Coffee Husk Highly Available in Ethiopia as an Alternative Waste Source for Biofuel Production

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Abstract— Coffee is one of the top commodities produced and commercialized worldwide, and the processing of coffee generates significant amounts of agricultural waste, ranging from 50% to 60% the weight of the total coffee produced, depending on the type of processing. Coffee husks are the major solid residues from the processing of coffee, for which there are no current profitable uses, and their adequate disposal constitutes a major environmental problem. In Ethiopia 192000metric tons of coffee is Husk cast adrift as by-product per year and there is 134,400 metric ton of coffee husk disposed per year in Jimma area. Thus, in compliance with the concept of sustainable development, innovative techniques and products for the profitable and adequate use of this type of residue are being sought. Several research works presenting proposals for such endeavors have been published in the literature and are reviewed herein. The alternative uses of Coffee Husk are producing bioethanol by biochemical conversion processes (pretreatment, hydrolyses, fermentation and distillation). There were six lab experiments conducted depending on size of the sample and diluted sulfuric acid concentration. From those experiments three of them were depending on three different size (0.5mm,1mm and 2mm).Three of such samples were hydrolyzed by 4%(v/v) $H_2SO_4$and the remained were hydrolyzedby1%(v/v)H_2SO_4.The output is flammable, clear and colorless liquid fuel. The yield of each experiment was calculated and the result was characterized by PH value, UV-spectroscopy, physical and chemical properties. Based on this characterization, from all samples, sample two (S_2) of size 0.5mm and 1 %(v/v) of dilute H_2SO_4 used is optimized. Thus from 100gm of coffee husk powder, 69%of the yield is obtained.

Index Terms—Coffee husk, H_2SO_4, Pretreatment, Hydrolysis, Fermentation, Distillation, Bioethanol, Biofuel, UV-visible spectroscopy.

1 INTRODUCTION

Coffee is one of the world's most popular beverages and important produces. It has grown steadily in commercial importance over the last 150 years. Globally, 25 million small producers rely on coffee for their living. Brazil, Vietnam and Colombia account for more than half of the world's production. The global coffee production per year on average accounts to approximately 7 million metric tons [1]. According to the International Coffee Organization (ICO), output of coffee brew in the 2011-12 seasons is estimated at 130 million bags [2]. The top five countries that produces coffee in 2015/16: Brazil, Vietnam, Colombia, Indonesia and Ethiopia are produces; 2592000, 1650000, 810000, 660000, 384000metric tons respectively. Ethiopia is the fifth country of coffee producer in the world and is considered to be the original habitat of Arabica coffee and Central Africa is reckoned to be the native of robust a coffee. Jimma zone produces about 70% of coffee in Ethiopia [3].

Coffee generates large amount of coffee by-products/residues during processing. Depending upon the method of coffee cherries processing, different residues are obtained. Coffee husks are the major solid residues from the handling and processing of coffee, since for every 2Kg of coffee beans produced, approximately 1kg of husks are generated [4,5]. In Ethiopia 192000metric tons of coffee is Husk cast adrift as by-product per year. Coffee grounds are highly pollutant due to the presence of organic material that demands a great quantity of oxygen in order to degrade. Proposed alternative uses for coffee husks include employing this solid residue as a supplement for animal feed, direct use as fuel, and fermentation for the production of a diversity of products (enzymes, citric acid and flavoring substances), use as a substrate for growth of mushrooms and use as adsorbents [1].

However, considering the high amounts generated, there is still a need to find other alternative uses for this solid residue. Given that, such residue consists mainly of the pulp and hull of the coffee fruit, it presents a high concentration of carbohydrates and thus can be viewed as a potential raw material for bio-ethanol production [6].There is 134,400 metric ton of coffee husk disposed per year in Jimma area. So this project is conducted in this area, in order to convert coffee husk wastes in to bio-ethanol. Thus, bio-ethanol is used for vehicles (car, lorry, bicycles, motors and etc.)

