



No : 042/ ICOOPChE 2<sup>nd</sup>-2017/JTK-UR/XI/2017

Subject: Letter of Acceptance

### LETTER OF ACCEPTANCE

Dear Mrs. Istiqomah R,

We are pleased to notify that your article which entitled “**Glucose, Temperature, and Time Reaction Effects: Quantitative Study of Furfural Decomposition**” is accepted as an oral presentation. Therefore, please complete our **registration form** for certificate’s requisite.

Your manuscript had been reviewed and hopely it would be revised as reviewer comments then you should send it back to the committee due to **14<sup>th</sup> November 2017**. For your information, we enclosed another hotel address around our conference place.

After receiving this acceptance letter, you could start paid the registration fee until the conference day to **Bank Negara Indonesia (BNI) Pekanbaru, Account No : 0438929025 c.q Komalasari, Swift code:BNINDJA** and please confirm it to **WA No. +6281365327330**.

Therefore, you are invited to attend the seminar

Date : Thursday / November 30<sup>rd</sup> , 2017  
Time : 07.30 am – Done  
Venue : Hotel Pangeran, Jl. Jend. Sudirman No. 371-373, Pekanbaru,  
Riau-Indonesia

Please prepare your presentation softcopy, short curriculum vitae (CV) and transfer payment receipt. We are sorry for the inconvenience. Thank you for your attention.

Best Regards,

ICOOPCHE 2017 Committee

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## REVIEWER COMMENTS

<b>Title of Manuscript:</b>	Glucose, Temperature, and Time Reaction Effects: Quantitative Study of Furfural Decomposition
<b>Reviewer Recommendation Term (Accepted Without Revision/Minor Revision/Mayor Revision/Rejected):</b>	Accepted with major revision
<b>Reviewer Comments to Authors</b>	<p>Title not very clear focused, it should be revised Abstarct is not very clear. Abstarct should adressed a background, method and result. It is not common to put figure or referred figure in abstarct. English is seemed should be improved Conclusion should in passive voice, and no referred to any figure anyway. It seem a quite similar research method of this paper and the other paper (title: The Role of xylose in Furfural Decomposition)</p>

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# Glucose, Temperature, and Time Reaction Effects: Quantitative Study of Furfural Decomposition

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**Abstract:** The utilization of biomass as renewable energy is potential to become the fuel and intermediate chemicals supplier. Furfural is one of important derivative chemical substances which is obtained from biomass degradation. Regarding the importance of furfural and its decomposition, this research would investigate several factors which could affect the furfural production and its decomposition. The presence of glucose addition which is one of sources/materials of furfural would be analyzed the interaction as a factor affected the quantity of furfural decomposition product beside of temperature and time factor in a decomposition reaction. We can conclude the effect from parameter analyzed in this study. The presence of glucose is one of factors which could affect the quantity of furfural decomposition product (i.e. levulinic acid (LA)). The effect of reaction conditions such as temperature (140–180 °C), glucose loading (0–0.6 M), and reaction time (0–120 min) on LA yields were determined experimentally. HPLC was used to identify and quantify the liquid product from the reactions. The highest experimental LA yield was 62 wt% and was obtained at 160 °C, a glucose loading of 0.2 M, and a reaction time of 120 min.

**Keywords:** Biomass; Glucose; Furfural; Sulfuric acid; decomposition mechanism

## 1. Introduction

The energy demand for the world development relies on the existence of mineral resources. Meanwhile, renewable energy, such as wind and solar cell guarantee sustainability of the energy available to produce electricity. However, the availability of renewable energy still depends on the supply from the industry of chemicals and fossil fuel. The facts, around 170 chemicals produced in the US with the volume exceeded  $4.5 \times 10^6$  kg, 98% is the derivative of oil and natural gas. Sophisticated synthesis product is also the derivative from oil, consequently, has the high cost [18]. Biorefinery technology gives the suitable alternative through biomass utilization, an organic chemical which is abundant on the ground. The utilization of biomass as renewable energy has a potency to become the fuel supplier and intermediate chemicals. The challenge to utilize the natural resources is to develop the cost-effective method to convert biomass into valuable chemicals.

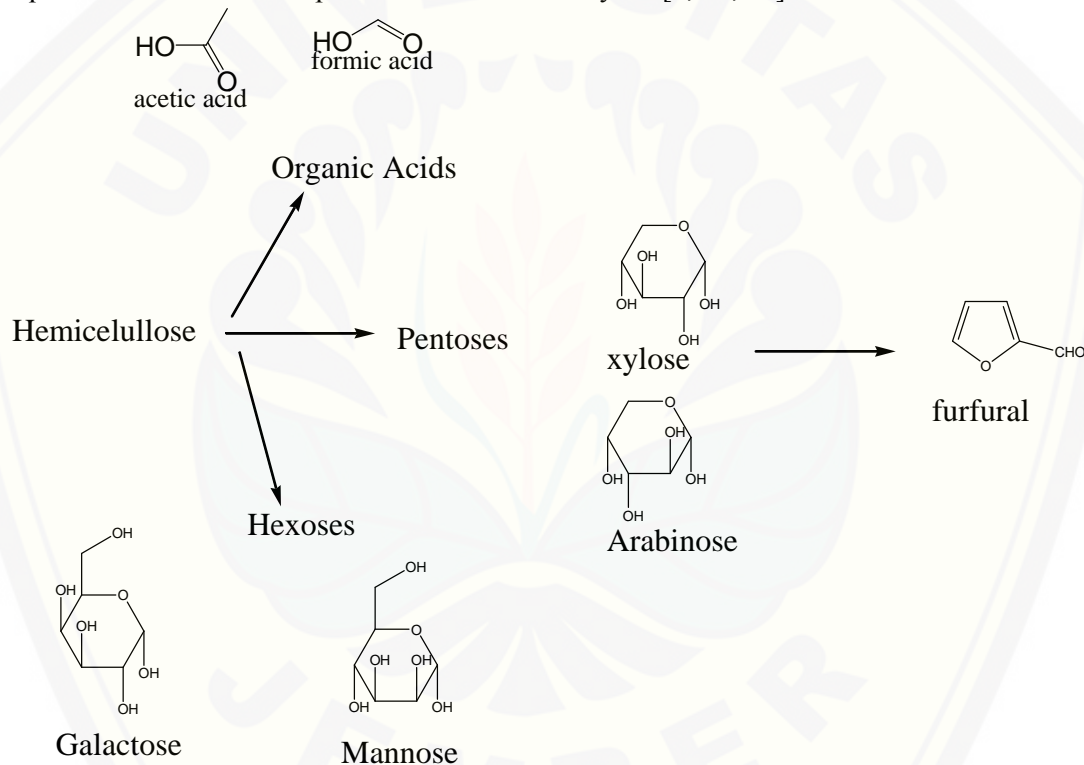
Inulin is one of biomass components had fructose polymer which majority have around 35 fructose monomer bond each other in a linear chain by  $\beta$ -2,1 glycoside bond, with the end of the chain is glucose. One of decomposition components from inulin forming is furfural. There is no synthesis pathway for furfural, therefore furfural exclusively is produced from biomass resources which are renewable using acid-dehydration [3]. Caused by unsaturated bond and aldehyde function, furfural is capable and a key of the derivative of important chemical substances, and can be implemented widely in oil refinery, plastic, pharmacy, and agrochemical industry.

Furthermore, many research had shown that HMF is one of furfural decomposition product and a precursor of liquid alkane production (C7 – C15) which is diesel fuel component [16]. HMF also can produce another substance, which are important substances such as levulinic acid. Levulinic acid (LA) is a short chain organic acid and precursor of chemical substances, has a potency to be implemented in textile coloring, anti-frozen ingredients, feed additive, ink and coating material, solvent, food flavoring, and drugs, resin, synthesis fiber battery material, and cosmetics. Chemical application of LA includes a chiral reagent, oil, adsorbent, and broadly used in the polymer industry. Furthermore, LA is mostly decomposed into 2-methyltetrahydrofuran which used as fuel [2].

Regarding the importance of furfural and its decomposition, this research would investigate several factors which could affect the furfural production and its decomposition. The presence of glucose addition which is one of sources/materials of furfural would be analyzed the interaction as a factor affected the quantity of furfural decomposition product beside of temperature and time factor in a decomposition reaction.

## 2. Literature Review

Furfural has no synthesis pathway except the biomass degradation. It is produced solely from hydrolysis of hemicellulose into monomeric sugar and further dehydration process could be seen from fig. 1. Furfural has unique production mechanism because furfural can be degraded and formed in the same condition. Formic acid and resinous tars [1, 15, 19] are formed during degradation, the degradation occurred could be broken down the carbon chain or polymerization. The role of formic acid has been argued, [14] however, in the majority of the studies, it can be catalyzed the reaction [12] and few quantities of formic acid presence have been analyzed [1, 19, 20].



