

# MICROWAVE ASSISTED SYNTHESIS OF VALUE ADDED CHEMICALS VIA IN-SITU ACETALISATION REACTION OF FURFURAL DERIVED FROM DEFATTED RICE BRAN WITH GLYCEROL

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**Abstract:** Defatted rice bran is a promising biomass residue comprises of furfural, a desirable intermediate bioplatfrom chemical acts as a potential raw material aims for sustainable production of value added chemicals and also for generating renewable fuels, as an alternative source of energy to fossil fuel. The main focus of the work is to develop novel microwave assisted synthetic approach which is one of the greener and eco-friendly method for the conversion of furfural to produce value-added products through in-situ acetalisation reaction with glycerol in presence of hydrogen peroxide as oxidizer. Many researchers have begun exploring new uses for glycerol, including its conversion to fuel additives, due to the overproduction of glycerol as byproduct from bio-refineries. In this paper, glycerol a renewable waste substrate obtained in excess has been reacted with furfural to yield the corresponding acetal and value added chemicals such as carboxylic acids, esters, aldehydes, dienes, amides, ketones, alcohols, cycloalkanes and so on confirmed by GC-MS analysis. This microwave assisted in-situ acetalisation reaction synthesis with different molar ratios of glycerol and minimal amount of 30% H<sub>2</sub>O<sub>2</sub> solvent leads to highly energy efficient, low cost and eco-friendly process carried out under optimum conditions with variation of reaction time and temperature has profound influence on the reaction rate, product yield and percentage conversion. These value-added chemicals used as fuel additive and have wide applications in polymer, agrochemical and pharmaceutical industries.

**Index Terms** - Defatted rice bran (DRB), Furfural, Glycerol, Acetalisation reaction, Microwave irradiation.

## 1. INTRODUCTION

Sustainability of society is intimately related to the utilization of renewable resources to meet the demand for energy and chemicals. Renewable energy is a focus because of the issue of global warming. Bio-based feedstocks may present a greener route to fulfill these demands. Regardless, the methods used in future bio-refineries will vary from those of petrochemical industries. Such procedures may breakdown sugars, their oligomers, and different biomass segments into fundamental molecules that practically resemble derived petro-chemical intermediates. Subsequently, the approach of fossil and biomass building blocks link and be upgraded into valuable synthetic chemicals [1].

The worldwide generation of biodiesel as a fuel by the trans-esterification of vegetable oils or animal fats came to 37 billion gallons in 2016[2]. Thus, glycerol therefore that tends to 10 wt % of biodiesel creation and different regular glycerol age from the reaction of C<sub>6</sub> or even C<sub>5</sub> sugars and exceptional compound aggregation by reaction[3] has saturated the commercial market. High-esteem bio-based substances got from over-created glycerol have impact throughout a heap of thought in current years. With the presentation of substantial volumes of glycerol originating from biodiesel generation, it is imperious to discover new applications for this chemical [4]. Glycerol acetals and ketals are among the promising synthetic substances that could be framed from the reaction of glycerol with different aldehydes. They can be utilized as fuel added substances to enhance low-temperature stream properties, aroma, season, beginning materials for surfactants and solvents for therapeutic applications [5]. These situations demonstrate that the business plausibility of the biodiesel program is straightforwardly connected with the business utilization of the glycerol. The fundamental utilization of glycerol is in cosmetics, cleansers and pharmaceuticals [6].

The fundamental issues are the utilization of proper catalysts and maintaining optimum reaction conditions for the complete conversion of glycerol to achieve a high yield of target items. Industrial considerations have distinguished sugars from sugarcane, sugar beets, grain starches (e.g., corn and wheat), lignocellulose and additionally vegetable oils from palm, soybean and oilseeds, as promising feed stocks for renovating. Be that as it may, new change forms are expected to enhance synthetic and warm properties and increment the vitality densities of these feedstocks on the prospect that they are to replace those developed from oil. While a large group of various reactions have been utilized for biomass redesigning, buildup reactions, for example, aldol condensation, acetalisation, etherification, ketonization and esterification are particularly vital substance changes, since they take into consideration the renovating of bio-determined feed stocks [7].

### 1.1 Glycerol and its properties:

Glycerol is one of such relevant biomass derived platform chemicals which has a significant potential in terms of conversion to valuable products. Glycerol is currently generated in large quantities as a by-product from the biodiesel industry [8]. Both crude glycerol and increased quantities of glycerol produced as a byproduct during biodiesel production serves as a fuel for research for the conversion of glycerol to value-added products.

Being a polyol with three hydroxyl groups with different reactivity, multiple chemistries ranging from redox (e.g. oxidations, hydrogenolysis) to acid-catalysed processes (e.g. etherifications, esterifications), dehydrations and oligomerisations can be designed to a number of useful products with applications in the polymer, agrochemical and pharmaceutical industries [9]. A number of recent overviews on glycerol conversion are available in the literature. For further information, readers are kindly referred to key recently reported overviews in the field [10-14, cross ref.1].

### 1.2 The role of Furfural in Acetal formation:

Current efforts are focused on developing new methodologies to obtain furfural in an inexpensive and environmentally sustainable manner. The aromatic character of the furan ring and its polarity provide furfural with good solvent selectivity towards aromatics. The high chemical reactivity of furfural is due to the presence of two functionalities (aldehyde group and the aromatic ring). The aromatic furan ring can be subjected to alkylation, hydrogenation, oxidation, halogenation, and nitration reactions [15, 16].