Statement of the Problem: Coffee is an important plant which provides an essential fruit for human being. Jimma is an area which produces coffee mainly as an agricultural product. During the processing of coffee bean to the final product there are a number of wastes generated that are harmful to health of human being (i. e it causes environmental pollution). Some people burn the generated coffee husk, while others disposes it on the field. This is improper way of handling the waste. Large amount of potential that could be utilized from coffee husk cannot be achieved in this way. So producing bio-ethanol from coffee husk is important to solve: environmental pollution problem, reduce cost rise of oil by producing

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bio-ethanol as an alternative fuel [7-10].

The main objective of this thesis is to produce bio-ethanol from coffee husk wastes, to establish fermentation setup to produce bioethanol from coffee husk, to evaluate yield of bioethanol by varying size of sample and diluted sulpheric acid concentration in hydrolysis stage and characterize bio-ethanol produced from coffee husk by different methods. Significance of this research work: Energy is one of the most fundamental parts of our universe. Energy powers our vehicles, trains, planes and rockets. Energy warms our homes, cooks our food. Energy powers machinery in factories and tractors on a farm. All energy sources have an impact on the environment. Concerns about the greenhouse effect and global warming, air pollution, and energy security have led to increasing interest and more development in renewable energy sources such as bio-fuel, solar, wind, geothermal, and hydrogen. In the worldwide economy much focus has been laid on the rising oil price which has become a hot topic. The rising oil price has increased the interest of finding other possible ways to produce fuel.

2 Materials and Method

2.1 Materials

The coffee husk was collected from the area of Jimma town. The coffee husk then dried in oven at 60°C for 48 hours). 98% Sulfuric Acid (H_2SO4) used for pretreatment and hydrolysis the coffee husk. Sodium Hydroxide (NaOH) used to adjust the pH of soluble cellulose and hemicelluloses before fermentation. Yeast extracts Agar used as media preparation. Urea used as media preparation. Dextrose sugar used for media preparation.Yeast (Saccharomyces cerevisiae) used as a catalyst.

2.2 Equipments

The Plastic bags used to collect and transport samples to the laboratory. Crushers used to crush the dried sample. Sieves used for sieve the crushed sample to the particle size of 3mm. PH meter to measure the pH of the hydrolysis before fermentation. **Distillations (simple distillation**) used to separate ethanol from water by boiling point difference. **Vessels** to hold samples and additives for hydrolysis, fermentation and distillation experiments. Centrifuge used to separate the soluble liquid from non-soluble part. Autoclave used for sterilization and hydrolysis. Fermentation and distillation set ups used to ferment and distill respectively.

2.3 Biochemical Conversion Process

The technology of ethanol production from biomass feed stocks consists of several steps, and varies depending on the type of raw materials used. It becomes more sophisticated as the raw materials turn from sugars to starches and cellulosic materials. Unlike starch, the specific structure of cellulose favors the ordering of the polymer chains into tightly packed, highly crystalline structures those are water-insoluble. For production of ethanol from cellulosic feed stocks, four major unit operations are required: Pretreatment, hydrolysis, fermentation, and distillation [7,8].

Pre-treatment: The purpose of pretreatment is to remove lignin, reduce cellulose crystalline, and increases the porosity of the materials. Pretreatment must meet the following requirements: improve the formation of sugar, avoid the degradation or loss of carbohydrate, avoid the formation of by-product inhibitors and must be cost effective. Objective of coffee husk pretreatment (preparation): To reduce level of impurities, to obtain better performance & yield, to reduce scale formation and Lower steam consumption.

Any sugar containing substances can be used as a raw material for alcohol production. The raw material preparation process differs from substance to substance [11,12].

Dilute Acid Hydrolysis: The carbohydrate polymers in lignocellosic materials need to be converted to simple sugars before fermentation, through a process called hydrolysis. Various methods for the hydrolysis of lignocellosic materials have recently been described. The most commonly applied methods can be classified in two groups: chemical hydrolysis and enzymatic hydrolysis. Even though there are many types of hydrolysis types, dilute acid hydrolysis is an easy and productive process and the amount of alcohol produced in case of acid hydrolysis is more than that of alkaline hydrolysis. This process is conducted under high temperature and pressure, and has a reaction time in the processing [13].

