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A review on conversions of Furfural: Gateway for numerous applications

Glanish Jude Martis[a], O. Sneha[a], D Rajkumar Bhat[a] and Praveen S Mugali[a*]

[a]*Department of P.G. Studies in Chemistry, Alva's College (Autonomous), Moodubidire, Dakshina Kannada, Karnataka 574227, India*

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A B S T R A C T

Due to highly functionalized molecular structure, furfural has made a huge impact in renewable platforms. As there is a big need of producing renewable sources, this review reveals several attempts made by researchers distributed in every nook and corner of the world to produce furfural-based derivatives. As furfurals are converted to various forms, they reflect many applications both with respect to economy and environment friendly approach. In this review, a summarized detail of various reactions of furfural substrates are provided which includes Diels-Alder reactions, Oxidations, Hydrogenations, Hydrogenolysis, Conversions to C6 Carboxylic acids, Furfuryl amines, Cyclopentenones and other useful compounds. These compounds and their applications in various fields are mentioned systematically. The chemical as well as biocatalytic conversions are explained in detail which will help to know the future challenges and hurdles when design and optimization of furfural derivatives are concerned.

1. Introduction

Furfural is the most widely discussed heterocyclic compound since its isolation in the year 1832. The core structure contains a Furan ring and a reactive aldehydic group at C2 position. Furfural grabbed its great attention from its production from branched pentose and hexoses.[1] Xylose when dehydrated gives Furfural in batch mode using microporous catalysts in solid phase.[2] Rice hull when hydrolyzed using sulphuric acid formed furfurals with lower rate of production whereas metallic oxides and chlorides when used as catalysts increased the production.[3] Carbon-based sulfonated catalysts when used under continuous distillation of *Acacia mangium* derived xylose transformed into furfural with optimal catalytic dosage.[4] 5-hydroxymethylfurfural (HMF) is a promising compound in biofuel and petroleum industry [5] which is produced from the conversion of fructoses and glucoses by the use of N-heterocyclic carbenes as catalysts as well as proficient ligands over solid support.[6] Another utility of producing HMF was from lignocellulosic biomass in a single step reaction using *N, N'*-dimethylacetamide (DMA) containing LiCl as solvent. [7]. We know that, Lignocellulose is composed of hemicellulose, cellulose and lignin and it is known for its renewability as it is distributed worldwide.[8,9] From the similar lignocellulosic biomass, furfurals are derived to give C4 and C5 chemicals.[10] When nature is concerned, the fuel reserves are depleting day by day for which there is a huge demand for developing novel renewable as well as sustainable resources.[11–15] Hence, a genuine attempt was made to present this review highlighting the advantages of furfurals.

2. Furfurals giving Diels Alder Reaction:

Direct Diels Alder (DA) reaction was fetched from biomass as starting material in aqueous medium wherein the reaction takes place in between furfural **1** and a diene **2** to give adduct **3** via **4**. Compounds **3** and **4** exhibit *endo-Z-endo-Z*-4,5-cis or *exo-Z-exo-Z*-4,5-cis configuration.[16] This is a consequence of the DA mechanism. New and classical approaches were developed with obtained furfural **1** to give adduct **6** from the DA reaction of compound **5.** (**Scheme 1**). Since a major limitation of poor sustainability was offered, furfural **1** was then tried to act as diene reacting with *N*-methyl maleimide **7**

* Corresponding author
E-mail address praveen.mugali@gmail.com (P. S. Mugali)

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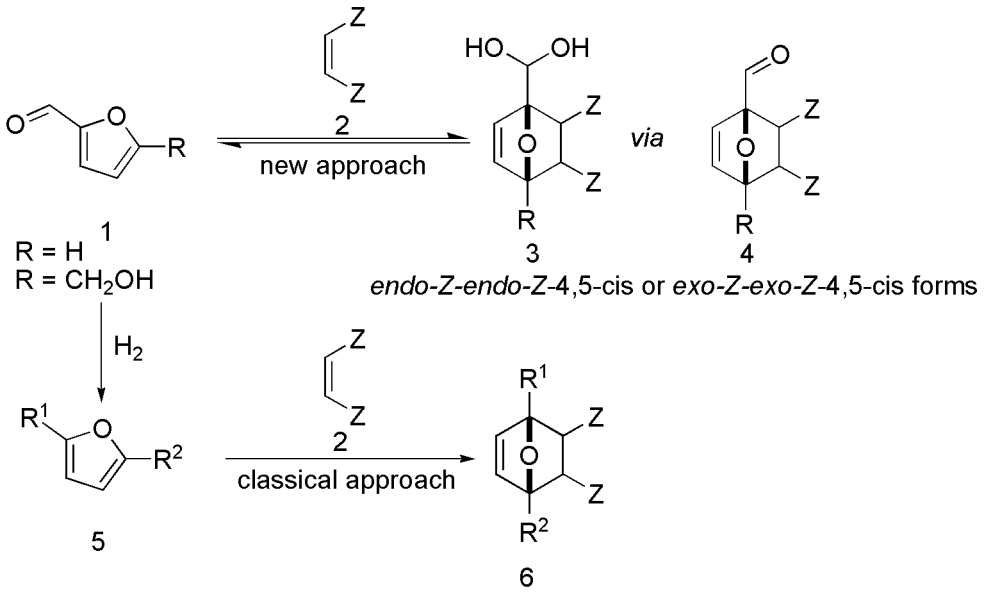
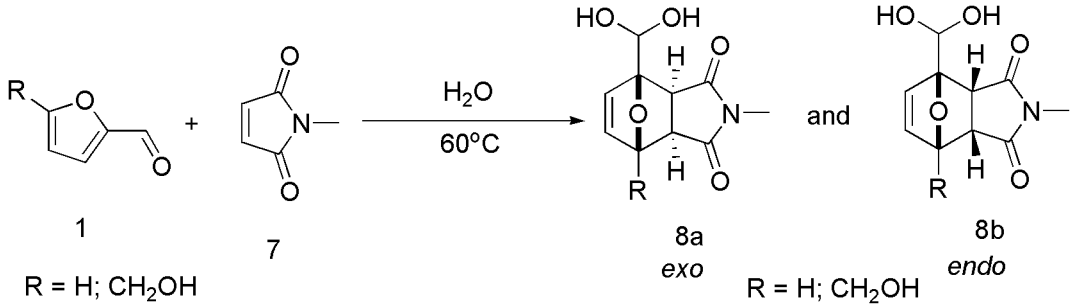
serving as dienophile under aqueous conditions at 60°C to combat the problem of sustainability. This reaction yielded both *exo*- as well as *endo*- products **8a** and **8b** respectively. (**Scheme 2**).[17]

