The Development of Bioethanol from *Iles-Iles* Tubers Using Hydrolyzed Fermentation Method for Alternative Fuels

Agus Sulistiawan[1], Denny Nurdiansyah [2]
[1]*Nahdlatul Ulama Sunan Giri University, Department of Mechanical Engineering, Bojonegoro*
[2] *Nahdlatul Ulama Sunan Giri University, Department of Statistic, Bojonegoro*
[1]*Agus.dmc354@gmail.com*, [2]*denny.nur@unugiri.ac.id*

Keywords: Fermentation; Bioethanol, Hydrolysis, *Iles-iles* tubers.

Abstract: Along with the increasing usage of fuel oil (BBM), namely gasoline, kerosene and diesel fuel, as a result, sources of fuel oil began to decrease. This condition urges the discovery of alternative energy that is converting *iles-iles* tubers containing ethanol for alternative fuels, which are very possible to be developed and important to be observed. *Iles-iles* tubers (Amorphophallus muelleri) are plants that live in tropical and subtropical areas including Indonesia, one of which is in Bojonegoro area. Experimental research in the laboratory was carried out in two stages, those are hydrolysis and fermentation. The aim of the study was to obtain better ethanol content of iles-iles tubers by hydrolysis of distillation fermentation, by varying the ratio of sulfuric acid in 7 ratios 4:1, 5:1, 6:1, 7:1, 8:1, 9:1, and 10:1. The volume / weight (v / w) of the dried *iles-iles* tubers were crushed and sieved. The *iles-iles* powder was hydrolyzed using H2SO4. From the results of bioethanol characteristic test on *iles-iles* tubers as an alternative fuel development, the most optimal obtained after the hydrolysis process is a sample ratio of 5:1, which had the characteristics of a total sugar content 6.72%, fermentation with 9 grams of yeast in each sample in 6 days' fermentation and distillation process. Bioethanol made from *iles-iles* tubers has good potential to be developed because the quality of the bioethanol produced reaches 62% in the initial distillation, has a density value of 0.8107 gr/ml in accordance with the bioethanol quality standard, which is 0.8215 gr/ml at a ratio of 5:1 of the raw material which has a viscosity value of 1.0780 cP, and has a calorific value of 2.121 Kcal/kg bioethanol quality standard and a flash point value of 7.0 0C.

1 INTRODUCTION

Along with the increasing usage of fuel oil (BBM), namely gasoline, kerosene and diesel fuel, as a result, sources of fuel oil began to decrease. This condition urges the discovery of alternative energy that is converting *iles-iles* tubers containing ethanol to become alternative fuels which are very possible to be developed and important to be observed. Along with few discoveries of renewable energy while energy sources derived from petroleum will increasingly run out due to continuous usage without being balanced with the discovery of renewable energy (Ika, 2009). This ethanol will later be used as a renewable energy source and as an alternative energy source. Ethanol itself is the result of fermentation of sugars derived from tuber starch with the help of bacteria to become alcohol, starch, or cellulose (Mariskian M. Sadimo, 2016)

Iles-iles tubers are widely grown in Indonesia. The plant -which is also called *Amorphophallus sp*- is commonly used as one of the ingredients for beauty or food products. Moreover, through the hydrolysis process, *iles-iles* tubers can be used as bioethanol. Meanwhile, to convert starch into sugar requires a hydraulic process. Hydrolysis of starch itself is the process of converting starch into glucose with the help of acid (Primata Mardina, 2014). One of the acids that can be used is sulfuric acid or can be called H2SO4, while the yeast Saccharomyces Cereviceae is used to convert sugar into ethanol. Fermentation is a process of glucose breakdown which produced ethanol and CO2, that occurs under anaerobic conditions through the activity of microorganism species called yeast.

2 REVIEW OF RELATED LITERATURE

Some concepts that is used in supporting this research include the followings:

2.1 Bioethanol

Bioethanol is a compound derived from plants in the form of starch which is converted into glucose then fermented and produced ethanol and carbon dioxide (CO2) (Huda, 2017). Bioethanol is very easy to be obtained from plants that contain high starch such as rice, corn and tubers. Apart from plants that contain high starch, it can also be obtained from cellulose materials such as wood, or agricultural waste such as rice straws. It can also be obtained from plants that contain high saccharin such as sugarcane or fruits.

