Bio-hydrocarbon Production from Sorbitol using FeI$_2$ to accelerate HI regeneration reaction

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Abstract. Petroleum reserve decrease, while the dominant fuel used for daily needs and industry comes from petroleum. These fossil fuels cause global warming. Because of the above, hydrocarbon fuel from biomass is needed. On the laboratory-scale (1990’s), bio-hydrocarbon can be produced from sorbitol. This was obtained in 2 steps. The first step uses HI and H$_3$PO$_4$ and the second step is heating using KOH-ethanol. In the first step, HI regeneration using H$_3$PO$_4$ produces H$_3$PO$_4$$_3$, which is difficult to be reduced back to H$_3$PO$_4$. It makes the process become expensive, so it needs modification. The modification is done by using HCOOH for regenerating iodine into HI. In order to reduce the need for HI in the system, HI regeneration reaction is expected not slower than sorbitol reduction reaction by HI, so it requires FeI$_2$ catalyst. Referring to the previous description, the purpose of this study is to investigate bio-hydrocarbon production from sorbitol using HI for sorbitol conversion, HCOOH for iodine regeneration, and FeI$_2$ as catalyst to accelerate HI regeneration reaction. A sorbitol, HI, HCOOH and FeI$_2$ mixture is refluxed in three-neck flask. Bio-hydrocarbon (C$_6$H$_{12}$) can be produced from sorbitol. Maximum sorbitol conversion is 85.84%, maximum iodohexane yield is 62.41% and maximum bio-hydrocarbon (C$_6$H$_{12}$) yield is 64.2%.

Keywords: Sorbitol, Formic Acid, FeI$_2$, Iodohexane, Bio-hydrocarbon.

1. Introduction
One of the causes of global warming is the use of fossil fuels, because of this, the use of fossil fuels is increasingly shunned. Fossil fuels (petroleum) reserve is decreasing year by year. Today, transportation machines and power plants require liquid hydrocarbon fuels as the main fuel, so more research about hydrocarbon fuel from biomass is being conducted. Glucose heating with HI will produce bio-hydrocarbons. This has been known for a long time. Substitution of sorbitol for glucose can increase the quality and quantity of bio-hydrocarbon yields. Sorbitol is a sugar alcohol that has six carbon chains. By removing the oxygen atoms, sorbitol can be made into bio-hydrocarbons. The process can be done in relatively mild conditions. Sorbitol can produce bio-hydrocarbons through 2 steps. The first step is the conversion of sorbitol into iodohexane using HI. The second step is the conversion of iodohexane into bio-hydrocarbons by heating using KOH-ethanol.

The first step, converting sorbitol into iodohexane using HI. Sorbitol reacts with HI to produce iodohexane [Lv, 2014, Robinson, 1996, Nakamura, 1978, Willstatter, 1922, Beil, 1918, Vincent, 1890]. The use of HI is intended for iodide ion oxidized to I$_2$. I$_2$ regenerated into HI using H$_3$PO$_4$. The process produces phosphoric acid which is difficult to be reduced back to phosphorous acid [Lv, 2014, Robinson, 1996]. The process is an expensive process, so it needs modification. The modification is replacing H$_3$PO$_4$ by HCOOH. HCOOH is used to regenerate iodine into HI.

The reaction of sorbitol with HI will produce I$_2$. Sorbitol reduction reaction rate will decrease because the absence of reducing agents that produce HI (HI concentration decreased). I$_2$ can be reduced back to...
iodide acid in the presence of HCOOH as reducing agent [Krisnayana, 2019, Abdulloh, 2018, Gunawan, 2018, Kimura, 1989, Hiller, 1966, Mitchell, 1938, Chow, 1935, Hammick, 1926, Dhar, 1917]. The advantages of using HCOOH as a reducing agent compared to H$_3$PO$_3$ are that HCOOH can be produced from biomass processing, and also the result of formic acid oxidation in the form of carbon dioxide gas can be separated from the system into the environment after reducing I$_2$. The carbon dioxide formed is relatively environmentally friendly compared to H$_3$PO$_4$ from the H$_3$PO$_3$ reducing agent.