**Figure 1.** Hemicellulose hydrolysis and further dehydration process [12]

Several studies on the hexoses hydrolysis have been extensively studied by [6 – 9]. The kinetic modeling of [6 –9] have been used broadly as the fundamental approach of the hydrolysis and dehydration process. Furfural degradation process from the pure substance has been determined in acidic solution by [14, 15, 29]. The previous studies also discussed the mechanism of furfural degradation. The presence of sugars in the solution especially glucose has not been studied extensively, although the sugars would be always in the solution when either furfural degradation or production. In the study of [13], it concluded that the presence of glucose could enhance the decomposition rate of furfural but it could not describe the detailed mechanism. Distinctively from furfural, HMF is not produced in a large amount yet because of the selectivity gained during the hydrolysis and dehydration process or expensive recovery process [13].

Many researchers had been conducted of glucose decomposition mechanism in aqueous solutions [17]. Glucose experienced isomerization to fructose was determined under the mild acid

condition as an important reaction pathway. Isomerization takes place through enediol transformations called the Lobry de Bruyn-Alberda van Ekenstein transformation (LBET) [17]. Glucose in 0.5M sulfuric acid solution was heated at 100°C for 1.5 hours produced 5-hydroxymethylfurfural (5-HMF) and isotope exchange techniques were studied [10]. It concluded that intramolecular hydrogen transfer occurred. Several studies had been proposed a generalized decomposition mechanism for glucose, as shown in Fig. 2.

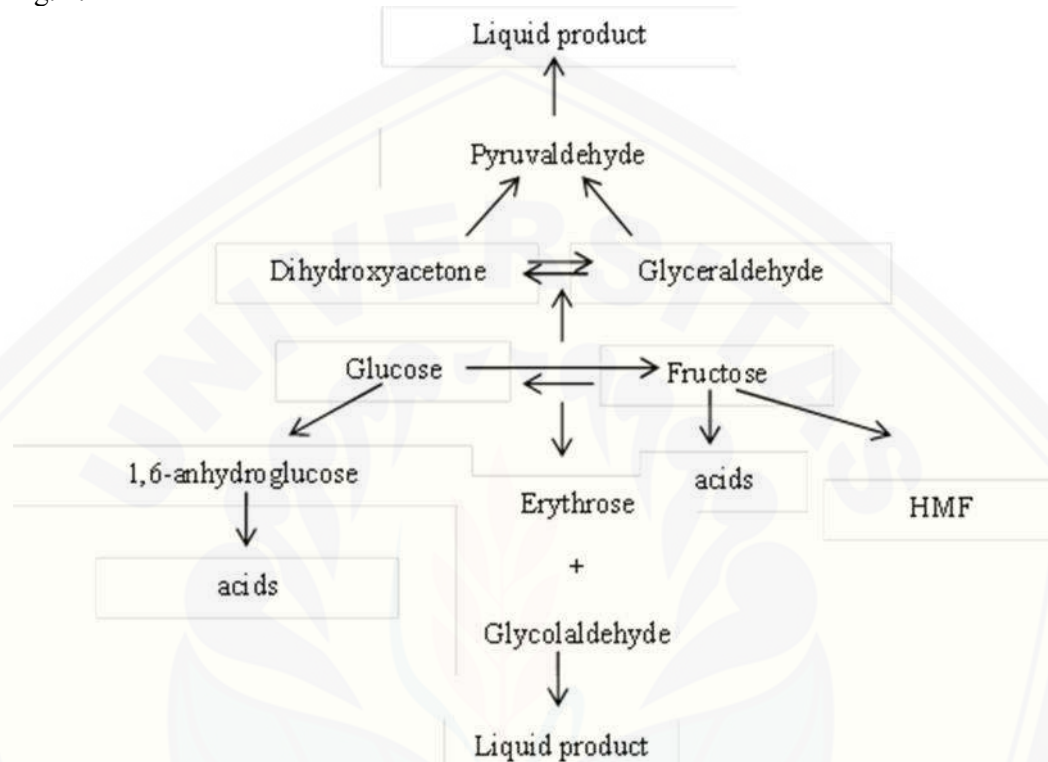


Figure 2. Generalize glucose decomposition mechanism without any catalyst [11]

### 3. Material & Methodology

#### 3.1. Chemicals

Furfural ( 99%) and Levulinic acid ( 97%) were obtained from Acros Organic (Geel, Belgium). Sulphuric acid (96-98 %wt), Formic acid ( 95%) and D-glucose ( 99.5%) were purchased from Merck KGaA (Darmstadt, Germany). 5-Hydroxymethylfurfural (HMF) ( 99%) was obtained from Sigma Aldrich (Steinheim, Germany). All chemicals were used without purification. De-ionized water was used to prepare the solutions.

#### 3.2. Method

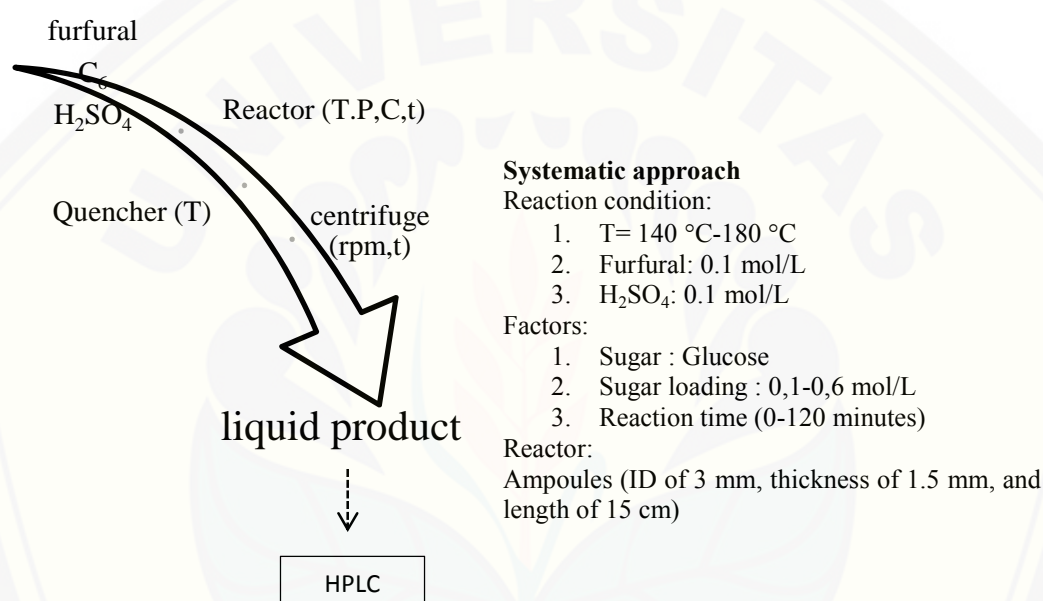
The experimental procedures are based on previous research by [4]. In a typical experiment, the pre-determined amount of glucose, and de-ionized water (4 mL) were loaded to glass ampoules with an internal diameter of 5 mm, a length of 15 cm and thickness of 1.5 mm. The ampoules were sealed with a torch.

For the exploratory experiments, a series of ampoules was placed on a rack in a heating oven (Heraeus Instruments Type UT6060) at a constant temperature. At different reaction times, an ampoule was taken from the oven and quickly quenched in cold water to stop the reaction. The experiments carried out in the framework of the experimental design were individually performed in an oven (Heraeus Instruments Type UT6060) at the pre-set temperature. After the reaction, the

ampoules were opened and the reaction mixture was taken out and centrifuged for about 10-30 minutes to remove the solids. The liquid product was diluted with demin water before analysis.

### 3.3. Analysis

Commonly, the analysis follows method by [5]. The HPLC was used to identify and quantify the liquid product from the reactions. The HPLC system consisted of a Hewlett Packard 1050 pump, a Bio-Rad organic acid column Aminex HPX-87H and a Waters 410 differential refractive index detector. A very dilute aqueous sulfuric acid ( $H_2SO_4$ ) solution (5 mM) was used as the eluent with a constant flow rate of about  $0.55 \text{ ml}\cdot\text{min}^{-1}$ . The column was operated at  $60 \text{ }^\circ\text{C}$ . The HPLC was calibrated with solutions of the pure compounds at a range of concentrations. Using the chromatogram peak area and the external calibration curve, the unknown concentrations of components in the liquid phase was determined.



