

# Synthesis of Furfuraldehyde from Lignocellulosic biomass (Potato Peel) through acid hydrolysis

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## Abstract

Furfuraldehyde, a key platform chemical derived from lignocellulosic biomass, serves as a precursor in producing various specialty chemicals and fuels. It has traditionally been produced through the acid hydrolysis of agricultural residues. This study focuses on optimizing the production of furfuraldehyde from potato peel, an abundant and underutilized waste product, using acid hydrolysis. Potato peels, primarily composed of cellulose, hemicellulose, and a small amount of lignin, represent a promising feedstock due to their widespread availability and rich hemicellulose content. The hydrolysis process was carried out under certain conditions, employing a moderate temperature range (160-200°C) and varying the reaction time between 30 to 90 minutes to evaluate their effects on furfural yield. It successfully demonstrated the synthesis of furfural of about 15% conversion at 165 °C from lignocellulosic biomass, potato peel waste (PPW) is obtained at 165°C with an acid to feed molar ratio of 7:1, utilizing acid hydrolysis at a concentration of 0.125 gm/L solution mixture. The study highlights the importance of valorizing agricultural waste, contributing to waste reduction, and the creation of value-added chemicals in a circular economy.

**Keywords:** Furfuraldehyde, Lignocellulosic biomass, Potato Peel, Acid Hydrolysis, Pentosans.

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## 1 Introduction:

Furfural( $C_6H_5OCHO$ ) is a colourless liquid that usually becomes brownish while exposed to air. It is an organic compound that can be produced from agricultural byproducts like corncobs, oat hulls, and sawdust through the acid hydrolysis of pentosans (complex sugars)[1,2]. The synthesis of significant furfural derivatives, such as furfuryl alcohol and tetrahydrofurfuryl alcohol, is of considerable interest due to their versatility in various applications like fungicides, transportation fuels, resins, drugs, insecticides, bioplastics, lubricants, flavoring agents for foods and drinks, etc. [3, 4, 5]. Furfural manufacturing has garnered substantial attention in recent years due to its wide use in process industries[6, 7, 8].

In the last few decades, the world has seen an over-dependence on fossil-based fuels to meet its energy needs [9, 10, 11]. These fossil fuels are non-renewable, and their excessive exploitation will lead to an energy crisis. To minimize this dependence on fossil fuels, many other alternatives have been explored, and one such alternative is the bio-conversion of lignocellulosic materials into chemicals and fuels[12, 13]. This method has gained particular interest in recent years due to its inexpensive and renewable source, which is widely accessible [14,15]. The lignocellulosic materials that are available in abundance in nature consist of approximately 34% cellulose, 26% hemicellulose, and 28% lignin[16]. This biomass material refers to organic material produced by natural photosynthesis, with cellulose, hemicellulose, and lignin as its three primary components. Hemicellulose within the plant cell wall constitutes 20% to 35% of the

lignocellulose's weight. It is an amorphous polymer comprising five-carbon and six-carbon sugars, with xylan being the main component. Xylan is primarily composed of 1,4- $\beta$ -D-pyran-type xylose in its main chain. When xylan is fully hydrolyzed, it yields five-carbon monosaccharides such as xylose and arabinose, with xylose being the predominant product [17, 18, 19]. The solid residue from the degradation processes of hemicellulose yields many products, such as food, chemicals, medicines, polymers, etc. [20,21]. Concentrated or diluted acids break down the cellulose and hemicellulose polymers in lignocellulosic biomass to produce individual sugar molecules that may be fermented to produce ethanol [22]. It is observed that hemicellulose hydrolyzes more readily than cellulose [22, 23]. The acid hydrolysis process usually employs sulphuric acid and hydrochloric acid at 1–10% concentrations using a moderate temperature (in the range of 100–150 °C) [24]. Although acid hydrolysis has the benefit of being able to dissolve lignin without the need for pretreatment and breaking down glucose more quickly in acidic environments than enzyme hydrolysis, both processes have drawbacks [25].