The need for HI (and I$_2$) in the sorbitol reduction system must be relatively small because I$_2$ and HI are expensive compounds. In order that the need for HI (and I$_2$) in the sorbitol reduction system is relatively small, the HI regeneration reaction is expected to take place not later than the sorbitol reduction reaction by HI. In order to speed up the HI regeneration reaction, catalyst is needed. Potential catalyst for accelerating the I$_2$ reduction reaction by HCOOH such as CuI and FeI$_2$. Bio-hydrocarbons (C$_6$H$_{12}$) can be made from sorbitol by reaction with HI, under relatively mild conditions (T = 90 °C) by regenerating HI using HCOOH and for the reaction of HCOOH with I$_2$ accelerated by using a CuI catalyst [Krisnayana, 2019]. CuI catalysts are less economical, so require modification process. The modification is replacing the CuI catalyst with the FeI$_2$ catalyst.

The second step, converting iodo-hexane into bio-hydrocarbon which can be done by heating using KOH-ethanol [Lv, 2014, Robinson, 1996]. Six carbon chains owned by iodo-hexane can produce bio-hydrocarbons by removing the iodide atom. The conversion of iodo-hexane into bio-hydrocarbons by heating using KOH-ethanol can be carried out under relatively mild conditions and producing C$_6$H$_{12}$. Referring to the previous description, the purpose of this study is to investigate bio-hydrocarbon production from sorbitol using HI for sorbitol conversion, HCOOH for iodine regeneration, and FeI$_2$ as catalyst to accelerate HI regeneration reaction.

2. Materials
The chemicals used in this research are D-Sorbitol 98 % Emsure, Hydroiodic acid 57 % for analysis Emsure, Formic acid 85 % from PT Brataco Indonesia, CuI 99 % from Hebei Guanlang Biotechnology Co Ltd China analytical-grade, dichloromethane from PT Brataco Indonesia, KOH for analysis Emsure and Ethanol from PT Brataco Indonesia.

3. Methods
The schematic diagram for bio-hydrocarbon production from sorbitol using FeI$_2$ to accelerate HI regeneration reaction is shown in Figure 1. Sorbitol can produce bio-hydrocarbon by 2 steps. First step is sorbitol conversion into iodo-hexane using HI. Second step is iodo-hexane conversion into bio-hydrocarbon by heating using KOH-ethanol.

![FIGURE 1. The Schematic Diagram for Bio-hydrocarbon Production from Sorbitol using FeI$_2$ to accelerate HI regeneration reaction](image)

3.1 Sorbitol Conversion into Iodo-hexane.
D-sorbitol, hydriodic acid, formic acid and FeI$_2$ was added to a 100 mL three-neck flask at atmospheric pressure, and the mixture was then heated and was refluxed. Every 1-hour, formic acid was added to make iodine reduced back to hydriodic acid. To study the effects of conditions on the yield of bio-hydrocarbon and halocarbons, variations in the process are carried out on the mixture, namely
reaction time, molar ratio of sorbitol/ hydriodic acid and molar ratio of sorbitol/ HCOOH. Variation of reaction time were 4, 6, 8, 10 and 12 hours, variation of the molar ratios of sorbitol/hydriodic acid were 1:2 and 1:6, variation of the molar ratios of sorbitol/HCOOH were 1:8 and 1:16. The mixture was stirred with a magnetic stirrer at 1000 rpm and at maintained temperature around 90 °C. After the reaction finished, the mixture was left until the temperature of the mixture equal to room temperature, then we can determine the sorbitol conversion and iodohexane yield.

Titration method used to determine sorbitol conversion. Iodohexane yield was determined by gravimetric method. After the mixture equal to room temperature, the mixture was titrated with natrium thiosulfate and starch indicator until the blue color disappears to determine sorbitol conversion. Iodohexane yield was determined by gravimetric method, after the mixture equal to room temperature and diluted with dichloromethane, the mixture was distilled until the temperature reaches 135 °C, the distillation residue was then weighed.