2.2 Fermentation

Fermentation is a glucose breakdown process that produces ethanol and CO2, which occurs under anaerobic conditions through the activity of a species of microorganism called yeast (Emmanuela M. Widyanti, 2016). In the fermentation process, it does not require oxygen so the fermentation process is called anaerobic fermentation. During fermentation, glycolysis occured. In glycolysis, one glucose molecule is converted into 2 pyruvate molecules. In fermentation, these pyruvates are converted to ethanol.

2.3 Hydrolysis

Hydrolysis is the decomposition of a substance in a chemical reaction caused by water. Hydrolysis depends on the chemicals substance, solubility, acidity, and redox of each compound (Irma Ramadhani Febriaty, 2016). Hydrolysis of starch itself is the process of converting starch into glucose with the help of acid, one of the acids that can be used is sulfuric acid or can be called H2SO4. Sulfuric acid is used because the price is more affordable compared to others. The hydrolysis process itself can be carried out in two methods, namely chemical and enzymatic methods. This chemical process is carried out more often because it is cheaper than the enzymatic process. Meanwhile, the enzymatic method in terms of time is relatively slower than the chemical method (Fika Herlina Moede, 2017). Therefore, a faster process will make it easier to do the research.

2.3.1 Iles-iles tubers

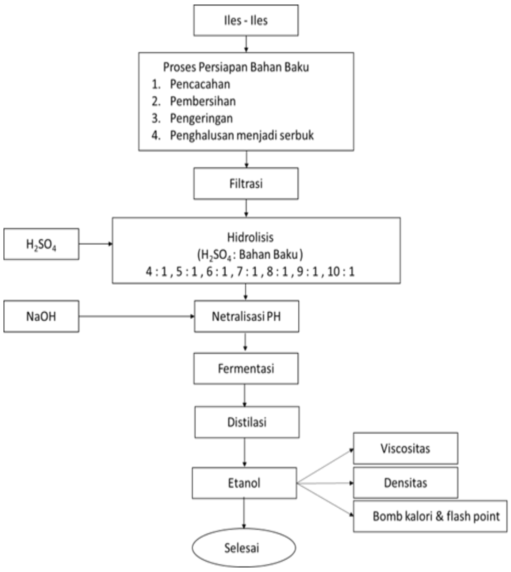
Iles-iles tubers (Amorphophallus muelleri) are plants that live in tropical and subtropical areas including Indonesia, one of which is in Bojonegoro area. This *iles-iles* tuber contains starch, in addition of being used for food, *iles-iles* is also used for beauty products (Supriati, 2016). This *iles-iles* plant contains starch so it can be used as an ingredient for bioethanol making. In addition, *iles-iles* tubers can also grow and develop in Bojonegoro with its climatic and weather conditions. *Iles-iles* tubers can also be cultivated and have a fast harvest period so they are very suitable to use without affecting the community as a food product. *Iles-iles* is included as a species that is rarely cultivated so it is often found in the wild nature (Supriati, 2016). In *iles-iles* tubers (A Amorphophallus muelleri) the highest glucomannan is obtained in about 67% (Siswanti, 2013)

3 METHODS

The experimental research was done in the laboratory. This study was conducted in two stages, those are hydrolysis and fermentation. The aim of the study was to obtain better ethanol content from *iles-iles* tubers than if it was carried out in one step. *Iles-iles* tubers were peeled then cut into small pieces. Next, the *iles-iles* tubers were washed using running water. After being washed, they were dried using the heat of the sun. The dried *iles-iles* tubers were then mashed using a blender until smooth and sieved using a manual sieve. After that, *iles-iles* tubers powder were dried in the sun for 5 hours then grounded again and filtered using a finer cloth filter. The *iles-iles* starch was then hydrolyzed using H2SO4.