Furfural is generally produced by two-stage processes: single-stage operation and double-stage operation. In the single-stage process, pentosan is broken down into xylose and subsequently dehydrated into furfural within the same reactor. This method results in a low furfural yield (0.7–3.3 wt %), and the solid residue is utilized for fuel manufacture[26, 27]. While compared to a single stage, the double-stage operation produces a higher yield of furfural with hydrolysis and dehydration stages occurring in the same reactor [28]. The solid residue from double-stage operation is generally used for manufacturing ethanol, glucose, etc, by the fermentation process. Kaur and Ni [29] investigated the utilization of pre-hydrolysis liquor from the Kraft process for furfural synthesis in a low concentration of about 5-7%. Hemicelluloses were enriched via membrane separation and converted to furfural in monophasic and biphasic reactor systems, demonstrating process viability. Baktashet et. al [30]utilized activated carbon adsorption and ion exchange resin treatment as pretreatment methods to enhance furfural yield up to 57% using H<sub>2</sub>SO<sub>4</sub> as the catalyst and using pre-hydrolysis liquor, aid lignin removal, streamline the process, and reduce side reactions. Li et. al [31]examined the influence of hemicellulose properties in corn stalks within an alkaline medium on furfural production. Results showed increased xylose content, fewer branching points, greater polydispersity, and higher hemicellulose crystallinity enhanced furfural yield. They observed the maximum furfural yield of 45.41% was achieved with a xylose conversion efficiency of 99.06% and a furfural selectivity of 45.84%, using oven-dried hemicelluloses precipitated at a 30% (v/v) ethanol concentration. Sangarunlert et al. [32]investigated the furfural production in the range of 45 -50% from rice husks using various experimental runs on hydrolysis and hydration processes.

So, it has been observed that hemicellulose, which is a key component of lignocellulosic biomass, holds significant potential as a sustainable alternative to finite fossil resources for furfural production, ensuring high atom efficiency. However, its complex structure and interactions with cellulose and lignin in lignocellulosic biomass present challenges for efficiently utilizing its natural form[33, 34, 35, 36, 37]. Potatoes (*Solanum tuberosum* L.) are one of the most important agricultural crops for human consumption after wheat (*Triticum* L.), rice (*Oryza* L.), and maize (*Zea mays* subsp. *mays* L.), with 376 million tons produced in 2013 (Compare data, 2013). In developed countries, up to 69.5% (in 2012) of the total potatoes produced are processed (U.S. per Capita, s.a.). Potatoes are usually peeled during processing, and production losses in the form of potato peel waste (PPW) can vary from 15 to 40%, depending on the peeling method [38, 39, 40]. Each year, vast quantities of PPW as a byproduct remain after

industrial potato processing [41]. Traditionally, potato peel waste is used for producing low-value animal feed and fertilizer, which causes waste of abundant nutritive materials having the properties of antioxidant, antibacterial, apoptotic, chemopreventive, and anti-inflammatory [42]. Due to its composition, abundance, and cost-free nature, potato peel wastes (PPW) serve as a renewable resource for producing high-value bioproducts. This study comprehensively reviews recent advancements in PPW valorization via biological and thermochemical conversion methods. PPW shows significant potential for biofuel and biochemical production through processes such as detoxification, pretreatment, hydrolysis, and fermentation [43,44, 45].

The principle of converting agricultural waste into furfural has been well-documented. Author Zeitschn [46] provides a comprehensive overview of the chemistry involved in transforming pentosan-rich biomass into furfural through acid hydrolysis. The process capitalizes on the dehydration of xylose, a five-carbon sugar prevalent in hemicellulose, under acidic conditions. The efficiency of this conversion is influenced by various factors, including the type of acid used, temperature, and the physical characteristics of the biomass. A study by K.J.Yong et al. [47] demonstrates the potential of various agricultural residues for furfural production, highlighting the significant influence of feedstock composition on yield.

The optimization of acid hydrolysis conditions for the efficient production of furfural is critical. Marcotullio and De Jong [48] emphasize the role of temperature, acid concentration, and reaction time in maximizing furfural yields while minimizing side reactions and energy consumption. Furthermore, the choice of acid—whether mineral (such as sulfuric acid) or organic (such as formic acid)—impacts not only the yield but also the selectivity and environmental footprint of the process. Peleteiro et al.[49] suggests that microwave-assisted hydrolysis could offer advantages regarding reaction speed and energy efficiency, presenting an area for potential innovation in furfural production from potato peels.