3.2 Iodohexane Conversion into Bio-hydrocarbon.

The weighed distillation residue was mixed with a mixture of KOH-ethanol with a ratio of distillation residue/KOH-ethanol is 1:3. Distillation residue reaction with KOH-ethanol made halocarbons converted into alkene (CnH2n). The mixture was then refluxed for 0.5 hour at 70 °C. After the reaction was finished, the mixture was distilled from 50 °C to 70 °C. The products were characterized by a programmable GC-MS (Shimadzu QP2010).

4. Results and discussion
4.1 Sorbitol conversion into iodohexane.
4.1.1 The effect of FeI2 Catalyst

The effect of FeI2 catalyst on sorbitol conversion into iodohexane can be seen in Figure 2 (a). Figure 2 (a) shows sorbitol conversion without the FeI2 catalyst at molar ratio sorbitol / HI / HCOOH 1:6:16 increased against time from 27.89 % at 4 hours to 80.52 % at 12 hours. Figure 2 (a) also shows sorbitol conversion with FeI2 catalyst, which FeI2 catalyst used to accelerate the reaction of HCOOH with I2, in the molar ratio of sorbitol / HI / HCOOH 1:6:16, sorbitol conversion increases against time from 28.94 % at 4 hours to 85.84 % at 12 hours. FeI2 catalyst used can increase sorbitol conversion.

The iodohexane yield is shown in Figure 2 (b). Molar ratio of sorbitol / HI / HCOOH 1:6:16 using the FeI2 catalyst, the FeI2 catalyst used to accelerate the reaction of HCOOH with I2, resulting in increased iodohexane production against time from 21.98% at 4 hours to 62.41% at 12 hours. Without using the FeI2 catalyst, iodohexane yield with the sorbitol / HI / HCOOH molar ratio 1:6:16 resulted in the increase of iodohexane production against time from 17.9 % at 4 hours to 51.81 % at 12 hours. Iodohexane yield using FeI2 catalyst is more increased than without using FeI2 catalyst.

The reaction of sorbitol with HI which produces iodohexane is a reduction reaction. The reduction reaction activity is influenced by the FeI2 catalyst. The FeI2 catalyst is a heterogeneous catalyst with Fe metals. Fe metals are easily coordinated with reactants. The FeI2 catalyst is a solid catalyst. FeI2 catalyst can be used several times. Based on these exposures, the FeI2 catalyst helps increase sorbitol conversion and iodohexane yield.

![FIGURE 2. The Effect of FeI2 Catalyst (a) on the Conversion of Sorbitol into Iodohexane (b) on the Yield of Iodohexane](image-url)
4.1.2 The effect of Molar Ratio of Sorbitol / HI

Sorbitol conversion using the FeI₂ catalyst to accelerate the reaction of HCOOH with I₂, where the molar ratio of sorbitol / HI affects sorbitol conversion is shown in Figure 3 (a). With a molar ratio of sorbitol / HCOOH 1 : 16, the conversion of sorbitol during the 12 hour reaction time increased against the molar ratio of sorbitol / HI from 55.79 % at 1 : 2 to 85.84 % at 1 : 6. The increase in sorbitol conversion shows that HI can convert sorbitol. The more HI used in the reaction the more sorbitol conversion. Equation (1) shows the reaction of sorbitol with HI. With a molar ratio of sorbitol / formic acid 1 : 16, the conversion of sorbitol also increases against the molar ratio of sorbitol / iodide acid for reaction times of 4, 6, 8 and 10 hours.

\[
\text{C}_6\text{H}_{14}\text{O}_6 + 11 \text{HI} \rightarrow 5 \text{I}_2 + \text{C}_6\text{H}_{13}\text{I} + 6 \text{H}_2\text{O} \quad (1)
\]

