No   : 041/ ICOOPChE 2nd-2017/JTK-UR/XI/2017

Subject: Letter of Acceptance

LETTER OF ACCEPTANCE

Dear Mr. Felix A Setiawan,

We are pleased to notify that your article which entitled **"The role of xylose in 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 **14th 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 30rd , 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

REVIEWER COMMENTS

Title of Manuscript:	The role of xylose in Furfural Decomposition
Reviewer Recommendation Term (Accepted Without Revision/Minor Revision/Mayor Revision/Rejected):	Accepted with major revision
Reviewer Comments to Authors	Title have a little bit revision. Abstarct structure should followed a backgrund, method and result. No need to have an explanation of the result in abstract. Conclusion should be in passive and no need to refer to any figure. English should be improved. It seem a quite similar research method of this paper and the other paper (title: Glucose, temperature and time reaction effects: Quantitative study of furfural decomposition)

Experimental Study on Role of Xylose on Furfural Decomposition in Acidic Composition

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Abstract: Sulfuric acid (H_2SO_4) is broadly used as a catalyst to accelerate hemicellulose and a considerable part of the lignin components decomposition to generate sugars as intermediate chemicals for further processing steps. Mainly, lignocellulose biomass such as hardwoods, agricultural waste, and grasses contains xylose as major constituent. Thus, the presence of xylose in decomposition of lignocellulose biomass is obvious. Meanwhile, the furfural production originates mainly from the hydrolysis of hemicellulose into monomeric sugars and the dehydration of sugars into furfural. Shortly, this works is to investigate the role of xylose in furfural decomposition. To gain insight into role of xylose in furfural decomposition, xylose and furfural were added into ampoules containing sulfuric acid. The investigated parameters were xylose concentration, T and t. During the reaction, the parameters were kept constant. The product was filtered, then, qualitatively and quantitatively analyzed by HPLC. The result shows that the presence of Xylose in sulfuric acid solution as a catalyst contributes the competition of decomposition mechanism. The xylose at the early time of the reaction, 15 minutes, needed time to initiate the reaction, so the furfural was exposed with the decomposition. Xylose would be mostly degraded into furfural and glyceraldehyde within 15 minutes of the reaction. This finding is obviously described in chromate profile which identified xylose, furfural, glyceraldehyde, and formic acid. The other component formed during the reaction was humins (insoluble polymer).
Keywords: Cellulose; Xylose; Furfural; Sulfuric acid; decomposition mechanism

1. Introduction

The substitution of fossil resources with renewable energy regarding environmental awareness has boosted the research to enhance suitable biomass-based production for particular chemicals [18]. Renewable biomass resources potentially could be converted to a sustainable supply of fuels and intermediate chemicals [14]. Lignocelluloses biomass, such as wood or agricultural waste, like wheat straw and corn Stover, is a widely available resource for chemical production. It contains cellulose, hemicellulose and lignin, and other minor components, like extractives and ash. Those substances become a great potential for largest feedstock to become value-added chemicals, such as monosaccharides, via acid hydrolysis process [27]. Acids, such as sulfuric acid, hydrochloric acid, etc., hydrolyze hemicellulose to produce sugars, which are glucose, galactose, hexoses, mannose, pentose, xylose, and arabinose. Xylose and arabinose are constituent materials of hardwoods, agricultural waste, and grasses (Table 1) and should be taken into account to make cost-efficient utilization for further biomass processing [1].

Acid-catalyzed hydrolysis has been reported as early as 19th-century, with commercial applications started at the beginning of 20th century [5, 6, 7, 17]. The use of sulfuric acid (H_2SO_4) as a catalyst is broadly used to hemicellulose and a considerable part of the lignin components decomposition to generate sugars as intermediate chemicals for further processing steps. Dilute sulfuric acid (H_2SO_4) processes with the concentration 0.5-1.5% and temperature 121-160°C becomes the most favored for industrial application because it will get feasible high sugar yields converted from hemicellulose [7, 13]. The use of sulfuric acid (H_2SO_4) as a catalyst has its own advantages and disadvantages. The advantages are the high catalytic activity, less energy requirement, and simple to be implemented, while the disadvantages are many undesired by-products, difficulty in acid recovery and recycling, corrosion in pipe and equipment, and pollution [5, 6].

Table 1. Percent dry weight of several biomass residue resources [24]

Sources	cellulose	hemicellulose	Lignin
Corn Stover	37.5	22.4	17.6
Corn fiber	14.28	16.8	8.4
Pinewood	46.4	8.8	29.4
Wheat straw	38.2	21.2	23.4
Switch grass	31.0	20.4	17.6

Note: Because minor components are not listed, these numbers do not sum to 100%.

One of the disadvantages of homogenous catalyst is undesired by-products when real biomass is used, a wide product spectrum is identified including compounds originating from both sugars and lignin [3] which complicate further processing such as recovery and separation. It is caused by the homogenous catalyst cannot control the mechanism of the reaction occurred in the solution and the compounds must have some interactions with furfural, sugars, or catalysts affecting furfural yields. Furfural is a chemical that is produced from pentoses, the 5-carbon sugars available in biomass hemicellulose. The presence of sugars derived from biomass is also took the responsibility of furfural yield and furfural decomposition. Moreover, the use of real biomasses or industrial streams would have resulted in complex reaction media, where the interactions between components would make the observation of the phenomena more difficult. However, an understanding of the reaction pathways of furfural degradation in sulfuric acid (H_2SO_4) solution in the presence of C5 sugar is a crucial issue to develop further efficient process. The research is to evaluate the role of xylose addition in furfural decomposition mechanism in sulfuric acid (H_2SO_4) solution as a catalyst. The study made an artificial solution with the addition of C5 (Xylose) and furfural in sulfuric acid (H_2SO_4) solution with the variation of sugar loading and temperature of the reaction.

2. Literature Review

Furfural is a chemical substance that is merely produced from biomass. As shown in Figure 2, the furfural production originates mainly from the hydrolysis of hemicellulose into monomeric sugars and the dehydration of sugars into furfural. The composition of some raw materials which are suitable for furfural production is given in Table 1. At the similar condition, Furfural can be both degraded and formed. Resinours tars and formic acid have been recognized as the degradation product [1, 25, 29]. The degradation could be decomposition or polymerization reaction or the combination of decomposition and polymerization. Formic acid could be catalyzed the reaction [16] but it has been argued by some researchers, [23] however, in the majority of the studies, few quantities of formic acid have been measured [1, 29, 30].