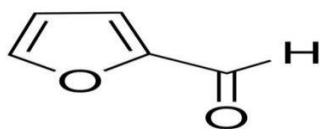


Figure 1 Furfuraldehyde (C<sub>4</sub>H<sub>3</sub>OCHO) structure

Despite the vast literature on biomass conversion, studies specifically targeting potato peels are relatively rare. Potato peels, as a residue from the food industry, are rich in cellulose and hemicellulose, which are ideal precursors for furfural synthesis. However, the unique composition of potato peels, including their moisture content and specific carbohydrate profile, necessitates tailored hydrolysis conditions for optimal furfural production. Early work by Kamm et al. [50] touches on the broader potential of potato waste in biorefinery applications but needs more detailed process optimization for furfural.

This study extends the existing knowledge on furfural production by focusing specifically on potato peels and optimizing the process parameters to enhance product efficiency. The experimental results demonstrate that potato peels are a viable and efficient source for furfuraldehyde production under optimized conditions, potentially contributing to a more sustainable biochemical industry by converting waste materials into valuable chemicals.

### 1.1 Reaction kinetics:

Furfural (Figure 2) is obtained through the acid-catalyzed dehydration of pentoses like xylose, which is derived from pentosans in the hemicellulose fraction of lignocellulosic biomass. Hemicellulose is abundant in agricultural residues, wood, and other plant materials, making furfural an important platform chemical in biomass valorization.

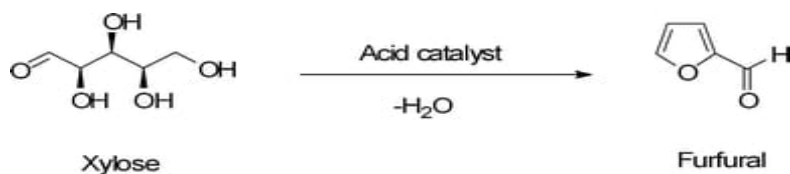


Figure 2: Furfural production reaction kinetics

### 2. Materials and Methods in Furfural production process:

The production of furfural typically involves processing lignocellulosic biomass, such as agricultural residues, through a series of steps. These steps are designed to first break down the complex biomass structure to release fermentable sugars and then dehydrate these sugars to produce furfural. The process can be broadly categorized into the following stages.