The effect of the sorbitol / HI molar ratio on iodohexane yield using the FeI₂ catalyst (FeI₂ catalyst used to accelerate the reaction of HCOOH with I₂) is shown in Figure 3 (b). Figure 3 (b) shows, with a molar ratio of sorbitol / HCOOH 1 : 16, the yield of iodohexane increases against the molar ratio of sorbitol / HI at a 12 hour reaction time from 36.70 % at 1 : 2 to 62.41 % at 1 : 6. With a sorbitol / HCOOH molar ratio of 1 : 16, iodohexane yield also increase with the sorbitol / HI molar ratio for reaction times of 4, 6, 8 and 10 hours. This phenomenon explains, HI can increase the yield of iodohexane, where the more HI is used in the reaction, the more iodohexane is produced.

The reaction of sorbitol with HI as shown in equation 1 is a reduction reaction which can be accelerated at relatively low temperatures. HI consists of H atoms and I atoms. I atom belongs to group VII A. Group VII A has the highest ionization energy and electronegativity in elements periodic. Based on this explanation, HI has a relatively high ionization energy and a high electronegativity. The higher the ionization energy, the greater the attraction between the nucleus and the outer electrons, so a large amount of energy is needed to release the outermost shell electrons that makes the reduction reaction easier. If the electronegativity is high, it means that the reduction reaction is easier and HI gives big impact on sorbitol conversion and iodohexane yield.

\[
\text{HCOOH} + \text{I}_2 \rightarrow 2 \text{HI} + \text{CO}_2 \quad (2)
\]
Iodohexane yield at 12 hours reaction time with molar ratio of sorbitol / HI 1 : 6 increased against molar ratio of sorbitol / HCOOH from 50.69% at 1 : 8 to 62.41% at 1 : 16. For reaction times 4, 6, 8 and 10 hours with sorbitol / HI molar ratio of 1 : 6, iodohexane yield increased against molar ratio of sorbitol / HCOOH. This explains that the reaction of sorbitol and HI produces I₂, the reaction of HCOOH with I₂ produces HI, where HI is used to produce iodohexane. The more HCOOH is used, the more HI is produced. With more HI being produced, more iodohexane is produced. The reaction of sorbitol with HI is shown in Equation (1) and the reaction of I₂ with HCOOH can be seen in Equation (2).

The reaction of sorbitol with HI will produce I₂. In the absence of a reducing agent, HI concentration will decrease and the reaction rate of sorbitol reduction will decrease too. Equation 2 means I₂ can be reduced back to HI with HCOOH as reducing agent. The reaction of HCOOH with I₂ is a reduction reaction. Based on the above explanation, HCOOH can increase sorbitol conversion and iodohexane yield. Other advantages of using HCOOH as a reducing agent are HCOOH can be produced from biomass processing, and also the result of formic acid oxidation in the form of carbon dioxide gas can be separated from the system into the environment after reducing I₂. The formed carbon dioxide is relatively environmentally friendly.

![Figure 4](image_url)

**FIGURE 4.** The Effect of Molar Ratio of Sorbitol / HCOOH (a) on the Conversion of Sorbitol into Iodohexane (b) on the Yield of Iodohexane

### 4.1.4 The effect of reaction time

The reaction time has an impact on the sorbitol conversion shown in Figure 5 (a). Sorbitol conversion increases against time from 28.94% at 4 hours to 85.84% at 12 hours with sorbitol / HI / HCOOH molar ratio 1 : 6 : 16. Sorbitol conversion increases against time at sorbitol / HI / HCOOH molar ratio 1 : 2 : 16. This phenomenon explains that collisions cause chemical reactions. According to the collision theory, chemical reactions occur because particles colliding with each other. The formation of iodohexane shows that there was a collision between sorbitol and HI. The collision between sorbitol and HI causes sorbitol to break its bond with O and form a new bond with I. With a longer reaction time, more collisions occur so that more sorbitol is converted.

In the molar ratio of sorbitol / HI / HCOOH 1 : 6 : 16, iodohexane production increases against time shown in Figure 5 (b) with a value from 21.98% at 4 hours to 62.41% at 12 hours. Iodohexane production also increases against time at the sorbitol / HI / HCOOH molar ratio 1 : 2 : 8. This means that the collision causes a chemical reaction. As reaction time increases, more collisions occur so that more iodohexane yielded.

