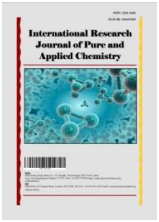

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Catalytic Conversion of Sugarcane Bagasse into 5-Hydroxymethylfurfural

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Authors' contributions

This work was carried out in collaboration among all authors. Author OAOI designed the study, performed experimental work, conducted the statistical analysis and wrote the first draft of the manuscript. Authors AMM, MMA and ABO reads and correct the manuscript. All authors read and approved the final manuscript.

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ABSTRACT

Furan derivatives especially 5-Hydroxymethylfurfural has recently been regarded as one important precursor for the production of biofuels and biobased compounds.
Aims: The aim of this study was to convert sugarcane bagasse into 5-hydroxymethylfurfural using Tetrabutylammonium bromide, Tetramethylammonium chloride and metal chlorides (chromium chloride, copper chloride, ferric chloride and cobalt chloride) in dimethylsulphoxide; using different reaction time and temperatures.
Study Design: The design of this study includes preparation of two different systems of catalyst to convert sugarcane bagasse into 5-hydroxymethylfurfural.
Place and Duration of Study: This study conducted at Department of Applied and Industrial Chemistry, International University of Africa- Sudan, between 2017 and 2019.

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Methodology: Two different catalytic systems were prepared to convert sugarcane bagasse into 5-hydroxymethylfurfaural, the first system Tetrabutylammonium bromide and metal chlorides (Chromium chloride, copper chloride, ferric chloride and cobalt chloride) in dimethyl sulphoxide; the second system tetrametheylammonium chloride and same metal chlorides in dimethylsulphoxide. The conversion of sugarcane bagasse was conducted at temperatures 100, 130 and 150°C; in addition to reaction times 60, 90 and 120 min.
Results: The obtained results indicated that the yield of 5-hydroxymethylfurfural was high 53.41% at 150°C and 120 min reaction time when chrome chloride was used in Tetrabutylammonium bromide and dimethylsulphoxide as co-solvent; although the highest yield 89.23%, was noted also at same condition, when chromium chloride was used in Tetramethylammonium chloride and dimethylsulphoxide as co-solvent.
Conclusion: Based on these results chromium chloride was considered the best catalyst for yielding 5-hydroxymethylfurfural from sugarcane bagasse in both Tertabutylammonium bromide and Tetramethylammonium chloride; but is best in Tetramethylammonium chloride.

Keywords: 5-Hydroxymethylfurfural; bagasse; biomass; dimethylsulphoxide; cellulose; lignin; hemicellulose; sugarcane.

