Chapter

Furfural: A Versatile Derivative of Furan for the Synthesis of Various Useful Chemicals

Kazeem Adelani Alabi, Rasheed Adewale Adigun, Ibrahim Olasegun Abdulsalami and Mariam Dasola Adeoye

Abstract

Furfural, a five-membered heterocyclic aromatic hydrocarbon derivable from acid hydrolysis of sugar cane bagasse, maize cob, rice husk or any cellulose-containing material, is useful in the synthesis of a range of specialized chemical products. Its condensation with nitromethane in basic medium yields 2-(2-Nitrovinyl) furan. This functional group (nitrovinyl) has been documented as a potent anti-microbial agent against gram-positive and gram-negative bacteria, with more potency against the gram-positive strains. The reaction of urea and thiourea with furfural yields bisimines-1,3-bis[(*E*)-furan2-yl)methylene]urea, and 1,3-bis[(*E*)-furan-2-yl) methylene]thiourea respectively. The two compounds are good antimicrobial agents in addition to the latter as a potential dye for wool and cotton fabrics with different hues. Also the reaction between acetophenone and furfural (an aldehyde) in a basic medium yields the chalcone: (*E*)-3-(furan-2-yl)-1-phenylprop-2-ene-1-one. This chalcone has been confirmed as a good antifungal agent and wood-protector against termite attack. Thus, chemical modification of the aldehyde functional group of furfural to nitro, imine and chalcone groups imparted different activities on furfural.

Keywords: Furfural, Furan, 2-(2-Nitrovinyl) furan, Bisimine, Chalcone, Extraction

1. Introduction

Furfural is a utility chemical. It is very simple to manufacture, the raw material is always agricultural waste, and the cost of production is reasonably low and affordable. It is fondly called "Gold from Garbage" [1]. Mostly, undiluted hydrogen tetraoxosulphate (VI) acid is used for the extraction of furfural from biomass that contain pentosans, which are aldose sugars, that composed of small rings formed from short five-member chains, that constitute a class of complex carbohydrates, present in the cellulose of many woody plants such as bagasse (sugar cane waste) corn cobs, rice, and oat hulls, etc. [2].

Furfural is a heterocyclic aldehyde with oxygen as the heteroatom replacing one of the carbon atoms of the five membered ring. It has a density of 1160 kg/m³ and boils at 161.7°C. It is though, colorless, but changes to dark brown when it reacts with the atmospheric oxygen. It has other names apart from furfural; furaldehyde, 2-furanaldehyde, and 2-carbaxylaldehyde [3].

The flexible production route of furfural makes it to be the most commonly produced chemical in the industry, in addition to its many industrial uses and renewability. It is one of the top value-added chemicals that is being produced from biomass. Furfural and its derivatives have been extensively used in plastics, pharmaceutical, agrochemical and it has been reported to be used as dye precursors in the synthesis of diazo disperse dyes [4].

Sometimes, it is used as a solvent or starting material for the production of various chemicals. In order to increase its production because of its usefulness, furfural is now synthesized in the laboratory through different sources [2].

The process of furfural production is well understood but it's limited by low yield, very high energy consumption, and environmental and safety risks attached to the corrosive homogenous acid catalysts being used [5]. Hence, the existing method is being modified to make it green by the use of heterogeneous catalysts. Westpro-modified Huaxia Technology and Supra Yield are two improved processes where high furfural yields, recovery of the catalysts and high purity are obtainable [6]. Hybrid solid acid catalysts are recently used to achieve better dehydration of xylose to form furfural [4]. Furfuryl alcohol has been used for the production of furan resin prepolymer material which is useful for the production of different thermoset polymers [4].

2. Reactivity of furfural

Furfural undergoes some chemical reactions to yield different products. Among the reactions are hydrogenation, oxidation, decarboxylation/decarbonylation and condensation reactions.

2.1 Hydrogenation of furfural

The hydrogenation of furfural is catalyst dependent. The carbonyl functional group (C=O) or the aromatic ring will be reduced [7]. Almost all hydrogenation catalysts are capable of hydrogenating the carbon–carbon double bonds (C=C) and the carbonyl group (C=O) of furfural. The control of the hydrogenation process of these bonds has been one of the most important subjects in the developmental study of catalysts [8]. The addition of hydrogen to the C=C bond is more thermodynamically and kinetically favorable compared to the C=O bond. There is a need for a chemoselective hydrogenation catalyst within a standard conditions [9]. Hydrogenation reaction takes place in liquid and gaseous phases. Despite the high yields reported for the liquid phase, industrial hydrogenation of furfural is mostly carried out in the gas phase because of its lower cost as compared to the liquid phase (**Figure 1**) [10].