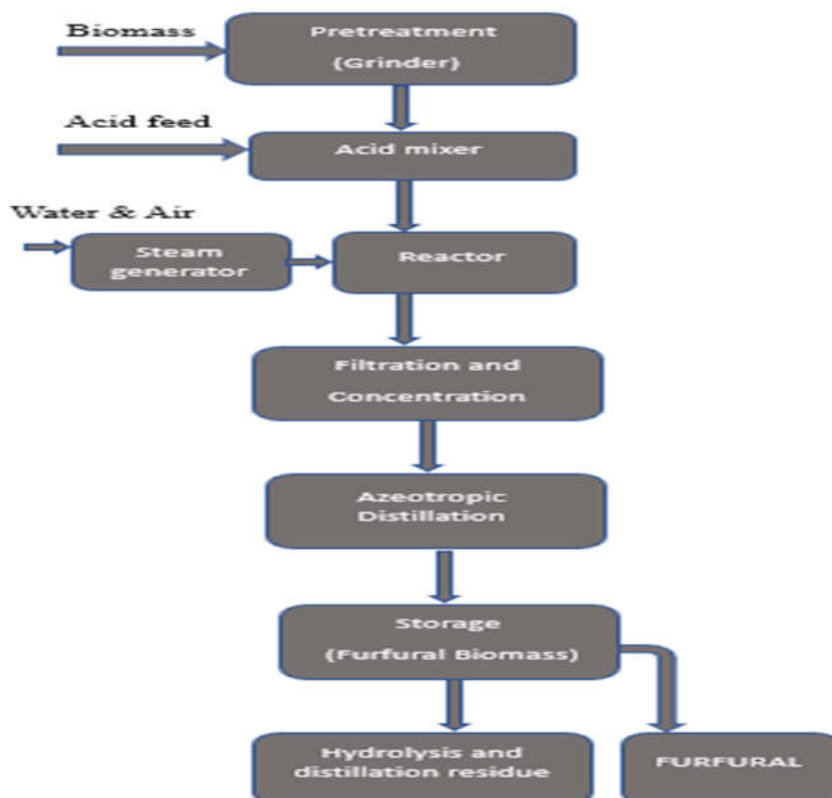


Figure 3: Flow process diagram for the production process of furfural

## 2.1 Preparation of Biomass

The process begins with the selection of an appropriate lignocellulosic feedstock rich in hemicellulose. Common materials include corn cobs, sugarcane bagasse, rice husks, and, for our study we selected potato peels as the source of hemicellulose for biomass. The biomass is mechanically reduced in size to increase its surface area, which enhances the efficiency of subsequent hydrolysis. This can be achieved through milling, grinding, or chopping. Then the dried biomass is crushed and sieved through 1 to 2 cm. The moisture content of the biomass is reduced (from 20% to 10%), typically through air drying or the use of drying equipment, to improve the efficiency of the acid hydrolysis step.

## 2.2 Acid Hydrolysis

The prepared biomass is subjected to acid hydrolysis to break down the hemicellulose into its constituent sugars, mainly xylose. This is done in a 10 litre batch reactor by treating the biomass with a dilute acid, sulfuric acid ( $H_2SO_4$  acid) at 1:5 solid to acid molar ratio with 100gm of prepared biomass with a stirring speed of 125 rpm. The conditions (temperature, acid concentration, and time) are optimized based on the type of biomass used to maximize sugar release.

## 2.3 Dehydration to Furfural

The hydrolysate containing the sugars is then heated, (the process can be conducted either in the presence of the same acid or after adjusting the pH to promote the dehydration of xylose to furfural) for dehydration. This step is typically conducted at temperatures between 160°C and 250°C to reduce the moisture content to around 5%.

## 2.4 Purification

The purification stage of furfural follows multiple distillation and extraction stages.

The furfural-solvent mixture is then distilled to separate furfural from the solvent and any by-products. It involves several stages of distillation, and the product is collected for a time of 1 hour. The distillate is double extracted with 50 ml of dichloromethane for 200 ml of distillate. (Dichloromethane is very toxic during inhalation.).The organic extract is then filtered and evaporated into dichloromethane. The product produced is diluted with little dichloromethane and dried Magnesium sulfate. After that, the product is filtered again, and dichloromethane and furfuraldehyde are distilled. The boiling point for furfural is 162 °C. Finally, distillate is collected from a range of 155 °C to 165 °C. It is observed that the distillate is colorless, but after a couple of hours, it turns black color.



Figure 4: Distillation laboratory setup for furfuraldehyde production

**3 Results:** Furfural was tested for identification by Molisch's test, Fehling's test and Schiff's test in the laboratory before characterization by spectroscopic methods (Fourier Transform Infra Red model)

### 3.1. Molisch's test:

In this test, to ascertain the furfural traces, two drops of sample were added in Molisch's reagent along with a few drops of concentrated acid ( $H_2SO_4$ ). Further 5% of  $\alpha$ -naphthol in 99% alcohol (Ethanol) was used as a reagent in the mixture. A dark violet ring between the acid and sample layer appears, which confirms the presence of carbohydrates, particularly pentose.



Figure 5: Laboratory Molisch's test for testing furfural

### 3.2 Fehling's test

Solutions of Fehling's A and Fehling's B are taken in a test tube and mixed, and after adding two drops of prepared sample, it is heated gently and after that it is left to cool down for some time (here in this test, Fehling's A (Aq.  $CuSO_4$ )+ Fehling's B ( $NaKC_4H_6O_6$ ) are used as a reagent). At the end of the test, a brick-red precipitate appears, confirming the presence of Aldehyde and reducing sugar.



Figure 6: Laboratory Fehling's test for identifying furfural

### 3.3 Schiff's test

A small amount of Schiff's reagent is taken, mixed, and shaken with the sample to react with the furfural present in it (which further reacts with the aldehydes). It produces a distinctive magenta colour, confirming and identifying aldehydes' presence.



