moron et al., 2023)

3.2.1. Preparation of CMF from 1,2-dichloroethane solvent

Cellulose from biomass is converted into CMF, with cellulose mixed into a LiCl/HCl solution reacting 1,2-dichloroethane (DCE). The mixture is continuously stirred for 18h at a temperature of 65 °C in a reactor. After that, additional LiCl/HCl is added to the reactor, and stirring continues for another 12h, resulting in a CMF yield of 71 mol%. Similar to cellulose, glucose and sucrose are also used under the reaction conditions above to produce CMF at 71 mol% and 76 mol%, respectively (Mascal & Nikitin, 2008). Mascal and Nikitin (2009) dissolved cellulose, glucose, sucrose, and

corn stover in a reactor, using DCE solvent and HCl catalyst, heated and stirred continuously for 3h at 100 °C. The extracted CMF molar yields were 83.5%, 81.2%, 89.7%, and 80.2%, respectively. Breeden et al. (2013) used a microwave heating oven with prepared mixtures of fructose, DCE, and HCl, stirred continuously at 80 °C for 0.25 h, achieving a CMF yield of 71 mol%. Similarly, using the same mixture but reducing the reaction time to 10 min at a temperature of 70 °C, the CMF yield increased to 85 mol%. When substituting fructose with glucose, polyfructan inulin, and cellulose in the above mixture, the CMF molar yields were 39%, 70%, and 24%, respectively, over a period of 0.75h at 80 °C.

Meller et al. (2016) studied CMF synthesised from a reactor with a quantity of carbohydrate in an HCl solution reacting LiCl and the organic solvent DCE, stirred vigorously at a temperature of 50-90 °C for 4h, with intermittent extraction. Using carbohydrates such as sucrose, glucose, and cellulose in HCl, LiCl at 70 °C, and DCE replaced every 0.5h, CMF molar yields of 35%, 7.4%, and 5.5% were formed in an open reaction vessel. With cellulose in HCl, DCE was added every 2h at a temperature of 110 °C. The total reaction time was 4h, yielding the highest CMF yield of 82.6 mol% in a closed reaction vessel.

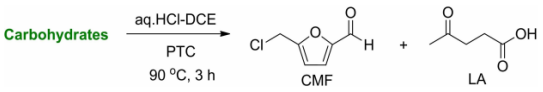

Figure 2. CMF obtained from carbohydrates using DCE solvent and PTC catalyst

(Source: Onkarappa & Dutta, 2019)

Onkarappa and Dutta (2019) synthesized a large amount of CMF from biomass-derived carbohydrates in a reactor under the influence of a phase transfer catalyst (PTC), as shown in Figure 2, catalyzed in HCl-DCE solution in the presence of benzyl-tributylammonium chloride (BTBAC) at a reaction temperature of 90 °C for 3h. Various types of carbohydrates were used to produce and obtain CMF molar yields, including starch (42%), cellulose (48%), fructose (62%), glucose (64%), and sucrose (73%). Additionally, a large amount of LA was also obtained along with CMF.

3.2.2. Preparation of CMF from deep eutectic solvent

The new solvent system, recently researched, deep eutectic solvent (DES), is developed as a low-melting-point solvent, easy to synthesize, and environmentally friendly. As a new type of ionic liquid, it operates under mild reaction conditions without requiring concentrated acid.

Zuo et al. (2016) extracted CMF derived from fructose in a biphasic reaction system, using DES as shown in Figure 3, $AlCl_3 \cdot 6H_2O$, and composed of methyl isobutyl ketone (MIBK), choline chloride (ChCl) catalyst at 110°C in 2h, the reaction afforded a CMF molar yield of 37%. Increasing the temperature to 120°C and extending the reaction time to 5h improved the yield to 50.3%. When MIBK was replaced with GVL (γ-valerolactone), the CMF yield decreased to 18.9%.

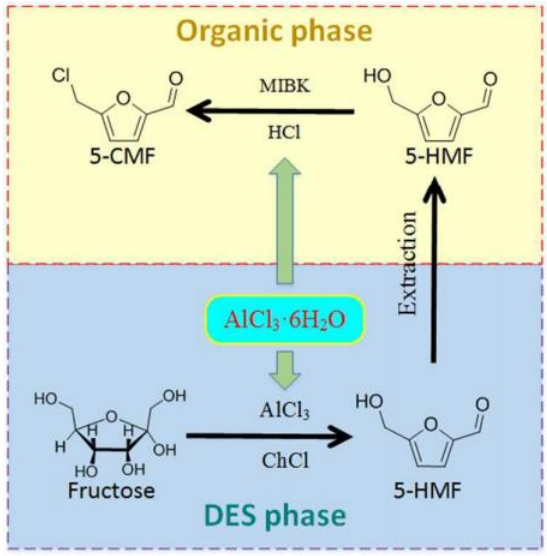

Figure 3. Mechanism of fructose to CMF conversion in DES/MIBK system catalyzed by $AlCl_3 \cdot 6H_2O$