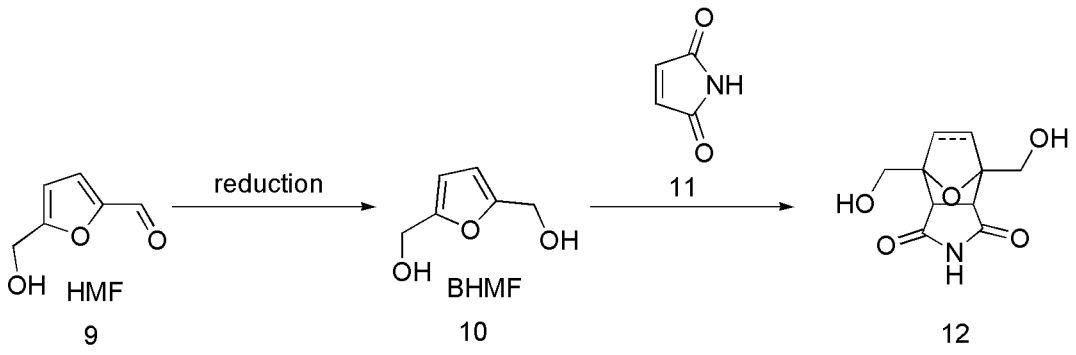


Scheme 1. A new approach to direct DA reaction of biobased formyl-functionalized furans.



Scheme 2. Direct DA cycloaddition of Furfural and 5-HMF with *N*-methyl maleimide

In order to produce sustainable polycyclic systems, Kucherov *et al.,* first reduced HMF **9** to BHMF **10** which was subsequently subjected to undergo water mediated DA reaction with maleimide **11** to yield sustainable norcantharimide core **12**. (**Scheme 3**)[18].

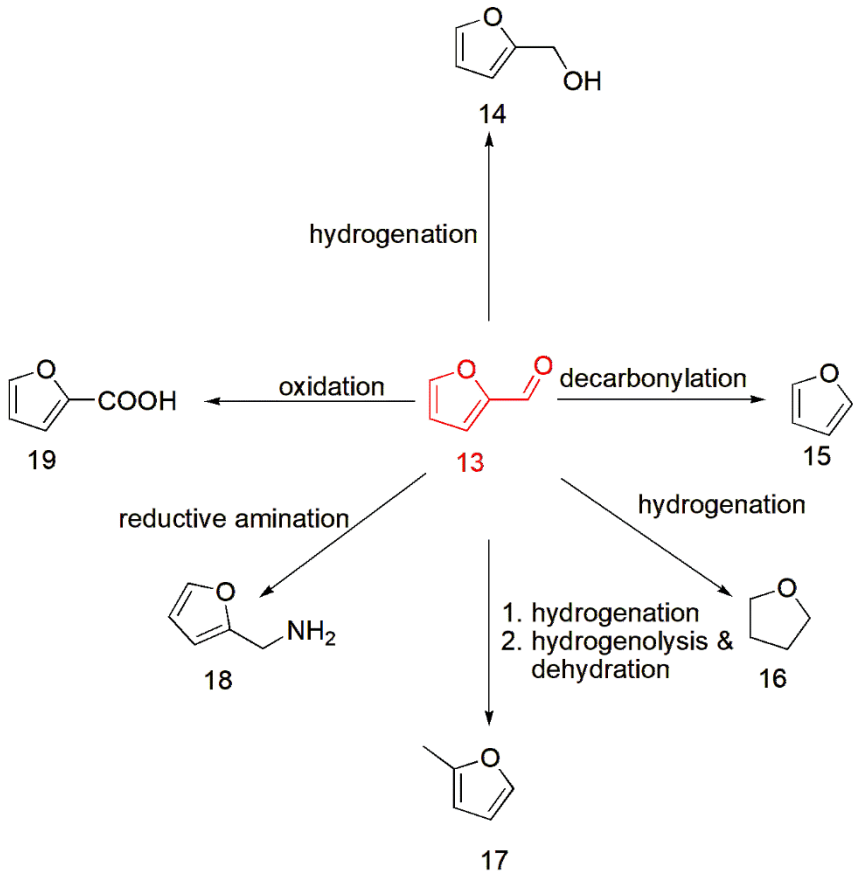


Scheme 3. Sustainable production of tricyclic non-planar compounds from HMF via DA reaction.