The acetalization reaction is a process intensely used in organic synthesis to protect the carbonyl group of ketones and aldehydes and it is of great synthetic utility [17-19].

Acetals are prepared by the reaction of aldehydes (or ketones) with alcohols (or orthoformates) in the presence of an acid catalyst [17-20]. Most of the acetalization process is the reaction of carbonyl compounds with ethylene glycol in the presence of an appropriate catalyst [17,21]. Acetal derived compounds have been used as a solvent in fragrance industries, cosmetics, food and beverage additives, pharmaceuticals, used in the synthesis of enantiomerically pure compounds and polymer chemistry [22,23].

Acetals have been used in motor oils, lubricating oils and hydraulic fluids [24] have evaluated various acetals as more biodegradable chemical. Acetalisation of glycerol can be a good alternative to the glycerol produced by biodiesel. The products derived from acetalisation of glycerol have wide industrial applications.

Owing to the high polarity and instability of furfural due to the presence of aldehyde group, it can't be incorporated explicitly into gasoline or diesel. Therefore, to secure a molecule of low polarity and long-term stability, furfural is converted into fuels. Furthermore, the octane number or cetane number of the products should be considered to meet the need of gasoline or diesel. Such molecules as alkane, ether, ester, and acetal have been synthesized from the typical reactions of aldehyde [25].

### 1.3 Hydrogen peroxide as Oxidizer:

During the course of the reaction, hydrogen peroxide acts as an oxidizer. A compound is said to be oxidized at an explicit carbon center if that center either gains electronegative substituents or loses hydrogen substituents. These processes will rise the formal oxidation number of the carbon molecule. Basic solutions of hydrogen peroxide are employed in the oxidative workup of the oxidation reaction method, to convert a C-B bond to a C-OH grouping [26]. Haber and Weiss<sup>96</sup> projected that decomposition of H<sub>2</sub>O<sub>2</sub> leads to the chemical generation of O<sub>2</sub><sup>•-</sup>, is potentially the most convenient technique as no byproducts are formed. The procedure is relatively simple, time-efficient with short reaction time. Among all available strategies, electrochemical processes have been used for O<sub>2</sub><sup>•-</sup> generation [27-30], which successively initiated research on both O<sub>2</sub> reduction and energy conversion [31-34]. During the past 50 years, especially from the 1960s to 1980s the study of reactions between electro generated O<sub>2</sub><sup>•-</sup> with different substrates has attracted increasing attention due to its nucleophilic and oxidative properties [35, 36, 37, 41].

O<sub>2</sub><sup>•-</sup> can selectively act as an oxidant for receptive substrates for undergoing oxidation through a hydrogen atom transfer mechanism [42]. The reduction in utilizing the solvents is widely recognized to be of great environmental concern and one of the most important aims of green chemistry. In addition to this, the appropriate selection of solvent for a process can greatly improve the sustainability of a chemical production process. There has also been extensive research into the application of so-called green solvents, such as ionic liquids and supercritical fluids. Extensive research on the application of green solvents such as ionic liquids and supercritical fluids etc. are in progress [43].

Hydrogen peroxide considered as greener and protic solvent have O-H bond and it can participate in a powerful intermolecular force i.e hydrogen bonding. Additionally, these O-H bonds can serve as a source of protons (H<sup>+</sup>) [44].

In this present work, we have utilized defatted rice bran (DRB) as biomass residue for the first time to derive furfural as an intermediate platform chemicals followed by insitu-acetalisation reactions with glycerol in presence of H<sub>2</sub>O<sub>2</sub> protic solvent under microwave irradiation which is highly efficient, low costs, short reaction times, high yields and eco-friendly greener method to produce high value added chemicals having wide commercial applications in industries.

## 2. Experimental Section

### 2.1 Raw materials and chemical compounds:

Rice bran was purchased from a nearby rice mill, Bangalore. The chemicals such as conc. HCl, acetone, glycerol and hydrogen peroxide was supplied by S D Fine Chemicals Ltd, Bangalore and used without further purification.

### 2.2 Procedure to derive Defatted rice bran:

Defatted rice bran was derived after removal of rice bran oil from rice bran through Soxhlet extraction by refluxing with acetone for eight hours at 55-60°C. The Obtained defatted rice bran residue was dried thoroughly and well-preserved for further analysis. The extraction process was repeated batch wise in similar manner.

### 2.3 Analytical procedure to synthesize intermediate chemical:

DRB was subjected to acid hydrolysis batch wise by refluxing with the mixture of Conc. HCl and 500ml distilled water in round bottom flask for eight hours at 55-60°C. The reaction mixture was filtered by Buckner filtration unit and the filtrate was collected and analyzed by GC-MS technique which indicated the presence of intermediate chemical i.e 2-furanmethanol which is one of the important platform molecule to derive various value added chemicals having wide synthetic applications.

Further continuation of my previous work, the DRB filtrate confirmed the presence of furan compound, which is an important platform molecule used as a starting material to derive value added products by in-situ aldol condensation reaction as a synthetic strategy, here in this present work, we made an attempt to derive various value added chemicals from furfural through in-situ acetalisation reaction using glycerol as the solvent in presence of H<sub>2</sub>O<sub>2</sub> by microwave irradiation.