(Source: Zuo et al., 2016)

Chen et al. (2020) used CMF synthesized from glucose based on a three-component deep eutectic solvent system (3C-DES) consisting of ChCl, $AlCl_3 \cdot 6H_2O$ and oxalic acid ($H_2C_2O_4$) under reaction conditions at 120 °C for 30 min. A CMF molar yield of 70% was achieved, as shown in Figure 4. In addition, intermittent extraction of CMF phase from the above mixture could increase the yield to 76%. Using the same catalyst solvent mixture, when glucose was replaced by fructose, sucrose, cellulose, bamboo and bamboo powder, the CMF molar yields were 86%, 80%, 30%, 29% and 35%, respectively.

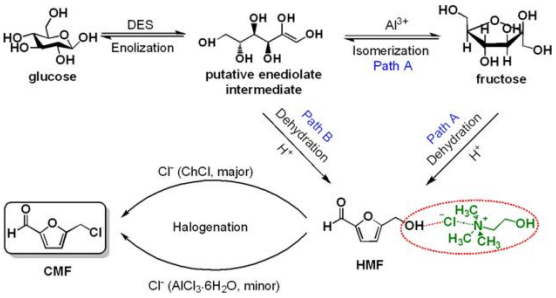

Figure 4. Glucose catalysis in 3c-DES system for CMF production

(Source: Chen et al., 2020)

3.2.3. *Preparation of CMF from chloroform solvent*

A very reliable solvent can extract a large amount of CMF and significantly reduce by-product formation during the reaction. Chloroform ($CHCl_3$) is a great solvent for extracting CMF from the acidic aqueous phase, promoting the reaction process, making it a very promising solvent.

Gao et al. (2013) used a biphasic HCl-H_3PO_4 catalyst-$CHCl_3$ solvent mixture to convert carbohydrates into CMF via a one-pot route. Specifically, a quantity of fructose at 45°C for 20h resulted in a CMF yield of 35.8 mol%. Increasing the amount of HCl in the catalyst mixture increased the CMF yield. Specifically, at a HCl-H_3PO_4 volume ratio of 4:1, the CMF yield reached 46.8 mol%. Indeed, the authors' group compared the results when changing the solvent from $CHCl_3$ to DCE and 1-chlorobutane, the molar yield of CMF obtained was only 28.8% and 22.6%, significantly lower than that of $CHCl_3$. Also in this study, another result reported by the authors' group showed that when lignocellulose (Eucalyptus Kraft pulp, Norway Spruce softwood TMP, Eucalyptus hardwood) was converted in the same mixture, the corresponding CMF molar yields were 21.3%, 33.7%, and 47.4%.

Wu et al. (2015) synthesized monosaccharides (glucose and fructose) in a $ZnCl_2$/HCl solution at a ratio of 1:3 (this is the ratio that achieves the optimal yield), in $CHCl_3$ solvent for 10h at 45 °C in a sealed container. The results showed that glucose yielded 11.2 mol% CMF, and fructose yielded 52.5 mol% CMF. According to this study, using $CrCl_3$ as a catalyst instead of $ZnCl_2$ increased the CMF yield. Specifically, glucose, fructose, sucrose, and cellulose produced molar yields of 13.9%, 61.6%, 71.7%, and 5.5% CMF, respectively, while bamboo pulp, eucalyptus pulp, and bagasse produced molar yields of 21.3%, 27.32%, and 39.14% CMF. The authors researched the combination of two metal chlorides, resulting in a significantly improved CMF yield, specifically achieving CMF molar yields of 80%, 32.7%, 36.2%, and 50.1% for fructose, bamboo pulp, eucalyptus pulp, and bagasse, respectively, as shown in Figure 5.