3. Furfurals as a source of economy:

One of the most extensively used furfural derivatives is furfuryl alcohol **14** which is produced through hydrogenation of Furfural **13** by Audemar *et al.,*[19] is found to have profound uses as building blocks in the synthesis of drugs, strippers in paint, modification of wood and viscosity condenser in epoxy resins.[20] For removal of dienes from other hydrocarbons, furfural is used as a solvent in petrochemical refining. Due to the polarity of furan ring, furfural is known for its extreme selectivity towards aromatic compounds as well as unsaturated molecules.[21] Apart from all these, it is susceptible to various

aldehydic reactions like production of furan **15** by decarbonylation, Tetrahydrofuran **16** by hydrogenation, 2-methyl furan **17** by hydrogenation and subsequent hydrogenolysis with dehydration, furfurylamine **18** by reductive amination and furoic acid **19** by oxidation. (**Scheme 4**)[22]

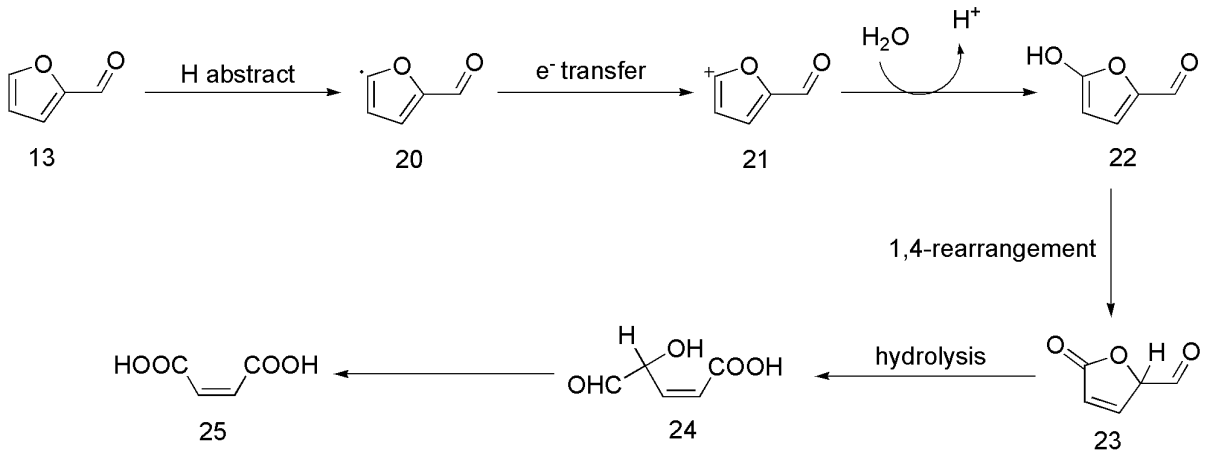


Scheme 4. Derivatives of Furfurals

4. Furfural Chemistry

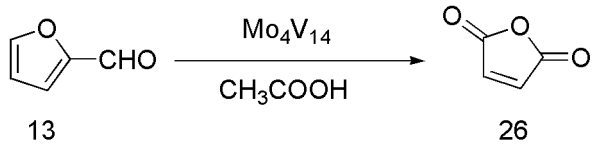
4.1 Various oxidations of Furfurals

Guo *et al*., proposed a detailed mechanism of conversion of furfural **13** to maleic acid **25** catalyzed by phosphomolybdic acid in different biphasic systems. As per the mechanistic approach, first and foremost the abstraction of a proton takes place to give a radical furfural **20** which undergoes an electron transfer giving a positively charged species **21**. This cation is then attacked by H_2O to give compound **22**, later undergoing 1,4-rearrangement to give compound **23** whose hydrolysis will yield compound **24**. Final oxidation of compound **24** produced maleic acid **25** which is an important value-added chemical. (**Scheme 5**)[23]



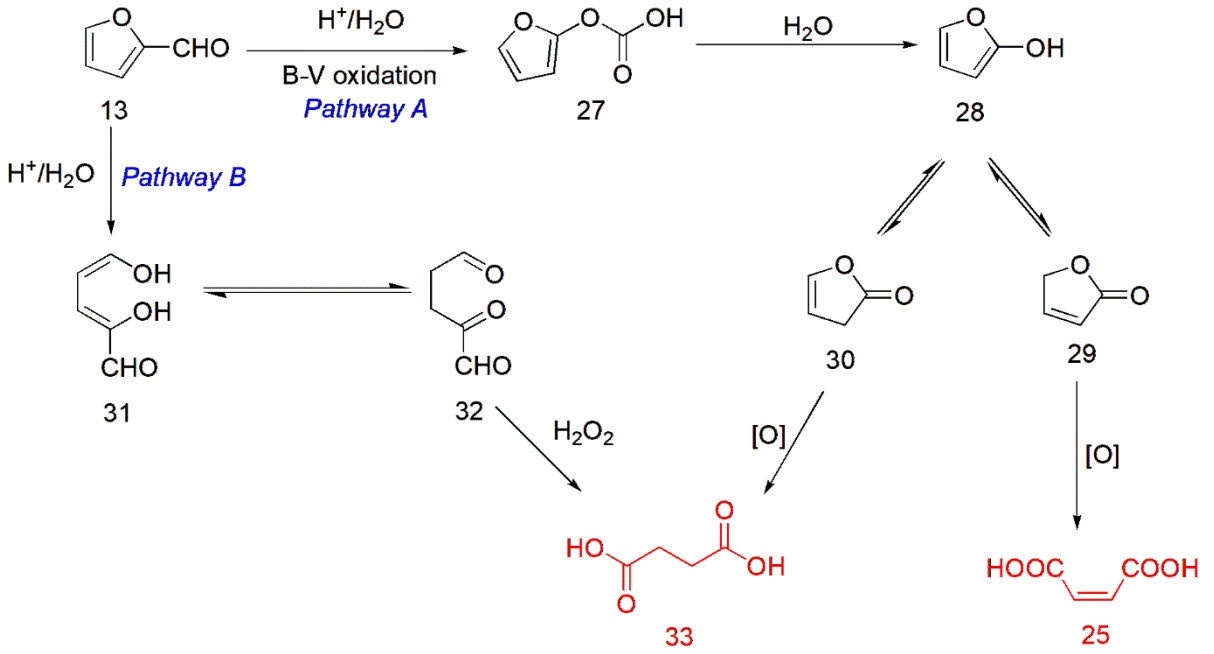
Scheme 5. Proposed mechanism of Furfural conversion to Maleic Acid