2.2 Oxidation of furfural

Furfural is easily oxidized even with atmospheric air to 2-furoic acid which is a heterocyclic carboxylic acid (**Figure 2**). Its name is derived from the Latin word furfur, meaning bran. The salts of this acid is called furoate. Many scientists/



furan-2-carbaldehyde

tetrahydrofuran-2-carbaldehyde

Figure 1. *Hydrogenation of furfural.*



furan-2-carbaldehyde

furan-2-carboxylic acid

Figure 2.

Oxidation of furfural to furoic acid.

researchers have worked in this area (oxidation of furfural). In addition, oxidative condensation of furfural with alcohol has produced biofuel useful in the aviation industry [11].

Gold supported catalyst condensed furfural with ethanol in the presence of oxygen molecule under mild conditions [12]. Au/ZrO₂ catalysts was also used in another reaction to produce furoate ester [13]. Oxidative esterification of furfural with oxygen and methanol was also performed [14]. The effect of catalyst was evaluated in oxidative esterification of furfural [15]. The furfural can also be oxidized to produce maleic acid in the presence of hydrogen peroxide and a solid catalyst. The maleic acid can be dehydrated to maleic anhydride. These compounds have many applications in the industry. They are used in the manufacture of polymers, resins, pharmaceuticals, and biofuels. The oxidation of furfural into maleic acid involves two steps; decarbonylation and oxidation.

2.3 Decarboxylation/decarbonylation reaction

Two methods are well known to transform furfural into furan. When furfural is passed over metal surface in the complete absence of air and water at a very high temperature; furan and carbonmonoxide are obtained. The other process involves passing furfural together with excess steam over chromite or soda-lime catalyst, in the presence of hydrogen but absence of oxygen. Furan, carbondioxide and hydrogen are the resulted products. Alternatively, furfural may be converted to furoic acid at 300–500°C, on the surface of any higher oxide of heavy metals in the presence of oxygen, and the acid is immediately decarboxylated to give furan.

Generally, furoic acid is the product of oxidation of furfural. Decarboxylation of this acid or decarbonylation of furfural with the help of catalysts is the prevailing way for furan production in both gas and liquid phases [16]. Furan is costlier than furfural. In industry, the gas phase method is more attractive because of its advantages of simple operation and the easy separation and recycling of catalysts. Due to the high energy barrier for the C–C bond cleavage, the decarbonylation of furfural is preferably conducted at high temperatures. Additionally, hydrogen is always present in this reaction to maintain the metallic state of catalysts. Furfural is highly stable because of its high resonance energy [16]. Decarbonylation of furfural with high efficiency still presents considerable challenges. Among the various catalysts employing different metals and supports, Pd catalysts loaded on carbon, alumina and silica have demonstrated superior activity due to the favorable formation of acyl surface species, the key adsorption intermediate for decarbonylation [16]. Recently, Platinum has attracted much attention due to its cheapness and good activity [16].

2.4 Condensation reaction

Furfural condenses with nitromethane in the presence of a base to yield Nitrovinyl furan derivative [2]. Any compound containing active alpha-hydrogen in the presence of a base (ammonia or amines) as a catalyst will condense with furfural according to Knoevenagel condensation [2]. The aldol condensation of acetone and furfural has been deeply studied using different homogeneous and heterogeneous catalysts, highlighting different hydrox-ides (NaOH and Ca(OH)₂) and mixed oxides such as Mg-Zr or Mg-Al [17].

Cyclopentanone can undergo condensation with furfural or 2-methyl furan through an alkylation reaction to produce oxygen-containing compounds, which can in-turn, be converted into high-density diesel or jet fuel by hydro deoxygenation. Aldol condensation of cyclopentanone and furfural by sodium hydroxide in a solvent-free system produced a high yield of 2,5-bis (2-furylmethylidene) cyclopentanone [18].

3. Imine

Imine or azo-methine is derivable from nature and also synthesized in the laboratory. The azo-methine functional group (C=N) is responsible for a number of biological activities [19]. Apart from the bioactivities, there are enough reports in the literature that azo-methine also exhibits pigments and dyes properties, used as catalysts, and as stabilizers in polymeric products [20]. Azo-methine also possesses herbicidal activities [21]. The formation of metal-complexes with imine as ligand improves its uses [22].