## 3. Results and discussions

In this present research we made an attempt to establish novel approach of synthesizing cyclic acetals and other value added chemicals from bioplatfrom intermediate chemical derived from defatted rice bran by microwave assisted technique which is highly efficient, low costs, less reaction times, high yields and eco-friendly greener method having wide commercial applications in industries.

In further continuation of my previous research work, the presence of furan compound in the DRB filtrate was confirmed by GC-MS characterization of the filtrate [45]. Here, furfural of the DRB filtrate, being recognized as one of the important raw material [46-53] to derive cyclic acetals and other value added chemicals via in-situ acetalisation reaction. There are various established chemical routes that has been reported in literature for the conversion of furfural to value added chemicals, biofuels and fuel additives such as hydrogenolysis, oxidation, dehydration, esterification, etherification, aldol condensation, carboxylation, acetalization, and chlorination [54].

We know that, acetalisation reaction includes the formation of acetals (or ketals) from an alcohol or ortho ester with carbonyl groups (e.g., ketones or aldehydes) within the sight of acid catalysts. [55]. Many years of work also demonstrated that glycerol got from hydrolysis of fats, reacts with carbonyl compounds to create blends of cyclic acetals comprising of six-membered ([1,3]dioxin-5-ols) and five-membered ([1,3]dioxolan-4-ylmethanols) rings[58]. In recent years microwave irradiation is more attractive in the area of chemistry[59]. The usage of microwave heating to control chemical transformations has increased over the last years [60].

### 3.1 Experimental set up and Methodology:

In this present work, new microwave induced synthetic approach was incorporated for the first time to bring the conversion of C=O compounds present in DRB to value added chemicals by in-situ acetalization reaction of furfural with glycerol taken in different molar ratios (1:1, 1:3, 1:5) in presence of hydrogen peroxide, a greener protic solvent. The reaction was carried out in a domestic micro oven by microwave irradiation which enhances the selectivity of the reaction with high efficiency at short reaction time [56] and also energy intensive. This methodology does not require excessive amount of solvents and hence provides a clean and green approach towards glycerol acetalisation [57].

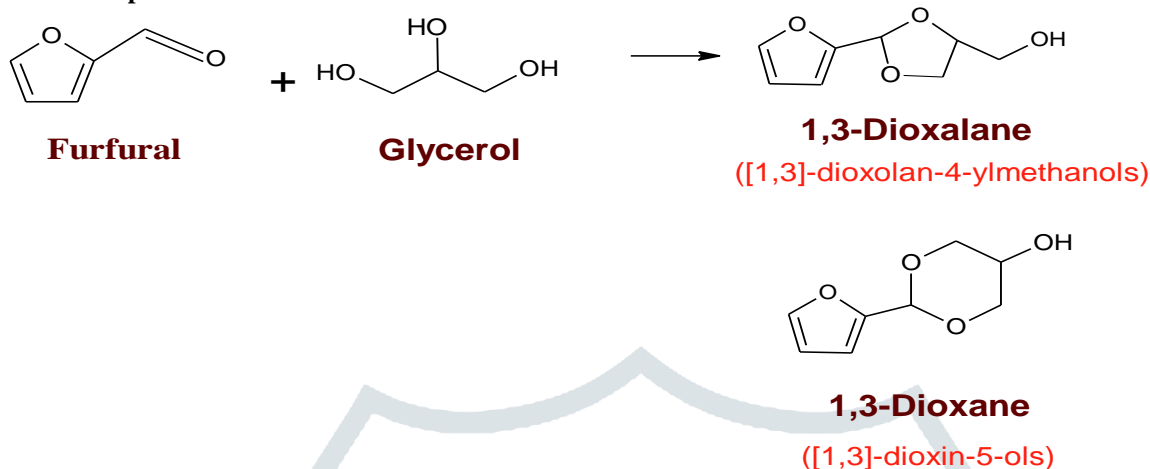
This process stimulates highly efficient energy transfer and selectivity that reduces reaction time significantly [61,62]. A latest contemporary work [63] reported high yields of cyclic acetals or ketals, including commercially valuable hyacinth fragrance and fuel additive precursors was derived from microwave-assisted acetalization of glycerol and carbonyl compounds. Due to the extended lifespan of hemiacetal intermediate with enormous amount of water in the reaction medium, none of the synthetic routes has been reported, the complete conversion and high yield from glycerol acetalization, with no isomeric six-(1,3-dioxane) being produced [64].

Microwave stimulated in-situ acetalisation reaction arose by controlling the reaction parameters like temperature and time with varying concentration of glycerol. The reaction mixture was microwave irradiated for 5mins to synthesis cyclic acetals and other value added chemicals. This microwave irradiation induces heat within the centre of the substrate through a direct energy conversion, making a reaction faster by raising the energy of the substrate material. This also improves selectivity of the products and energy efficiency, short reaction time and reduces the capital cost compared to conventional heating method [64].

Such glycerol-derived blend of cyclic acetals is the major products derived by insitu acetalisation reaction. It is noted that the DRB filtrate not only contains furfural but also other aliphatic, aromatic and cyclic compounds that undergoes secondary reactions such as oxidation, dehydration, condensation, esterification, etherification, hydrogenolysis, carboxylation etc that leads

to the formation of various minor products like alkene, esters, ethers, carboxylic acids as value added chemicals (shown in table-2) having wide commercial applications. These synthetic cyclic acetals can be significantly utilized as additives for diesel fuels and as building obstructs for surfactants.