After that the effect of *iles-iles* tubers with the ratio of sulfuric acid was treated in 7 steps 4:1, 5:1, 6:1, 7:1, 8:1, 9:1, and 10:1. Volume / weight (v / w). [12]. Next, the starch was weighed as much as 15 grams for 7 times. Furthermore, H2SO4 was added into the starch according to the ratio. Then, it was hydrolyzed at 100°C for 2.5 hours. At this stage, the highest sugar yield is achieved. Then NaOh was added as neutralizer. Once it is neutral, the results of the hydrolysis were added with distilled water until it reaches a solution of 500 ml then the filtered filtrate can be measured with a hydrolyzed sugar spectrometer and fermented after it is given 4 grams of urea and ammonium sulfate. Next, it was heated for 2.5 hours and given 9 grams of baker's yeast (Saccharomyces cereviseae) into a sugar solution that has been hydrolyzed and let it stand for 6 days in closed state.

After fermentation, the results were filtered to separate the pulp, then a distillation process is carried out to separate the bioethanol from the water at a temperature of 80°C, then the distillation results are tested for the bioethanol content produced by analyzing the flash point and heating value.

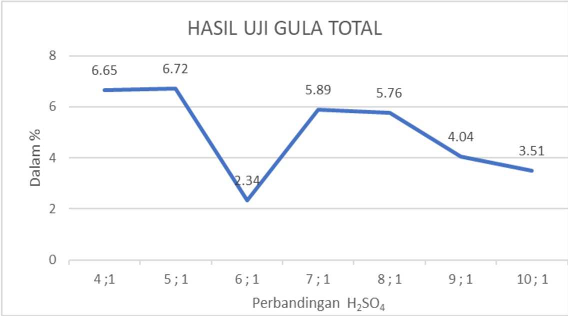


Picture 3.1. Schematic of Bioethanol Making Process from *iles-iles*

4 RESULTS AND ANALYSIS

No	(H_2SO_4) ml : (Iles – Iles) gr	PH
1	A (4 :1)	2.3
2	B (5 :1)	2.1
3	C (6 :1)	2.0
4	D (7 :1)	1.8
5	E (8 :1)	1.6
6	F (9 :1)	1.4
7	G (10 :1)	1.3

The method of data analysis in this research is using descriptive quantitative statistical methods. Descriptive statistical method is a statistic by collecting information or data from each result of changes that occurs through direct experiments. Descriptive statistics explain how the data is presented, the data will be presented in the form of tables and graphs accompanied by a distributive explanation.



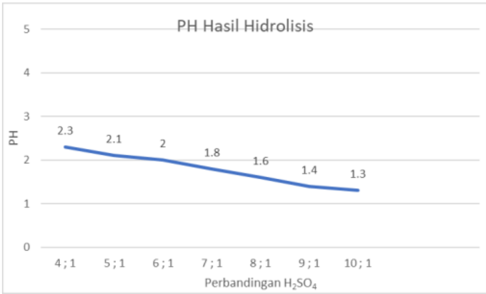
4.1 PH Test Result

Hydrolysis and Neutralization of PH

In the hydrolysis stage, dried *iles-iles* starch is dissolved with acidic liquid, that is H2SO4 with a concentration of 10%, the adding of the acid is in accordance with the ratio of each sample, then after adding the acid solution, a reflux process is carried out at a temperature of 1000 ℃ for 2,5 hours. This process aims to get the total sugar results in each sample.

In this process, the pH produced in each sample varies that can be seen in the table below, this neutralization process uses alkaline liquid, NaOH with a concentration of 5% and after neutralization, Aquades is added until the solution reaches 500 ml.