1. INTRODUCTION

As the fossil fuel reserves are threatening to be depleted and with the increase of the global climate change, there is an urgent demand for alternative energy sources such as biomass feedstock. The conversion of biomass to useful chemicals and fuels represents, in general, a major challenge. This turns out to be particularly ambitious if the process should be made in a sustainable and economical way, in order to replace fossil fuels [1]. 5-Hydroxymethylfurfural (5-HMF), an important biomass derived platform chemical, has received increasing attention as a key bio-refining building block. 5-HMF can be obtained from biomass by dissolution, hydrolysis or isomerization and conversion of mono-, di- and polysaccharides, using acid catalysts or base catalysts in mild conditions [1]. 5-HMF can be converted to a promising biofuel (2,5-dimethylfuran) and a broad range of useful derivatives, which are currently produced from petroleum [1,2,3,4]. The catalytic oxidation of 5-HMF can give rise to 2,5-furandicarboxylic acid (FDCA), which was listed as the top 12 value-added bio-based chemicals by the US Department of Energy. FDCA can be used as a substituent for terephthalic acid in the production of polyethyleneterephthalate and poly butyleneterephthalate. The reduction of 5-HMF can lead to products without consumption of H_2 such as 2,5-bis(hydroxymethyl)furan, and 2,5 bis(hydroxymethyl) tetrahydrofuran, which serve as alcohol components in the production of polyesters. Moreover, 5-HMF and its derivatives can be coupled with other chemicals such as acetone through aldol-condensation/ hydrogenation to obtain long chain liquid alkanes for diesel fuel [5]. 5-HMF is a yellow low-melting solid and having solubility in a range of solvents, viz., water, methanol, ethanol, benzene, acetone, chloroform, formaldehyde and ethyl acetate. It consists of a furan ring, containing both aldehyde and alcohol functional groups. The 5-HMF molecule with an odor of chamomile flowers possesses a boiling point between 114 and 116ºC and a density of 1.2062 g/cc at 25ºC [6]. It is generally produced by the acid catalysis of a hexose material resulting in loss of three molecules of water and in this process several side reactions including the re-hydration of the 5-hydroxymethylfurfural to levulinic acid and formic acid, and cross-polymerization to soluble polymers and insoluble humins also is of utmost importance. Several mechanisms are involved in the formation of 5-HMF from fructose by acyclic and cyclic pathways. Some groups followed the cyclic route concerning a fructofuranosyl cationic intermediate and others nodded towards the acyclic route involving an enediol intermediate. Researchers have also pointed out that 5-HMF synthesis is relatively more efficient and selective from fructose than from glucose [6]. Lignocellulosic biomass is the most abundant and non-edible biomass in nature, and is considered as a promising feedstock for the production of some chemicals. The efficient transformation of cellulose, which account for 40%–50% of lignocellulosic biomass, to chemicals is essentially important for the efficient utilization of biomass and has received much attention in recent years [5]. Cellulose is a crystalline polymer of D-glucose linked by β-1,4-glycosidic bonds. Owing to the huge amounts of

hydroxyl groups in cellulose, extensive intra- and intermolecular hydrogen bonding networks exist, which make its crystalline structure robust. Furthermore, cellulose macromolecules possess various kinds of C–C and C–O bonds. The selective cleavage of specific C–C or C–O bonds under mild conditions to obtain target chemicals remains a big challenge [5,7]. The crystallinity and complexity of cellulose super-molecular structure cause complete insolubility in aqueous and most of common organic solvents. The capability of ionic liquids to dissolve cellulose is a key for a promising selective method to produce 5-HMF [8,9]. Previous studies showed that ionic liquids combined with several metal chlorides are efficient on converting cellulose and even untreated biomass into 5-HMF [10,11,12,13,14]. Ionic liquids have some specific properties, such as very low vapour pressure, non-flammability, high thermal and chemical stability, and efficient solvent power for organic and inorganic substances [15]. Fructose is a good starting material, which can be smoothly converted into 5-HMF in varieties of reaction media, such as water/organic solvent biphasic system, dimethylsulfoxide (DMSO), dimethylacetamide (DMA)-lithium chloride (LiCl), and ionic liquid, with many different catalysts, including mineral acid, organic acid, metal salt, solid acid, and functionalized ionic liquid. However, fructose is of limited abundance in nature and typically at a higher cost than glucose [16,17,18,19]. Glucose is the most abundant monosaccharide in nature and, as the main monomer, can be produced from the hydrolysis of starch and cellulose. However, the obstacle of effectively converting glucose into 5-HMF has slowed the development of 5-HMF based biorefinery [19,20]. Sugarcane bagasse is a residue obtained from sugarcane after it is crushed to obtain the juice used for sugar and ethanol production [21]. Sugarcane bagasse contains appreciable amount of cellulose and hemicellulose, which can be de-polymerized by chemical or enzymecocktails into simple sugar monomers (glucose, xylose, arabinose, mannose, galactose, etc.) [22]. Conversion of the simple sugar monomers from sugarcane bagasse into 5-HMF in one bot reaction using low cost catalyst will support the development of production of value-added bio-based chemicals. The aim of this study was to convert sugarcane bagasse into 5-HMF using Tertabutylammonium bromide (TBAB), Tetramethylammonium chloride (TMAC) in DMSO and metal chlorides (chromium chloride, copper chloride, ferric chloride and cobalt chloride); in different reaction times and temperatures.