### 3.2 Schematic representation of Acetal formation:



### Cis and Trans 5-and 6- membered furans

#### Scheme.1: Acetal products from Acetalisation of Furfural with Glycerol

#### 4. Characterization of the filtrate (FDRB-Filtrate Defatted rice bran)

The filtrate was well characterized by Gas chromatography-Mass spectroscopy technique using Spectrum Two model PerkinElmer FT-IR spectrophotometer having resolution of  $4\text{cm}^{-1}$  fitted with a  $\text{LiTaO}_3$  detector was used to get Fourier transform infrared (FT-IR) spectra recorded in the range  $4000\text{-}400\text{cm}^{-1}$ . UV-Visible spectra of the filtrate sample in the range  $190\text{-}950\text{nm}$  was obtained using UV-VIS system-Cary 8454, Agilent T Technologies. All this characterization of the filtrate confirms the presence of furan compound.

#### 4.1 GC-MS Spectral Analysis of the filtrate:

The maximum peak in the GC-MS spectra indicates the relative abundance of furan compound in the DRB filtrate as shown in the Fig.1 (a) and (b). FT-IR spectra and UV-Visible spectra also confirm the presence of intermediate compound.



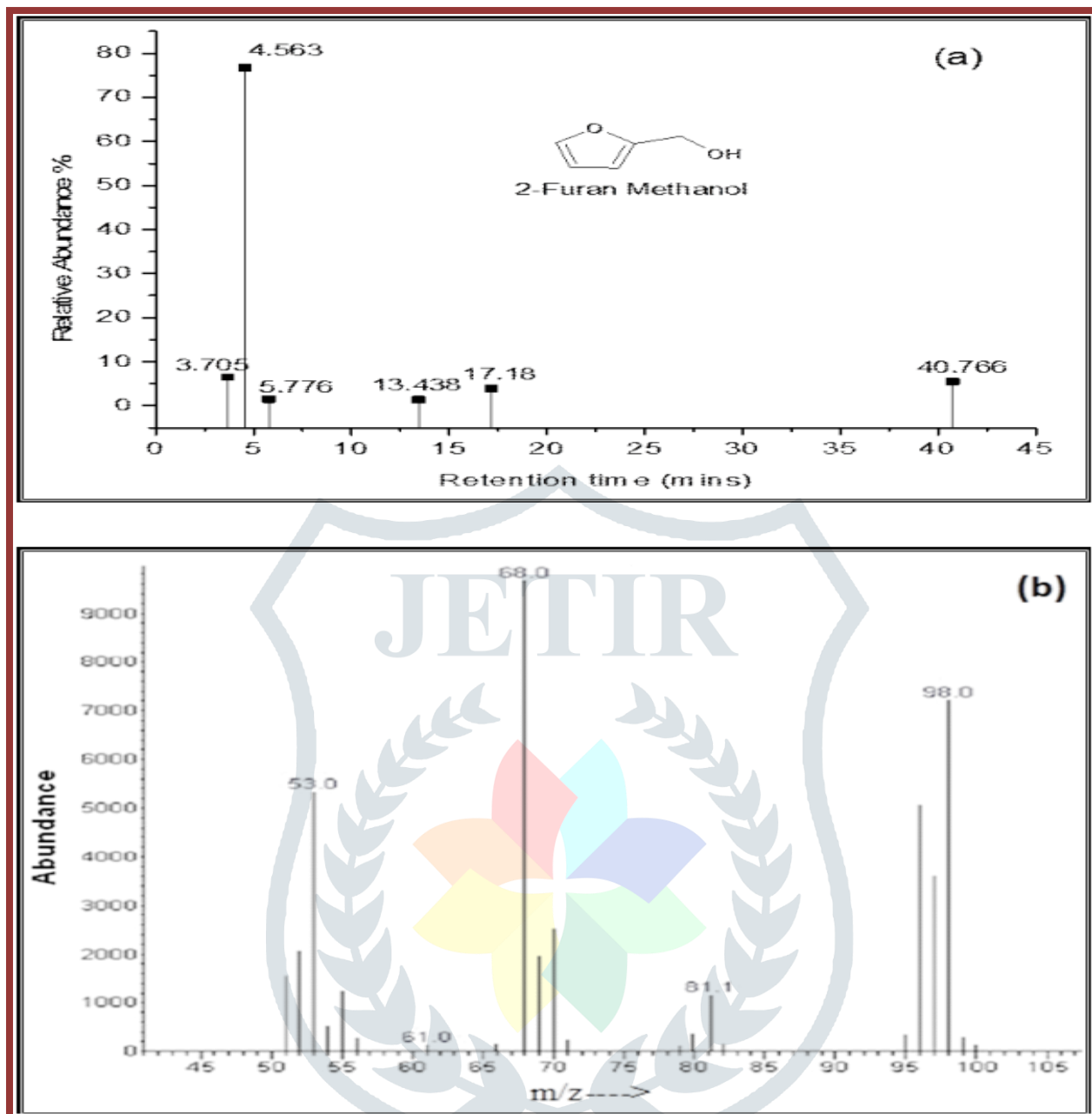


Fig.1 Shows the relative yield of the value added products for 1:1 ratio

#### 4.2 FT-IR Spectral Analysis of the filtrate:

The "Figure 2 (a)" shows the FT-IR spectra of FDRB indicates the presence of various functional groups present in the DRB filtrate. The strong broad absorption band at  $3369\text{cm}^{-1}$  and  $3592\text{cm}^{-1}$  indicates the stretching vibration of the hydroxyl groups. The absorption bands at  $1049\text{cm}^{-1}$ ,  $1153\text{cm}^{-1}$  and  $1414\text{cm}^{-1}$  attributed to C-O stretching vibrations confirms the presence of esters. The absorption bands at  $1634\text{cm}^{-1}$  indicates the presence of alkenes. The absorption bands at  $1725\text{cm}^{-1}$  indicates the stretching vibrations of carbonyl, carboxylic acid and aldehyde groups. The absorption band at  $2103\text{cm}^{-1}$  is attributed to alkyne, the absorption band at  $2942\text{cm}^{-1}$  is attributed to stretching vibrations of C-H bond and also the vibrations in the range  $1,650\text{cm}^{-1}$  to  $2,000\text{cm}^{-1}$  confirms the presence of conjugation.

#### 4.3 UV-Visible Spectral Analysis of the filtrate:

The "Figure 2(b)" shows UV-Visible spectra for the FDRB in which the major peaks with maximum absorbance ( $\lambda_{\text{max}}$ ) at  $196\text{nm}$  attributes for the presence of acids, esters, the minor peak at  $270\text{nm}$  attributes for the presence of carboxylic acids, aldehydes/ketones carbonyl groups, dienes and alcohols and also absorbance peak at  $309\text{nm}$  shows the presence of lignoglucoside in the DRB filtrate which inspired to make an attempt for the synthesis of various value added chemicals via acetylation of glycerol in different molar ratios with DRB filtrate.