**Figure 3.** The Research Methodology

## 4. Results and Discussion

### 4.1. Separation result

The experiment was performed at  $160 \text{ }^\circ\text{C}$ , the initial concentration of respectively  $0.1 \text{ M}$  glucose, furfural, and  $H_2SO_4$ . Based on chromatography profile (Figure 4), glucose and furfural are still identified and several new products are detected in the liquid phase which is HMF (the retention time holds at  $24^{\text{th}}$  minutes), levulinic acid (the retention time holds at  $18^{\text{th}}$  minutes, and formic acid (the retention time holds at  $16^{\text{th}}$  minutes). HMF is the decomposition product of glucose before it decomposes further to LA. LA is also produced by furfural decomposition process as the main product. In furfural decomposition process, some formic acid is also produced as a by-product. Some solid by-products known as humins were also formed in this experiment.

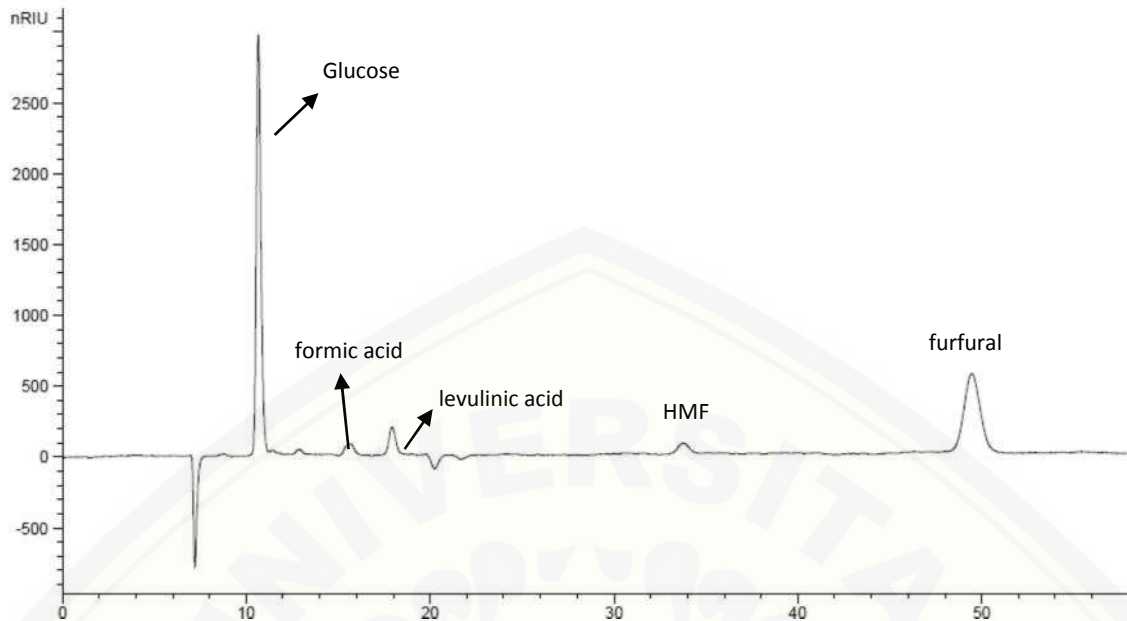


Figure 4. Chromatogram profile at reaction time 90 minutes and 160°C.

Figure 5 shows the relationship between the concentration and reaction time. It is clearly seen that the concentration of glucose and furfural decreases as a function of reaction time because they decompose to HMF and formic acid in acidic condition. The concentration of LA increases significantly over time because LA is the main decomposition product of glucose and furfural. The decomposition product, LA, can be generated for 15 minutes. It triggered by formation product and followed by ring-opening reaction of HMF. The concentration of HMF is remained constant due to it is an intermediate product of glucose decomposition before it transforms to LA.

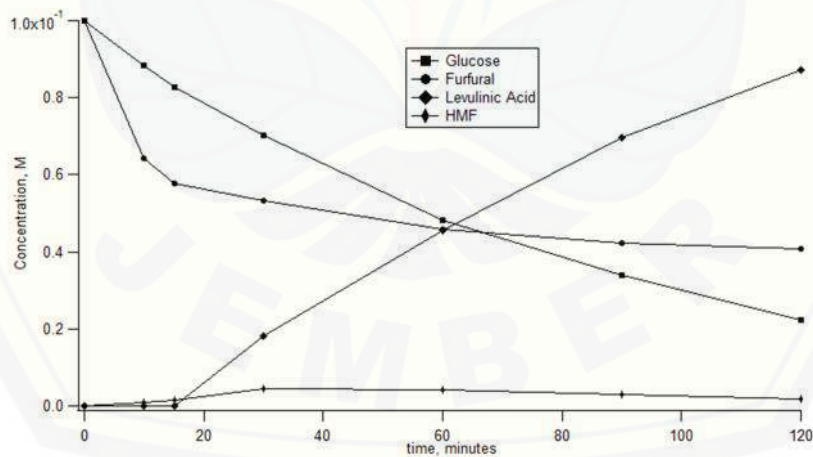
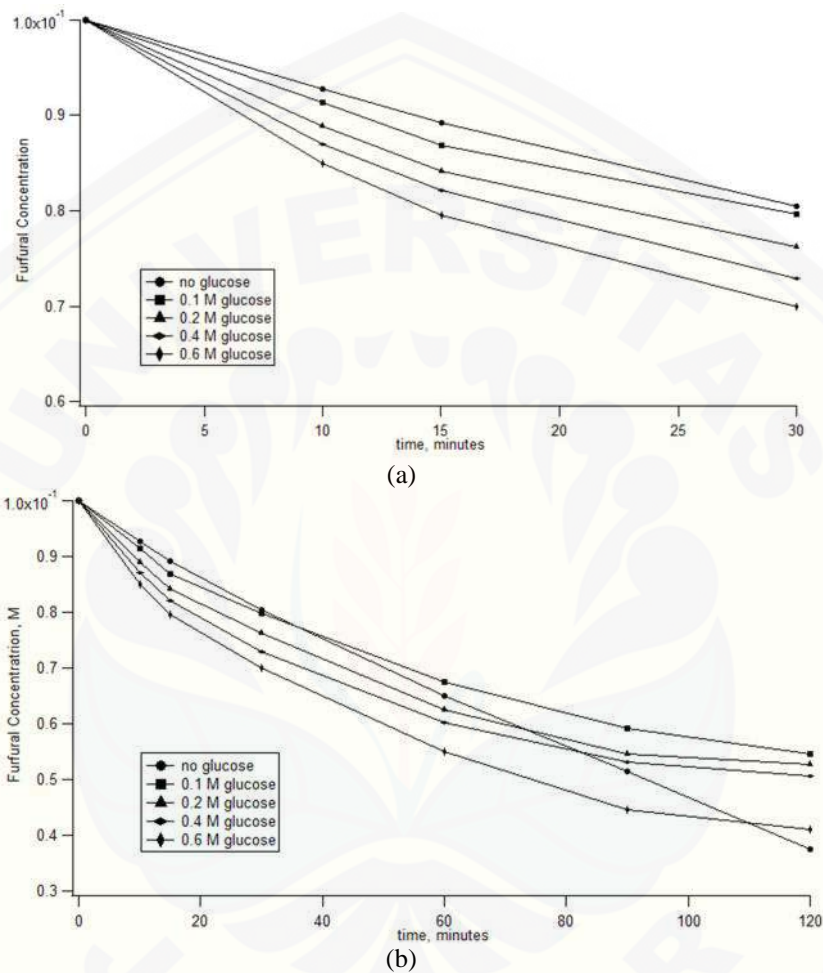


Figure 5. The concentration profile of interaction result between glucose and furfural in presence of sulfuric acid.