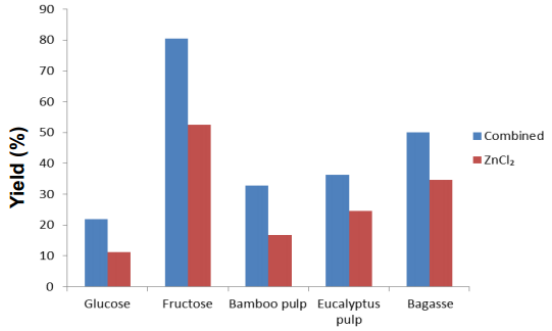

Figure 5. CMF yield when combining 2 metal chlorides and $ZnCl_2$

(Source: Wu et al., 2015)

Singh et al. (2020) extracted aromatic biomass from several aromatic species of the genus Cymbopogon to develop treatments for this aromatic waste biomass to obtain highly selective CMF. Specifically, the aromatic waste biomasses include lemongrass (Cymbopogon flexuosus), citronella grass (C. winterianus), and palmarosa (C. martini), along with the catalyst HCl + NaCl, in $CHCl_3$ solvent in a high-pressure glass reactor at a temperature of 100 °C, continuously stirred for 1h, resulting in CMF molar yields of 72.4%, 65.8%, and 76.5%, respectively.

3.2.4. *Preparation of CMF from cyclopentyl methyl ether solvent*

Although when using the solvent $CHCl_3$, DCE extraction yields very good results for CMF, the chemical properties of these two solvents are very hazardous, potentially causing acute toxicity to humans when ingested or inhaled, and are highly carcinogenic (Sigma-Aldrich, 2019, 2020a). Therefore, the solvents cyclopentyl methyl ether (CPME) and toluene are proposed as replacements. Toluene is less hazardous to humans than the solvents $CHCl_3$, DCE but still poses a threat to the ecosystem (Sigma-Aldrich, 2020b). CPME is a green solvent, with a high flash point, low volatility, and stability in the reaction environment (Watanabe, 2013).

Smith et al. (2021) studied the use of a more environmentally friendly solvent system consisting of CPME and toluene. The initial biomass fuel was fructose, which was heated and continuously stirred in HCl in the CPME and toluene solvents at 100 °C for 1h, yielding a CMF molar yield of 51% with toluene and 46.3% with CPME. The next step in this study was to implement a semi-batch flow system with toluene as the solvent for 4h at 80 °C (the temperature for optimal CMF yield), achieving a CMF yield of 79 mol%, as shown in Figure 6. Finally, when replacing the initial raw material fructose with sucrose, glucose, cellulose, Ulva lactuca, and larch sawdust in HCl/toluene at 100 °C for 4h, the corresponding CMF molar yields were 67%, 65%, 39%, 21%, and 30%.

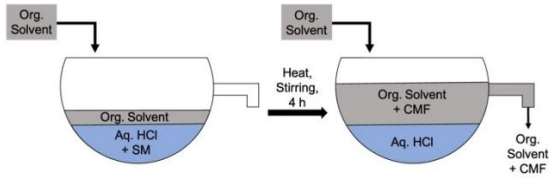

Figure 6. Semi-batch flow reaction system

(Source: Smith et al., 2021)

3.2.5. *Preparation of CMF from dichloromethane solvent*

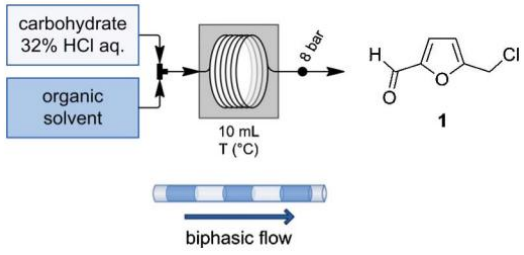

Figure 7. A biphasic continuous flow reactor converts carbohydrates to CMF

(Source: Brasholz et al., 2011)

Brasholz et al. (2011) used a continuous flow reactor to separate water, converting carbohydrates into CMF. D-glucose, D-fructose, and sucrose were initially dissolved in HCl, and the mixture was then blended into a dichloromethane (CH_2Cl_2) solvent at a reactor pressure of 8 bar and a temperature of 100°C, yielding CMF yields that varied with reaction time. Specifically, with a reaction time of 150s, the molar yields of CMF from D-glucose, D-fructose, and sucrose were 15%, 80%, and 51%, respectively as shown in Figure 7.

3.2.6. *Preparation of CMF from chlorobenzene solvent*

Szmant and Chundury (1981) studied high-fructose corn syrup (HFCS), D-fructose, D-glucose, and corn starch in HCl solution and in chlorobenzene (C_6H_5Cl) solvent, continuously stirred at high speed in a reactor filled with nitrogen gas. For HFCS, with a reaction time of 1h at a temperature of 75ºC, the CMF yield reached 90 mol%; D-fructose, with a reaction time of 1h at a temperature of 75ºC, achieved a CMF yield of 95 mol%; D-glucose, with reaction times of 2h and 24h at a temperature of 75 ºC, resulted in CMF molar yields of 30.1% and 35.6%, respectively; corn starch with reaction times of 2h and 24h at a temperature of 75 ºC yielded CMF molar yields of 14.2% and 17%. Moron et al. (2023) reported the highest CMF yield of 96.7 mol%, achieved under mild conditions of low temperature and ambient pressure, through an initial hydrolysis of aspen wood chips followed by downstream saccharification.