Adjustment of PH: Before addition of any microorganism to the diluted hydrolyzed sample, pH of these samples has to be adjusted. Otherwise the microorganism was died in hyper acidic or basic state. A pH of around 5.0 - 5.5 will maintain. The hydrolyzed samples were primarily checked for pH using a digital pH meter. The pH then adjusted to 5.0 - 5.5. When the pH went below 5.0 - 5.5, sodium hydroxide solution was added drop wise to the flask with constant stirring until the pH reaches to a range of 5.0 - 5.5. When the pH went beyond 5.0 - 5.5, concentrated sulfuric acid was added drop wise to maintain the pH in the range.

Fermentation: This is the chemical transformation of organic substance into simpler compounds by the action of enzymes. Originally the term fermentation was used to mean the enzymatic breakdown of carbohydrates in the absence of air. In industrial practice, fermentation refers to any process by which raw materials are transformed by the controlled action of carefully selected strains of organisms into definite products. Louis Pasteur used the term in a narrower sense to describe changes brought about by micro-organisms growing in the absence of air. However, for the cause of this thesis it is a biological method of producing ethanol. The fermentation reaction is caused by yeast or bacteria which need on simple sugars. The glucose produced from the hydrolysis described above is fermented with yeast to produce ethanol [14-16].

Distillation: is a method of separating liquid mixtures based on differences in their volatilities in a boiling liquid mixture. Distillation is a unit operation, or a physical separation process, and not a chemical reaction. It is broadly defined as the separation of more volatile components from less volatile components by a process of vaporization and condensation. The term distillation is properly applied only to those operations where vaporization of a liquid mixture yields a vapor phase containing more than one constituent and desired to recover one or more of these constituents in a nearly pure state. The process of distillation is affected based on the relative volatilities of the

liquids in the mixture and taking advantage of their different boiling point. Distillation is the most widely used method of separating liquid mixture and is at the heart of separation process in many chemical and petroleum plants. The basic requirement of separation of components by distillation is that the composition of the vapor be different from the composition of the liquid with which it is in equilibrium. Theoretically, distillation can never yield a component in absolutely pure form, although practically the product may be made of any purity that is economically warranted. In distilleries there is certain limit of alcohol production during fermentation. Usually the alcohol obtained by fermentation is within the range of 6.0 – 12% by volume. The alcohol obtained from fermentation process should be strengthened to the desired quality to be used for specific purpose. A distillation system is used to separate the bioethanol from water in the liquid mixture. All distillation experiments will be carried out at a temperature of 85°C and a distillation time of 3 hours by rotary evaporator.

2.4 Experimental Procdure

Coffee husk: The coffee husk was collected from the area of Jimma town from coffee been extracting machine that was dumped as waste material in land. The coffee husk then dried in oven at 60°C for 48 hours). The dried coffee husk was placed in mortar and the maximum particle sizes of 3 mm. The sample of larger particle size of greater than 3 mm was grinded over and over again until all particle size became 3 mm or less than 3mm. The sample that was acquired had to be prepared and conditioned for pretreatment, hydrolysis, fermentation and distillation. Sample preparation process include: drying, manual size reduction (mortal grinding) and sieving after the samples were collected from Jimma area, disposed as waste material during coffee bean processing. After drying, each of the samples is milled separately. The sample was kept at low temperature until the next stage of experiment. Grinding of coffee husk powder form increased the surface area of the sample which enhanced the contact between hemicelluloses and cellulose with dilute acid to reduce cellulose Crystallinity.

The coffee husk was grinded in different sizes. The experiment has been conducted in three different sizes of coffee husk powder (fig.1). Each of different sizes of coffee husk experiments should be hydrolyzed in two different concentration of sulpheric acid.

Fig.1. Grinded Coffee husk powder for pre-treatment.

Experiment 1:

- The size of coffee husk powder is equal to 0.5mm
- 33.5 ml of 1% (v/v) diluted sulfuric acid was added to the insoluble component which come from pretreatment steps

Experiment 2:

- The size of coffee husk powder is equal to 0.5mm
- 8.5mlof 4% (v/v) diluted sulfuric acid was added to the insoluble component which come from pretreatment steps.

Experiment 3:

- The size of coffee husk powder is equal 1mm.
- 33.5ml of 1% (v/v) diluted sulfuric acid was added to the insoluble component which come from pretreatment steps.