The several kinetic studies performed by [8–11] for the hexoses hydrolysis and dehydration can be a fit example for the kinetic cascade approach. Pure furfural has been previously determined with consideration of kinetics parameter in dilute sulfuric acid (H_2SO_4) solution [23, 25, and 29]. The previous studies about the mechanism of furfural production or degradation have been conducted by several researchers. The reason why the mechanism of furfural production and degradation are not so popular is mostly caused by furfural as an inhibitor substance for fermentation process (ethanol production) and as an intermediate substance to produce more beneficial substances [15]. Furthermore, A deeper study on the mechanism of furfural degradation with the presence of xylose is a mandatory to acquire a plan for further processing steps with economical consideration.
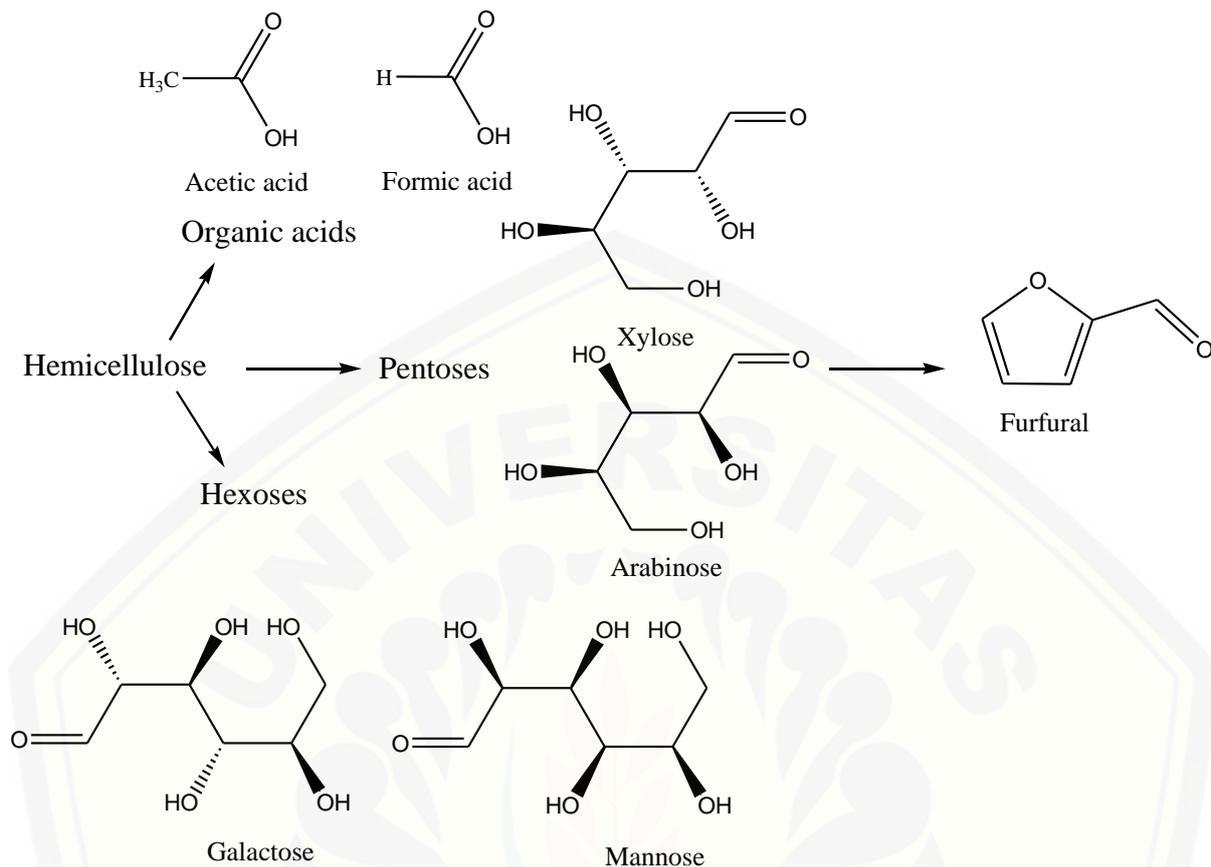
Figure 2. Furfural formation from hemicellulose [16]

Several studies had proposed different processing mechanism with the variation of reaction conditions. The first reaction was convinced to start from the acyclic form the pentose, either via a 1, 2-enediol intermediate 2 and subsequent dehydration (see Fig. 3a) [21, 26] or directly via a 2, 3-(α, β-) unsaturated aldehyde 4, see Fig. 3b [2, 28]. Latterly, the researchers convinced that the reaction to take place starting from the pyranose to form the pentose, by the action of H^+ on the O-2 of the pyranose ring, leading to the 2,5-anhydroxylose furanose intermediate 7 which is subsequently dehydrated to furfural, see Fig. 3c [4, 19, 20, 31].

A homogeneous catalyst, such as sulfuric acid and hydrochloric acid, was widely used during the furfural production. Based on [32], It compared some different homogeneous acid catalysts on furfural formation from xylose and xylan. The best catalyst resulting in the highest furfural yields from xylose and xylan was hydrochloric acid, 37.5%, and 34.3%, respectively. Sulfuric acid (H_2SO_4) was the second best of the studied acids as a catalyst. Another study conducted by [16], it had similar results with previous study, where better yield and selectivity was gained by HCl compared with H_2SO_4 when the conditions remained the same.

In this research, the mechanism of furfural decomposition and production was observed during the addition of xylose in sulfuric acid (H_2SO_4). An artificial solution was made in this research to make a limitation which is only pure xylose and pure furfural in sulfuric acid (H_2SO_4) solution. The limitation was made to minimize the complex reaction occurred during the time. After the limitation was implemented, the role of xylose addition in furfural decomposition mechanism could be studied extensively. If the real biomass was implemented in the study, the researcher would have difficulty to describe the mechanism of furfural decomposition in the presence of C5 (xylose) because there were many substances in the solution including glucose, galactose, hexoses, pentose, xylose, arabinose, etc.
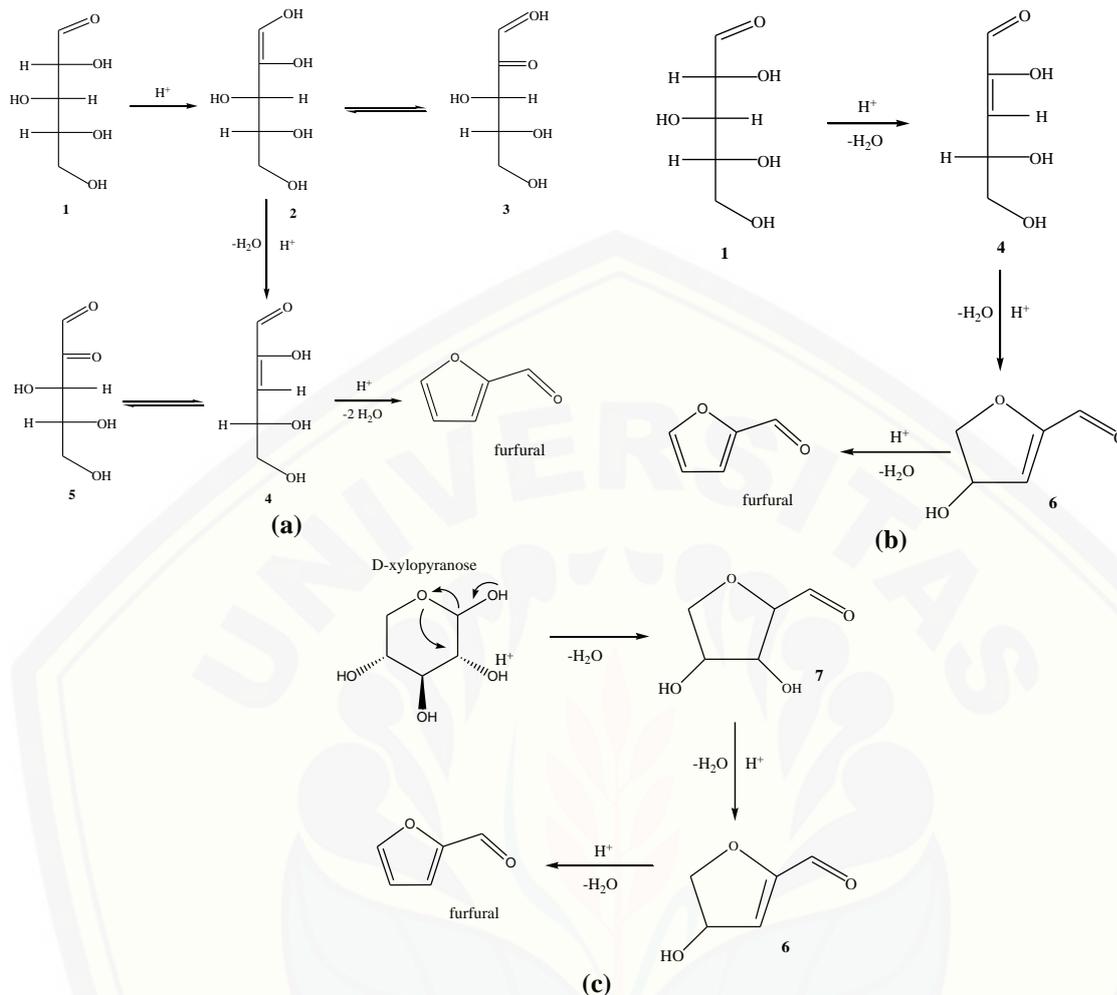
Figure 3. Xylose dehydration mechanism (a) via enolization [21, 26]; (b) via β-elimination [2, 28]; and via cyclic intermediates [4, 19, 20, 31]

3. Material & Methodology

3.1. Chemicals

Furfural (≥99%) and Levulinic acid (≥ 97%) were obtained from Acros Organic (Geel, Belgium). Sulfuric acid (96-98 %wt) and Formic acid (≥ 95%) were purchased from Merck KGaA (Darmstadt, Germany). D-Xylose (≥ 99%) and (5-Hydroxymethylfurfural (HMF) (≥ 99%) were 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 [5]. In a typical experiment, the pre-determined amount of sample (mixture of xylose and furfural) 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 [6]. 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 ml.min^{-1}. The column was operated at 60 °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.