Compared with halogenated solvents such as DCE, $CHCl_3$, CH_2Cl_2, and chlorobenzene, greener alternatives like CPME and DES present distinct trade-offs in terms of performance, cost, and recyclability. CPME offers clear advantages in terms of safety, lower toxicity, and alignment with green chemistry principles. However, reported CMF yields in CPME are generally lower than those obtained with halogenated solvents, which may limit its application unless further process optimization is achieved. DES are inexpensive, biodegradable, and potentially recyclable, thereby reducing environmental impact. Nevertheless, their long-term stability and recovery efficiency require further validation, and, in many cases, the observed CMF yields remain moderate compared with those of DCE-based systems (Demir et al., 2025; Marco-Velasco et al., 2024; Soukup-Carne et al., 2024). Overall, while CPME and DES improve safety and sustainability, they may involve compromises in efficiency and scalability that should be carefully considered in future process development.

3.3. Method of synthesizing DMF from CMF

CMF, nano palladium catalyst, and tetrahydrofuran (THF) were added to a stainless steel closed reactor, and hydrogen replaced air for the catalytic reaction to produce DMF (Table 3). The conditions for the catalytic reaction are: reaction temperature from 20 to 50°C, hydrogen pressure from 0.1 to 2 MPa, and stirring speed from 400 to 1000 RPM (Xianhai et al., 2022).

Table 3. Influence of catalysts and process conditions on CMF hydrogenation yield

Substrate	Solvent	Catalysts	T/°C	t/h	P/MPa	DMF yield/mol%
CMF (0.144g)	THF (10mL)	Pd/CNTs (0.036g)	30	0.25	0.5	92.0
CMF (0.144g)	THF (10mL)	Pd/CNTs (0.036g)	20	1	0.5	91.0
CMF (0.144g)	THF (10mL)	Pd/CNTs (0.09g)	30	0.25	0.5	92.0
CMF (0.144g)	THF (10mL)	Pd/CNTs (0.09g)	30	0.25	1	92.0
CMF (0.144g)	THF (10mL)	Pd/CNTs (0.09g)	30	0.25	1.5	90.0
CMF (0.144g)	THF (10mL)	Pd/CNTs (0.09g)	30	0.25	2	91.0
CMF (0.144g)	THF (10mL)	Pd/C (4.1wt%)	30	0.25	0.5	63.0
CMF (0.144g)	THF (10mL)	Pd/Al_2O_3 (4.2wt%)	30	0.25	0.5	56.0

Li et al. (2020) used polymethylhydrogensiloxane, commercialized Pd/C and solvent n-butanol to catalyze the CMF reaction at 25-45°C for 10-30 min to obtain a DMF molar yield of 87-96%. This invention, although it achieves a high yield of DMF, still has drawbacks compared to Xianhai et al. (2022), such as the use of polymethylhydrogensiloxane, which is more expensive than hydrogen and generates many by-products during the reaction. The solvent n-butanol used also has a higher boiling point than THF, leading to a higher reaction temperature.

Dutta and Mascal (2014) directly reduced CMF to DMF using a Pd/C catalyst in an N,N-dimethylformamide/acetic acid mixture and hydrogen at room temperature for 85 min, achieving a 76% yield of DMF. Next, 5-methylfurfural (MF) obtained from the hydrogenation of CMF is prepared in a mixture of N,N-dimethylformamide/acetic acid using Pd/C at room temperature and 1 atm, reacting for 60 min to a molar yield of 73% DMF. In this study, CMF was treated with n-butanol in the presence of HCl at 0 °C for 1h to form the acetal 2-(chloromethyl)-5-(dibutoxymethyl)furan (98 mol%), which was subsequently hydrogenated over Pd/C to yield DMF.

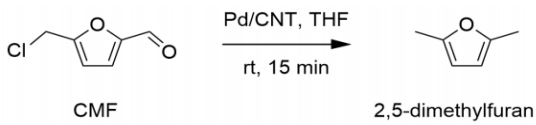

Figure 8. Hydrogenation of CMF to DMF under short reaction conditions

(Source: Chen et al., 2022)

Chen et al. (2022) prepared a stainless steel autoclave and reacted biomass-derived CMF on a Pd/CNTs catalyst for 15 min at 30°C, achieving 92 mol% DMF, as shown in Figure 8. The STY value (space-time-yield) of the DMF production process from CMF is higher than that of HMF. This is also the most successful case of synthesizing DMF from CMF with H_2 in a short reaction time.