The reaction of sorbitol with HI is a reduction reaction that can occur at low temperature. If a collision occurs, a reaction occurs. The reaction can convert sorbitol and produce iodohexane. The longer the reaction occurs between sorbitol and HI, the more collisions occur. The more collisions, the more sorbitol is converted and the more iodohexane yielded.
4.2 Iodohexane conversion into bio-hydrocarbon.

4.2.1 The effect of FeI\(_2\) catalyst

The effect of FeI\(_2\) catalyst on the bio-hydrocarbon yield from iodohexane is shown in Figure 6. The product characterization was carried out using GC-MS. The identified product is C\(_6\)H\(_{12}\). This means iodohexane turns into hexene. Hexene has a boiling point at 68 °C, so it is very possible to be separated by distillation. The reduction reaction of iodohexane to bio-hydrocarbons is shown in Equation (3). The yield of bio-hydrocarbons (C\(_6\)H\(_{12}\)) from iodohexane is calculated by comparing the actual mass of bio-hydrocarbons (C\(_6\)H\(_{12}\)) with its theoretical mass. The mass of the reaction product (3) increases against time with the sorbitol / HI / HCOOH molar ratio 1 : 6 : 16, which uses the FeI\(_2\) catalyst (FeI\(_2\) catalyst to accelerate the reaction of HCOOH with I\(_2\)). Yield of bio-hydrocarbons (C\(_6\)H\(_{12}\)) using FeI\(_2\) catalyst from iodohexane also increases against time from 23.27% at 4 hours to 64.2% at 12 hours. Yield of bio-hydrocarbons (C\(_6\)H\(_{12}\)) from iodohexane using a FeI\(_2\) catalyst is better than without using a FeI\(_2\) catalyst.

\[
\text{C}_6\text{H}_{13}\text{I} + \text{KOH} \rightarrow \text{C}_6\text{H}_{12} + \text{KI} + \text{H}_2\text{O} \quad (3)
\]

The FeI\(_2\) catalyst makes iodohexane formed more, where the more iodohexane is formed, the more bio-hydrocarbons are produced. The FeI\(_2\) catalyst is a solid catalyst with Fe metal. Fe metals are easily coordinated with reactants.
4.2.2 The effect of molar ratio of Sorbitol / HI

The sorbitol / HI molar ratio affects the bio-hydrocarbon yield \((C_6H_{12})\) from iodohexane using the FeI\(_2\) catalyst shown in Figure 7. The bio-hydrocarbon yield \((C_6H_{12})\) of iodohexane increases against the molar ratio of sorbitol / HI at the 12 hour reaction time with a molar ratio of sorbitol / HIcoOH 1 : 16 from 47.7 % at 1 : 2 to 64.2 % at 1 : 6. The more HI used in the reaction, the more sorbitol is converted and the more iodohexane is produced. If iodohexane production is more produced, the yield of bio-hydrocarbons \((C_6H_{12})\) will increase. The conversion reaction of iodohexane into \(C_6H_{12}\) is shown in Equation (3). Yield of bio-hydrocarbons \((C_6H_{12})\) from iodohexane also increases to molar ratio of sorbitol / hydriodic acid at 4, 6, 8 and 10 hours reaction times with molar ratio of sorbitol / formic acid 1 : 16.

HI makes more iodohexane formed, whereas the more iodohexane is formed, the more bio-hydrocarbons is produced. HI consists of H atoms and I atoms. I atom belongs to group VII A. Group VII A has the highest ionization energy and electronegativity in periodic elements. Based on this explanation, HI has a relatively high ionization energy and a high electronegativity. It means that reaction is easier to occur.