Li *et al.,* gave an exceptional method of producing maleic anhydride **26** from furfural **13** via heterogenous catalytic system involving binary Mo-V metal oxides. In this method, Mo_4V_{14} was used as catalyst in acetic acid solvent under optimal conditions. (**Scheme 6**)[24]



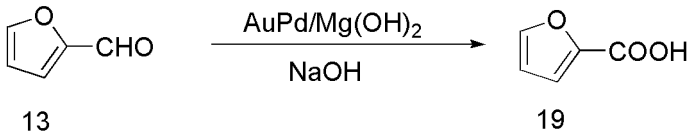
Scheme 6. Conversion of Furfural to Maleic Anhydride

Along with Maleic acid **25** and Maleic Anhydride **26**, Succinic acid **33** is also regarded as one of the promising value-added chemicals which finds its applications in the production of pharmaceuticals, fuel additives, biopolymers and many other chemicals to be produced from biomass.[25] For producing Succinic acid **33**, Wang *et al.,* selectively oxidized furfural **13** to 2(5*H*)-furanone **29**, succinic acid **33** and maleic acid **25** using formic acid as the catalyst and ethyl acetate as solvent. According to them, the first pathway shows the Baeyer-Villiger (B-V) Oxidation of furfural **13** takes place to give 2-formyloxyfuran **27** which is hydrolyzed to 2-hydroxyfuran **28** and its tautomers **29** and **30** wherein oxidation of **29** and **30** gave maleic acid **25** and succinic acid **33** respectively. In the second pathway, furfural **13** is opened up to form a dienol **31** whose keto-enol tautomerisation leads to the formation of a diketo aldehyde **32** and its oxidation gives succinic acid **33**. (**Scheme 7**)[26]



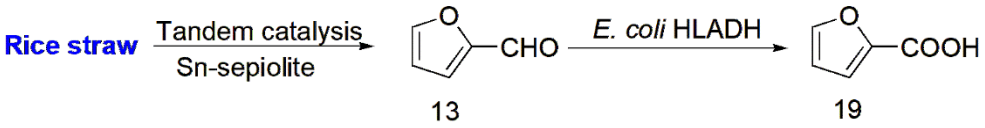
Scheme 7. Proposed reaction pathways of oxidation of furfural

Giordano *et al.,* proposed a method of converting furfural **13** to furoic acid **19** using Au/ZTC catalyst in the absence of a base.[27] Apart from this, selective oxidation is also reported using Ag_2O/CuO[28] and $AuPd/Mg(OH)_2$[29]. The importance of furoic acid **19** is found in the production of pharmaceutical drugs, fragrances and biofuels. Earlier, furoic acid was produced via Cannizaro reaction in the presence of a base. (**Scheme 8**)[30].



Scheme 8. Conversion of Furfural to Furoic Acid

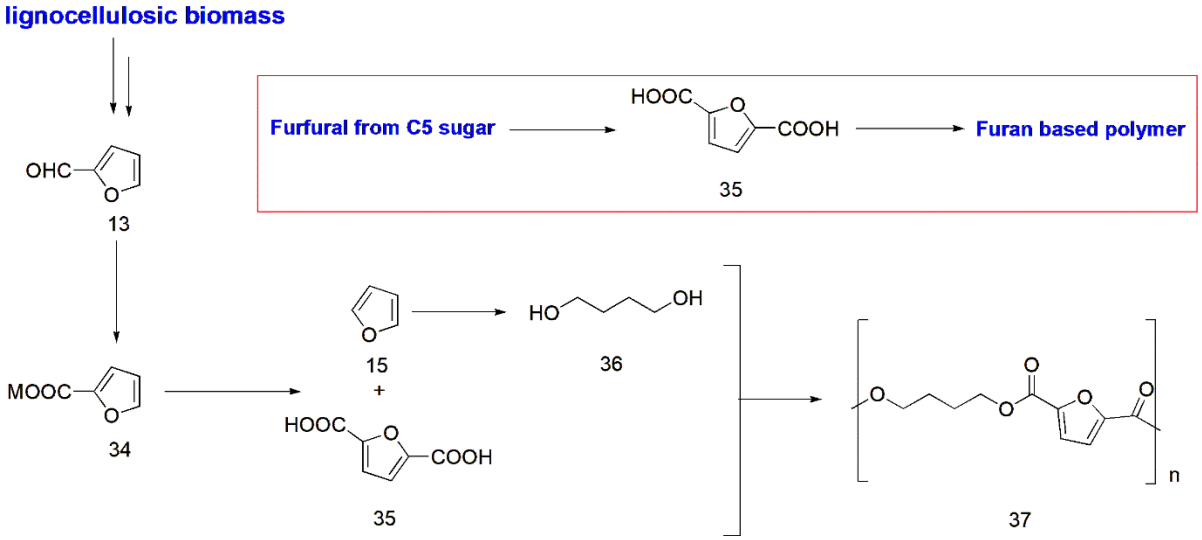
Peng *et al.,* gave a biocatalytic method of producing furoic acid **19** via furfural **13** from rice straw through tandem catalysis with Sn-sepiolite. In this method, conversion of rice straw to furfural **13** takes place via tandem catalysis. Later the oxidation of furfural **13** with *E. coli* harboring horse liver alcohol dehydrogenase (HLADH) enzyme was done at 170°C for 20 minutes to give furoic acid **19**. (**Scheme 9**)[30] Mutti *et al.,* showed a similar type of oxidation using three recombinant aldehyde dehydrogenases (ALDHs) [31].