4. Results and Discussion

The variable used in the research were xylose concentration (0, 0.1, 0.2, 0.4, and 0.6 mol/L) and temperature (140,160, and 180°C). Zero xylose concentration was used to be the base line of comparison with the others. The chromatogram profile after the reaction occurred at 60 minutes could be seen in Fig. 4. The detected substances were xylose, glyceraldehyde, formic acid, and furfural. This information could be used to propose mechanism (reaction pathways). The insoluble polymer, called humins, were produced during reaction, which is insoluble by-products, in all experiments, though the amount was more pronounced at prolonged reaction times. Some substances were the reactants, xylose and furfural. The furfural degradation mechanism, which results glyceraldehyde and formic acid, should be proposed to describe the entire reaction mechanism. The chromatogram is smooth with a few noise, the noise indicates several substances which is not known. The others data are needed to describe the mechanism of the degradation pathway such as the profile of Xylose and Furfural versus time.
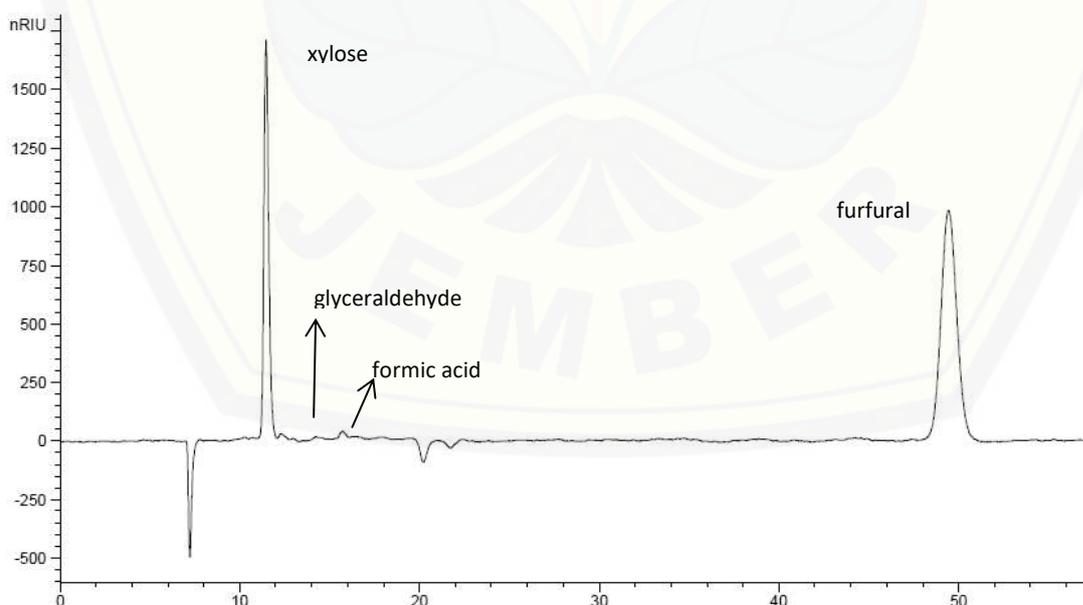
Figure 4. The chromatogram profile at reaction time 60 minutes

The presence of formic acid makes the catalytic process of the furfural and xylose decomposition become more complex. It was proved with the research conducted by [16] which varied furfural,

xylose, and formic acid concentration resulting that formic acid could catalyze the decomposition of furfural and xylose. Formic acid was formed due to furfural decomposition from Fig. 4 and it was supported by the mechanism described by [15], Fig. 5. Furfural itself is an intermediate product, thus it is sensitive to acidic compounds (which refers to acid catalyzed cleavage of the furan structure) and auto-oxidation [1] leading to decomposition of formic acid and to polymeric products [23]. The presence of glyceraldehyde was largely derived from xylose decomposition.
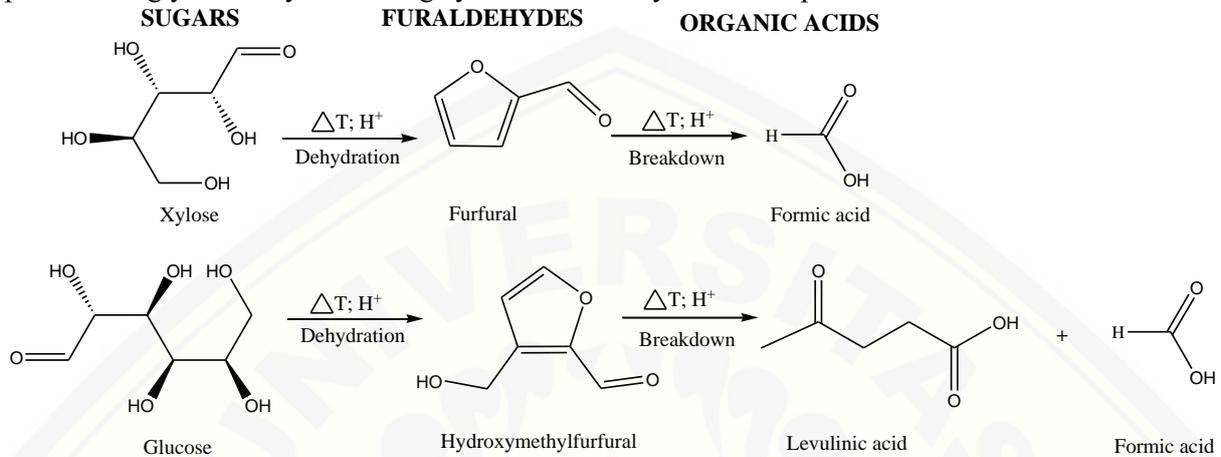
Figure. 5 Degradation mechanism of xylose and glucose [15]

In Figure 6, it could be seen that the xylose concentration was depleted rapidly until 30 minutes, from 0.1 M to 0.0481 M. The xylose concentration decreased slower from 30 – 120 minutes, from 0.0481 M to 0.0085 M. Reversely for the situation of furfural concentration, the depletion of furfural concentration only took place until 15 minutes, from 0.1 M to 0.0759 M. After 15 minutes, the concentration was increasing slowly within time 15 – 90 minutes, from 0.0759 M to 0.1021 M. The concentration of furfural was decreasing slowly within time 90 – 120 minutes, from 0.1021 M to 0.0935 M. It can be concluded that at early reaction time, 0 – 15 minutes, both of furfural and xylose were exposed by decomposition. Xylose had been degraded into furfural and other substances at 15 – 120 minutes, meanwhile, furfural could be degraded but the higher rate of furfural addition from xylose decomposition made the furfural decomposition being shadowed.
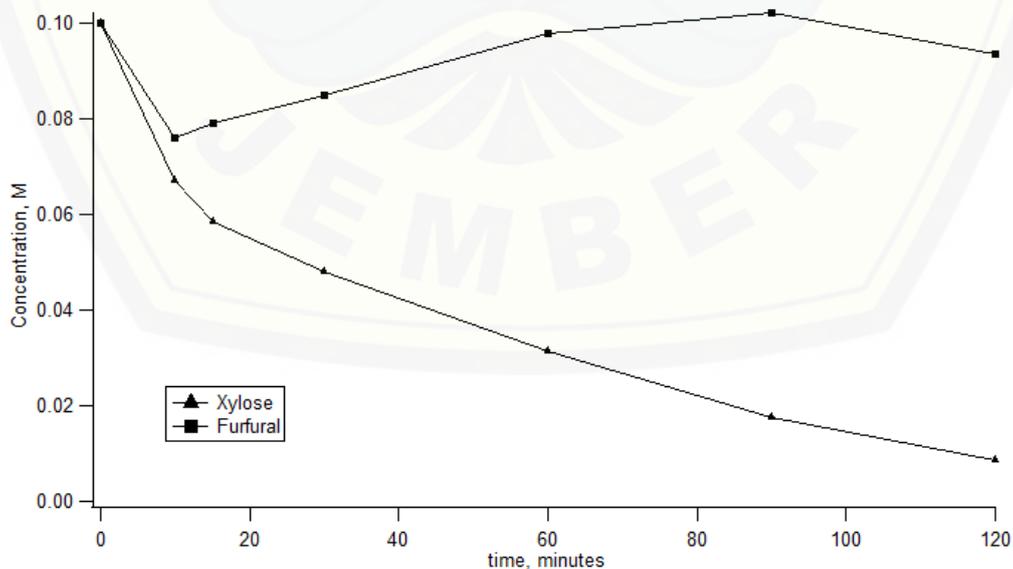
Figure 6. The furfural and xylose concentration profile within time at 160 °C
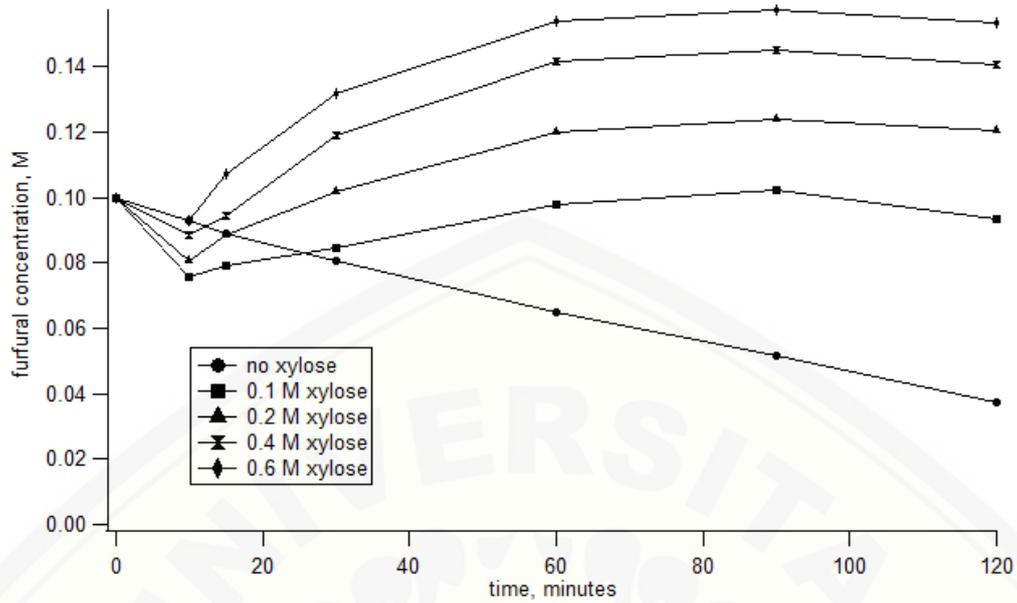
Figure 7. The furfural concentration profile within time at 160 °C in the several concentration of xylose