Figure 7: Laboratory Schiff's test for testing furfural

### 3.4 Fourier Transform Infrared Spectroscopy (FTIR)

Fourier-transform infrared spectroscopy (FTIR)(Make: Shimadzu, Japan, Thermo Fisher Scientific, USA, Model: IRAffinity-1, Thermo Nicolet iS50 with inbuilt ATR) characterization is done to confirm the presence of furfural in a sample. Figure (8) represents the furfural spectrum from FTIR analysis, and each peak illustrates each functional group of furfural that is present in the sample characterized. Furfural has characteristic absorption bands in the infrared spectrum, which FTIR analysis has detected and shown in the result. Furfural has several distinctive peaks due to its functional groups. Carbonyl Stretch: Look for a strong absorption peak around 1590-1670  $\text{cm}^{-1}$ , which is characteristic of the  $\text{C}=\text{O}$  stretch in the aldehyde group. C-H Stretch: Aromatic compounds like furfural show C-H stretch vibrations in the 3100-2900  $\text{cm}^{-1}$  region. C-O Stretch: There should be an absorption band around 1260-1000  $\text{cm}^{-1}$ , indicative of the C-O single bond stretch in the furan ring. Aromatic Ring: Absorption bands due to the aromatic ring vibrations can appear in the 1700-1600  $\text{cm}^{-1}$  region. Furan Oxygen: The furan ring in furfural contributes to a peak at around 800-500  $\text{cm}^{-1}$  due to the C-O-C asymmetric stretch.

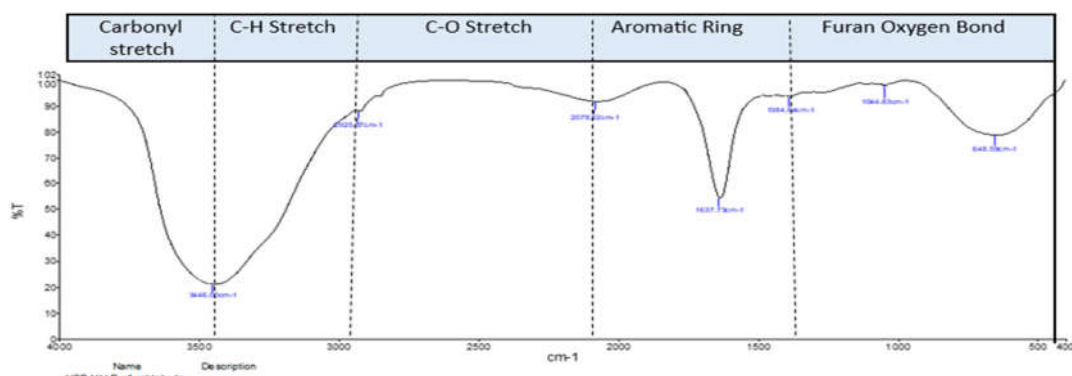


Figure 8: FTIR spectrum for furfural

### 3.5 Factors influencing the yield of furfural:

The synthesis of furfural from lignocellulosic biomass, such as potato peel waste (PPW), is significantly influenced by various factors during the acid hydrolysis process. These factors include the temperature of the reaction, the concentration of the acid used, the acid-to-feed ratio, and the presence of salts. Each of these variables can affect the yield and purity of furfural produced.

#### 3.5.1 Effect of Temperature on Product

Temperature plays a significant role in furfural production. The choice of temperature depends on the specific catalyst and reaction conditions used in the furfural production process. Experimentation and optimization are often necessary to determine the ideal temperature for maximizing furfural yield while maintaining process efficiency. The effect of temperature on the production yield is shown in figure (9) and table (1) which clearly reveals that the furfural yield sharply increases from 150°C to 165°C and suddenly decreases at 180°C, after reaching the highest yield at 150°C. The furfural yield gradually increased as the temperature rise (150–165 °C), likely due to the initial hydrolysis of cellulose and hemicellulose into hexoses and pentoses, followed by the dehydration of the hexoses and pentoses to produce furfural. Elevating the reaction temperature generally increases the rate of hydrolysis of hemicellulose to xylose and the subsequent dehydration of xylose to furfural. This can lead to a higher furfural yield up to an optimal temperature. Beyond the optimal temperature, the rate of side reactions, including the degradation of furfural and further decomposition of sugars, increases which can conclude in a decrease in furfural yield and the formation of unwanted by-products. Thus, higher temperatures require more energy input, which can affect the overall sustainability and cost-effectiveness of the process.