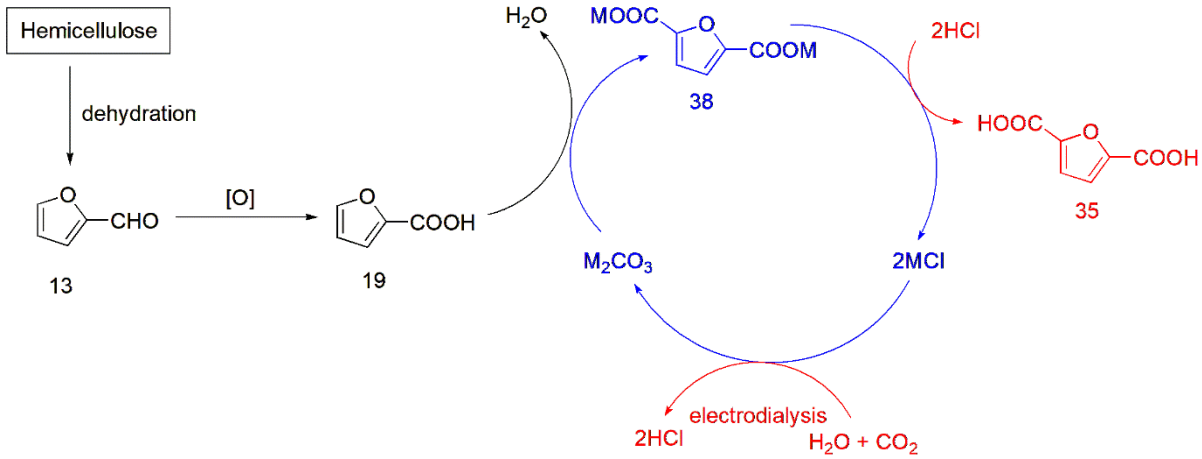
Scheme 9. Biocatalytic conversion of furfural to furoic acid

4.2 Furfurals into C6 Carboxylic acids

C6 carboxylic acids like 2,5-furandicarboxylic acids are prepared from furfural **13** as reported by Fu and coworkers. Poly(butylene 2,5-furandicarboxylate) (PBF) **37** was obtained via multistep catalytic transformation where disproportionation of furoate **34** to give furan **15** and 2,5-furandicarboxylate (FDCA) **35** takes place with the complete utilization of carbon. Furan **15** obtained is hydrogenated and hydrolyzed to yield 1,4-butanediol (1,4-BDO) **36** which polymerizes with 2,5-FDCA **35** to give final compound **37**. (**Scheme 10**).[32] Similarly, Kanan *et al.,* produced 2,5-FDCA **35** from 2-furoic acid **19** via CO_3^{2-} promoted C-H carboxylation in high yield. Here the reaction is carried out by heating a salt mixture containing cesium carbonate under CO_2 atm. After 5h, the reaction was completed and final protonation was done using strong acids such as HCl. This method is considered to be eco-friendly when compared to that of converting HMF **9** to FDCA **35** (**Scheme 11**)[33]

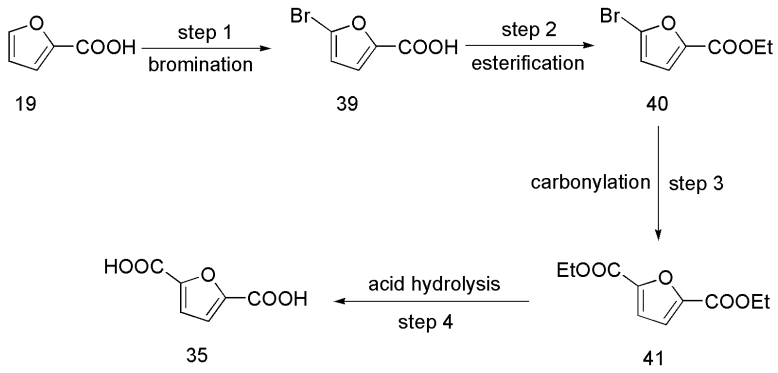


Scheme 10. Production of FDCA from lignocellulosic biomass



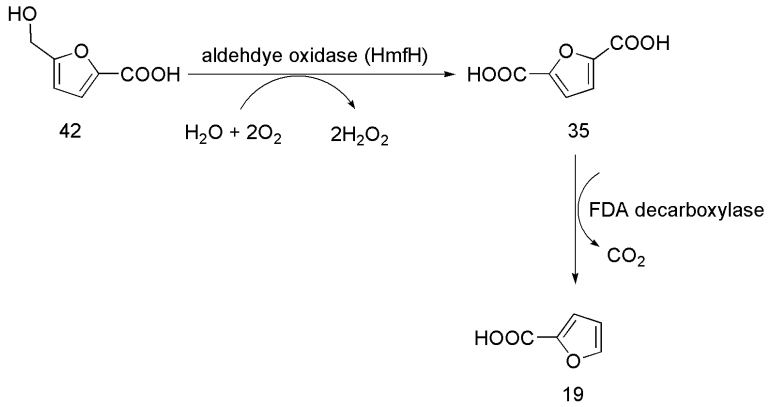
Scheme 11. Production of FDCA from hemicellulose through furfural using CO_3^{2-} promoted C-H carboxylation