#### 4.2. Effect of glucose loading

A typical furfural concentration versus reaction time profile is shown in figure 6. In reaction time between 0-30 minutes, as glucose concentration and reaction time increase, furfural concentration decreases. This phenomenon indicates that the reaction produces less humin. On the other hand, after 30 minutes reaction, more humin were produced. Based on Figure 6 b, the effect of glucose addition is obviously noticed. In the absence of glucose, no humin is produced, the relationship between furfural concentration and reaction time can be drawn as a linear line. The bigger glucose concentration,

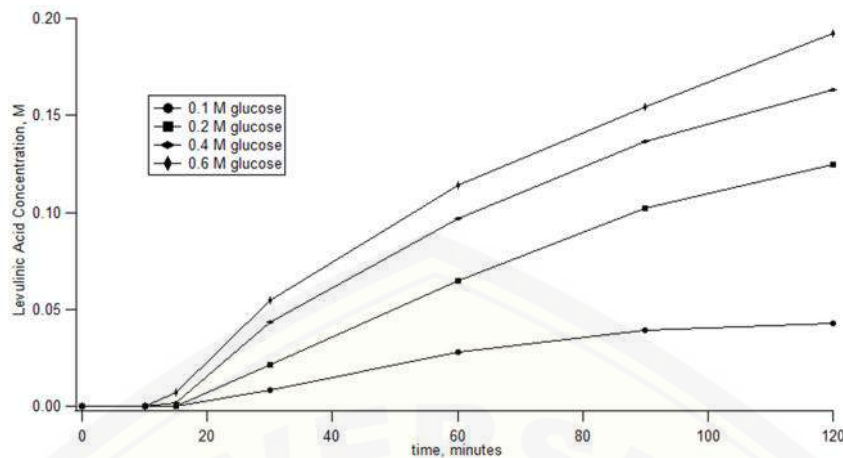
furfural concentration diminishes rapidly early 30 minutes. After 30 minutes, the decomposition reaction with presence of 0.1 M glucose is slower compared to blank (no glucose), perhaps either glucose was decomposed into furfural or glucose was decomposed to another substance. After 90 minutes, the decomposition reaction with presence of 0.1 M, 0.2 M, and 0.4 M glucose are slower compared to blank (no glucose). After 120 minutes, all decomposition reactions with the presence of glucose are slower compared to blank (no glucose).



**Figure 6.** Relationship between furfural concentration and reaction time (a) 0-30 and (b) 30-120 minute in various glucose concentrations.

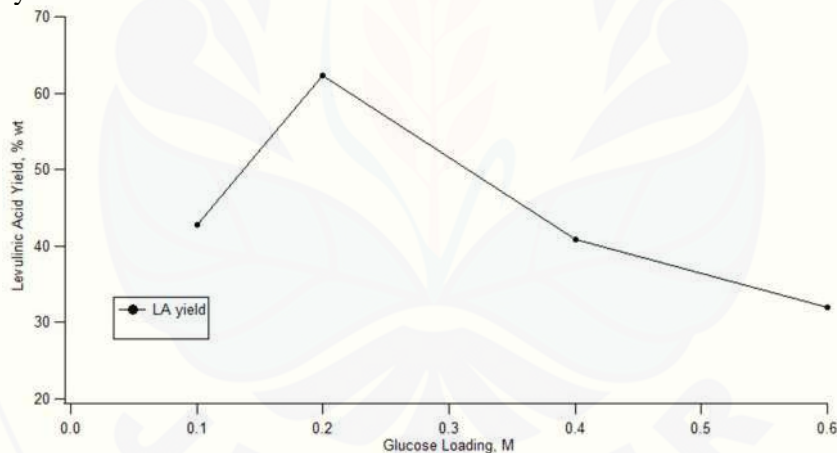
The effect of glucose concentrations on the furfural conversion is presented in Figure 7. It is found that LA was produced after 15 minutes reaction. Figure 7 also clearly implies that high glucose concentration loading leads to increase LA concentration produced. This because glucose can be converted to furfural which feeds the production of LA.





**Figure 7.** The relationship between levulinic acid concentration and reaction time in various glucose concentrations.

Based on Figure 8, the highest experimental LA yield is about 60 wt%, obtained at a glucose concentration of 0.2 M. The LA yield decreased significantly at higher glucose concentration (more than 0.2 M). There is a competition reaction between LA formation and the humin formation, which the reaction of humin formation was more favourable at the higher concentration of glucose. This explanation was supported by the data, where the addition of glucose concentration (0.6 M) showed a decrease of LA yield.



**Figure 8.** The relationship between levulinic acid yield and glucose concentration at 120 minutes.

#### 4.3. Effect of reactor temperature

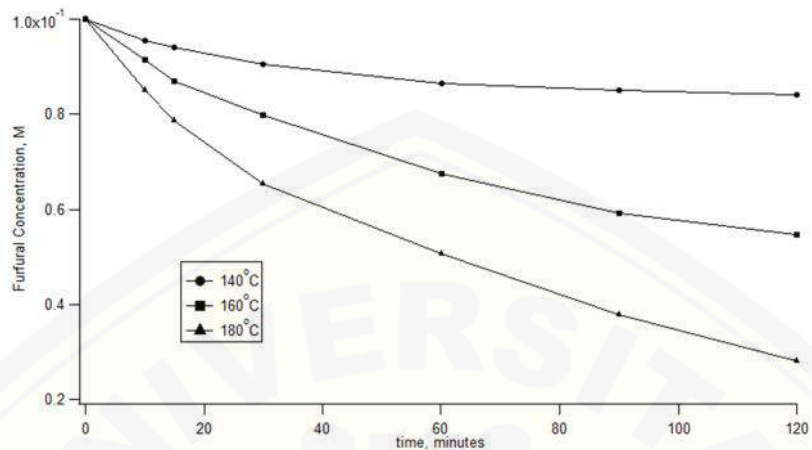
Figure 9 shows the effect of different temperature to furfural decomposition process. In line with to previous results, the concentration of furfural decreases as a function of reaction time. As the reactor temperature increases, the decomposition process occurs more rapid which due to the higher value of reaction rate constant ( $k$ ) at an elevated temperature [20]. In an acidic aqueous solution (e.g.  $H_2SO_4$ ), the following kinetic equation of furfural decomposition was written as a pseudo-first-order reaction with an assumption that the concentration of acid remained essentially constant and presence of water is in overwhelming abundance [20].

$$\frac{d[\text{furfural}]}{dt} = -k[\text{furfural}]$$

The following Arrhenius-type equation correlated the experimental results over the temperature range 150-210 °C,

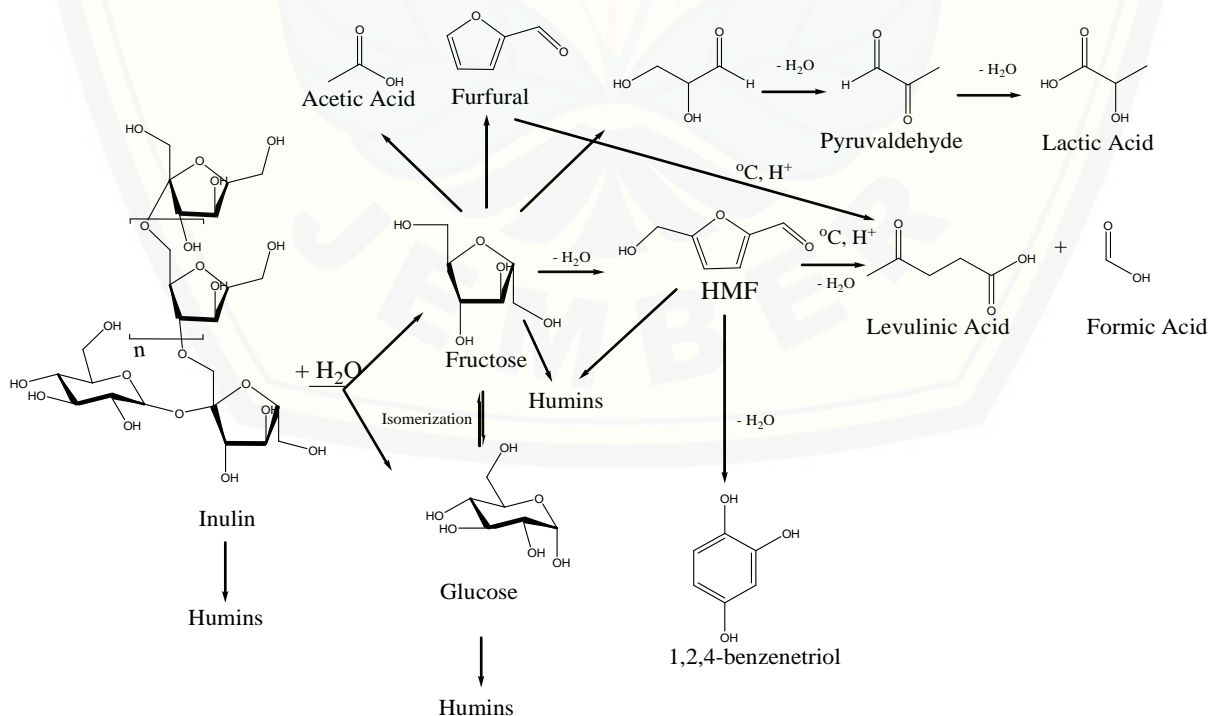
$$k = (1.396 \times 10^7) e^{-83.6/RT}$$

Where the rate constant  $k$  is in  $\text{min}^{-1}$ , the universal gas constant  $R$  in  $\text{kJ/mol K}$ , and the absolute temperature  $T$  in  $\text{K}$ .