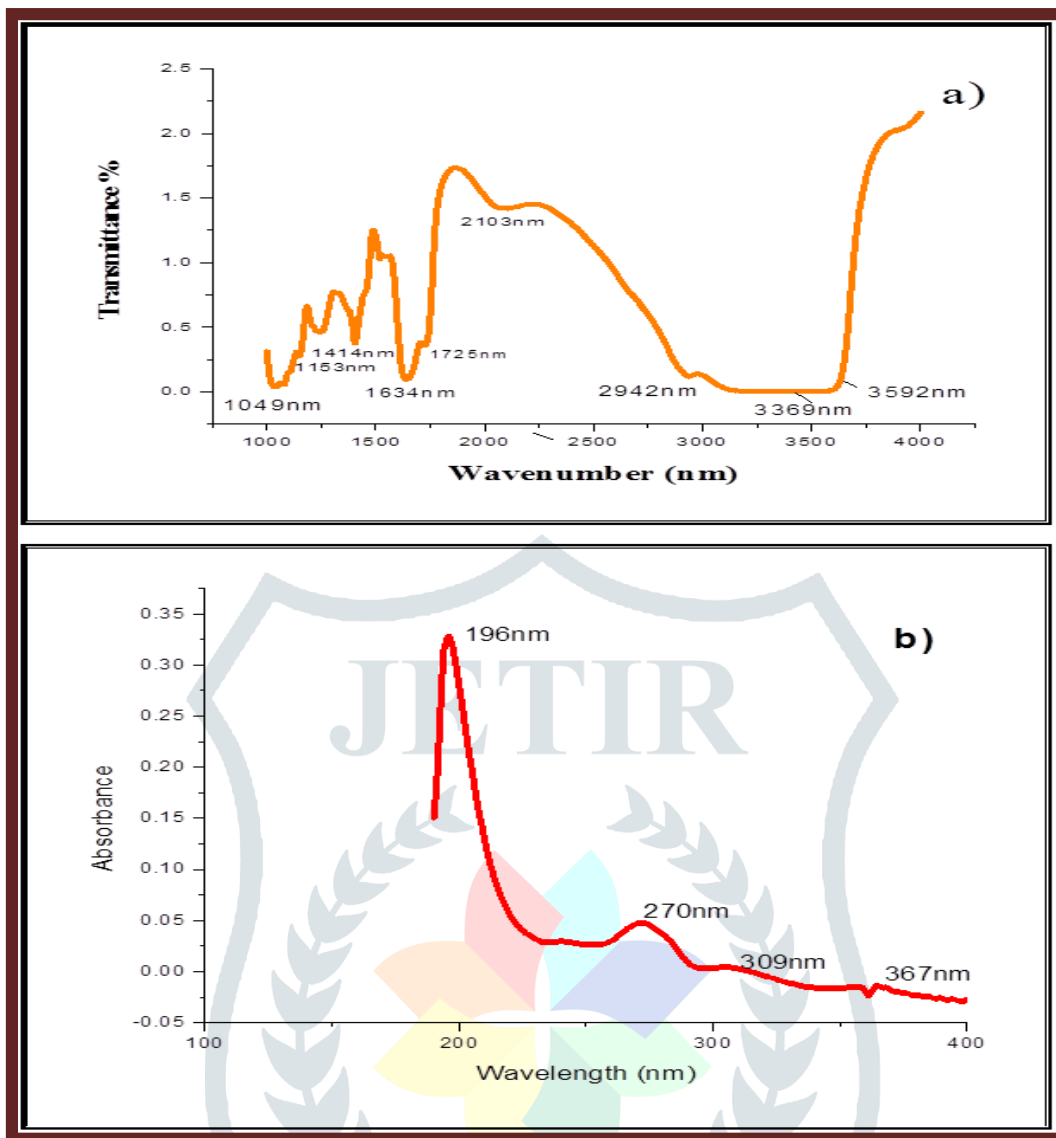


Fig.2. Shows the relative yield of the value added products for 1:1 ratio

**4.4 Microwave assisted in-situ acetalisation reaction:**

In-situ acetalisation reaction was performed in domestic microwave oven between furfural and glycerol in presence of hydrogen peroxide where the reaction mixture was prepared by varying molar ratios of Furfural/Glycerol and controlling all the reaction parameters like time and temperature identical throughout the process.

Table. 1 Comparative study of reaction parameters i.e., molar ratios, reaction temperature and time on the yield of the product derived from in-situ acetalisation reaction

DRB Filtrate(ml)	Glycerol (ml)	Molar Ratios	30% H <sub>2</sub> O <sub>2</sub> (ml)	Batch-1			Batch-2		
				Temp (K)	Time (mins)	Yield of the Product (g)	Time (mins)	Temp (K)	Yield of the Product(g)
10	---	Blank	---	325	1.5	0.749	5	303	0.693
10	----	-----	5	325	1.5	0.390	5	303	0.281

10	10	1:1	5	325	1.5	12.6	5	303	1.205
10	20	1:2	5	325	1.5	22.912	5	303	6.567
10	30	1:3	5	325	1.5	34.143	5	303	26.813
10	40	1:4	5	325	1.5	46.273	5	303	37.389
10	50	1:5	5	325	1.5	61.058	5	303	50.398

#### Optimum Reaction conditions:

1. Molar ratios of the sample:

- 1:1 ratios: 10ml filtrate+10ml Glycerol +5ml H<sub>2</sub>O
- 1:3 ratios: 10ml filtrate+30ml Glycerol +5ml H<sub>2</sub>O<sub>2</sub>
- 1:5 ratios: 10ml filtrate+50ml Glycerol + 5ml H<sub>2</sub>O<sub>2</sub>

2. Reaction time: 1.5mins, 5mins

3. Reaction Temperature: 325K, 303K

The reaction was performed in two batches for all the molar ratios of the mixture including blank and filtrate with H<sub>2</sub>O<sub>2</sub> by varying reaction time and temperature in each batch as shown in the table-1. Increase in the concentration of reactant i.e glycerol in the reaction mixture will drive the reaction in forward direction allowing for an increase in % conversion and the yield of acetal product [65]. If these reactants are provided in any other amounts, one of the reactants will nearly always be entirely consumed, thus limiting the amount of product that may be generated [67].

As per Le Chatelier's principle, the position of reaction equilibrium can be altered by using a large excess of glycerol with respect to furfural [66].

According to the collision theory, as the concentration of glycerol is increased in the reaction mixture, frequency of successful collisions of that glycerol would increase as well, allowing for an increase in the forward reaction [65].