2. MATERIALS AND METHODS

2.1 Sample Collection and Preparation

Sugarcane bagasse sample used in this study was collected from Al-gunide sugar factory, Aljazeera- Sudan; during the production season of 2017. Sample was shade dried, milled and kept in polyethylene bags for further studies.

2.2 Chemical Compositions

Chemical composition of sugarcane bagasse was determined according to Tappi Standards Methods; which were T 207om93, T-204, T-211 and T-222; as described by Farhad Zeinaly et al. [23].

2.3 Catalyst Preparation

About 1 g of either TBAB or TMAC was weighted into clean 50 mL conical flasks, 0.2 g of metal chloride ($CrCl_3$, $CuCl_2$, $FeCl_3$ and $CoCl_2$) was added and then 15mL of DMSO was added too in each flask. The flask content was heated 100ºC on hotplate magnetic stirrer for 20 min, cooled and then kept for further studies.

2.4 5-HMF Production Procedure

About 1g of prepared sample was accurately weighted and added into flak containing the prepared catalyst. The flask content was heated at different temperatures (100, 120 and 150°C) and different reaction times (60, 90 and 120 min) on hotplate magnetic stirrer 20,000 rpm. After cooling 10mL of deionized water were added into the flask, stirred and centrifuged at 20,000 rpm. The product was filtered using 0.45 µm syringe filter and then kept for analysis.

2.5 Analytical Methods

The 5-HMF was quantified using HPLC (Shimadzu LC-20AD) with an ultraviolet detector at 284 nm and an Inertsil ODS-SP column (4.6×250 mm) at 35ºC. The mobile phase was methanol (20%v/v) at a flow rate of 1 mL/min. The 5-HMF product calculated based on standard curve methods.

3. RESULTS AND DISCUSSION

The prepared sample of sugarcane bagasse was subjected to chemical composition analysis and the obtained result was shown in Table 1.

Table 1. Chemical composition of sugarcane bagasse

Component	Percentage %
Cellulose	43.00
Hemicellulose	42.00
Lignin	23.17

*Values are means of triplicate

Comparing the results showed in Table 1 above with results obtained by Guimarães et al (2009) [21] we noted that our obtained result of cellulose was lower than their result 54.87 ± 0.53%; while hemicellulose result 16.52 ± 0.56, was lower than our result, but for lignin their result 23.33 ± 0.02%, was close to our obtained result. Misook Kim and Donal F. Day [24] their obtained results indicated that cellulose 42%, hemicellulose 25% and lignin 20%; where the cellulose result was closed to our obtained result, while lignin result was slightly lower than our result, but hemicellulose result was very low than our obtained result. Farhad Zeinaly et al. [25] their obtained results revealed cellulose 44.09%, hemicellulose 23.89% and lignin 21.42%; where cellulose result was closed to our result; also lignin result was slightly lower than our result; but for hemicellulose result it was very low compared to our obtained result. Cueva-Orjuela et al. [26] from their work reported that cellulose 59.1%, hemicellulose 24.3% and lignin 6.8%; where our results of cellulose was low compared to their obtain result; also our results of hemicellulose and lignin were high compared to their obtained results; these may be due to environmental issues. In general the results obtained for cellulose, hemicellulose and lignin in this study were found in the range of previous studies.