**Figure 9.** Relationship between furfural concentration and reaction time in various temperatures

We can draw a plausible mechanism from the above description to show the role of glucose addition for furfural decomposition. The mechanism occurred in the solution is mainly caused by the degradation of both glucose and furfural. However the furfural degradation itself gives a major contribution in the solution proved by the decrease of furfural concentration for the entire time of the reaction. Moreover, the presence of glucose increases the number of furfural in the reactor because glucose can be converted to furfural via isomerization process to become fructose as an intermediate species first. TBased on obtained experimental data and literatures in this reference, we can obtain the mechanism pathway of furfural decomposition with the presence of glucose below (Fig. 10)



**Figure 10.** A plausible mechanism of furfural decomposition with the presence of Glucose.

## 5. Conclusion

The rate of furfural decomposition has been accelerated in the presence of glucose. The result indicated that glucose addition may increase decomposition rate in the early reaction. The reaction rate was declined after 30 minutes. The decomposition reaction rate has been influenced by temperature in accordance with Arrhenius equation.

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# **Glucose, Temperature, and Time Reaction Effects: Quantitative Study of Furfural Decomposition**

**Istiqomah R, Fachri B A, Felix A S, Bektı P, Meta F R and Susanti A**

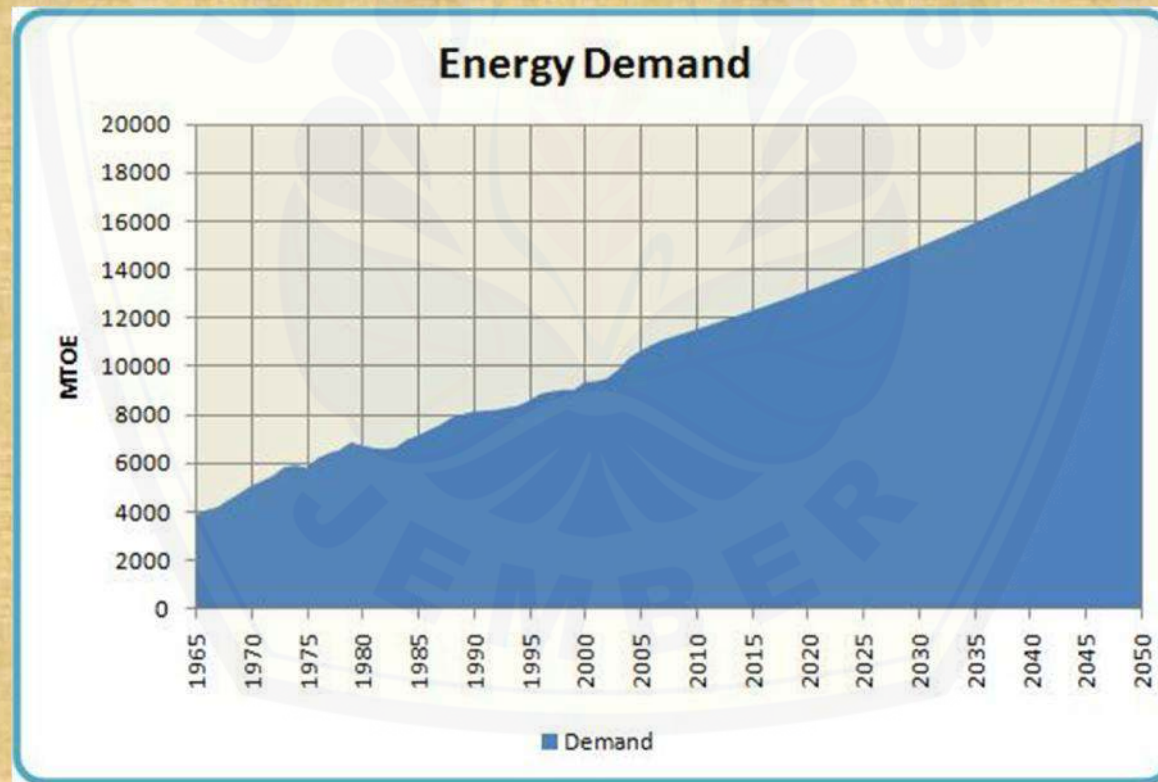
**Chemical Engineering Department, University of Jember, Indonesia**

University of Riau, Riau

29 – 30 November 2017

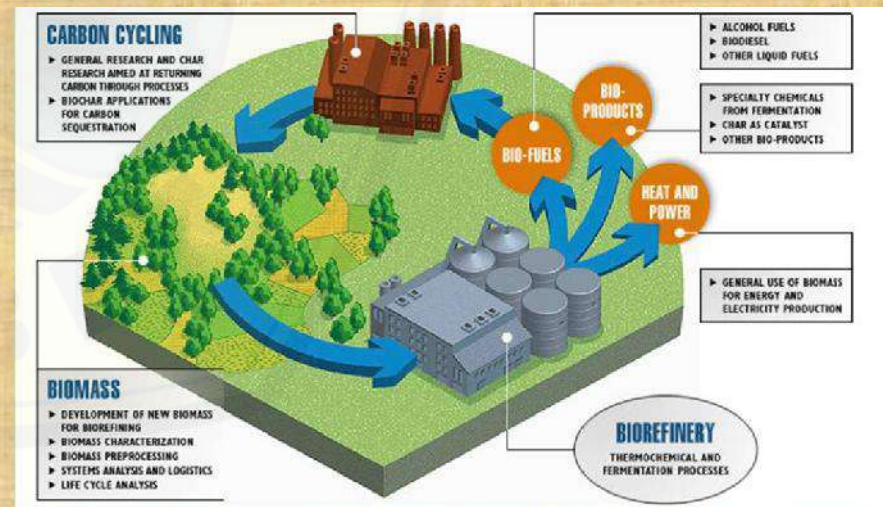
# Introduction

- The energy demand for the world development relies on the existence of mineral resources



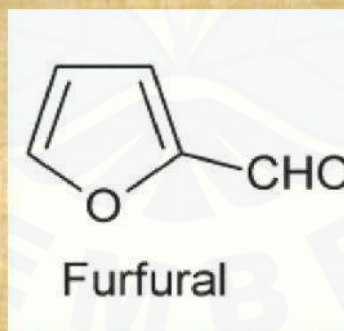
# Biorefinery

- Biorefinery technology gives the suitable alternative through biomass utilization, an organic chemical which is abundant on the ground
- The utilization of biomass as renewable energy has a potency to become the fuel supplier and intermediate chemicals
- The challenge is to develop the cost-effective method to convert biomass into valuable chemicals



# Furfural

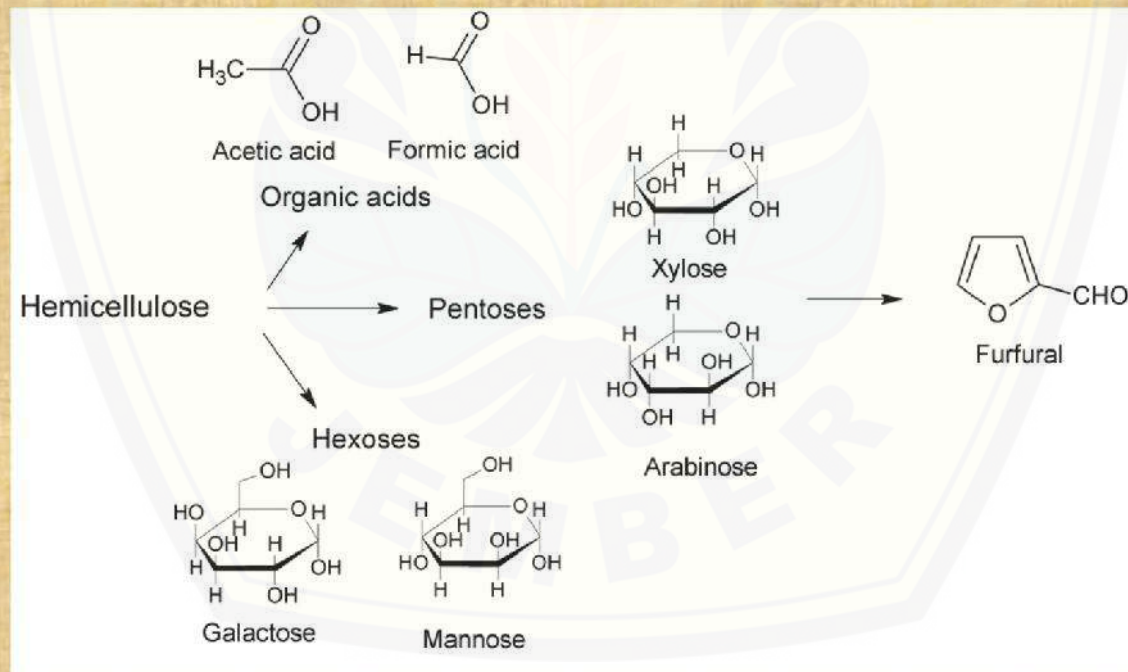
- Obtained from biomass degradation
- Key of the derivative of important chemical substances
- Can be implemented widely in oil refinery, plastic, pharmacy, and agrochemical industry.