The reaction was performed by microwave irradiation technique which has become an attractive tool for high speed synthesis of organic and inorganic compounds. Microwave radiation is a type of electromagnetic energy lies in the frequency range of 0.3 to 300GHz having wavelengths approximately in the range 30cm to 1mm. The energy is transferred rapidly in reaction containers normally made up of microwave-transparent substances, like borosilicate glass, quartz or Teflon by direct coupling of microwave radiation with the particles that are present in the reaction mixture, resulting quick instantaneous localized superheating [68, 69].

Microwave technique results in an inverted temperature gradient compared to conventional heating method. Microwave radiation consists of both electric field and magnetic field. Heating in microwaves is caused by electric field component and interactions with magnetic field normally do not occur. The two fundamental methods i.e., dipolar polarization and ionic conduction transfers energy from the electric component of microwaves to the substrate effectively so that completes the reaction fast with good product yield. It is evident from the above table that product yield and % conversion also depends on the temperature and reaction time. The batch-1 reaction was done at 325K with reaction time of 1.5mins whereas batch-2 reaction was done at 303K with reaction time of 5mins also has profound influence on the product yield.

## 4.5 GC-MS analysis of the synthesized product:

Table.2 comparison of relative yield and retention time of the derived products for the molar ratios 1:1, 1:3 and 1:5

1:1 ratios			1:3 ratios			1:5 ratios		
Name of the compound	Retention time (mins)	Relative Yield (%)	Name of the compound	Retention time (mins)	Relative Yield (%)	Name of the compound	Retention time (mins)	Relative Yield (%)
2-Propanone, Methyl hydrazone 1,3-Dioxane,	3.270	17.60	9-Octadecen-1-ol	26.301	20.99	Cis-9-Hexadecenal	26.301	26.328
3-cyclopentyl -6-methyl-1-Pentanol 3,4-Heptadien-2-one	23.097	11.242	Silicic acid	31.811	79	13-Octadecenal	26.301	26.328
7-Hexadecyn-1-ol, Cyclohexanone	25.460	13.7162	Diethyl bis(trimethylsilyl) ester	31.811	79	Isothiazole, 4-methyl-2-Butene	3.276	47.34
9,12-Octadecadienoic acid	26.295	57.4407	----	----	----	Methylester 2-Myristinoyl glycinamide	31.857	26.328
----	----	---	----	----	----	1,2 Bis(trimethylsilyl)benzene,11-Hexadecenoic acid	31.857	26.328
----	----	-----	----	----	-----	11-Hexadecenoic acid	31.857	26.328
----	----	----	-----	-----	-----	15-Methyl-methylester	31.857	26.328