Complexes formed by reacting imine with transition metal have numerous applications. They have biological, industrial, catalystic applications [23]. These compounds are very important pharmaceutical precursors for their wide spectrum of biological activities [24].

They are very much applicable in dyes industries also; they perfectly dye leathers, food packages and wools with different hues [25].

Copper complexes are the most potent transition metal-imine complexes ever formed. They are very useful in chemical sciences like the food industry, dye industry, analytical chemistry, catalysis and many other fields [26].

The complexes formed with zinc metal exhibit good photo luminescent and electroluminescent activities. These compounds show good color purity and could be used in the production of full-colored organic light-emitting diodes [27]. They could also be used as dopant materials [28].

4. Synthesis and potential uses of 1,3-bis((E)-furan2-yl)methylene)urea (UBI)

In our recent study furfural was redistilled, separately mixed with urea and thiourea in the presence of glacial acetic acid (drops) under reflux to obtain

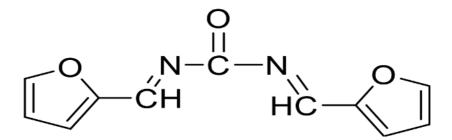


Figure 3. *Structure of 1,3-bis((E)-furan-2-ylmethylene)urea.*

1,3-bis((E)-furan2-yl)methylene)urea and 1,3-bis((E)-furan2-yl)methylene) thiourea respectively. The products were filtered, washed with water and recrystal-lized in alcohol (**Figure 3**) [4].

A study has shown that 1,3-bis(furan-2-yl) methylene) urea (UBI) possesses hepatotoxic and nephrotoxic effects [19]. UBI has been reported to be a potential inhibitor of *P. aeruginosa* and *S. bovis* [4].

5. Potential uses of 1,3-bis((E)-furan-2-yl) methylene) thiourea (TBI)

Loomis and Hayes stated that any substance that causes harm to an animal with a dose below 5 mg/ kg could be regarded as a highly toxic substance [29]. Therefore, 1,3-bis((E)-furan-2-yl) methylene) thiourea cannot be regarded as being toxic since it did not cause harm to any of the mice administered with doses of 50 and 150 mg/kg.

TBI has been referred to as multifunctional dye when applied on wool and cotton fabrics. Because of its very high and moderate inhibitions rate on fungi and bacteria. The dyed fabrics can be utilized in the production of medical face masks. Excellent Ultraviolet Protection Factor (UPF) of the dyed fabrics with TBI is an indicator of its potency as a sun-screen protector, which may protect the users from harmful rays of sun burn. Its high fastness enhances the use of the dyed fabric for a very long time without fading. Also, it was reported that its mechanical properties make it a potential materials for engineering laboratory coats production [29].

6. Toxicity and the potential uses of 2-(2-nitrovinyl) furan

2-(2-Nitrovinyl)furan is a lipid-soluble substance that has been discovered to have antimicrobial properties, against skin-related microbial infections [30].

The compound 2-(2-Nitrovinyl) furan significantly reduced alkaline phosphatase, alanine, and aspartate aminotransferase activities in male rat liver and kidney with a corresponding increase in serum. The activities of superoxide dismutase, catalase, glutathione peroxidase, glutathione reductase, and levels of reduced glutathione/glutathione disulfide (GSSG) ratio in the liver and kidney of 2-(2-nitrovinyl) furan-treated rats decreased significantly. In contrast, GSSG, protein carbonyl, conjugated dienes, lipid hydroperoxides, malondialdehyde, and fragmented DNA (%) in 2-(2-nitrovinyl) furan-treated rats increased significantly. 2-(2-nitrovinyl) furan exhibited its toxicity by decreasing the antioxidant systems [31].

2-(2-nitrovinyl) furan exhibited additive interactions with chloramphenicol, erythromycin, lincomycin and gemifloxacin [32].

It has been reported that malondialdehyde and fragmented DNA level increased significantly in the bacterial cells treated with 2-(2-nitrovinyl) furan when compared with dimethylsulfoxide (DMSO) treated cells. The colonyforming unit (CFU) of *Escherichia coli*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* following exposure to 2-(2-nitrovinyl)furan increased significantly (p < 0.05) in the presence of 2,2' bipyridyl, an iron chelator, when compared with only 2-(2-nitrovinyl)furan suggesting the involvement of hydroxyl radical in the cell death. This study showed that 2-(2-nitrovinyl)furan induced oxidative stress in *Escherichia coli*, *Pseudomonas aeruginosa* and *Staphylococcus aureus* as evident from elevated levels of superoxide anion radical nitric oxides and antioxidant enzymes (**Figure 4**).