# Furfural (cont'd)

- Has unique production mechanism
- Can be degraded and formed in the same condition

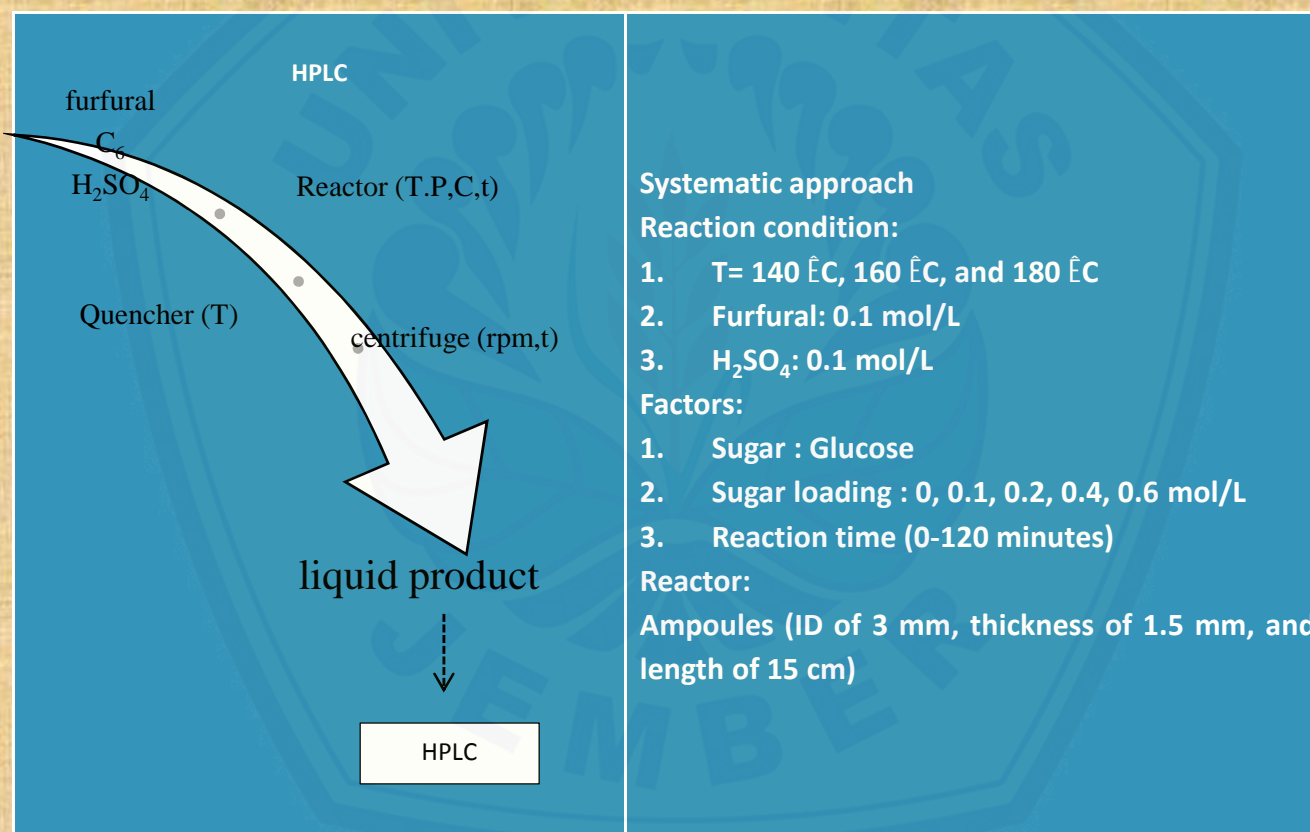


# Aim of research

Investigate several factors which could affect the furfural production and its decomposition

1. Glucose concentration
2. Reactor temperature
3. Reaction time

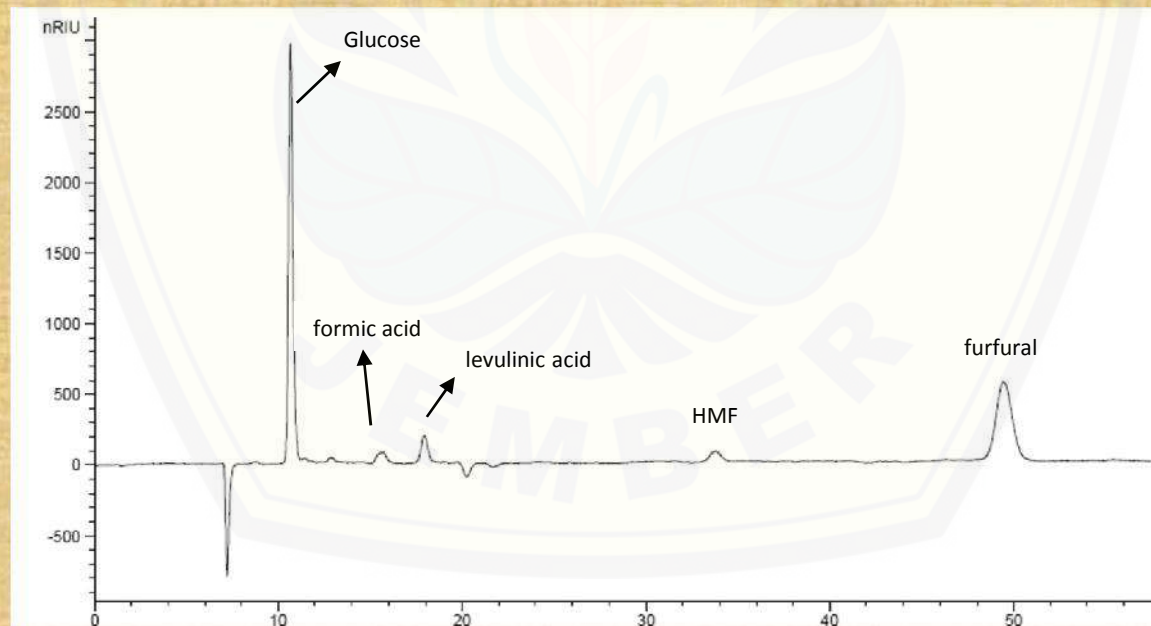
# Method scheme



# Results and discussions

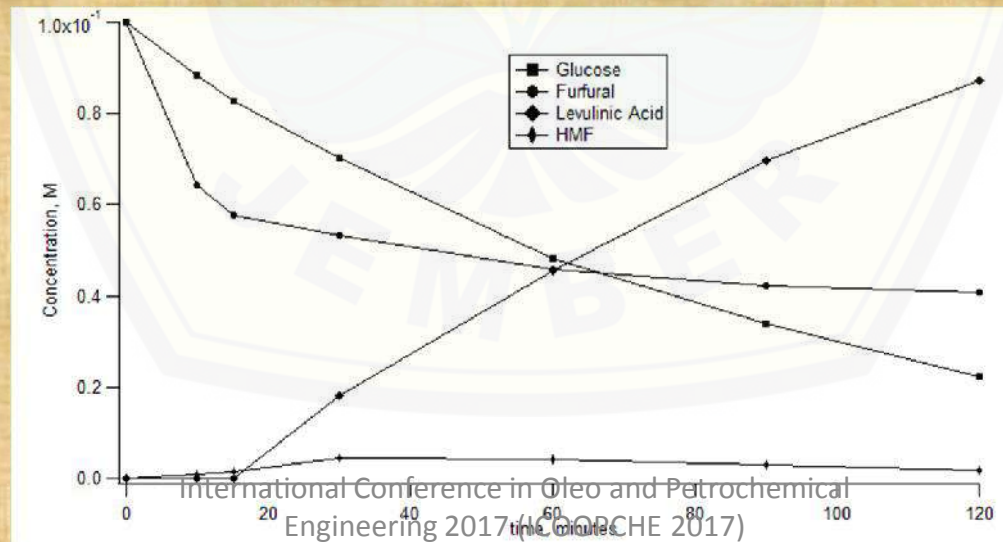
# Separation result

- Glucose and furfural are still observed and several new products are detected in the liquid phase (HMF, levulinic acid (LA), and formic acid)
- Some solid by-products known as humins were also formed