![FIGURE 7. The Effect of Molar Ratio of Sorbitol / HI on the Yield of Bio-hydrocarbon from Iodohexane](image)

4.2.3 The effect of molar ratio of Sorbitol / HCOOH

The molar ratio of sorbitol / HCOOH affects the bio-hydrocarbon \((C_6H_{12})\) yield from iodohexane using FeI\(_2\) catalyst as shown in Figure 8. The amount of HCOOH used has an impact on the bio-hydrocarbon yield \((C_6H_{12})\) from iodohexane. The yield of bio-hydrocarbons \((C_6H_{12})\) from iodohexane at a reaction time of 12 hours with a molar ratio of sorbitol / HI 1 : 6 increased against molar ratio of sorbitol / HCOOH from 53.5% at 1 : 8 to 64.2% at 1 : 16. Yield of bio-hydrocarbons \((C_6H_{12})\) from iodohexane also increased against molar ratio of sorbitol / HCOOH for reaction times 4, 6, 8 and 10 hours against molar ratio of sorbitol / HI 1 : 6. That means, the sorbitol and HI reactions produce \(I_2\). The \(I_2\) from the reaction then reacts with HCOOH to produce HI, HI is used for the conversion of sorbitol. The more HCOOH used in the reaction, the more HI is produced. The more HI is produced, the more sorbitol is converted and the more bio-hydrocarbon \((C_6H_{12})\) yield from iodohexane.

The more iodohexane produced, the more bio-hydrocarbon yield \((C_6H_{12})\). The conversion of iodohexane into \(C_6H_{12}\) is shown in Equation (3). HCOOH makes more iodohexane formed, where more iodohexane is formed, more bio-hydrocarbons are produced. HCOOH will reduce \(I_2\) to HI, where HI is needed to produce iodohexane. In the absence of reducing agents, the concentration of HI decreases and the rate of reduction of sorbitol decreases.
4.2.4 The effect of the reaction time
The reaction time has an impact on the bio-hydrocarbon (C₆H₁₂) yield of iodohexane shown in Figure 9. For the molar ratio of sorbitol / HI / HCOOH 1 : 6 : 16, the bio-hydrocarbon (C₆H₁₂) yield of iodohexane increases against time from 23.27% at 4 hours to 64.2% at 12 hours. The yield of bio-hydrocarbons (C₆H₁₂) from iodohexane also increases against the molar ratio of sorbitol / HI / HCOOH 1 : 2 : 8. That means, the collision causes a chemical reaction. The longer the reaction, the more collisions occur. The more collisions, the greater the yield of iodohexane. The more iodohexane produced, the more bio-hydrocarbons (C₆H₁₂) yielded. The conversion of iodohexane into C₆H₁₂ is shown in Equation (3).

5. Conclusions
Sorbitol and HI reaction at T = 90 °C with HI regeneration using HCOOH and for the reaction of HCOOH with I₂ accelerated by FeI₂ catalyst can produced bio-hydrocarbon (C₆H₁₂). Sorbitol conversion, iodohexane and bio-hydrocarbon (C₆H₁₂) yield increased with the longer reaction time, the more HI, the more HCOOH and the use of FeI₂. Maximum sorbitol conversion is 85.84 %, iodohexane yield is 62.41 % and bio-hydrocarbon (C₆H₁₂) yield is 64.2 % at a temperature of 90 °C, reaction time 12 h, molar ratio of sorbitol / HI / HCOOH is 1 : 6 : 16 and use FeI₂ catalyst to accelerate the reaction of HCOOH with I₂. The product of iodohexane conversion to bio-hydrocarbon is hexene (C₆H₁₂).

The process in this study gives KI as a side product, which KI produced in second step from reaction of iodohexane with KOH-ethanol. This side product (KI) makes the process expensive because KI difficult to be covert back into KOH so the modification process is needed to make the process more efficient and effective. The modification is changing the second step process to pyrolysis method. Iodohexane conversion into bio-hydrocarbon can be done by pyrolysis. Benson (1963), Maccoll (1963) and Ogg (1934) did pyrolysis for iodoalkane conversion into hydrocarbon and I₂. Pyrolysis optimum temperature at 295°C.
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