Experiment 4:

- The size of coffee husk powder is equal 1mm
- 33.5ml of 4% (v/v) diluted sulfuric acid was added to the insoluble component which come from pretreatment steps.

Experiment 5:

- The size of coffee husk powder is 2mm.
- 33,5ml of 1% (v/v) diluted sulfuric acid was to the insoluble component which come from pretreatment steps.

Experiment 6:

- The size of coffee husk powder is 2mm.
- 1 liter of 4% (v/v) diluted sulfuric acid was added to the insoluble component which comes from pretreatment steps.

In general, "Experiment 1" and "Experiment 2" used the same size of coffee husk powder (equal to 0.5mm), but different concentration of dilute sulpheric acid used in hydrolysis stage. "Experiment 3" and "Experiment 4" the same size of coffee husk powder equal to 1mm, but different concentration of dilute sulpheric acid used in hydrolysis stage. "Experiment 5" and "Experiment 6" used the same size of coffee husk powder is equal to 2mm, but different concentration of dilute sulpheric acid used in hydrolysis stage.

Therefore the following procedure is common for all six experiments except size of the sample and concentration of dilute sulpheric acid needs for hydrolysis

Pre -treatment of coffee husk: Pretreatment must meet the following requirements: improve the formation of sugar, avoid the degradation or loss of carbohydrate, avoid the formation of by-product inhibitors and must be cost effective.

The main purpose for pretreatment are to: Destroy lignin shell protecting cellulose and hemicelluloses, Decrease crystallinity of cellulose, Increase porosity, and Must break this shell for enzyme to access substrate

Procedure for pretreatment:

- 100g of grinded coffee husk powder was placed in to 2000 ml conical flasks.
- Then, 1000ml of distilled water was mixed with powder
- The conical flasks capped with the help of rubber plugs.
- The sample was placed in Autoclave (fig. 2) at a temperature of 121 °C for 15 minutes
- After finishing the given pretreatment time and temperature, the sample in autoclave and allowed to cool

and separate soluble from the insoluble portion. The soluble portion was hydrolyzed in the next steps and put the soluble solution in another 2000ml conical flask.

Fig. 2. Autoclave for the pre-treatment of coffee husk.

Procedures for Acid Hydrolysis:

- 1 liter of 0.5 %(v/v) diluted sulfuric acid was added to the insoluble component from pretreatment steps and soaked for 24hr.
- The coffee husk was then hydrolyzed in the reactor at 121 ^{o}C for 15 min.
- After hydrolysis, neutralized with 10 M NaOH until the pH became around 7.
- Separate the solid particles from the liquid in the hydrolyzed sample by centrifugation (to remove the non-fermentable lignin portion).
- After separate the solid part, the solid part was washed with distilled water for two times. The washing was performed in order to extract all soluble sugars from the solid coffee husk material.
- The soluble component was mixed with the previously filtered solution from the pretreatment step for the next procedure.

PH Adjustment:

- Pretreated and hydrolyzed solution was mixed, filtered, and checked for pH using a digital pH meter. The pH then adjusted to 5.0-5.5.
- Mix samples (pretreated and hydrolyzed) were acid hydrolyzed, so it needs highly basic solution to bring the pH in the range of 5.0-5.5.
- Sodium hydroxide solution was added drop wise to the other flask with constant stirring until the pH reaches to a range of 5.0-5.5.
- Since the pH goes beyond 5.0-5.5, concentrated sulfuric or hydrochloric acid was added drop wise to maintain the pH in the range.

Media Preparation:

Chemicals for media preparation for fermentation process are: For preparing 100 ml media - Sugar (Dextrose) = 10gm, Yeast extract = 0.2gm, Urea = 1.0gm, Make up water = 100 ml, and yeast, Saccharomyces cerevisiae = 0.5gm.

Procedures in Media Preparation:

- To the above 100ml media, 0.5gm of yeast, Saccharomyces cerevisiae was added in a 250 ml conical flask.
- The conical flasks were properly covered with aluminum foil.
- The conical flask was placed in a shaking incubator for 24 hours, at temperature of 30 ^{o}C and 2000rpm.

FermentationProceture:

Major processing steps in alcoholic fermentation (fig. 3) are: Raw material (substrate) preparation, Yeast propagation (inoculums preparation), and Final fermentation.