The Fig.3 shows the relative yield of the products for 1:1 ratio confirms the presence of major aldol product as methyl hydrazone 1,3-Dioxane by GC-MS spectra with less relative yield signifies the incomplete conversion of glycerol into aldol product whereas 2-Propanone, 4,6-dimethyl-Isothiazole, 5-methyl-3,4-Heptadien-2-one, 3-cyclopentyl -6-methyl-1-Pentanol,5-cyclopropylidene-2,4-Hexadien-1-ol,7-Hexadecyn-1-ol,9,12-Octadecadien-1-ol,cyclohexanone compounds are the minor products. Value added products such as 9,12-Octadecadienoic acid as major peak.

The Fig.4 shows the relative yield of the products for 1:3 ratio indicates the presence of products such as 9-Octadecen-1-ol, 14-Methyl-8-hexadecen-1-ol,18-Nonadecen-1-ol compounds as major peaks and Silicic acid, diethyl bis(trimethylsilyl) ester, 1,1,1,3,5,5,5 Hepta methyltrisiloxane Benzene as minor peaks.

The Fig.5 shows the relative yield of the products for 1:5 ratio indicates the presence of products such as Cis-9-Hexadecenal,2-Methyl-Z,Z-3,13-octadecadienol,13-Octadecenal compounds as major peaks and Isothiazole, 4-methyl-2-Butene, methyl ester,11-Hexadecenoicacid, 2-Myristinoylglycinamide, 1,2-Bis(trimethylsilyl)benzene compounds as minor peaks. Hence the GC-MS spectral studies for 1:1,1:3 and 1:5 ratios indicates the presence of various value added chemicals such as carboxylic acids, esters, aldehydes, diene, amide, ketones, alcohols, cycloalkenes etc derived from in-situ acetalisation reaction of DRB filtrate containing furfural with glycerol in different molar ratios by microwave irradiation.

Hence the data's shown in table-2 evidently reveals that relative yields and selectivity of the product differs with increase in the furfural to glycerol ratios in the reaction mixture.