Table 1: Effect of Temperature on Furfural yield

Parameters	Sample 1	Sample 2	Sample 3
Mass of sample (potato peel)	100	100	100
Mass of salt	15	15	15
Amount of diluted acid (ml)	500	500	500
Temperature (°C)	150	165	180
Time (min)	60	60	60
Amount of product obtained	8	12.5	9.6

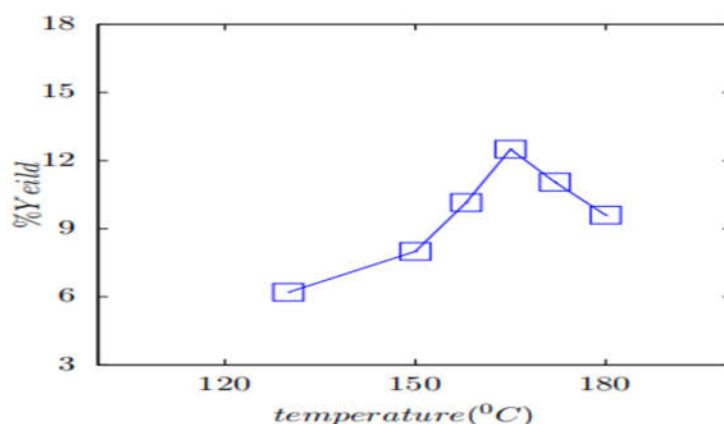


Figure 9: Effect of temperature on furfural yield



### 3.5.2 Effect of Acid Concentration on Product

The concentration of acid plays a crucial role in furfural production from biomass, typically using a process called acid hydrolysis. Higher acid concentrations can increase furfural yield but may also lead to side reactions or degradation of furfural into unwanted by-products. It's a balance between maximizing yield and minimizing undesirable outcomes. Table (2) illustrates the effect of acid concentration on the furfural yield which clearly demonstrates that with increasing acid normality from 0.05 to 0.1, the furfural yield also increases and also increasing acid concentration slows the furfural production due to formation of some unwanted byproducts. Increasing the concentration of the acid improves the hydrolysis of hemicellulose, leading to more xylose available for dehydration to furfural. However, there is an optimal concentration beyond which the benefits plateau or decrease due to the increased formation of by-products. Higher acid concentrations can lead to increased corrosion of equipment and raise safety concerns, requiring more robust and expensive materials for construction. Higher acid concentrations often accelerate the hydrolysis reaction, leading to faster furfural production.

Table 2: Effect of Acid concentration on Furfural yield

Parameters	Sample 1	Sample 2	Sample 3
Mass of feed taken(gm)	100	100	100
Normality of Acid(N)	0.05	0.125	0.5
Amount of acid taken	500	500	500
Temperature( $^{\circ}$ C)	165	165	165
Amount of Product obtained (ml)	5.5	12.5	11.2