- The sample was conditioned at temperature of 30^{o}c before fermentation step was started.
- The adapted media with the proportion of 1:10 to the soluble sample wasAutoclave set at 30 ^{o}C and 200 rpm and then mixed the prepared sample with the media prepared into the autoclave using sterilized funnel.
- **T**he parameters of fermentation i.e. fermentation time, yeast concentration (yeast proportion) and fermentation temperature were set to be at 72 hour, 10% (with the proportion of 1:10 that was the prepared media and sample respectively) and 30 ^{o}C respectively. And after 72 hours of fermentation, the samples were taken out and distilled.

This kind of fermentation setup has many advantages such as: It is important to keep equipment safety by appropriate removal of carbon dioxide, It is simple to decide whether fermentation time is reached or not by looking the distilled water only, and The setup is exactly an aerobic process that gives the maximum yield.

Fig.3. Fermentation process setup.

Distillation:

Distillation is the last step in the production of ethanol from coffee husk experiments. It is the Purifications steps. Distillation is the method used to separate two liquid based on their different boiling points. However, to achieve high purification, several distillations are required. In this experiment separation are used by simple distillation at a temperature of 85 ^{o}C for 3hrs. Finaly, the amount of six samples are measured.

3 RESULT AND DISCUSTION

The process consists of four parts: pretreatment to remove lignin, reduce cellulose crystalline, sterilize the coffee husk and increase the porosity of the materials, dilute acid hydrolysis and fermentation to produce ethanol, distillation to remove the ethanol. After following the above series of procedure, the experimental outcomes of those particular results are measured to know the yield of ethanol concentration. There were six experiments were conducting by varying size of sample and diluted sulfuric acid concentration. The amount of prod-

uct obtained for each sample was measured and shown in table 1.

Table 1: Bio-ethanol product yield for different seze of the particle.

Samples	Size of sample (mm)	H_2SO_4 %(v/v)	Amount of bioethanol obtained (g)	Yield (%)
S1	0.5	4	33.3402	65.40
S2	0.5	1	35.2659	69.00
S3	1	4	27.5903	54.00
S4	1	1	29.9014	58.50
S5	2	4	25.2821	49.47
S6	2	1	27.0711	53.00

3.1 Yield analysis of different particle size and acid concentration.

The size of the sample has great effect on bio-ethanol production. If the size of sample increases the amount of ethanol production decreases. This is due to that, some portion of cellulosic portion is not changed to sugar or glucose. The results represented in fig.4and 5for different concentration of acid. Ths graphical representation briefly indicates that for increasing of sample size, the yield be decreasing.

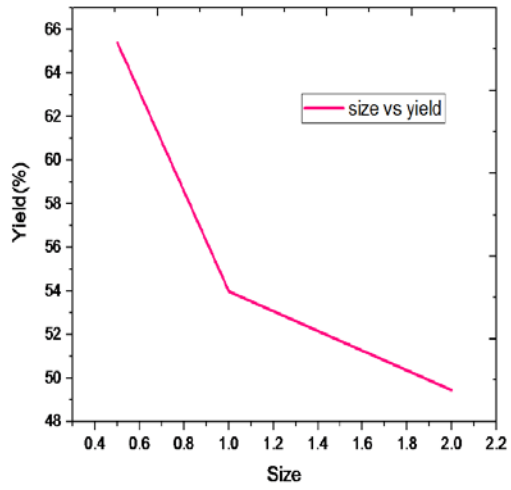
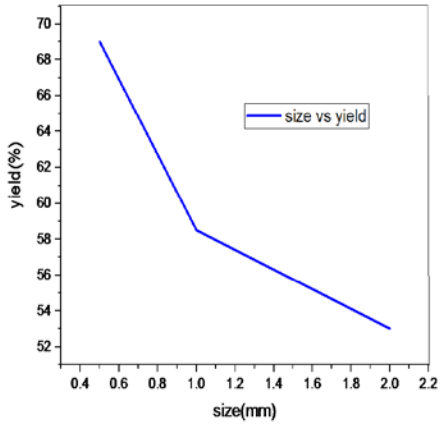


Fig.4. Bio-ethanol yield for various size of particle with 4 % (v/v) H_2SO_4 concentration.