Table 2 showed the 5-HMF yield when using TBAB and TMAC in DMSO as co-solvent in addition to four metal chlorides, in different reaction times and temperatures; all values are means of triplicate. The results obtained when using TBAB and metals chlorides in DMSO gave high variation; high yield 53.41% was noted at 150ºC and 120 min reaction time when using the chromium chloride and TBAB in DMSO, then the yield 45.06% and 30.81% at same temperature and reaction times 90 and 60 min respectively. Also the yield 18.76%, 17.23% and 15.59% were noted at 130ºC and reaction times 120, 90 and 60 min respectively. The low yield 3.12%, 2.95% and 2.08% were noted at temperature 100ºC and reaction times 90, 120 and 60 min respectively. These results showed that increasing temperatures and reaction times was result in increasing the yield of 5-HMF as shown in the obtained results (Table 2); beside the best condition for high yield of 5-HMF was 150ºC and 120 min reaction time. For using copper chloride and TBAB in DMSO the yield was very low at all temperatures 100, 120 and 150ºC and all reaction times 60, 90 and 120 min; compared to the results obtained for chromium chloride above. This indicated that the copper chloride can be considered not a good catalyst for conversion of sugarcane bagasse into 5-HMF at this conditions and it may need more work to increase 5-HMF yield. However, the 5-HMF yields when using ferric chloride and TBAB in DMSO was also very low as when we used copper chloride at all conditions compared to chromium chloride results. In addition using cobalt chloride and TBAB in DMSO for production of 5-HMF; the highest yield noted 1.67% at 100ºC and reaction time 90 min and 0.91% at reaction time 60 min at the same temperature; while for 120 min no yield was observed. Also for 130ºC all reaction times showed no yield; in addition to 150ºC in reaction time 60 min was also showed no yield of 5-HMF. But for 150ºC and reaction times 90 and 120 min the 5-HMF yield were very low. These results were very low Comparing to the results of chromium chloride. Based on this result chromium considered the best for conversion of sugarcane bagasse into 5-HMF in TBAB and DMSO. The best condition for the conversion was 150^{o}C temperature and 120 min reaction time; also it was noted that as temperature increased the yield of 5-HMF increased.

The combination of $SnCl_4$ with a quaternary ammonium salt also realized the efficient conversion of glucose directly to 5-HMF. Tian et al. [27] obtained a 69% yield of 5-HMF using the $SnCl_4$-TBAB system in DMSO at 373 K. while during the conversions of starch and inulin using this bifunctional system, the yields of HMF were 21.5% and 62%, respectively. Cunshan et al. [1] studied synthesis of 5-HMF from glucose in H_2O, DMSO and 1-butyl-3methylimidazoliumchloride ([Bmim]Cl) catalyzed by metal chloride(III) ($FeCl_3.6H_2O$, $CrCl_3.6H_2O$ and $AlCl_3$). Under the optimal reaction conditions, 5-HMF yields of

Table 2. The yield of 5-HMF from sugarcane bagasse using TBAB and TMAC

NO.	Solvent	Catalyst	Temp (^oC)	t (min)	Yield (%)*	NO.	Solvent	Catalyst	Temp (^oC)	t (min)	Yield (%)*
A1	DMSO	TBAB	100	60	2.08	B1	DMSO	TMAC	100	60	6.52
A2		$CrCl_3$	100	90	3.12	B2		$CrCl_3$	100	90	10.28
A3			100	120	2.95	B3			100	120	9.80
A4			130	60	15.59	B4			130	60	29.38
A5			130	90	17.23	B5			130	90	35.18
A6			130	120	18.76	B6			130	120	37.04
A7			150	60	30.81	B7			150	60	63.27
A8			150	90	45.60	B8			150	90	74.99
A9			150	120	53.41	B9			150	120	89.23
A10	DMSO	TBAB	100	60	0.14	B10	DMSO	TMAC	100	60	0.69
A11		$CuCl_2$	100	90	0.10	B11		$CuCl_2$	100	90	0.69
A12			100	120	0.12	B12			100	120	1.02
A13			130	60	0.20	B13			130	60	5.13
A14			130	90	0.16	B14			130	90	0.64
A15			130	120	0.21	B15			130	120	7.00
A16			150	60	0.44	B16			150	60	14.58
A17			150	90	0.30	B17			150	90	11.86
A18			150	120	0.74	B18			150	120	43.45
A19	DMSO	TBAB	100	60	0.14	B19	DMSO	TMAC	100	60	-
A20		$FeCl_3$	100	90	0.13	B20		$FeCl_3$	100	90	0.14
A21			100	120	0.15	B21			100	120	0.10
A22			130	60	0.18	B22			130	60	0.74
A23			130	90	0.15	B23			130	90	0.92
A24			130	120	0.16	B24			130	120	32.91
A25			150	60	0.42	B25			150	60	59.76
A26			150	90	0.84	B26			150	90	27.30
A27			150	120	0.70	B27			150	120	25.31
A28	DMSO	TBAB	100	60	0.91	B28	DMSO	TMAC	100	60	-
A29		$CoCl_2$	100	90	1.67	B29		$CoCl_2$	100	90	-
A30			100	120	-	B30			100	120	-
A31			130	60	-	B31			130	60	-
A32			130	90	-	B32			130	90	0.54
A33			130	120	-	B33			130	120	1.19
A34			150	60	-	B34			150	60	0.34
A35			150	90	0.04	B35			150	90	4.33
A36			150	120	0.25	B36			150	120	7.04