Yin *et al.,* gave a new method for the production of FDCA **35** from furoic acid **19** via a series of reactions involving bromination using Br_2 in the presence of CCl_4 in acetic acid at 60°C for 24 h. This Step 1 gave 86% of brominated compound **39**. This compound **39** was then esterified in step 2 using H_2SO_4 and refluxed for 60 h in EtOH to give 87% of compound **40**. In step 3 compound **40** was made to undergo carbonylation using CO in the presence of $Pd(dppf)Cl_2$ catalyst with $NaHCO_3$ in EtOH to give 91% of diester **41** which was finally hydrolyzed with MeOH in the presence of H_2SO_4 at 160°C for 8h to give 96% of FDCA **35** in step 4. (**Scheme 12**)[34]



Scheme 12. Synthetic strategies to obtain FDCA from furoic acid

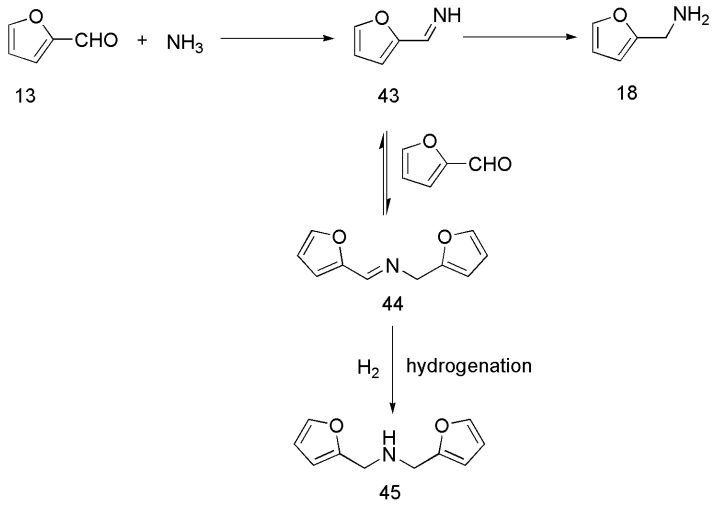
Leys *et al.,* put forward an enzymatic approach of synthesizing FDCA **35** from HMF acid **42**. HmfH, an aldehyde oxidase from *Pelotomaculum thermopropionicum* is used to transform HMF acid **42** to FDCA **35** which later with the aid of FDCA decarboxylase gives furoic acid **19**. This is one of the best reported biocatalytic methods for the production of FDCA **35**. (**Scheme 13**)[35]



Scheme 13. Biocatalytic approach of producing FDCA

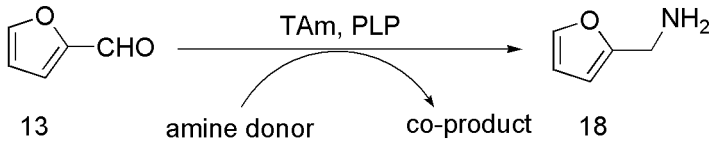
4.3 Furfural into Furfurylamine

Selective reductive amination of furfural **13** leads to the formation of furfurylamine **18**. This has grabbed its utmost attention due to its application in the development of pharmaceuticals, pesticides, antihypertensives and many more. Furfurylamine **18** is manufactured by making use of aqueous solution of ammonia and molecular hydrogen in an ecofriendly manner (**Scheme 14**)[36] Pt, Ir, Au based catalysts were employed by Martinez and coworkers to selectively reduce furfural with aniline[37] Apart from the above-mentioned metal catalysts, Raney Cobalt[38], Nickel over Al_2O_3 with LaO_x[39], Ru/HZSM-5[40] were also proved with highly efficient yields.



Scheme 14. Formation of furfurylamine from furfural

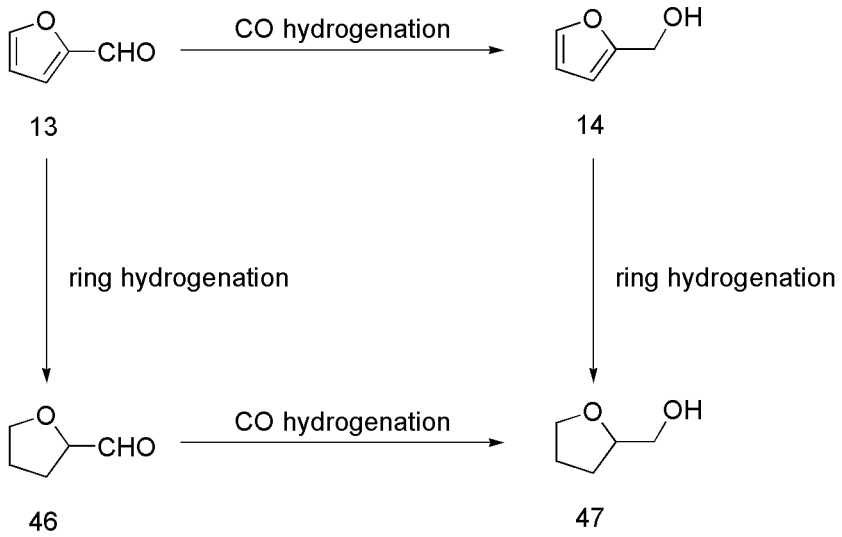
Hailes *et al.,* gave a biocatalytic method for the selective reductive amination of various furfural derivatives using Transaminase (TAm) with co-factor pyridoxal-5-phosphate (PLP) (**Scheme 15**)[41].