The presence of xylose varied from 0.1 M to 0.6 M was to describe the role of xylose in furfural decomposition mechanism in sulfuric acid (H_2SO_4) solution as a catalyst, see Fig. 7. The presence of xylose inhibited the furfural decomposition with the increasing of xylose concentration in the solution. It proves that the xylose chain is more fragile to be breakdown rather than furfural structure. Interestingly, the decreasing of furfural concentration without the presence of xylose was merely similar to the presence of 0.6 M xylose concentration in 15 minutes in preliminary reaction, with the same gradient of the concentration within time. It shows that furfural was mostly degraded without any competition with other substances at those conditions. The graph shows the bigger the xylose initial concentration the slower the furfural degradation at the early of the reaction.
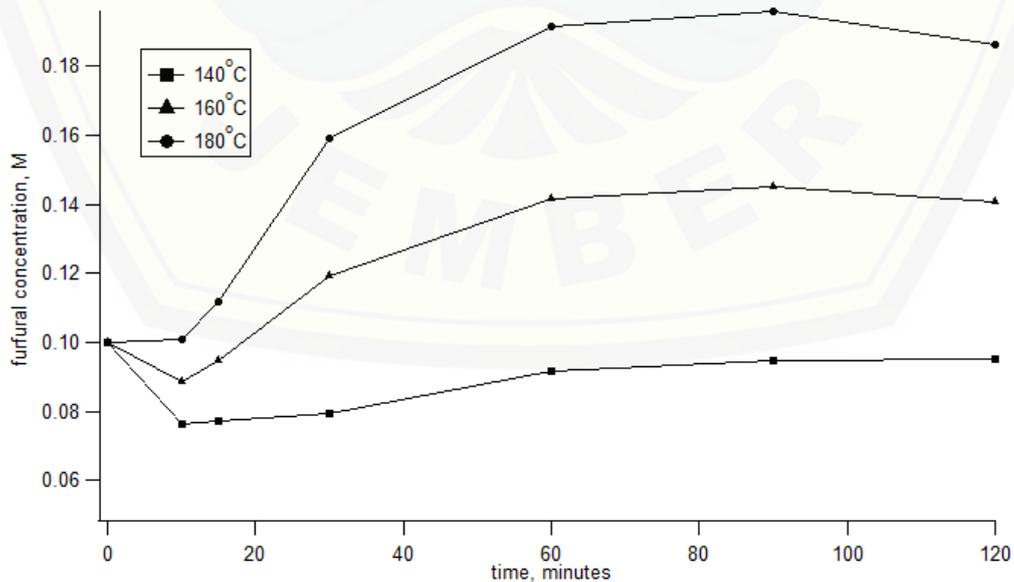
Figure 8. The furfural concentration profile within time at 0.4 M of xylose concentration in the vary of reaction temperature

The reaction rate of furfural decomposition in the presence of 0.4 M of xylose could be seen in Fig. 8 which shows a comparison of reaction rate in temperature difference. The similar pattern of the furfural concentration within time describes that furfural decomposition rate had the same effect regarding with temperature. The reaction mechanism with the presence of xylose proposed in this research is the decomposition of xylose into furfural and furfural into organic acid (formic acid) and humins.

After the description above, it could be drawn the mechanism of the role of xylose addition in furfural decomposition in sulfuric acid (H_2SO_4) as a catalyst. The mechanism occurred in the solution are caused by the degradation of xylose and furfural. The xylose degradation had a significant part in the solution because of the furfural concentration was increasing for after 15 minutes after the reaction took place. The mechanism pathway is drawn with consideration of other literature in this references and the data gained from the experiment. It could be seen in Fig. 9.
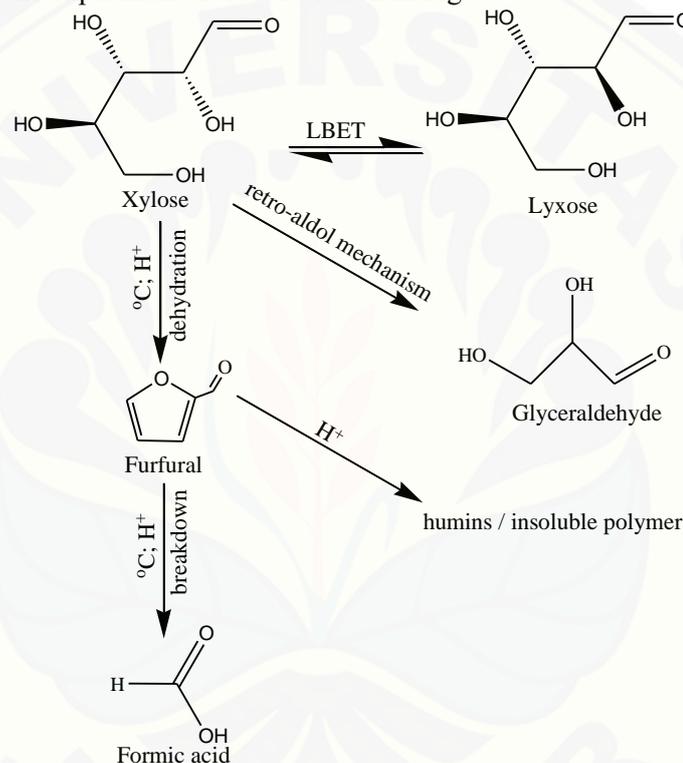
Figure 9. Plausible mechanism of furfural decomposition with the presence of Xylose in sulfuric acid solution as a catalyst; (LBET: Lobry de Bruyn–van Ekenstein transformation)

5. Conclusion

We can conclude from this study that the competition of decomposition mechanism had been contributed in the presence of xylose in sulfuric acid solution as a catalyst. Preliminary time of the reaction, around 15 minutes, was needed by xylose to initiate the reaction, otherwise the furfural was exposed with the decomposition reaction. The reaction time took place 15 minutes, the xylose would be mostly degraded into furfural and glyceraldehyde and few furfural, also, would be degraded into further constituents. The chromatogram data which contains xylose, furfural, glyceraldehyde, and formic acid at 60 minutes was a clear evidence of decomposition process. Humins (insoluble polymer) was also formed during the decomposition reaction. Furfural concentration pattern had not been distinct with the variation of temperature, which means that the reaction rates of both xylose and furfural decomposition have the same sensitively with the temperature.

