

# A laboratory scale synthesis

*by Fiki Hidayat*

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**Submission date:** 30-Mar-2023 10:11PM (UTC+0700)

**Submission ID:** 2051075835

**File name:** A\_Laboratory\_Scale\_Synthesis.pdf (517.04K)

**Word count:** 4775

**Character count:** 24218

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To cite this article: M K Afdhol *et al* 2020 *IOP Conf. Ser.: Mater. Sci. Eng.* **854** 012017

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# A Laboratory Scale Synthesis of Ethanol from Agricultural Waste as Bio-based Solvent for Waxy-Paraffinic Crude Oil Mitigation

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**Abstract.** Paraffin is a problem commonly faced in petroleum production. The presence of paraffin can inhibit the process of oil flow. Mitigation methods commonly used to overcome this problem is by using chemical injection, which is solvent. In this research, a laboratory experiment was conducted to make bioethanol from agricultural waste that are rice husks and corn cobs. This bioethanol functions as a solvent that is used to reduce the pour point in a waxy-paraffinic crude oil sample. Oil samples were mixed with bioethanol with different mixing ratios, including 5%, 10%, 15%, 20% 50%. The results showed that the alcohol content of the Husk and Corn cob rise was 11% and 9% respectively. Then, the best result from mixing of bioethanol with oil reduced the pour point from 43.3 to 41 at mixing ratio of 50%.

## 1. Introduction

### 1.1. Wax Paraffin

The problem of paraffin wax is a very crucial problem in the oil field because it can cause oil to not flow when the temperature is below the pouring point (Abdurrahman, Rizal, Husna, & Pangaribuan, 2018). Paraffin wax is part of hydrocarbons, which cannot be dissolved by crude oil under normal conditions, where the normal conditions of crude oil that have been tested are at a temperature of 70-150 °C and at a pressure of 55-103 Mpa (Wang, Wu, Creek, Shuler, & Tang, 2003).

There are several methods to overcome paraffin wax, one of which is the preventive method of heating and using chemicals, one of the most effective methods is using a hydrocarbon solvent (Afdhol, Lubis, & Siregar, 2019; Khaibullina, 2016). According to Al-yaari & Fahd (2011) for examples of injection schemes using solvents can be seen in Figure 1. The solvent injection device proposed by C-FER Technologies.





**Figure 1.** Wax (Huang, Wang, Li, Ren, & Zhu, 2017)

### 1.2. Bioethanol Production

Ethanol is produced through several processes, namely hydrolysis and fermentation using food ingredients that contain starch carbohydrates, such as rice and tubers (Cai, Liu, He, Chai, & Cai, 2018). Biomass containing lignocellulose can be used as a source of reducing sugars such as glucose, xylose and maltose (Mahboubi et al., 2017). In the next step, the alcohol produced must be turned into steam through a distillation process with a temperature of 78.5°C (Salehi, Taghizadeh-alisaraci, Shahidi, & Jahanbakhshi, 2018). According to (Cai et al., 2018) several stages of the biomass process will be carried out to produce ethanol, namely pretreatment, hydrolysis and fermentation. Biomass pretreatment is important because it is to get high ethanol yield. The purpose of pretreatment is to break the structure of lignocellulose so that cellulose is separated from lignin and thus cellulose. Hydrolysis is the process of breaking down polysaccharides in lignocellulosic biomass, which are cellulose and hemicellulose which will be broken down into sugar monomers. Perfect cellulose hydrolysis will produce glucose. Hydrolysis can be done chemically using acids or enzymatically. There are several factors that influence the hydrolysis process, namely carbohydrate content of raw materials, pH of hydrolysis, hydrolysis time, temperature and pressure. Fermentation is microbial activity in food so that the desired product is produced. Common microbes involved in fermentation are bacteria, yeast and mold.