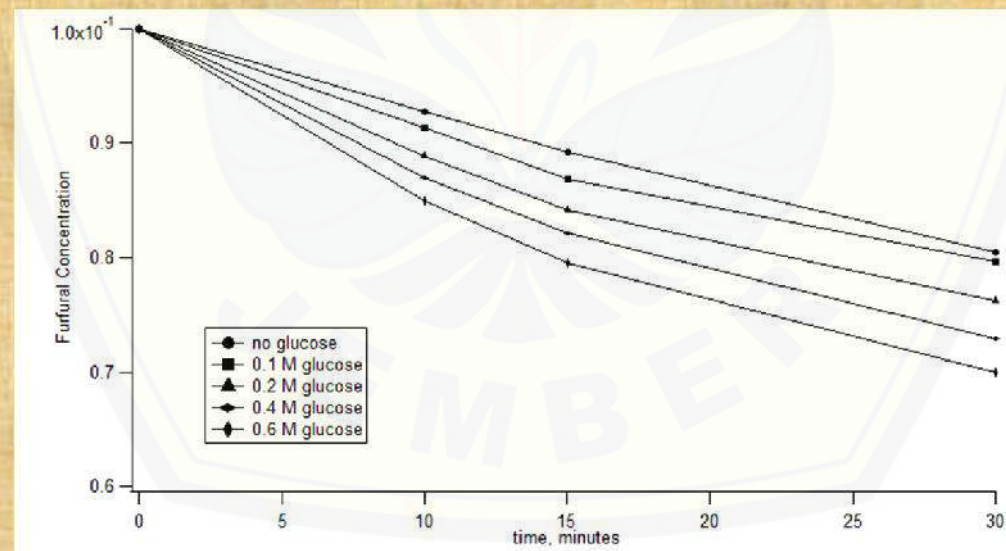
# Separation result (cont'd)

- The concentration of glucose and furfural decreases over time
- The concentration of LA increases dramatically over time
- The production of LA takes 15 minutes of reaction due to the formation of HMF as an intermediate product before.
- The concentration of HMF is remained constant



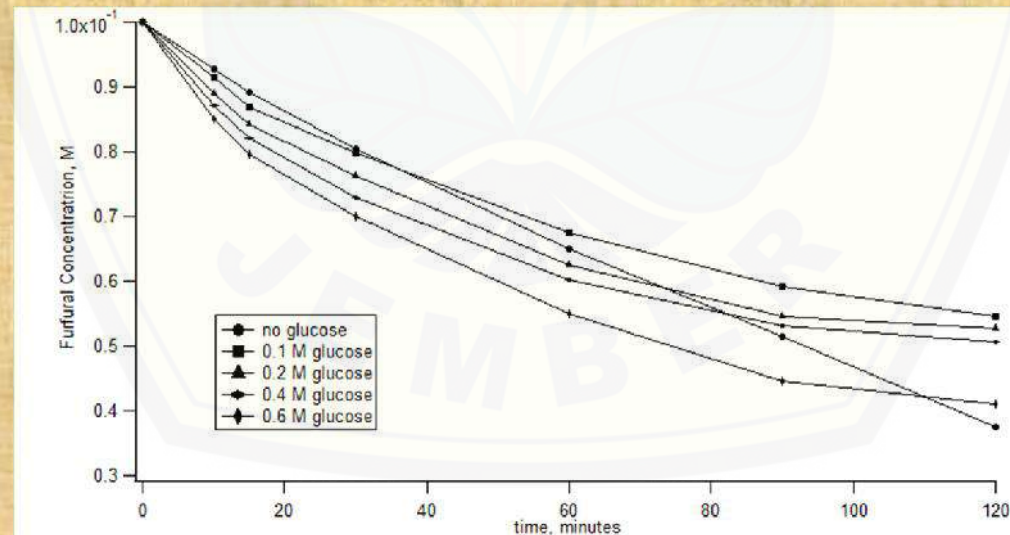
# Effect of glucose loading

- In reaction time between 0-30 minutes, as glucose concentration and reaction time increase, furfural concentration decreases.
- This phenomenon indicates that the reaction produces less humin.



# Effect of glucose loading (cont'd)

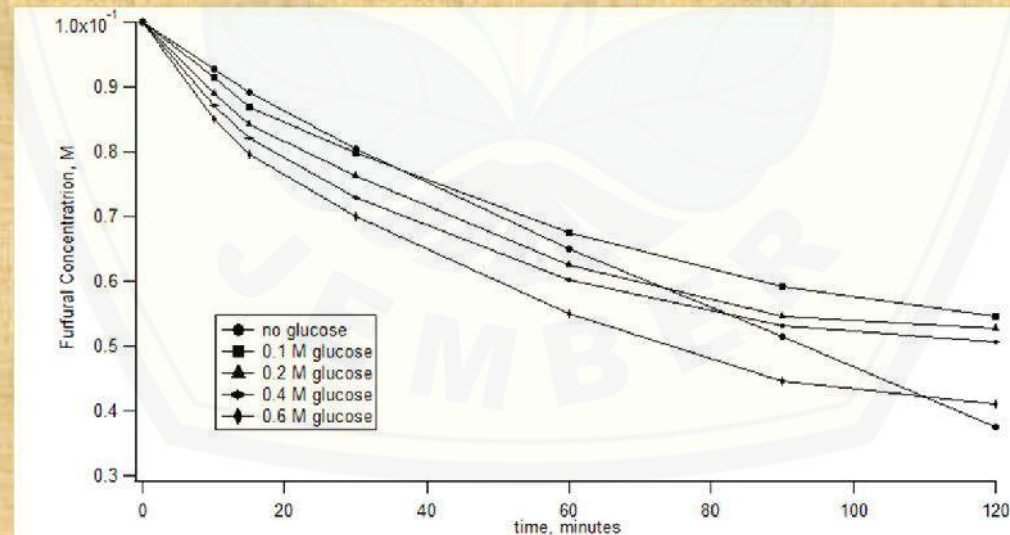
- After 30 minutes, the decomposition reaction with presence of 0.1 M glucose is slower compared to blank (no glucose)
- After 90 minutes, the decomposition reaction with presence of 0.1 M, 0.2 M, and 0.4 M glucose are slower compared to blank (no glucose)





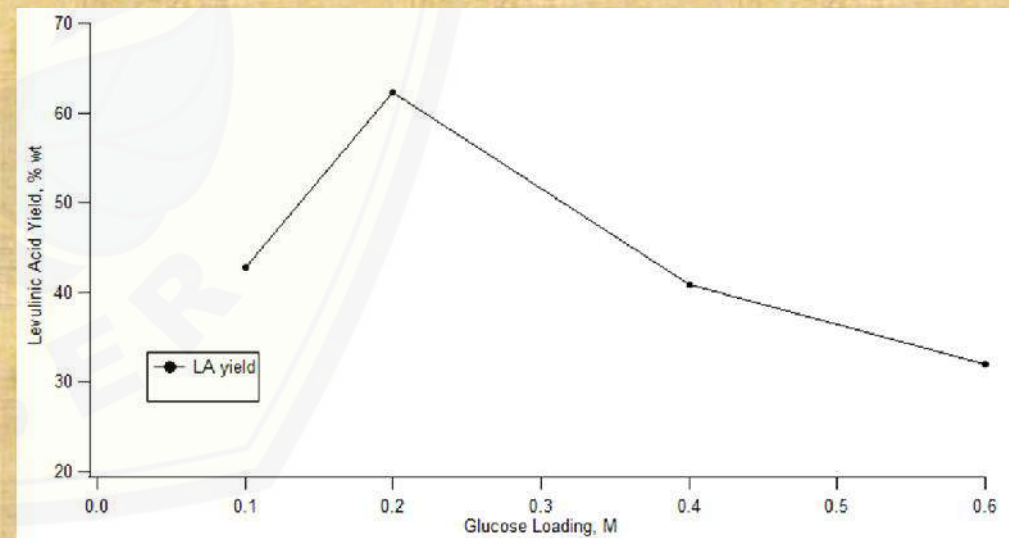
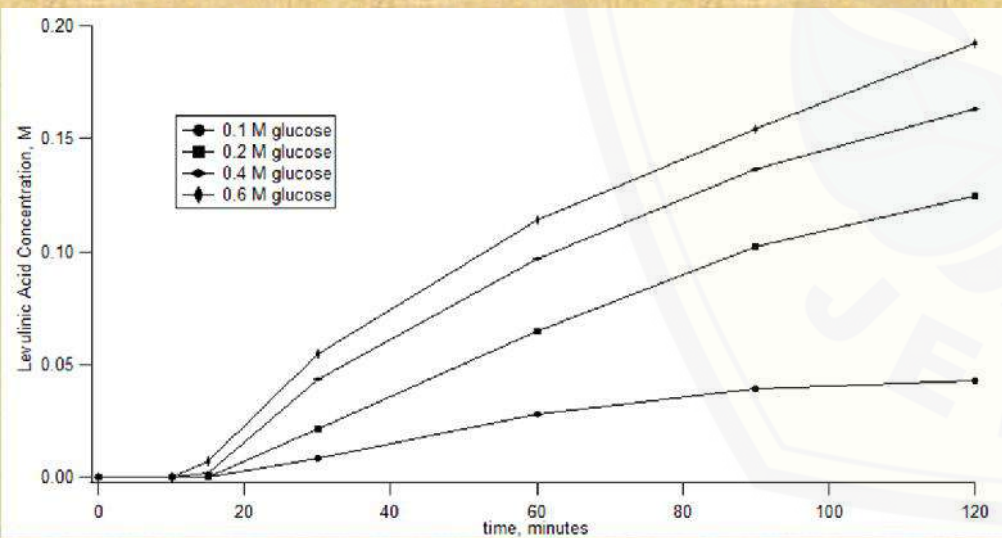
# Effect of glucose loading (cont'd)