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International Conference in Oleo and Petrochemical Engineering 2017 (ICOOPChE 2017)

Chemical Engineering
Research Team

Present in University of Riau
29 – 30 November 2017

INTRODUCTION

Energy demands

Source: BP Statistical energy review 2017
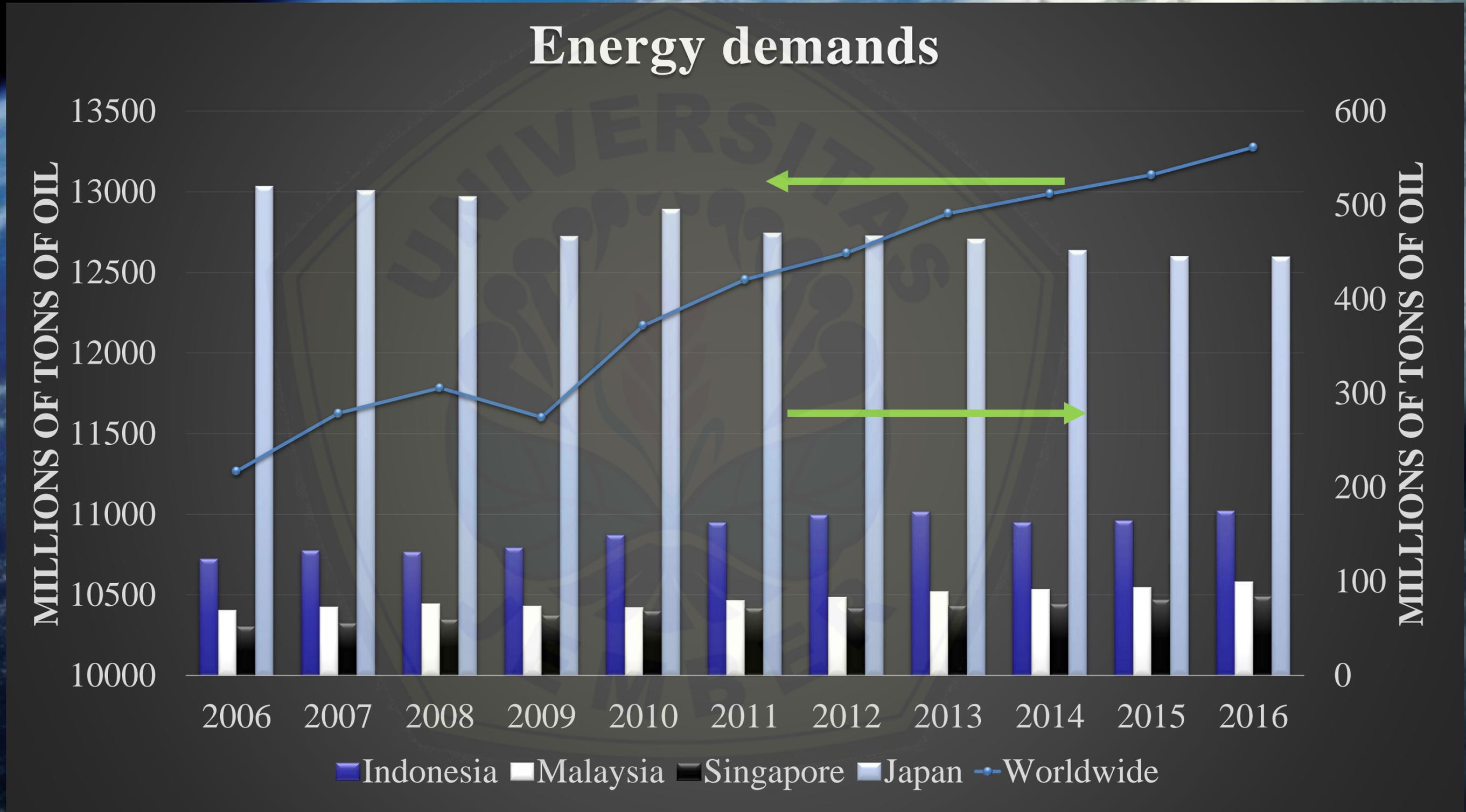
BIOREFINERY

- Bio-refinery 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
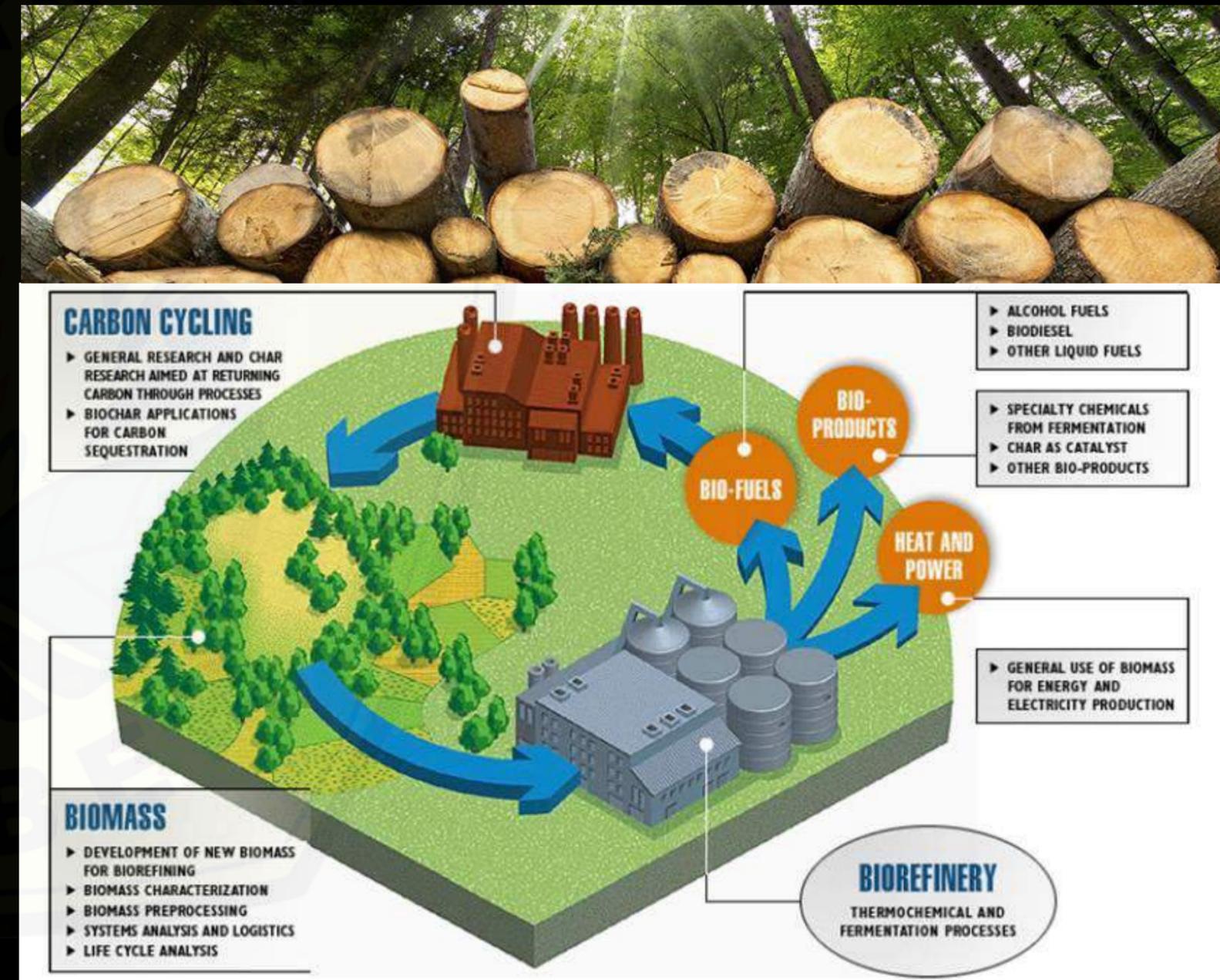
Furfural

- Obtained from biomass degradation

- Key of the derivative of important chemical substances

- Can be implemented widely in oil refinery, plastic, pharmacy, and agrochemical industry.