There are important factors that will affect ethanol yield and efficiency. First, under physiological conditions the microbes are added to the media, which depends on the optimal optimum growth conditions of the microbes to be used. Second, the environmental conditions during the fermentation, i.e. pH and temperature. Bioethanol raw materials can be derived from cellulose source materials such as rice straw, bagasse, corncobs and rice husks (Said, Wahid, Diah, & Sabang, 2014). Utilization of biomass has been done a lot such as oil palm shells (Yuliusman, Nasruddin, Afdhol, Amiliana, et al., 2017), coffee grounds and others (Yuliusman, Nasruddin, Afdhol, Haris, et al., 2017). The production of bioethanol from cellulose waste has been developed, rice husk is one of the renewable raw materials used for making bioethanol as it is easily available and very economical (Kouteu Nanssou, Jiokap Nono, & Kapseu, 2016).

#### 1.2.1. Rice Husk

Rice husk (Figure 2) contains several organic compounds namely, lignin, cellulose, hemicellulose, nitrogen compounds, vitamin B and organic acids and contains inorganic compounds in the form of silica (Ebrahimi, Caparanga, Ordone, Villaflores, & Pouriman, 2017). Rice Husk (RH), a broad covering of rice grain which accounts for around 20% of rice, has the potential to produce about 0.4 sugar per 1 gram of dry RH and the sugar process can further be fermented into bioethanol (Ebrahimi et al., 2017).



**Figure 2.** Rice husks (Cai et al., 2018)

**Table 1.** Composition of Rice Husks

Component	Rice Husks	Rice Bran	Broken Rice
starch	6.9	28.5	68.6
Cellulose	38.4	4.6	1.8
Ash	15.9	8.0	1.5
Hemicellulose	16.1	8.4	3.7
Lignin	16.7	2.8	-
Protein	3.4	12.9	9.9

Source : (Favaro et al., 2017)

It can be seen in Table 1 that the cellulose and hemicellulose content in rice husk is the largest at 38.4% and 16.1% so that it can be used as a source of bioethanol production.

### 1.2.2. Corn Cobs

In corn plants consist of corn kelobot, corn hair, corn cobs and corn kernels. Corn plants based on systematics (taxonomy) of plants are classified as follows (Sain, 2016).

**Table 2.** Taxonomic status of maize (Sain, 2016)

<i>Regnum</i>	<i>Plantae</i>
<i>Devisio</i>	<i>Spermatophyte</i>
<i>Sub Devisio</i>	<i>Angiospermae</i>
<i>Classis</i>	<i>Monocotyphyta</i>
<i>Ordo</i>	<i>Graminae</i>
<i>Familia</i>	<i>Graminaceae</i>
<i>Genus</i>	<i>Zea</i>
<i>Species</i>	<i>Zea mays L.</i>

Corn contains crude fiber which is composed of complex compounds of lignin, hemicellulose and cellulose (lignocellulose), and each one is a compound that can potentially be converted into other compounds biologically. The dry solid content of Corncob by using alkaline, solvent or ammonia based pretreatments produces the greatest amount of ethanol (19-22%), whereas mushroom based pretreatments produce much less (11%) (Zhao, Damgaard, & Christensen, 2018).



Figure 3. Corncob waste (Fachry, Astuti, & Puspitasari, 2013)

The complex compounds of lignin, hemicellulose and cellulose (lignocellulose) each are compounds that can potentially be converted into other compounds biologically and the following table compositions of these compounds from several materials:

Table 3. Composition of Cellulose, Hemicellulose and Lignin from different Lignocellulose (Byadgi & Kalburgi, 2016)

Lignocellulosic Material	Cellulose (%)	Hemicellulose (%)	Lignin (%)
Miscanthus	40	18	25
Coastal Grass	25	35.7	9-18
Corncob	35-40	17-35	7-18
Wheat Straw	30	50	15

From the table above it can be seen that corn is a plant-based material with a compound that is easily converted into alcohol, so this study takes corn as a raw material for making bio-ethanol solvents.