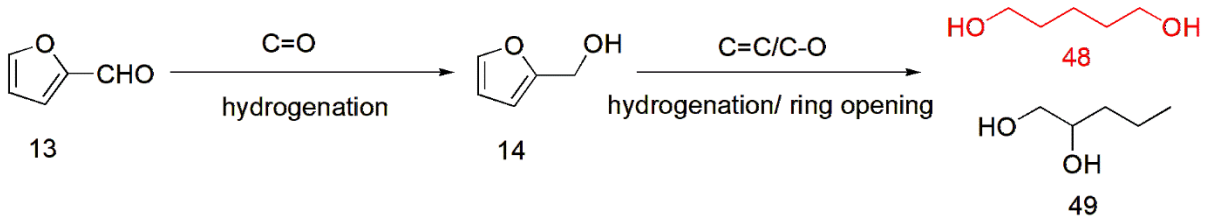
Scheme 15. Biocatalytic conversion of furfural to furfurylamine

4.4 Hydrogenation of Furfural

The most significant product obtained after hydrogenation of furfural **13** is furfuryl alcohol **14**. In industrial production, selective hydrogenation is performed in liquid and gas phases using copper chromite.[42] Wang *et al.,* used Pd/UiO-66 for the aqueous phase hydrogenation of furfural **13** which gives tetrahydrofurfuryl alcohol **47**. (**Scheme 16**)[43] This compound **47** is also obtained by making use of Pd catalyzed reaction supported by Si-MFI molecular sieves.[44] Halilu *et al.,* reported one pot synthesis of furfuryl alcohol using magnetic $Fe(NiFe)O_4$-SiO_2 nano catalyst.[45] On the other hand, some different catalysts were employed for the hydrogenation purpose like Cu-Fe[46], Ni-Fe layered double hydroxide[47] to give furfuryl alcohol **14** whose subsequent ring opening reaction with CuCo bimetallic catalysts gave 1, 5-pentanediol **48** and 1,2-pentanediol **49**. (**Scheme 17**)[48]



Scheme 16. Reaction network of furfural hydrogenation

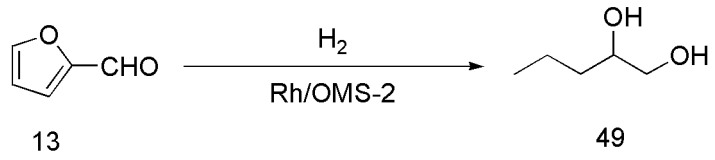


Scheme 17. Reaction pathway for the hydrogenation of furfural to 1,5-pentanediol

Instead of using H_2 for hydrogenation, catalytic transfer hydrogenation (CTH) can be used for obtaining furfuryl alcohol **14** with the aid of carbon encapsulated Fe_3O_4[49], core-shell magnetic zirconium hydroxide[50], carbon coated CuNi[51] and Zr based MOF-88 viz., metal organic framework[52].

4.5 Hydrogenolysis of Furfural

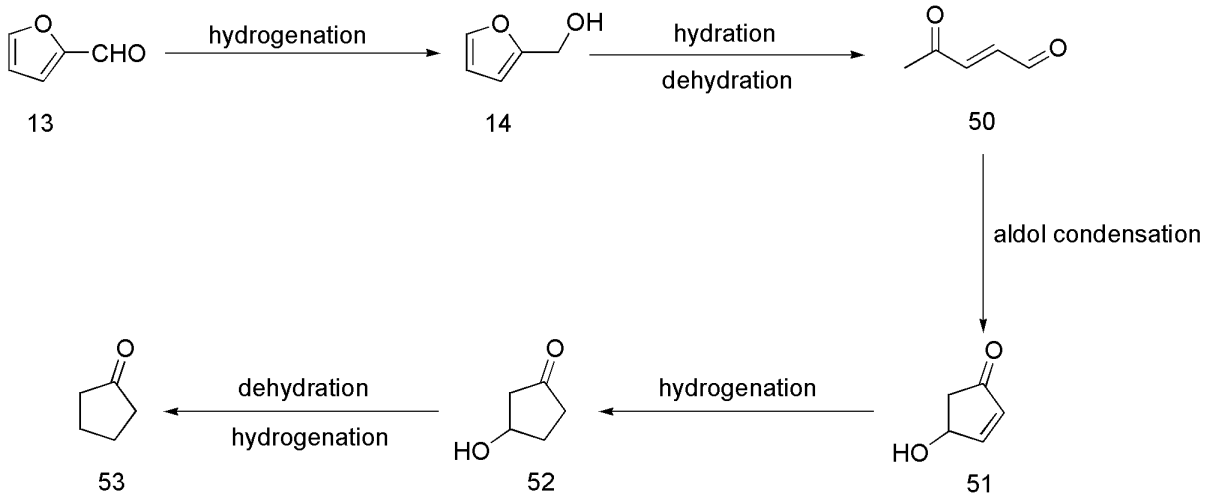
The direct conversion of furfural to pentanediol is difficult as it yields many byproducts and low sensitivity[53,54]. Thus, Tetrahydrofurfuryl alcohol (THFA) **47** was hydrogenolyzed to Pentanediol as it is an important source of biofuel, fine chemical intermediate and monomer of several polyesters. In order to obtain Pentanediol, Pt/Co_2AlO_4 was employed with THFA 47 as an intermediate by Lu *et al.,*[55]. Yadav *et al.,* developed a method of producing specific 1,2-pentanediol **49** by using a bifunctional Rh supported over octahedral molecular sieve (OMS-2) catalyst. (**Scheme 18**)[56]