Hamada et al. (2001) obtained a high-purity CMF yield of 87 mol% after extracting fructose, followed by a process in which a mixture of CMF, cyclohexene, and Pd/C was heated to 80°C and then hydrogenated with stirring for 9 h. After the reaction was complete, the mixture was distilled using a rapid condensation method, with the main product obtained being DMF at a yield of up to 81 mol%; with the same mixture and a reaction time reduced to 8h at a temperature of 60°C, the DMF yield increased to over 88 mol% (Figure 9).

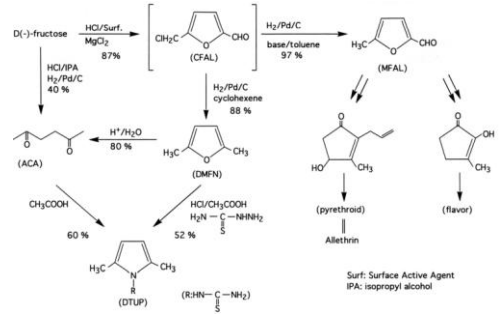

Figure 9. Synthesis of reaction methods of furan compounds

(Source: Hamada et al., 2001)

In the process of producing purified terephthalic acid (PTA) (Pacheco, 2015), p-xylene (PX) is a very important substance for the oxidative synthesis of PTA. Cellulose biomass is converted into intermediate products (HMF or CMF) using HCl as the acid catalyst, then undergoes H_2 reduction to produce a large amount of pure DMF, as shown in Figure 10.

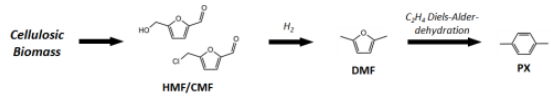

Figure 10. Pathway from biomass to PX

(Source: Pacheco, 2015)

3.4. Sustainability Considerations

While many studies have reported high CMF/DMF yields across various catalytic systems and solvents, the environmental and economic implications of these processes are equally important in evaluating their sustainability (Osman et al., 2021; Wiranarongkorn et al., 2023). Reactions performed at lower temperatures (≤100 °C) and shorter durations (≤3 h) are generally more energy-efficient and preferable for scale-up than those requiring higher temperatures (>150°C) and extended reaction times.

Solvent choice also plays a critical role: halogenated solvents such as DCE provide efficient extraction and high yields but are associated with toxicity and persistence. In contrast, DES such as ChCl, $AlCl_3 \cdot 6H_2O$, and $H_2C_2O_4$ systems offer more biodegradable and recyclable options (Demir et al., 2025; Marco-Velasco et al., 2024).

From an economic standpoint, the use of inexpensive raw materials (e.g., cellulose, agricultural residues) and common mineral acids is more feasible than processes that require costly reagents such as polymethylhydrosiloxane or precious-metal catalysts. Sensitivity and cost analyses indicate that feedstock cost and yield are dominant factors (Wiranarongkorn et al., 2023; Lee et al., 2024).

Although full life cycle assessments (LCAs) or techno-economic analyses (TEAs) specifically focused on CMF/DMF production remain limited, existing reviews suggest that applying green chemistry metrics such as atom economy, E-factor, and PMI is essential for rigorous evaluation (Soukup-Carne et al., 2024). Future research should integrate LCA and TEA to guide the selection of the most viable and sustainable synthesis routes.

4. CONCLUSIONS

Recent advances in furan chemistry have highlighted the growing potential of CMF alongside conventional platforms such as HMF and furfural. While early studies focused on monophasic systems that often led to unwanted by-products, the adoption of biphasic systems has significantly improved selectivity, particularly in CMF synthesis, where their hydrophobicity favours efficient partitioning. Compared to HMF, CMF can be obtained under milder conditions, often below 100 °C, which reduces energy demand and minimizes side reactions. Moreover, the presence of the –Cl functionality in CMF provides versatile synthetic opportunities, enabling transformations such as esterification and hydrogenation. These reactions expand its utility not only as a precursor for advanced biofuels like DMF but also as a building block for bioplastics, speciality chemicals, and other high-value products. Overall, CMF emerges as a promising intermediate for the sustainable valorization of carbohydrate-derived waste into renewable fuels and materials, and further exploration of complex conversion pathways is expected to drive progress in this field.

CONFLICT OF INTEREST

The authors declare no conflict of interest.

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