## 2. Material and Methods

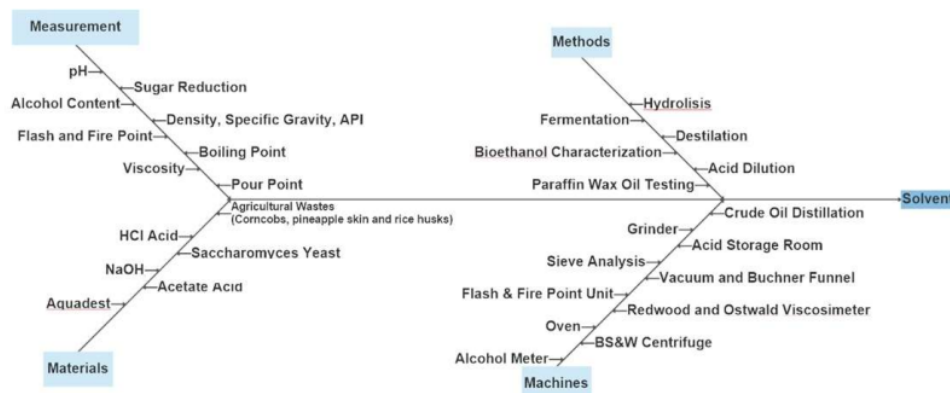


Figure 4. Research's Fishbone



### 2.1. Raw Materials

Materials used in this study comprises of rice husks, comcobs, acetic acid, chloric acid, sulfuric acid, NaOH, saccharomyces and distilled water.

### 2.2. Hydrolysis

Prepare materials that are ready for hydrolysis with sizes 30, 50, 80, 100 and 200 mesh. Enter the ingredients as much as 15 grams into a beaker (Hayuningtyas, Sk, & Sla, 2014). Then add a solution of sulfuric acid and hydrochloric acid as much as 150 ml each with a temperature of 100°C for 2.5-3 hours (Hayuningtyas et al., 2014) at 5% acid concentration. Repeat steps 2 and 3 for acid concentrations of 10%, 15% and 25%. The rice husk from hydrolysis is then filtered, where the hydrolyzed solution is the main product for the fermentation process (Osvaldo, S, & Faizal, 2012)

### 2.3. Fermentation

The rice husk hydrolysis substrate was ready to be fermented. Prepare anaerobic bacteria, the bacteria used are *Saccharomyces Cerevisiae* with pH conditions 3-4 (Azizah, N., Al-Baarri, A.N., dan Mulyani, 2012). Entering the results of hydrolysis of rice husk into the fermentation series that has been prepared. Add bread yeast (Utami & Windiyati, 2014) with a mass of 1.5 grams (Jayanti, 2011). Fermented for 5 days at 30°C. (Azizah, N., Al-Baarri, A.N., dan Mulyani, 2012)



Figure 5. Fermentation Process

### 2.4. Distillation (ASTMD1160)

The process of distillation (purification) is the stage of separating a substance based on its boiling point. The following stages of distillation: Take a sample of the fermentation that has been done. Weigh flash containing sample + thermometer. Clean and dry the measuring cup, place the measuring cup so that the tip of the outlet goes into the measuring cup of the distillate. Close the measuring cup to prevent the steam from coming out of the tube. Circulate coolant through the condenser. Run to warm up and adjust the temperature slowly so that the distillation liquid drips properly. Stop heating at the end boiling point and allow the distillate liquid to drip on the measuring cup. After cooling, record the volume of the distillate produced.