4.5.1 GC-MS Spectras for the reaction mixture with molar ratios 1:1, 1:3 and 1:5.

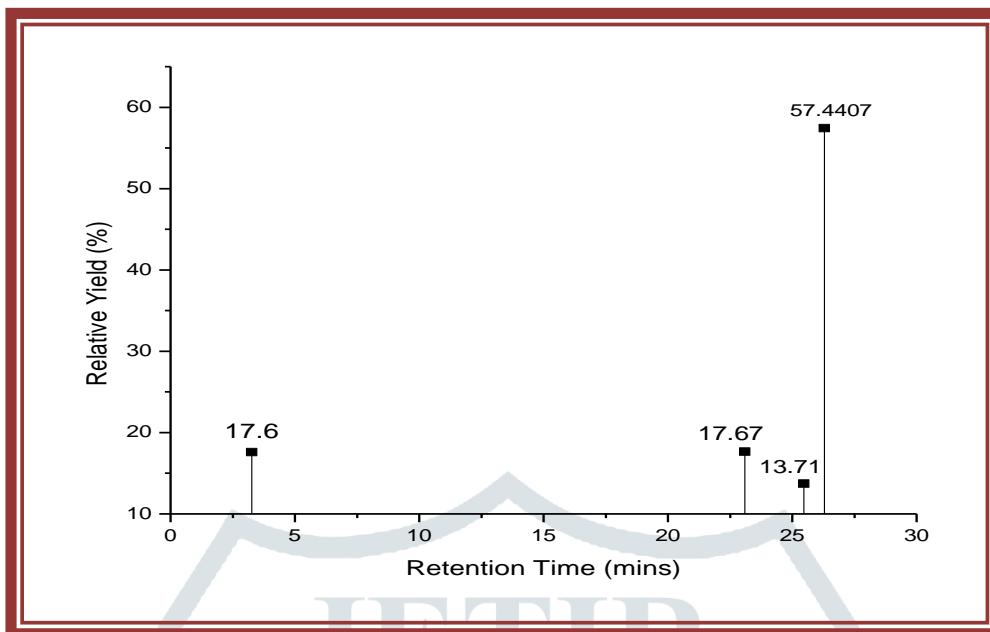


Fig.3. Shows the relative yield of the value added products for 1:1 ratio

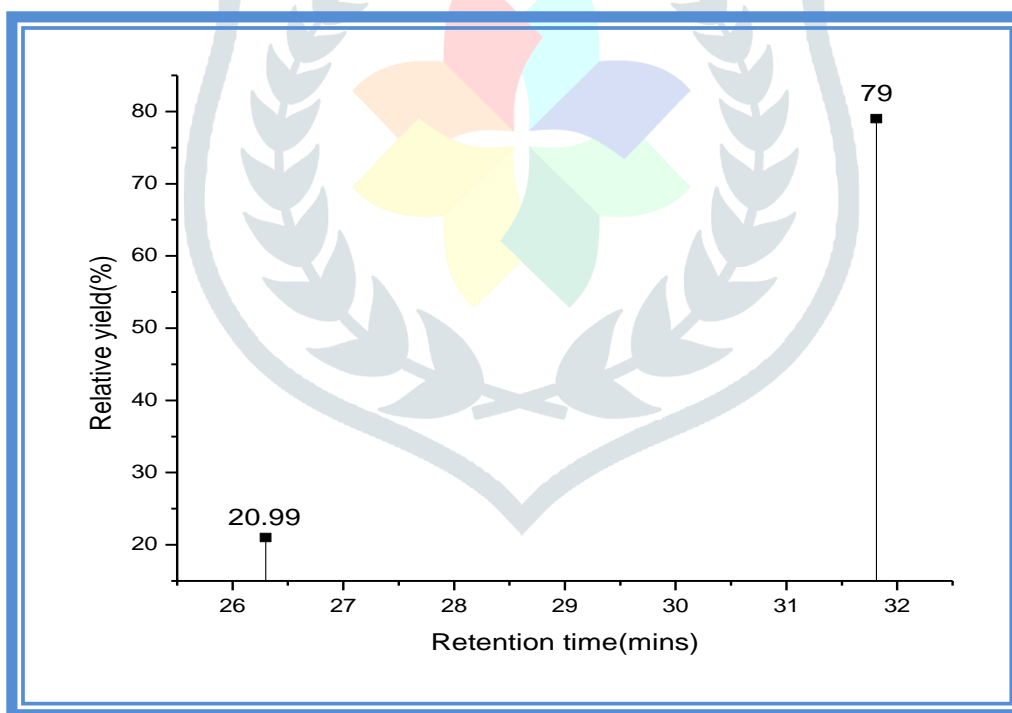


Fig.4. Shows the relative yield of the value added products for 1:3 ratios

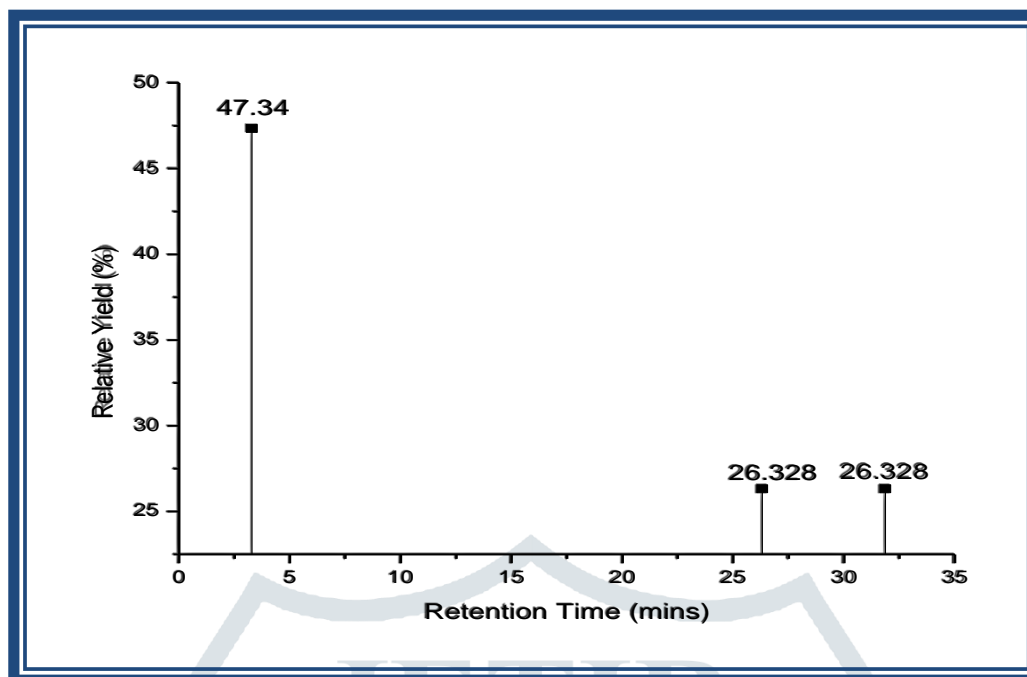


Fig.5. Shows the relative yield of the value added products for 1:5 ratios

## 5. CONCLUSION

The main focus of the work is to develop a greener and eco-friendly method to enforce and promote more sustainable practices for a bio-based future society. Here, glycerol available in excess as by-product from the biodiesel production is utilized as a renewable waste substrate for the conversion of furfural derived from DRB residue. The novel approach of microwave assisted in-situ acetalisation reaction of furfural with glycerol, leads to the formation the acetal i.e 1,3-dioxane and value added chemicals such as carboxylic acids, esters, aldehydes, dienes, amides, ketones, alcohols, cycloalkanes and so on derived by rapid and selective heating, cost effective and energy intensive method. The work aims at optimizing the reaction parameters like reaction time, temperature and molar ratios of substrates that has great impact on yield of the products. These acetals are used as fuel additive for diesel fuels and also value added chemicals have wide applications in polymer, agrochemical and pharmaceutical industries.

## 6. ACKNOWLEDGMENT

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