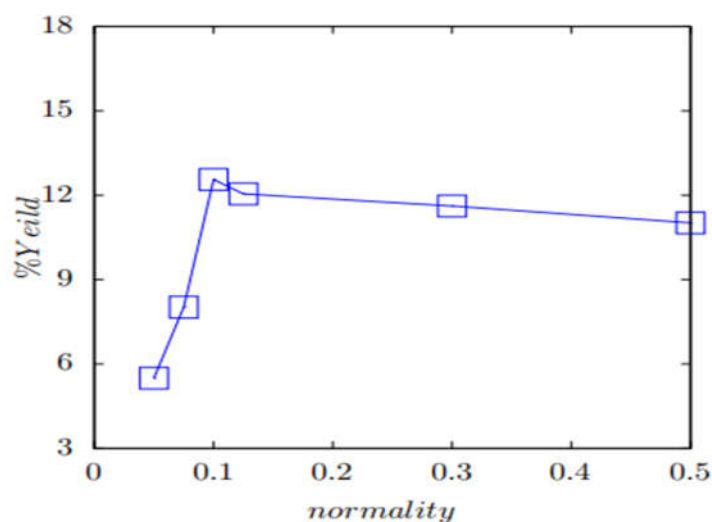


Figure 10: Effect of acid concentration on fufural yield

### 3.5.3 Effect of Acid-Feed Molar Ratio on Product Yield

The acid feed ratio, which refers to the ratio of acid to biomass in furfural production, significantly influences the process. Here are some effects. An optimal ratio must be determined experimentally, as too low a ratio may lead to incomplete hydrolysis, while too high a ratio may not be economically feasible. Higher acid-feed ratios can also result in more significant amounts of acidic waste, which must be neutralized and treated, increasing the process's environmental footprint. Maintaining the optimal acid feed ratio is essential for process control and consistency in furfural production. Table (3) illustrates the effect of acid to feed molar ratio on the furfural yield which is clearly illustrated in the figure which states that increasing the acid to feed ratio from 5:1 to 7:1 will increase the furfural yield from 11 to 15 ml but generally increasing from 7:1 to 10:1 will slow down the reaction of furfural yield with production of some acidic byproducts. A higher acid feed ratio often accelerates the hydrolysis reaction, leading to faster furfural production. However, excessively high ratios may also increase the likelihood of unwanted by-products.

Table 2: Effect of Acid - Feed molar ratio on Furfural yield

Parameters	Sample 1	Sample 2	Sample 3
Amount of feed taken	100	100	100
Normality of Acid(N)	0.1	0.1	0.1
Acid feed ratio(ml/g)	5:1	7:1	10:1
Amount of acid taken	500	700	1000
Temperature( $^{\circ}$ C)	165	165	165
Time(min)	60	60	60
Amount of Product obtained (ml)	11.5	15.2	10.1

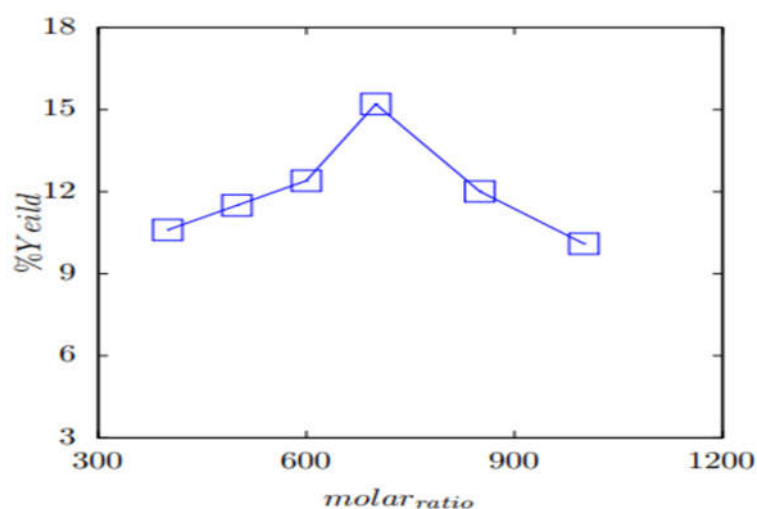


Figure 10: Effect of acid feed ratio on fufural conversion

#### 4.CONCLUSION:

This study successfully demonstrated the synthesis of furfural from lignocellulosic biomass, specifically potato peel waste (PPW), utilizing acid hydrolysis. Through systematic experimentation, it was established that parameters such as reaction temperature, acid concentration, acid-to-feed ratio, and the presence of salts significantly influence the yield and purity of the furfural produced. So from the above experimental results, it is concluded that with a reaction temperature of 165<sup>0</sup>C with acid to feed molar ratio of 7:1 and acid normality of 0.125, the highest furfural yield of 15% conversion is obtained. Optimal conditions were identified that maximize furfural production while minimizing by-products and waste, underscoring the potential of PPW as a viable and sustainable feedstock for furfural synthesis. The study highlights the importance of valorizing agricultural waste, contributing to waste reduction, and the creation of value-added chemicals in a circular economy.

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