**Figure 6.** Distillation Tool Series

### 3. Calculation

#### 3.1. Paraffin Wax Measurement

In this study using crude oil samples taken from field X and the following is the oil properties data in the field:

**Table 4.** PVT data and crude oil properties in field X

Information	Value	Unit
Oil density	30,8	API
Specific Gravity	0,8713	-
Water phase density	1,00028	gr/cm <sup>3</sup>
Oil Viscosity	14,4	cp
Water Viscosity	0,5	cp
Pour Point	100	°F
Flash Point	170	°F
Reservoir Temperature	136	°F
Bottom Hole Pressure	200-400	psig
Reservoir Pressure	532	psig
Bubble Point Pressure	113	psig
Oil formation volume factor	1,049	-
Initial Oil Saturation	63,1	%
Depth Formation	1100-1200	ft

Reservoir fluid composition is also one important parameter that needs to be known in order to be able to choose the type of solvent used and also to predict the formation of paraffin wax. The following is a table of reservoir fluid composition in field X:



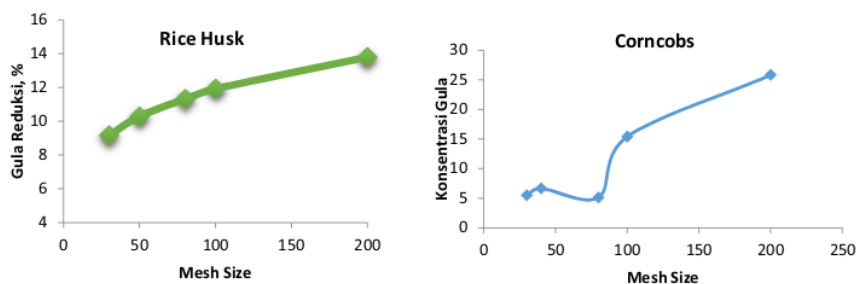
**Table 5.** Composition of fluids in field X

Component	Percent of Mol	Percent Weight
Hydrogen sulphide	0,00	0,00
Carbon dioxide	0,56	0,08
Nitrogen	0,00	0,00
Methane	0,67	0,04
Ethane	0,67	0,07
Propane	1,51	0,22
Iso-Butane	0,90	0,18
n-Butane	1,57	0,31
Iso-Pentane	1,56	0,38
n-Pentane	1,50	0,36
Hexane	0,35	0,10
Heptane Plus	90,71	98,26
Total	100,00	100,00

The data above becomes a reference for laboratory experiments conducted to find out indications of paraffin wax formation by the methods / tests carried out as described below referred to ASTM D97; Pour the sample into a tube of 10 cc. Insert the tube in the oven and heat it to the test temperature. Remove the tube containing the sample from the oven while the sample is still hot. Observe temperature changes when all samples can be poured

#### 4. Results and Discussion

##### 4.1. Sugar Reduction

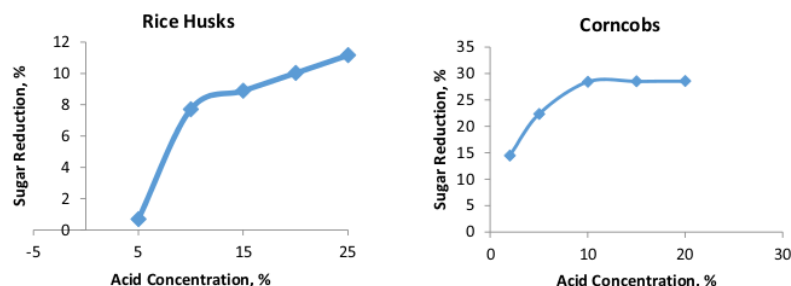


**Figure 7.** Results of Reducing Sugar Based on Mesh Size Differences

Analysis of the results of reducing sugars on different mesh sizes such as Figure 7 rice husk shows that the smaller the size of the mesh used, the resulting reducing sugars are getting bigger. It was proven that when the 30 mesh size produced reducing sugar 9.17, the mesh size 50 produced the reducing sugar 10.29, the mesh size 80 produced the reducing sugar 11.32, the mesh size 100 produced the reducing sugar 11.92 and the mesh size 200 produced the reducing sugar by 13.8. so it can be concluded that the smaller the mesh used it will produce a large reducing sugar.