Scheme 18. Hydrogenation of furfural to 1,2-pentanediol via Rh/OMS-2

4.6 Furfural to Cyclopentanone

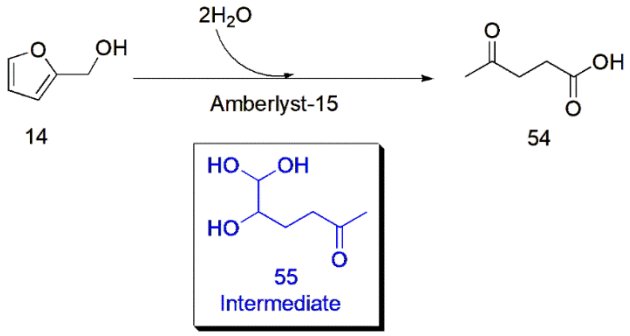
Cyclopentanone, derived from furfural **13** is known for its versatility as it is widely applicable in the production of fuels, pharmaceutical compounds, fragrances, flavors, fungicides etc.[57–59] The mechanism behind the conversion of furfural **13** to cyclopentanone **53** is still under debate and is it widely accepted that hydrogenative ring arrangement is the key step leading to the formation of the product. For this reaction mechanism, several routes and intermediates have been proposed which may be in dependence with the type of catalysts, reaction conditions etc.[60–63] Li *et al.,* gave the synthetic route for the production of cyclopentanone **53**. First, furfural **13** was hydrogenated to furfuryl alcohol **14** whose ring opening led to the formation of 2-pentene-1,4-dione (2-PED) **50**. Intramolecular Aldol reaction of **50** gave 4-hydroxy-2-cyclopentenone (HCPEO) **51** which was further hydrogenated to yield 3-hydroxycyclopentanone (HCPO) **52**. This hydrogenated product **52** was re-hydrogenated followed by dehydration and gave cyclopentanone **53**. (**Scheme 19**)[59]



Scheme 19. Reaction pathway for the production of cyclopentanone from furfural

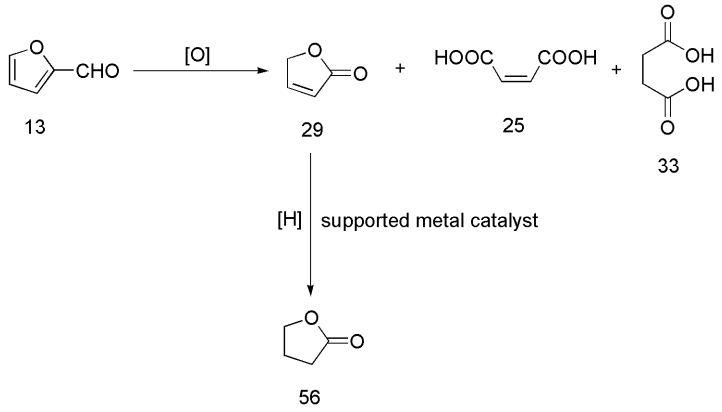
4.7 Furfural to other beneficial compounds

Levulinic acid **54**, derived from furfural **13** is one of the prominent building blocks for pharmaceutical and food industry[64] Maldonado and coworkers tried various experiments to know the actual protocol of hydrolysis of furfuryl alcohol **14** to levulinic acid **54** via intermediate 4,5,5-trihydroxypentan-2-one **55** with the help of Amberlyst-15 catalyst in aqueous phase. (**Scheme 20**)[65] Dumesic *et al.,* used sulfuric acid to hydrolyze furfural **13** from hemicullouse to levulinic acid **54** in a bi-phasic reactor having alkylphenol solvents.[66]



Scheme 20. Synthesis of levulinic acid from furfuryl alcohol

γ-butyrolactone (GBL) **56** is extensively used as a core material for fine medicine and chemical.[67-69] Wang *et al.,* studied the method of converting furfural **13** to GBL **56** where they first oxidized furfural **13** to 2(5H)-Furanone **29** along with maleic acid **25** and succinic acid **33** using formic acid as the catalyst and hydrogen peroxide as oxidant. Compound **29** was further hydrogenated over a metal catalyst to give GBL **56**. (**Scheme 21**)[70]



Scheme 21. Synthesis of γ-butyrolactone (GBL) from furfural

However, this work confirms the high importance of organic compounds in different fields due to their various uses as reported before.[71,72] Furthermore, EDG functionalised furans are especially active in the reaction with electrophilic alkenes. On the other hand, EWG functionalised furans are especially active in the reaction with nucleophilic alkenes. The source of this phenomenon was recently explained in the framework of the Molecular Electron Density Theory.[73]

5. Conclusion

Furfurals are known for their vast and wide applications due to which they draw great attention. Conversions of furfurals are important to give several value-added chemicals. Industrially, furfural derivatives are a great economic source. Their conversions with significant yields mesmerize industries to go for bulk-scale reactions. As pharmaceuticals play a pivotal role in the health sector of mankind, furfural derivatives contribute a lot in the production of medicinal targets which help in curing various diseases and in development of drugs. Due to all these benefits, it makes scientists and chemists to synthesize more novel furfural derivatives and enhance research. The chemistry behind furfural was not easy as discussed in the earlier sections, but now due to the continuous efforts made by the worldwide researchers they have made them more accessible and handier. The most influential fact is that the principles of green techniques employed for producing furfurals are of great advantages. As our nature is facing numerous problems namely, pollution, greenhouse effect, ozone depletion, global warming etc., it is our responsibility to combat these problems and give effective and eco-friendly methods in synthesizing furfurals. Thus, this review presents several biocatalytic methods reported in synthesizing various furfural-based derivatives.

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