Table 4.1 pH Hydrolysis Results of *iles -iles* in each sample



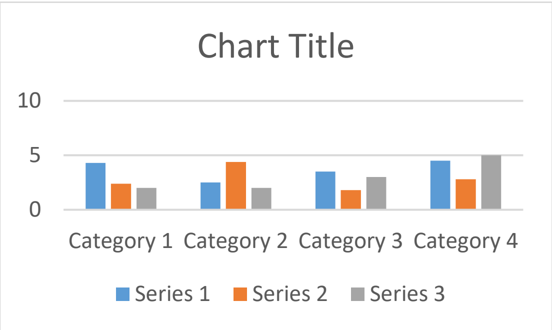
Picture 4.1 Graph of pH hydrolysis results in each comparison

4.2 Total Sugar Test Result

In this research, after preparing the raw materials, the next process is the hydrolysis process that is adding acid liquid H2SO4 according to the ratio of 4:1 , 5:1, 6:1, 7:1, 8:1, 9:1, 10:1. In this hydrolysis process, the temperature used was 1000℃ for 2.5 hours and after the hydrolysis process was completed, the hydrolysis solution was cooled down and the pH was neutralized with NaOH solution. After the *iles-iles* solution resulting from hydrolysis was neutral, it was added with distilled water until the solution reaches 500 ml then a spectrometer test is carried out to determine total glucose levels in each test sample, the results of the total glucose test obtained as follows:

(H_2SO_4) ml : (Iles – Iles) gr	Kadar Gula Total (%)
A (4 :1)	6.65
B (5 :1)	6.72
C (6 :1)	2.34
D (7 :1)	5.89
E (8 :1)	5.76
F (9 :1)	4.04
G (10 :1)	3.51

Table 4.2 Total Sugar level in each comparison



Picture 4. 2 Graph of total sugar level in each comparison

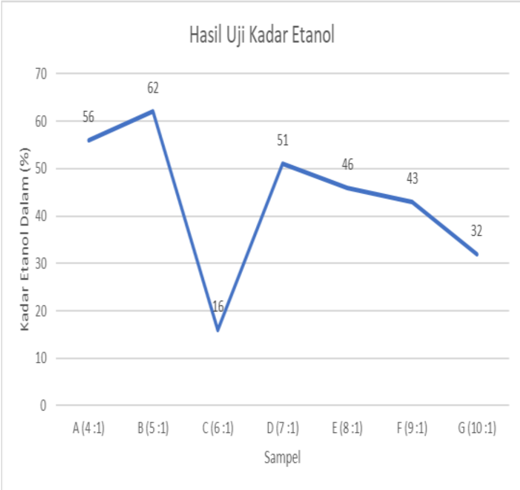
Picture 4.2 shows that the ratio of H_2SO_4 with iles-iles powder produces various sugar levels. In the sample with a ratio of 4:1, it produces 6.65% glucose, then in a 5:1 ratio it produces the highest glucose, which is 6.72%, while in a 6:1 ratio the lowest glucose is 2.34%, then a 7:1 ratio results in 5.89% glucose not that significant when it is compared to the results of a comparison of 8:1, which produces glucose by 5.76%, then at a comparison of 9:1 and 10:1, glucose is obtained at 4.04% and 3.51%.

4.3 Ethanol Level Test Result

After 6 days' fermentation process with the addition of Yeast (Saccharomyces cerevisiae) as much as 9 grams into the sample with a ratio that had the highest glucose content of 5:1 with a glucose level of 6.72%, the starch solution was then distilled to get ethanol. After that, the ethanol from this distillation was tested to determine the Ethanol content, and at this stage it was found that the highest ethanol content was 62%, while the lowest ethanol content is obtained at a ratio of 6:1, which is 16%.

Table 4.3 Total Ethanol Content in Each Comparison

No	Code	Ethanol Content (%)
1	A (4 : 1)	56
2	B (5 : 1)	62
3	C (6 : 1)	16
4	D (7 : 1)	51
5	E (8 : 1)	46
6	F (9 : 1)	43
7	G (10 : 1)	32