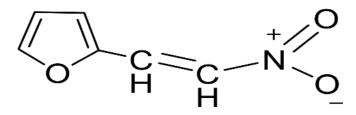


Figure 4. *Structure of 2-(2-nitrovinyl) furan.*

7. Natural chalcone, extraction and uses

The name "Chalcones" was given by [33]. Chalcone is a generic term used in describing compounds with the 1, 3- diphenylprop-2-en-1- one frame work (**Figure 5**).

They are natural compounds that are largely found in plants, fruits and vegetables [34]. They are mostly found in various plants species like Angelica, Glycyrrhiza, Humulus and Scutellaria, which are widely used as traditional remedies. Most of the aromatic rings of natural chalcones are found in the hydroxylated form. Chalcones, dihydrochalcones and aurones are composed of pigments whose color changes from yellow to orange in some plant species. Those compounds are found not only in flowers but also in lots of different tissues of plants. Free radical scavenging properties of phenol groups of chalcones increase the interest in consumption of the plants that contain chalcones. The chemistry of chalcones has generated intensive scientific studies throughout the world.

Chemically they consist of open chain flavonoids in which two aromatic rings are joined by a three-carbon α , β carbonyl system. To develop most of the biological and pharmacoligical agents, a series of chalcones were prepared by Claisen-Schmidt condensation of appropriate acetophenones with appropriate aromatic aldehydes in the presence of an aqueous solution of potassium hydroxide and ethanol at room temperature [35]. Chalcones either natural or synthetic are known to exhibit various biological and pharmacological activities.

Chalcone is an aromatic ketone and enone that forms the central core for a variety of important biological compounds [36].

8. Synthesis and potential uses of furfural based chalcone

In one of our studies, (E)-3-(furan-2-yl)-1-phenylprop-2-en-1-one was synthesized using Claisen-Schmidt condensation. Sodium hydroxide (3 g) was dissolved in 10 ml of methanol. Acetophenone (3 mL) and 3 mL furfural were dissolved in 10 ml of rectified ethanol in a 250 ml conical flask on a magnetic stirrer. Then 10 ml NaOH solution (3 g in 10 ml methanol) was added drop wise to the reaction mixture on vigorous stirring for 30 minutes until the solution became turbid. The reaction temperature was maintained between 20 and 25°C using a cold water bath on the magnetic stirrer. After vigorous stirring for 4–5 hours the mixture started forming precipitate. The reaction vessel was kept overnight in the refrigerator to give room for complete precipitation. The precipitate formed was filtered, air-dried and recrystallized in rectified ethanol to give the pure product (**Figure 6**) which was weighed and stored for further analysis.

The presence of a reactive α , β unsaturated ketone functionality in chalcones is found to be responsible for these activities. In recent years, many studies have been reviewed and chalcone is found to be useful for their cytotoxic, anti-cancer,

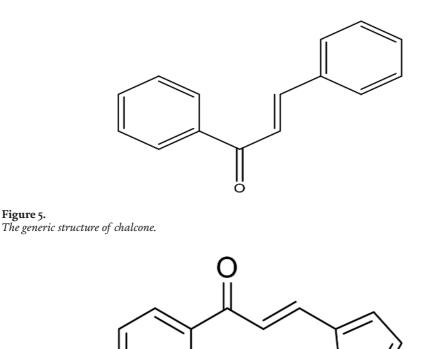


Figure 6. *Structure of (E)-3-(furan-2-yl)-1-phenylpro-2-en-10ne.*

anti-viral, anti-malarial, anti-oxidant, anti-leishmanial, anti-inflammatory, analgesic, anti-platelet, anti-ulcerative, anti-tubercular, anti-hyperglycemic, chemo-preventive, mutagenic, insecticidal and enzyme inhibitory properties [37].

9. Qualitative structure: activity relationship

The structures of the molecules have been modeled and geometry optimization as well as molecular orbital at DFT/B3LYP/6-31G* level of theory in vacuum. The optimized geometries of the studied molecules were then used to obtain the ground state molecular geometry parameters. The highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) energy values obtained were used to calculate other parameters [4].