- Has unique production mechanism

- Can be degraded and formed in the same condition
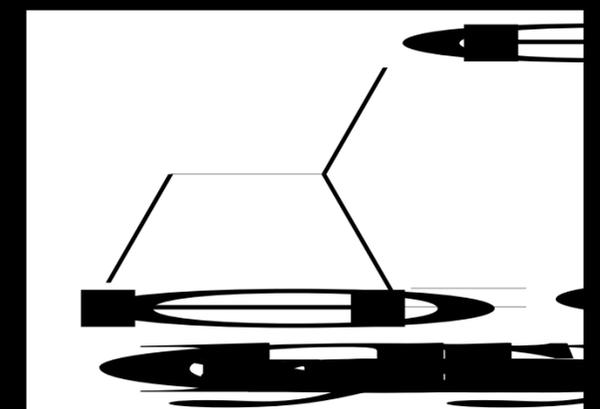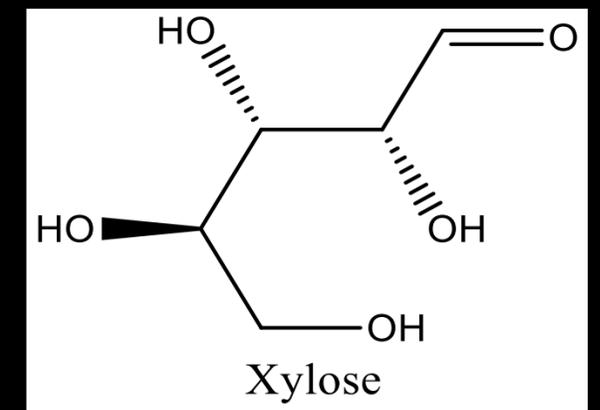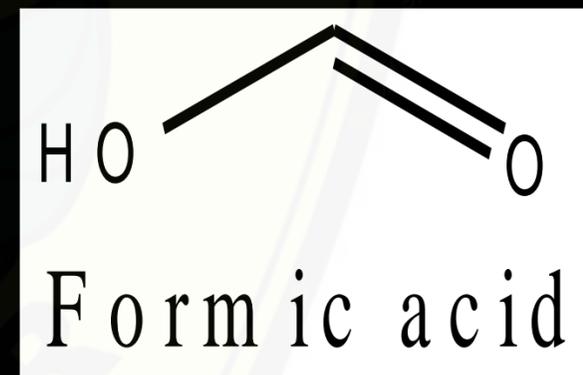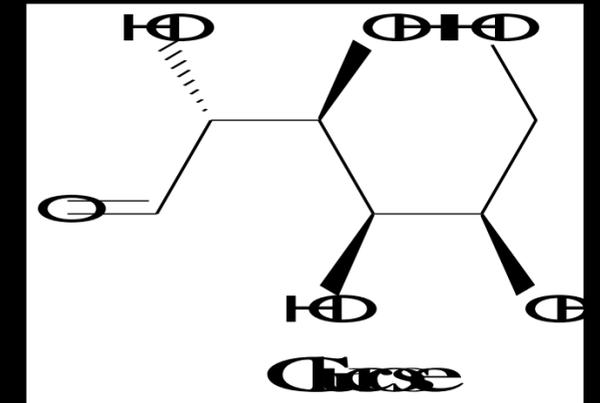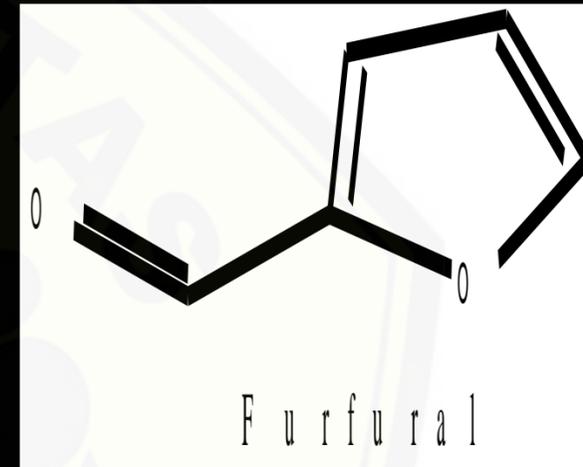
Research goals

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

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

Methodology

Systematic approach

Reaction condition:

- T= 140 °C-180 °C
- Furfural: 0.1 mol/L
- H_2SO_4: 0.1 mol/L

Factors:

- Sugar : Glucose / Xylose
- Sugar loading : 0.1-0.6 mol/L
- Reaction time (0-120 minutes)

Reactor:

- Ampoules (ID of 3 mm, thickness of 1.5 mm, and length of 15 cm)
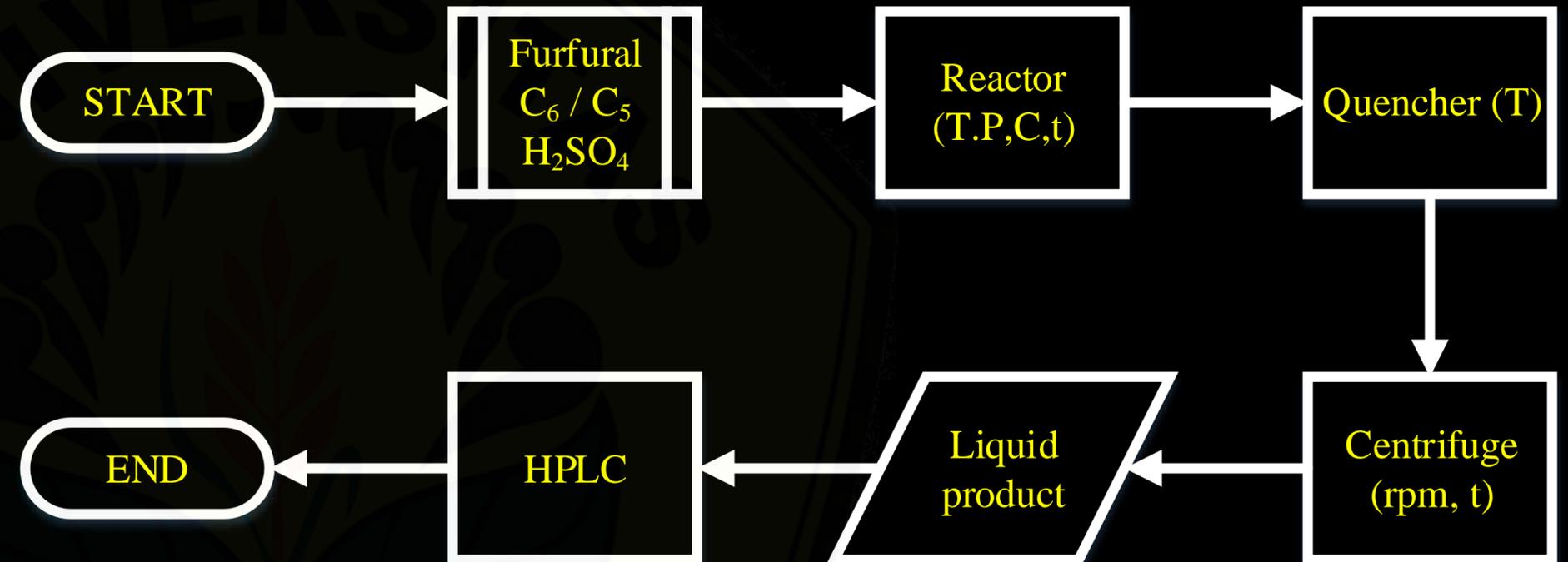
START → Furfural C_6 / C_5 H_2SO_4 → Reactor (T.P,C,t) → Quencher (T)

Centrifuge (rpm, t) → Liquid product → HPLC → END

Experimental Study on Role of Xylose on Furfural Decomposition in Acidic Composition

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Results and discussion
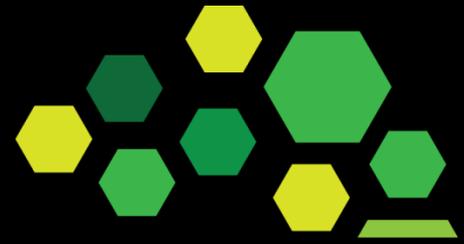
- There are several substances detected by HPLC