- After 120 minutes, all decomposition reactions with the presence of glucose are slower compared to blank (no glucose)
- This behavior occurs because more humin were produced



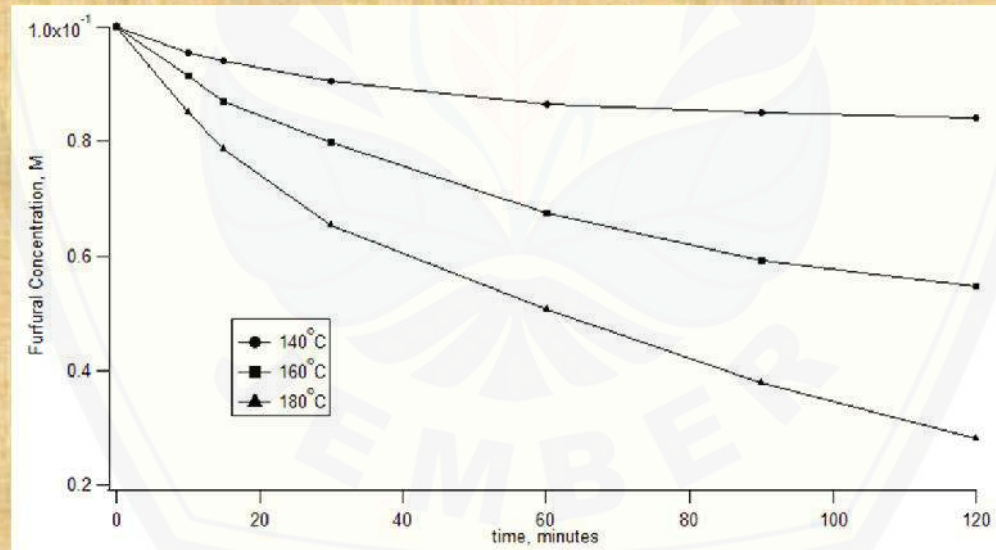
# Effect of glucose loading (cont'd)

- High glucose concentration leads to enhanced LA concentration which is produced after 15 minutes reaction.
- The highest experimental LA yield within the experimental window is 62 wt%, obtained at a glucose concentration of 0.2 M.



# Effect of reactor temperature

- The decomposition process occurs more rapidly when the reactor temperature increases which due to the higher value of reaction rate constant ( $k$ ) at elevated temperature



# Effect of reactor temperature (cont'd)

- In an acidic aqueous solution (e.g. H<sub>2</sub>SO<sub>4</sub>), the following kinetic equation of furfural decomposition was written as a pseudo-first-order reaction

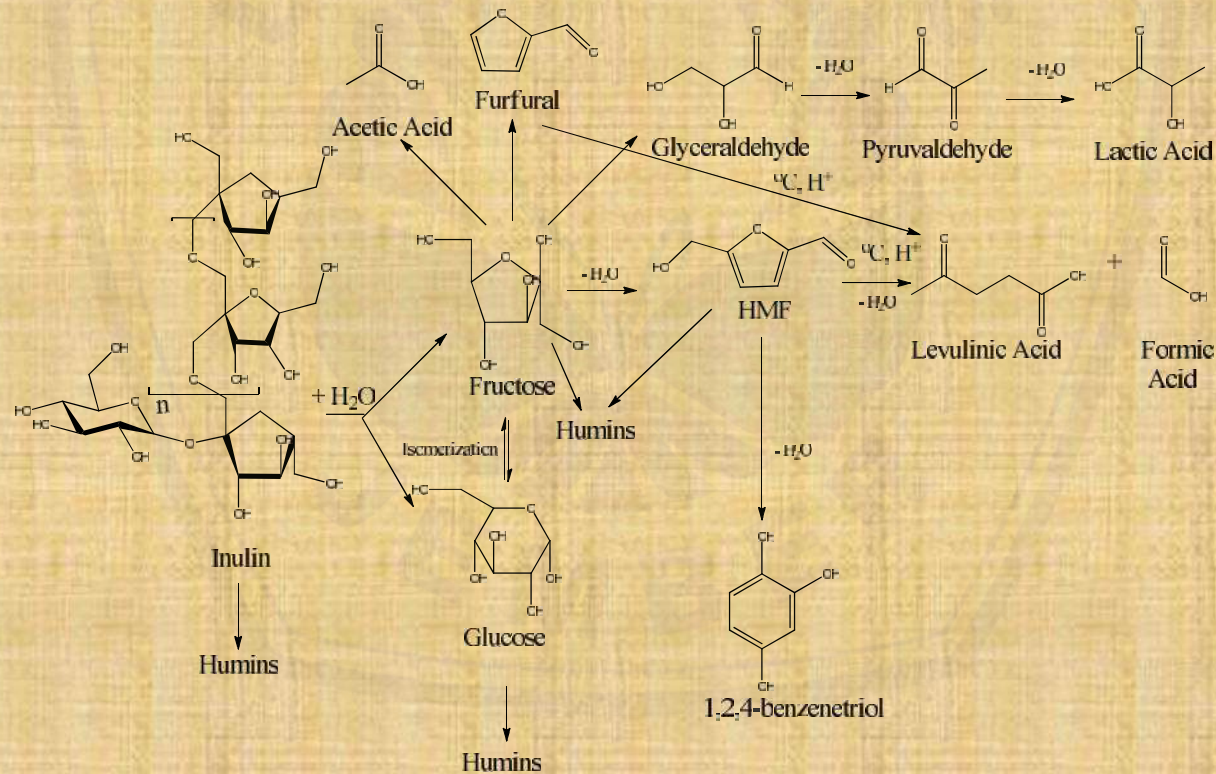
$$\frac{d[\textit{furfural}]}{dt} = -k[\textit{furfural}]$$

- The following Arrhenius-type equation correlated the experimental results over the temperature range 150-210 °C

$$k = (1.396 \times 10^7)e^{-83.6/RT}$$

# Proposed furfural degradation mechanism

The furfural degradation took a big part in the solution because of the furfural concentration was decreasing for the entire time of the reaction.



# Conclusions

- The presence of Glucose accelerates furfural decomposition in the solution for an early time of reaction (0 – 30 minutes)
- After the reaction time is increasing, it becomes slower.
- The temperature also accelerates furfural decomposition aligned with Arrhenius equation.
- The longer of reaction time is, the slower the decomposition reaction occurs.

# Thank you

# Certificate

of Appreciation

No : 036/STRF/ICOOPCHE/XI/2017

This Certificate is hereby presented to :

**ISTIQQOMAH RAHMAWATI**

as

**Presenter**

at

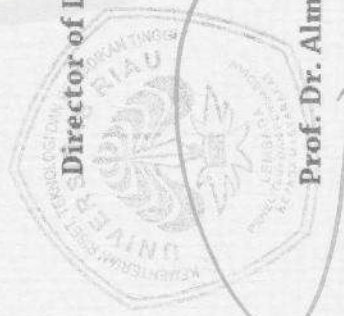
The 2<sup>nd</sup> International Conference on Oleo and Petrochemical Engineering (ICOOPCHE) 2017

“Chemical Engineering for Sustainable Development”

Hotel Pangeran, Pekanbaru-Indonesia, 30 November 2017

Director of LPPM Universitas Riau

Chairman of ICOOPCHE 2017



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