Figure 7 corncob shows that the higher the mesh size (the finer) the reducing sugar content will be even higher while the size of 80 mesh will decrease because it can be caused by several factors such as errors during hydrolysis and the sample when tested experiencing changes in properties and after testing as much as 2 times the researcher regards it as an anomaly in the study because viewed from the trendline has shown a graph that increases with the mesh size.

According to Osvaldo et al., (2012) the size of the raw material used will affect the size of the porosity so that it can maximize contact between the material and the acid to increase the results of cellulose hydrolysis so that the smaller the size of the material used, the greater the yield of reduced sugar.



**Figure 8.** Results of Reducing Sugar Based on Differences in Acid Concentration

Meanwhile, for the observation of Figure 8 rice husk regarding the results of reducing sugars which is influenced by different acid concentrations, it is known that the higher the concentration of sulfuric acid used in the hydrolysis process, the results of the reducing sugars are also greater. Can be seen in the picture when the 5% acid concentration results in reducing sugar by 0.7, 10% acid concentration produces 7.72% reducing sugar, 15% acid concentration produces 8.9% reducing sugar, 20% concentration produces 10.04 reducing sugar and 25% concentration produces reducing sugar equal to 11.17.

From Figure 8 it can be seen that the higher the concentration of acid used, the higher the level of reducing sugar. The higher the reducing sugar levels, the more ethanol will be produced. the highest acid concentration is 20% HCL and also the advantage of using strong acids at low concentrations is that there is no need for acid recovery and the absence of acid ions lost in the process.

#### 4.2. Alcohol Product from Fermentation



**Figure 9.** Distillation Circuit

In Figure 9 shows the entire distillation circuit consisting of a core series of sample containers, heaters, thermometers and measuring cups for the distillate reservoir. The boiling point of alcohol is around 78°C (Fachry et al., 2013; Jaya, Arthawan, & Sari, 2012; Salehi et al., 2018). Then the temperature in the distillation device is set to 78°C so that the resulting distillate is a type of bioethanol alcohol. The percentage of alcohol in the rice husk distillate sample is 8%. In previous studies using rice husk fermented for 5 days produced alcohol by 14.4227% (Utami & Windiyati, 2014)

The bioethanol that has been made is measured by using alcohol meter alcohol meter and after measuring the alcohol content obtained from bioethanol from corncob waste by 11% where this is a fairly high alcohol content which based on research that has been done before the dry solid content of Corncob produce the greatest amount of ethanol (19-22%) (Zhao et al., 2018).

#### 4.3. Alcohol Characterization of Synthetic Solvent and Bio-Solvent

**Table 6.** Characteristics of synthetic solvents and bioethanol

Properties	Bioethanol		Parasol II
	Rice Husk	Corn Cob	
Density, gr/cc	0.971	0,984	0.8528
SG	0.971	0,984	0.8528
°API	14.13	12.30	34.42
Color	Colorless	Colorless	Colorless
Odor	Aromatic	Aromatic	Aromatic
pH	4	4	9
Viscosity, cp	0.752	0.80	-
Flash Point, °C	48	45	16.1
Fire Point, °C	54	56	-

The advantage of bioethanol from agricultural waste is the value of the sample flash point where the rice husk flash point is 48°C, which means that at room temperature the sample has not been burned so it is safe to use on a laboratory scale. This is the advantage of bioethanol over parasol II, because parasol II has a flash point 16.1 which means below room temperature that is 30°C parasol II will be flammable so bioethanol is more safety than parasol II.

Bioethanol from corncob based on flash point and fire point testing can be said to be more safety for the application of production wells and production pipes, with a flash point of 45°C and a fire point of 56°C. This can be indicated that the bioethanol produced is non-flammable compared to synthetic solvents.

#### 4.4. Paraffin Wax Test Results

Tests carried out to identify the success of solvents in overcoming the problem of paraffin wax in oil, namely testing the pour point (ASTM D97). In this test, two tests were conducted, namely testing the pouring point of oil before mixing the solvent and the pouring point of oil after mixing the solvent. The following table shows the results of tests conducted on three oil samples.