- Some solid by-products known as humins were also formed

8
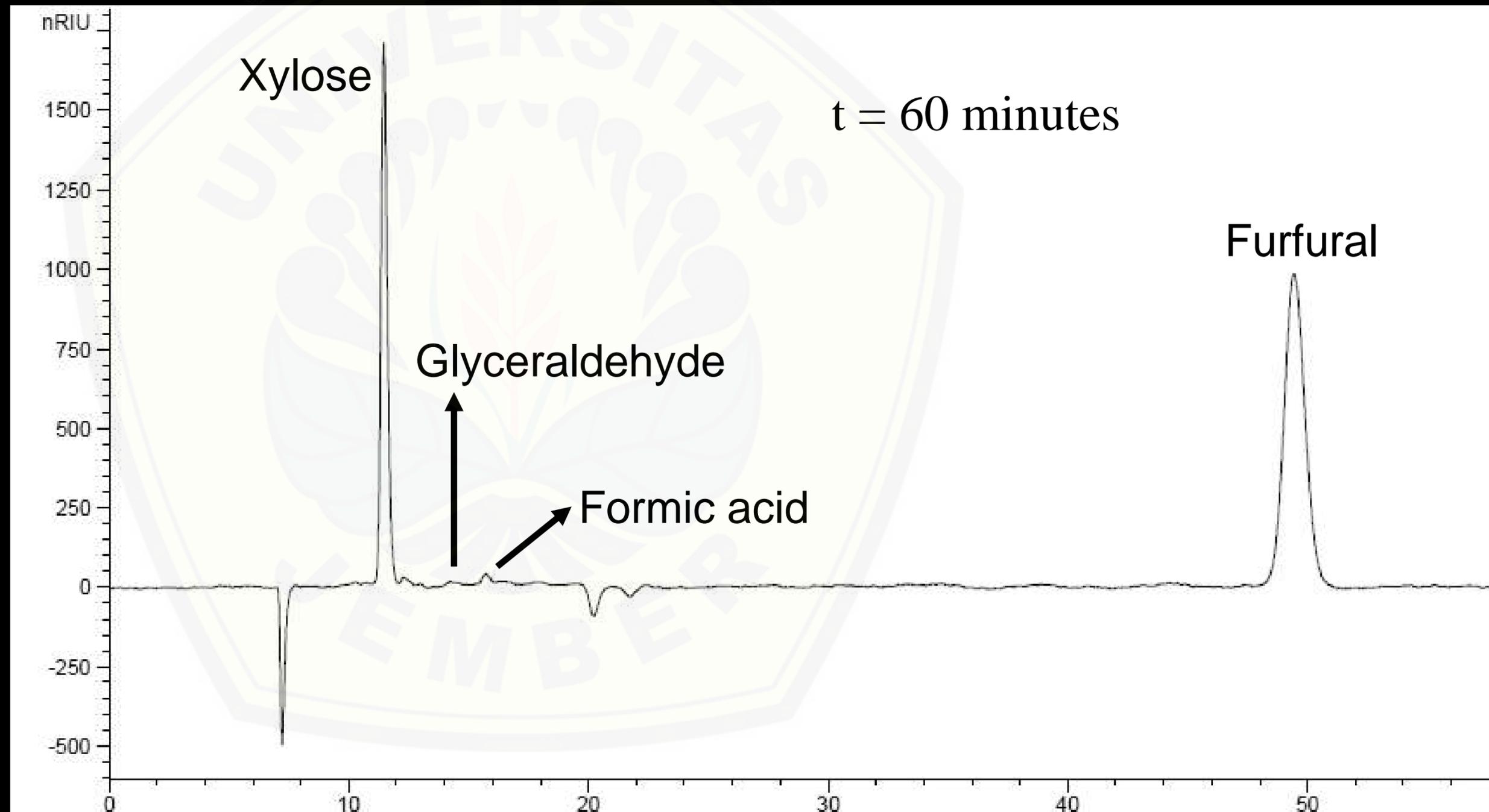
Results and discussion
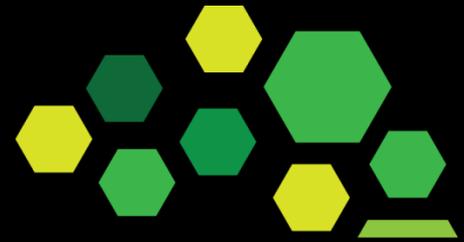
- Furfural concentration decreased over the time, but the xylose addition would produce some furfural to have further decomposition
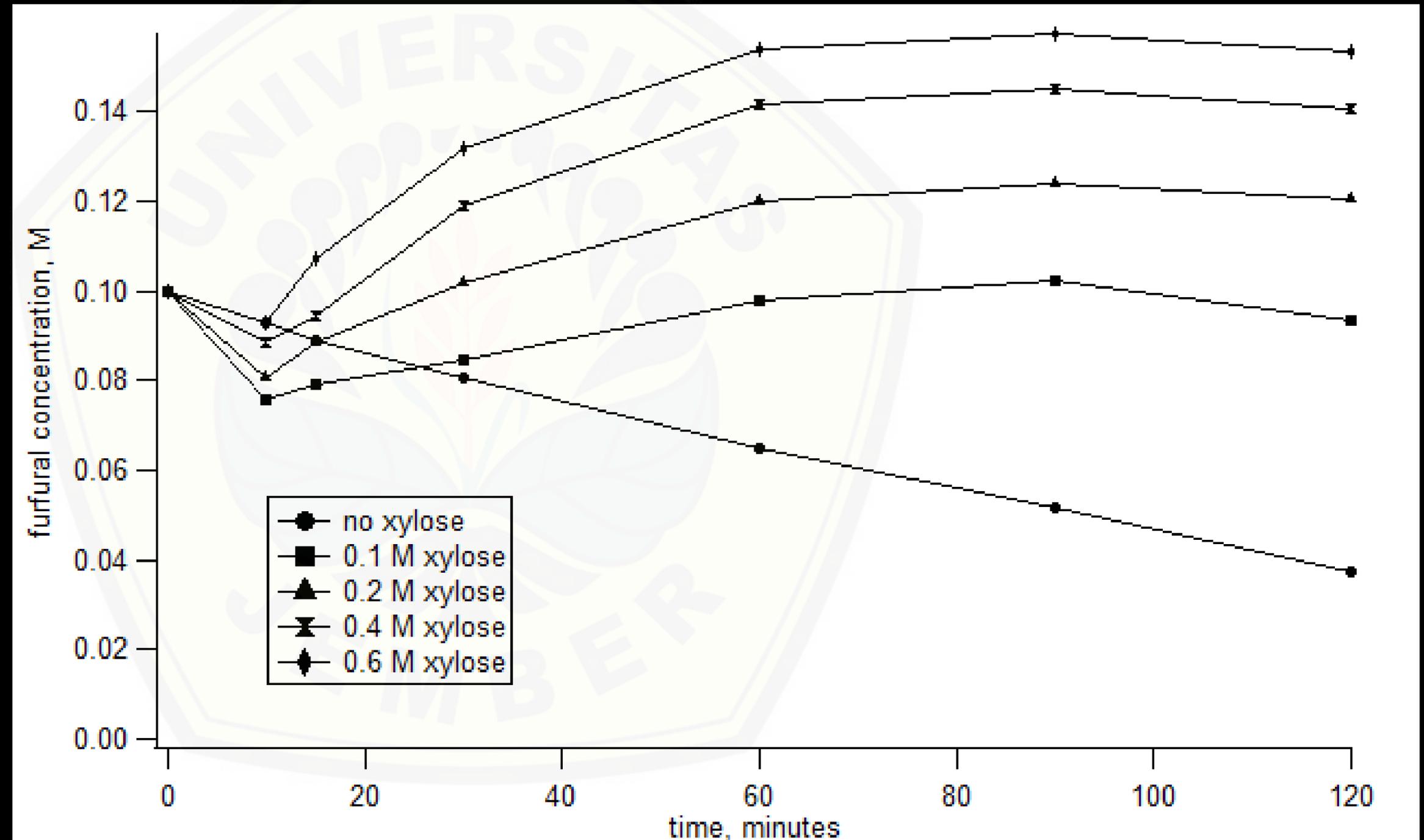
Results and discussion
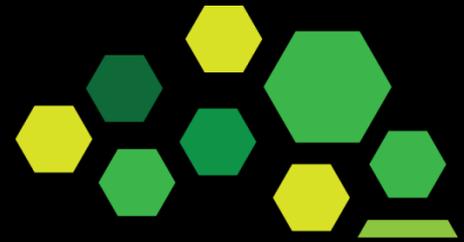
- The similar pattern describes that furfural decomposition rate had the same effect regarding with temperature in 0.4 M xylose addition.
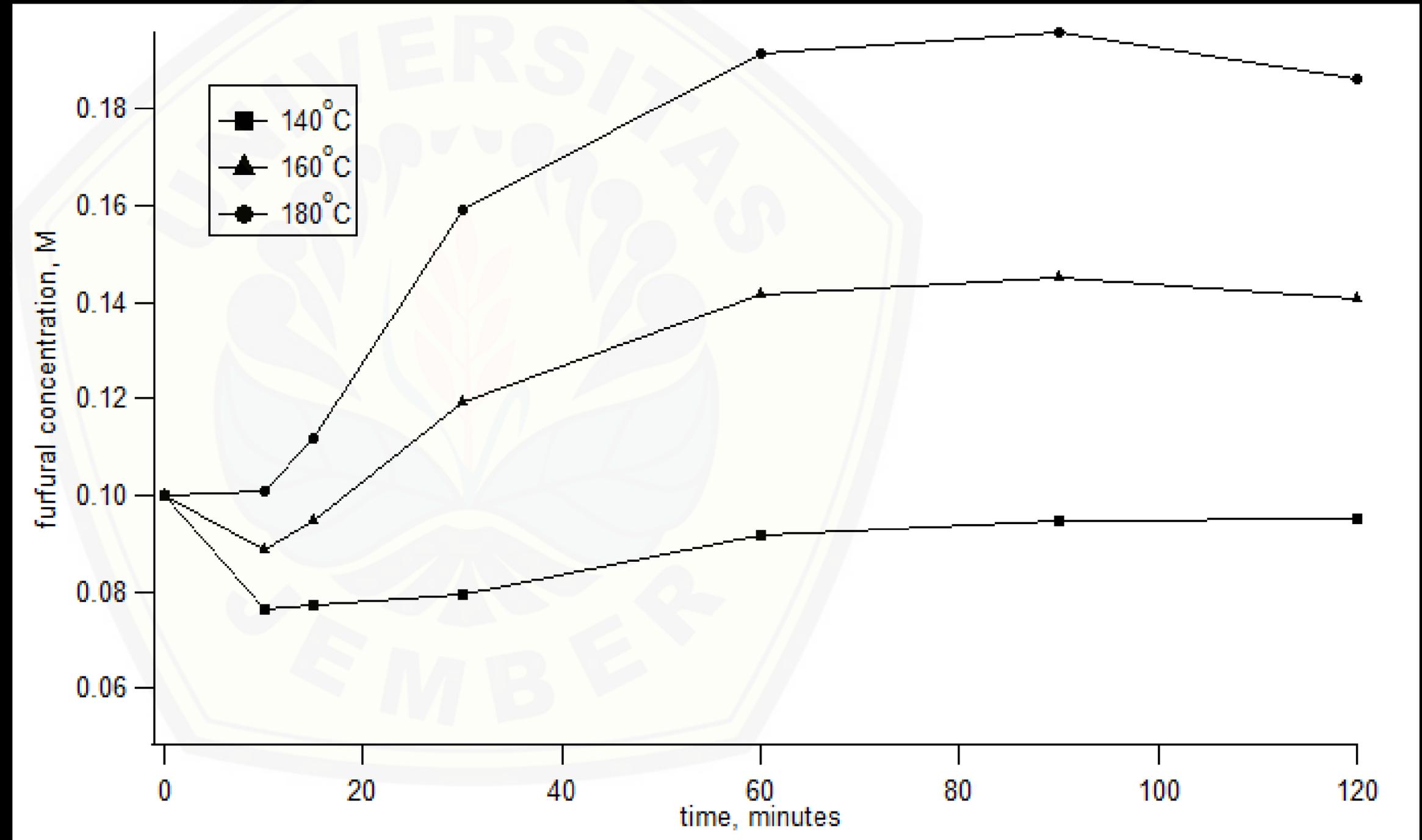
Results and discussion