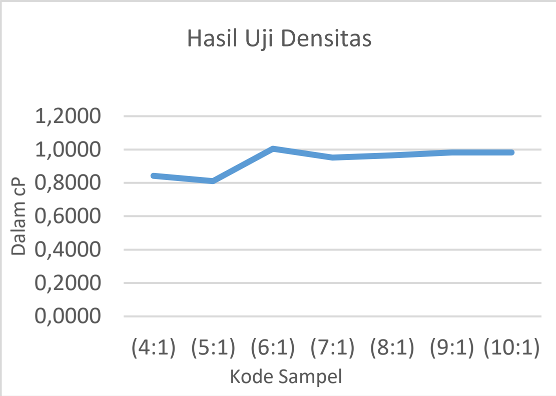


Picture 4.3 Graph of ethanol content in each comparison

4.4 Density Test Result

From the density test using a 10ml pycnometer was resulted several density values. The analysis od density was based on the maximum bioethanol quality requirements of 0.8215 gr/ml. Based on the density value produced in this density test, it meets the bioethanol quality requirements. It can be seen in the table below that there are differences in the results of the density values in each of the samples tested. The smallest density value was obtained in sample B (5:1) of 0.8107 gr/ml. While the highest density value was obtained in sample C(6:1) of 1.0050 gr/ml

Table 4.4 Density Values in Each Comparison



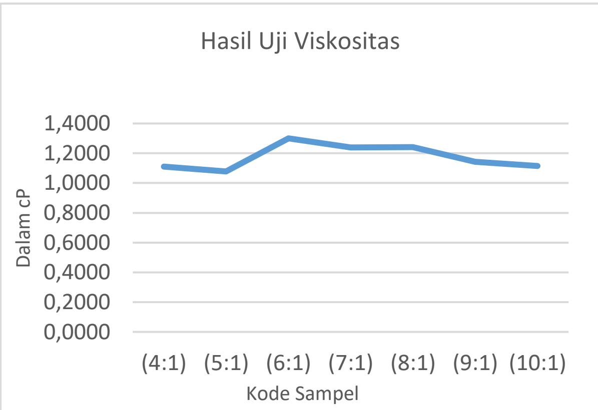
Picture 4.4 Graph of Density Value in Each Comparison

4.5 Viscosity Test Result

This stage of viscosity testing was carried out using an Ostwald viscometer. From the observation and calculation of viscosity with the Ostwald viscometer, which was carried out by measuring the time required for each sample of ethanol liquid to pass through the capillary tube from point A to B when flowing due to the influence of gravity, the highest viscosity value was 1.2414 cP in the 8:1 sample. and the lowest was in the 6:1 sample, which was 1,0002 cP.

Table 4.5 Viscosity Value in Each Comparison

No	Sample	Code	Viscosity (cP)
1	P. 27	A (4 : 1)	1,1103
2	P. 28	B (5 : 1)	1,0780
3	P. 29	C (6 : 1)	1,3002
4	P. 30	D (7 : 1)	1,2398
5	P. 31	E (8 : 1)	1,2414
6	P. 32	F (9 : 1)	1,1433
7	P. 33	G (10 : 1)	1,1143



optimal, approaching the fuel standard allowed, in the 5:1 comparison sample which had the highest ethanol content reaching 62% with LEL-ITS/BK testing

No	Sample	Code	Density gr/mL
1	P. 20	A (4 : 1)	0,8432
2	P. 21	B (5 : 1)	0,8107
3	P. 22	C (6 : 1)	1,0050
4	P. 23	D (7 : 1)	0,9527
5	P. 24	E (8 : 1)	0,9653
6	P. 25	F (9 : 1)	0,9826
7	P. 26	G (10 : 1)	0,9835

method, calorific value yields 2,121 cal/gr, which is close to the bioethanol quality standard set by the National Standards Agency (BSN) at a maximum calorific value of 5,000 kcal/kg and in flash point testing using the ASTM D92 method, the experiment performed shows 7.0 ℃

4.7 Discussion

From the experimental results above, there are various data obtained, starting from the total sugar content, pH level, ethanol content, density value, viscosity value and the value of the calorie bomb and flash point. All of those results were discussed according to the related literature which relevant to the experiment. Here are the discussions:

4.7.1 PH Value

After the process of adding acid liquid H2SO4 then continued with the hydrolysis process for 2.5 hours at a temperature of 100℃, the solution was neutralized and once it became neutral it was measured with a pH meter showing that the 4:1 sample had a pH value of 2.3 while the 10:1 sample yielded a pH value of 2.3. This pH 1.3 indicates that the more acidic liquid is given, the lower the resulting pH or the more acidic the solution (Betrisia, Novia, Dahrul, 2017)

4.7.2 Total Sugar Content

In the hydrolysis process using strong acid H2SO4 at a temperature of 100℃ with a predetermined ratio in each sample, it produced various total sugar, which was the highest in the 5:1 sample of 6.72% and the lowest in the 6:1 sample which was 2.34%. The addition of acid with a higher hydrolysis temperature on starch hydrolysis resulted an increase in the total sugar produced (Ni Kadek Ariani Dewi, 2018)

4.7.3 Ethanol Content

The lowest ethanol content was found in the 6:1 sample where in the sample, the sugar content produced was only 2.34% which produced 16% ethanol while the highest sugar content was found in the 5:1 sample of 6.72% by producing 62% ethanol. Thus, the more reducing sugar produced is utilized by Saccharomyces cerevisiae cells, the higher the ethanol concentration produced (Santi Andriani Putri, Fajar Restuhadi, Rahmayuni, 2016)

4.7.4 Density

The highest density value in sample 6:1 is 1.0050 gr/mL with a pure ethanol value of 16%, while the lowest is in sample 5:1, which is 0.8107 gr/mL. It occurs because the sample has the highest bioethanol content of 62 %. The density value of bioethanol decreases with increasing alcohol content in bioethanol (H.S. Tira, 2018)

4.7.5 Viscosity

The highest viscosity value was in the 6:1 sample that is 1.3002 cP with 16% ethanol content while the lowest was in the 5:1 sample that is 1.0780 with 62% ethanol content. These results indicate that the higher the alcohol content in bioethanol, the lower the viscosity value. (H.S. Tira, 2018)

4.7.6 Calorie Bomb and Flash point

The calorific value of fuel has a calorific value that can determine the amount of fuel consumption at each unit time. The higher the heating value indicates that the less fuel is used (Sinta Putri Anisa, 2021). Tests using the Bomb Calorimeter instrument showed that the heating value of bioethanol from *iles-iles* tubers resulted in a value of 2,121 kcal/kg.

5 CONCLUSIONS

The development of alternative fuels from *iles-iles* was done through several stages. The process of converting *iles-iles* tubers into bioethanol consists of several stages, those are the stages of chopping, drying, refining, hydrolysis using H2SO4 with various ratios of 4:1, 5: 1, 6:1, 7: 1, 8:1, 9:1, 10:1, fermentation with 9 grams of yeast in each sample with 6 days of fermentation and continued with the distillation process. Bioethanol made from *iles-iles* tubers has good potential to be developed because the quality of the bioethanol produced reaches 62% in the initial distillation, has a density value of 0.8107 gr/ml in accordance with the bioethanol quality standard, which is 0.8215 gr/ml at a ratio of 5:1 of the raw material. It also has a viscosity value of 1.0780 cP, and has a calorific value of 2.121 Kcal/kg bioethanol quality standard and a flash point value of 7.0 0C.

REFERENCES

Betrisia, Novia,Dahrul. (2017). *Korelasi Pengukuran Kadar Asam, Gula, dan pH pada Buah Belimbing, Jeruk, dan Tomat dengan Nilai Kapasitansi Elektrik.* bogor: Library of IPB University .

Emmanuela M. Widyanti, B. I. (2016). Proses Pembuatan Etanol Dari Gula Menggunakan Saccharomyces Cerevisiae Amobil. *METANA.* , Vol. 12(2):31-38.

Fika Herlina Moede, S. T. (2017). Pengaruh Lama Waktu Fermentasi Terhadap Kadar Bioetanol Dari Pati Ubi Jalar KUNING (Ipomea batata L). *Jurnal Akademika Kimia*, Volume 6, No. 2,: 86-91.