Values are means of triplicate

54.43% and 52.86% were obtained in DMSO with $CrCl_3.6H_2O$ at 403 K and 480 min and $AlCl_3$ at 393 K and 240 min, respectively; where his result agreed with our result for chromium chloride, which gave also high yield of 5-HMF, while ferric chloride gave low yield in our study. Hussein Abou-Yousef et al. [8] performed direct conversion of cellulose into 5-HMF using single or combined metal chloride catalysts in 1-ethyl-3-methylimidazoliumchloride ([EMIM]Cl) ionic liquid. They demonstrated that $CrCl_3$ was the most effective catalyst for selective conversion of cellulose into 5-HMF 35.6%; while for $FeCl_3$ the yield was 23.6%; In addition $CrCl_3/CuCl_2$ was the most selective combination to convert cellulose into 5-HMF 39.9%. Their result agreed with our obtained result for chromium chloride as the most effective catalyst. Harishandra et al. [28] studied the formation of 5-HMF from glucose using tetrabutylammoniumchloride (TBAC) and chromium chloride (II); they obtained a 56% yield of 5-HMF; while for chromium chloride (III) the yield was 49%. Their results were also proved that Chromium chloride was most effective for 5-HMF conversion. The obtained results above indicted that chromium chloride was the best compared to other metal chlorides used in TBAB for conversion of sugarcane bagasse into 5-HMF; also increasing temperatures and reaction times were result in increasing the yield of 5-HMF.

Table 2 also showed the 5-HMF yield when using TMAC and four metal chlorides (chromium chloride, copper chloride, ferric chloride and cobalt chloride) in DMSO with different reaction times and temperatures; all values are means of triplicate. The results obtained demonstrated high difference; the high yields were 89.23%, 74.99% and 63.27% were noted at 150ºC and reaction times 120, 90 and 60 min respectively; when using the chromium chloride and TAMC in DMSO. Also the yields 37.04%, 35.18% and 29.38% at temperature 130°C and reaction times 120, 90 and 60 min respectively. Beside the low yield 10.28%, 9.80% and 6.52% were noted at 100°C and reaction times 90, 120 and 60 min respectively. It's clear that increasing temperatures result in increasing the yield of 5-HMF, also increasing reaction times were result in increasing the yield too. Based on these results chromium chloride can be considered best catalyst for conversion of sugarcane bagasse into 5-HMF under these conditions and the best condition for high yield was 150ºC and 120 min reaction time. For copper chloride and TMAC in DMSO the high yields was 43.45%, 14.58% and 11.86%, which were noted at 150°C and 120, 60 and 90 min reaction times respectively; while for 130°C the yield was 7.00% and 5.13% on reaction times 120 and 60 min respectively, the rest of results were very low. The 5-HMF yields were low compared to the yields obtained when using chromium chloride under the same conditions. Increasing temperatures and reaction times for copper chloride were also result in increasing the yield of 5-HMF. The best condition for conversion sugarcane bagasse into 5-HMF was 150ºC and 120 min reaction time.