- The mechanism occurred in the solution are caused by the degradation of xylose and furfural.

- The xylose degradation had a significant part in the solution because of the furfural concentration was increasing for after 15 minutes after the reaction took place.
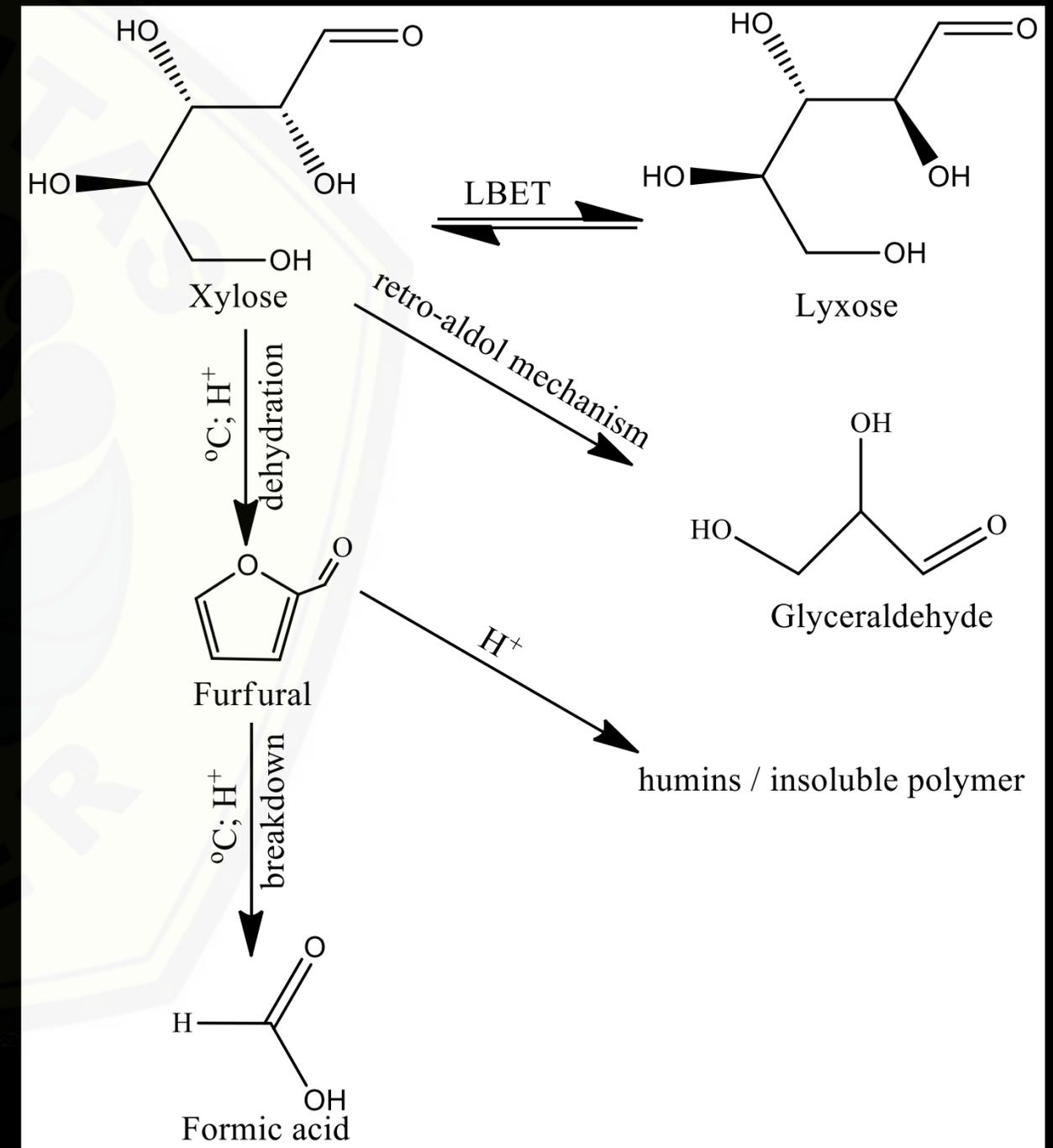
LBET: Lobry de Bruyn–van Ekenstein transformation

Conclusions

We can conclude from this study that:

- The presence of xylose had been contributed in the competition of furfural decomposition mechanism (Xylose becomes furfural).

- There were several substances detected by chromatogram data which contains xylose, furfural, glyceraldehyde, and formic acid at 60 minutes. Humins (insoluble polymer) was also formed during the decomposition reaction.

- Furfural concentration pattern had not been distinct with the variation of temperature.

Certificate

of Appreciation

No :035/STRF/ICOOPCHE/XI/2017

This Certificate is hereby presented to :

FELIX ARIE SETIAWAN

as

Presenter

at

The 2 nd International Conference on Oleo and Petrochemical Engineering (ICOOPCHE) 2017

"Chemical Engineering for Sustainable Development"

Hotel Pangeran, Pekanbaru-Indonesia, 30 November 2017

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