H.S. Tira, I. M. (2018). Uji sifat fisik dan kimia bioetanol dari jagung (Zea mays L) . *Dinamika Teknik Mesin 8*, 77-82.

Huda, N. (2017). *Proses Pembuatan Bioethanol.* Bandung : Eksismedia Grafisindo.

Ika. (2009, Maret 27). *www.ugm.ac.id.* Retrieved from www.ugm.ac.id/id/newsPdf/740-pemanfaatan-sumber-energi-terbarukan-atasi-krisis-energi: https://www.ugm.ac.id

Irma Ramadhani Febriaty, H. A. (2016). Perbandingan Metode Hidrolisis Asam Dan Basa Tandan Kosong Kelapa. *Jurnal Kimia Khatulistiwa*, Vol 5(4), halaman 22-28.

I. W. Arnata, D. Setyaningsih, and N. Richana, "Produksi Bioetanol Dari Hidrolisat Asam Tepung Ubi Kayu Dengan Kultur Campuran Trichoderma viride dan Saccharomyces cerevisiae (Ethanol Production From Acid Hydrolysate Cassava Flour with Mixed Culture Trichoderma viride and Saccharomyces cerevisiae)," J. Agritech, vol. 35, no. 04, p. 396, 2015, doi: 10.22146/agritech.9323

Mariskian M. Sadimo, I. S. (2016:). Pembuatan Bioetanol Dari Pati Umbi Talas (Colocasia Esculenta [L] Schott) Melalui Hidrolisis Asam Dan Fermentasi. *Jurnal Akademika Kimia*, Volume 5, No. 2, hal 79-84.

Ni Kadek Ariani Dewi, A. H. (2018). Pengaruh Suhu Dan Jenis Asam Pada Hidrolisis Pati Ubi Talas (Colocasia Esculenta L. Schott) Terhadap Karakteristik Glukosa. *Jurnal Rekayasa dan Manajemen Agroindustri*, Vol. 6, No.4, 307-315.

Primata Mardina, H. A. (2014). Pengaruh Waktu Hidrolisis Dan Konsentrasi Katalisator Asam Sulfat Terhadap Sintesis Furfural Dari Jerami Padi.

Jurnal Konversi UNLAM, Volume 3 No. 2, Oktober, 37-44.

Rb. Katri Anandito, G. Jati Manuhara, and S. Pengajar Jurusan Ilmu dan Teknologi Pangan, "Karakterisasi Edible Film Komposit dari Glukomanan Umbi Iles-iles (Amorphopallus muelleri Blume) dan Maizena," J. Teknol. Has. Pertan., vol. VI, no. 2, 2013

Santi Andriani Putri, Fajar Restuhadi, Rahmayuni. (2016). Hubungan Antara Kadar Gula Reduksi, Jumlah Sel Mikrob Dan Etanol Dalam Produksi Bioetanol Dari Fermentasi Air Kelapa Dengan Penambahan Urea. *Jom FAPERTA*, VOL. 3 NO 2 .

Sinta Putri Anisa. (2021). Produksi Bioetanol Dari Limbah Brem Sebagai Bahan Bakar Alternatif Dengan. *JPTM*, Volume 10 Nomor 02 Tahun , 11 - 20.

Siswanti, R. K. (2013). Karakterisasi edible film komposit dari glukomanan umbi iles-iles (Amorphopallus muelleri) dan maizena. *Jurnal Teknologi Hasil Pertanian,*, Vol. VI, No.2, Agustus.

Supriati, Y. (2016). Keanekaragaman Iles-Iles (Amorphophallus Spp.) Dan Potensinya Untuk Industri Pangan Fungsional, Kosmetik, Dan Bioetanol. *Jurnal Litbang Pertanian.*, Vol. 35 No. 2 juni : 69-80.

Z. S. Osvaldo, P. S. Putra, and M. Faizal, "Pengaruh Konsentrasi Asam dan Waktu Pada Proses Hidrolisis dan Fermentasi Pembuatan Bioetanol dari Alang-Alang," J. Tek. Kim., vol. 18, no. 2, pp. 52–62, 2012