10. Chemical-activities relationship result

Table 1 presents Highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) of furfural (starting material), 1,3-bis[(*E*)furan2-yl)methylene]urea (UBI), and 1,3-bis[(*E*)-furan-2-yl) methylene]thiourea (TBI), 2-(2-Nitrovinyl) furan (NVF) and (*E*)-3-(furan-2-yl)-1-phenylprop-2ene-1-one (FPP) (last two rows). Other parameters/properties were derived from these values using different formulas for each property. The energy of the HOMO is directly related to the ionization potential while that of the LUMO is related to the electron affinity [38]. The high value of HOMO in any molecule is an indication of its high tendency to donate electrons to molecules with empty and low-lying energy orbital acting as an acceptor.

Furan Derivatives - Recent Advances and Applications

Properties	Formulae	Furfural	UBI	TBI	NVF	FPP
Energy gap (eV)	E _L - E _H	10.17	4.54	3.50	3.89	2.45
Chemical potential (K)	¹ /2(E _H -E _L)	-5.09	-2.27	-1.75	-1.95	-1.23
Absolute Hardness (η)	$^{1/2}(E_{L}-E_{H})$	5.09	2.27	1.75	1.95	1.23
Softness (S)	$\frac{1}{2\eta}$	0.10	0.22	0.29	0.26	0.41
Electrophilicity	$\frac{K^2}{2\eta}$	2.55	1.14	0.88	0.99	0.62
Electronegativity	$\frac{I + EA}{2}$	9.29	4.38	4.12	4.57	4.25
Ionization potential, I (eV)	-E _H	9.73	6.65	5.87	6.51	3.03
Electron affinity, EA (eV)	-E _L	-0.44	2.11	2.37	2.62	5.48
Dipole moment, μ (D)		2.68	3.41	3.34	6.44	
Polarizability (α)			57.59	58.65	51.15	
E _{HOMO} (eV)		-9.73	-6.65	-5.87	-6.51	-3.03
E _{LUMO} (eV)		0.44	-2.11	-2.37	-2.62	-5.48

Table 1.

Global parameters.

The energy band gap of furfural the starting material was the highest (10.17 eV), followed by UBI (4.54 eV), then NVF (3.89 eV), TBI (3.50 eV) and the lowest was FPP (2.45 eV). Large HOMO–LUMO energy gap is an indication of high stability and consequently less activity for the molecule in chemical reactions [39]. Thus, furfural is the most stable with less activity and FPP is the least stable with the highest activities.

Also, the Chemical potentials follow the same trend with furfural having the highest value (-5.09 eV), followed by UBI (-2.27 eV), next was NVF (-1.95 eV), then TBI (-1.75 eV) and least was FPP (-1.23 eV). Energy-gap determines the chemical potential of a compound. The higher the energy-gap, the more stable the compound; more energy would be required to overcome the barrier and consequently the lower would be the chemical activities of such compound [40].

These two properties go together, hardness is inversely proportional to the softness. It was discovered that the hardest was furfural (5.09) which is the least soft (0.10), again followed by UBI with hardness (2.27) and softness of (0.22). This was closely followed by NVF with hardness and softness values of (1.75) and (0.26) respectively, followed by TBI with (1.95) and (0.29) for hardness and softness. FPP is also the least hard (1.23) with the highest softness value (0.41). A hard compound will not solvate easily in a solvent and therefore the activities will be low and vice versa. The softness of a compound is an indication of increased movement of a system towards another compound and vice versa [41]. FPP with the highest softness value (0.29) will ionize easily and all its activities (biological and Chemical) would be very high.

The ionization potential for the furfural compound was greatest and the least value was for FPP molecule. This result indicates that furfural molecule needs high energy to become cation compared with the synthesized compounds (UBI, NVF, TBI and FPP) while FPP needs the smallest energy to overcome the barrier.

The strength of an acceptor molecule is measured by its electron affinity (EA) which is the energy released when adding one electron to the LUMO of a compound. An acceptor must have a high EA. Electron affinity (EA) for the molecule FPP was the largest (**Table 1**). The calculated properties for both electron affinity and ionization potential reveal that FPP molecules have a tendency to receive electrons and as well as donating electrons for reaction to occur.

11. Conclusion

Furfural without a doubt is versatile as a starting material for chemical synthesis. The activities of its derivatives; 1,3-bis[(E)-furan2-yl)methylene]urea (UBI), 1,3-bis[(E)-furan-2-yl) methylene]thiourea (TBI), 2-(2-Nitrovinyl) furan (NVF) and (E)-3-(furan-2-yl)-1-phenylprop-2-ene-1-one (FPP) are different from one another and far better than that of the starting material (furfural). Therefore, it is a good starting material for numerous beneficial industrial chemicals.

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