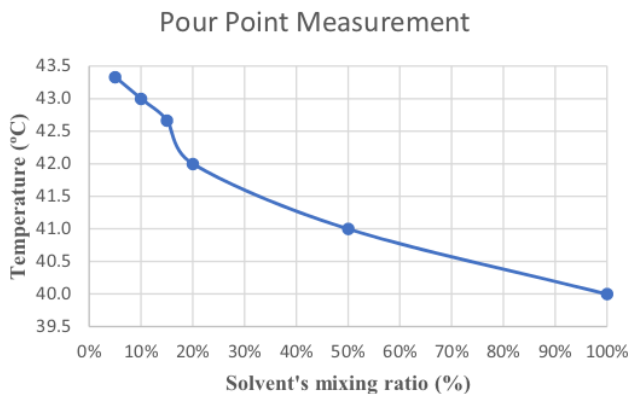
**Table 7.** Testing of Pour Oil Before Mixing Solvent

Simple Name	Pour Point	Unit
LGK (11-07-2018)	44	°C
LGK (24-04-2018)	43	°C
LGK (28-06-2018)	43	°C
Average	43,3	°C

In the results table above is the initial pour point temperature of oil containing paraffin wax before the solvent is mixed. Furthermore, to test the feasibility of the solvent from the corncob material that has been made, the solvent is mixed with the oil sample above with the amount of oil sample of 10 ml with a percentage of many solvents added namely 5%, 10%, 15%, 20%, 50% and 100% so we get the test results as in the table below:

**Table 8.** Testing of the Pour Point of Oil After Solvent Mixing

Sample Name	Pour Point					
	5%	10%	15%	20%	50%	100%
Sample I	44°C	44°C	44°C	43°C	42°C	41°C
Sample II	43°C	43°C	42°C	42°C	41°C	40°C
Sample III	43°C	42°C	42°C	41°C	40°C	39°C
Average	43,3°C	43°C	42,7°C	42°C	41°C	40°C



**Figure 10.** Pour Point Testing After being mixed with Solvent

Seen from the table and figure above, it can be seen that there is a change in value at the pouring point test before and after the solvent is mixed. Changes in the temperature of the pouring point of this oil can indicate the effect of mixing the solvent on the oil containing paraffin wax where the oil in the sample is average with an initial temperature of 43.3°C which means that oil for the first time can flow and by mixing the solvent with the pouring temperature oil into 40°C which shows that after being mixed with oil solvent it is easier to flow at a lower temperature than the initial temperature of the oil so that this can overcome the problem of paraffin wax on oil where with paraffin wax the oil becomes more difficult to flow but with the addition the oil pour point solvent decreases and even at low temperatures the oil can still flow more easily than oil without mixing with the solvent.

### 5. Conclusion

Based on the results of the analysis in the manufacture of bioethanol and the application of bioethanol to oils with high paraffin levels, the following conclusions are obtained:

1. The characteristics of the bioethanol produced can be seen from the density, SG, viscosity, PH, aroma and physical form that is almost the same as synthetic solvents and corncob itself has a match as bioethanol raw material because it produces an alcohol content of 11% and a risk husk of 9%.
2. Parameters that affect the manufacture of bioethanol such as reducing sugar content, particle size, acid concentration and cellulose content where the highest corncob reducing sugar content is 28.61%, optimal particle size is 200 mesh, optimal acid concentration is 20% HCl and cellulose content is corncob by 30-40%.
3. The average pouring point test results before mixing bioethanol is 43.30C then the oil sample is mixed with bioethanol with a mixing ratio of 5%, 10%, 15%, 20%, and 50% of the sample weight

so that the point temperature has decreased pour at the highest mixing ratio 100% to 40 °C or a difference of 3.3 °C.

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