However, the highest 5-HMF yield when using ferric chloride and TMAC in DMSO was 59.76%, which was noted at 150°C and reaction time 60 min; then 32.91% which was noted at 130°C and 120 min; also 27.30% and 25.31% were at 150°C and reaction times 90 and 120 min respectively. The rest of results were very low, although comparing these results with the result of chromium chloride, they were considered low; while they were high and better results compared with the copper chloride results. For Ferric chloride increasing temperatures result in increasing the yield of 5-HMF; but increasing reaction times not so. Increasing reaction time at high temperature 150ºC result in decreasing the yield of 5-HMF. The best condition of conversion sugarcane bagasse into 5-HMF using ferric chloride was 150ºC and 60 min reaction time. In addition using cobalt chloride and TMAC in DMSO for production of 5-HMF; the high yields 7.04% and 4.33% were noted at 150°C and reaction times 120 and 90 min respectively. Also at 130°C and 120 min reaction time the yield 1.19% was noted; at 100°C showed no yield of 5-HMF at all reaction time. Furthermore at 130°C and reaction time 60 min there was also no yield of 5-HMF. Comparing these results to the results obtained when using chromium chloride, copper chloride and ferric chloride yields were considered very low. Increasing temperatures and reaction times for cobalt chloride result in increasing the yield of 5-HMF; this indicted that for conversion sugarcane bagasse into 5-HMF using cobalt chloride high temperature must be used. Xinhua et al. [7] conducted efficient two-step process for converting microcrystalline cellulose into 5-HMF with ionic liquids under mild conditions. In the first step, high glucose yields of above 80% could be obtained from the cellulose hydrolysis by a strong acidic cation exchange resin in 1-ethyl-3-methyl imidazolium

chloride ([EMIM][Cl]) with gradual addition of water. In the second step, the resin was separated from the reaction mixture and CrCl3 was added which lead to a 5-HMF yield of 73% based on cellulose substrate. Yu et al. [29] presented a single- step process for cellulose conversion into 5-HMF by using an ionic liquid solvent system with CuCl2-CrCl2 catalyst, and obtained a 5-HMF yield of 55% under relatively mild conditions of 120ºC in 8 h reaction time.

Based on these results chromium chloride considered the best for conversion of sugarcane bagasse into 5-HMF in TMAC and DMSO compared to other chlorides in this study; and the best conditions for the conversion were 150°C temperature and 120 min reaction time. In addition comparing TBAB and TMAC; chromium chloride gave high yield of 5-HMF in TMAC than TBAB. As TMAC and chromium chloride in DMSO gave high yield of 5-HMF from sugarcane bagasse, it can be utilized in large scale as catalyst for yielding 5-HMF from cellulosic biomass. Beside TMAC cost is lower than the cost of other catalyst such as [Bmim] Cl which gave high 5-HMF yield but its cost was very high also.

4. CONCLUSION

Recently 5-HMF has been regarded as one important precursor for the production of biofuels and biobased compounds. The obtained results showed that temperatures, reaction times and metal chloride types affect the yield of 5-HMF form sugarcane bagasse. The high yield of 5-HMF 53.41% was at 150°C and 120 min reaction time when chrome chloride was used in TBAB and DMSO; also the high yield 89.23% was noted at the same condition when chromium chloride was used in TMAC and DMSO. Other metal chlorides showed low yields in Tetrabutylammonium bromide; while better yields when using Tetramethylammonium chloride; copper chloride gave better yield, then ferric chloride and cobalt chloride. These results proved that chromium chloride is better catalyst for yielding 5-HMF from sugarcane bagasse in both TBAB and TMAC chloride; but is best in TMAC.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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