Fig. 5. Bio-ethanol yield for various size of particle with 1 % (v/v) H_2SO_4 concentration.



3.2 Effect of Sulfuric Acid Concentration

Diluted sulfuric acid concentration has less effect on the bio-ethanol production from coffee husk with relative to the size of the sample. Since the concentration of diluted acid increases, the sugar content in the coffee husk decreases.This result in the decrease of bio-ethanol yield. As illustrated in figure 5 for 1%(v/v)H_2SO_4, the yield of sample two (S2) is 69% and the yield of(a)4 %(v/v)H_2SO_4 used for same size of sample is 65.402% . Thus, the increasing of diluted sulfuric acid concentration directly affect the yield of bio-ethanol.

Since the concentration of diluted acid increases, the sugar content in the coffee husk decreases. Due to this suger degradation, the bio-ethanol yield decreases.

3.3 Characterization

Characterization of the laboratory product (i.e,bio-ethanol) is important to determine whether the product which obtained is similar or different in physical or chemical properties of those which are already produced in commercially. UV Spctroscopy result of all sample wasshown in fig.6.

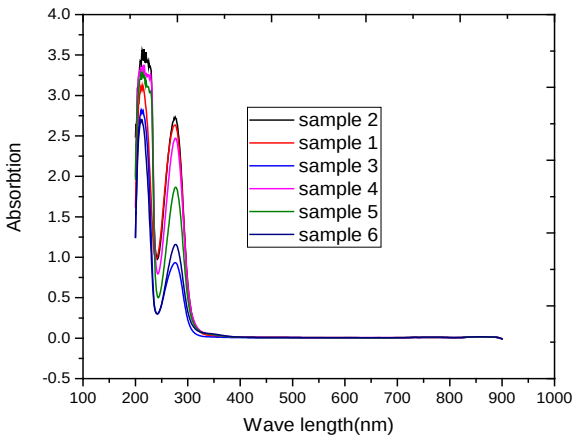


Fig.6. UV- Spectroscopy of Bio-Ethanol

UV spectrocopy used to determine the alcohol content of liquid solution. It is used to determine the alcohol content of liquid solution. Is most accurate and easily determine the concentration of ethanol. From the figure below illustrates absorption versus wave length. The instrument used in ultraviolet-

visible spectroscopy is called a UV/Vis spectrophotometer. The basic parts of a spectrophotometer are a light source, a holder for the sample, a diffraction grating in a monochromatic or a prism to separate the different wavelengths of light, and a detector.

From the fig. 6 illustrates absorption versus wave length. The plot of (S_2) indicates the maximum absorbtion occur on wave length of 200nm relative to others. Concentration is directly proportional to absorbition. Thus, the concentration of ethanol is directly depends on dilute acid concentration and size of the sample. The graphical plot shows that both effect of size and acid concentration in one X-Y plane. Thus, the UV spectroscope simply shows that, relationship of concentration and wave length depending on the size of sample and acid concentration.

The pH value is the one criterion to distinguish if the product is in its range or out of the range. The optimum value PH of the commercial ethanol from 5 to 5.5. table 2. Illustrate the pH value of bio-ethanol.

Table 1: The pH value of Bioethanol from different sample.

Samples	Size of sample(mm)	%(v/v) H2SO4	pH value
S_1	0.5	4	4.3
S_2	0.5	1	5.1
S_3	1	4	5.3
S_4	1	1	7.2
S_5	2	4	6.8
S_6	2	1	5.4

Commercials ethanol reacts with chromic acid, the color changes to pink. Then the reaction expressed above reacted with98% H_2SO_4, the color changes from pink to green. Bioethanol produced from coffee husk also reacted with chromic acid, the color changed to pink. And then this reaction mixed with 98%of H_2SO_4, the color changed to green.

4 Conclusion

Generally, coffee husks are the major solid residues from the handling and processing of coffee. In Ethiopia 192000metric tons of coffee is Husk cast adrift as by-product per year. Jimma produce 70% of coffee in Ethiopia .From the 2kg of coffee fruit 1kg coffee husk will be produced. To precede the fusibility of coffee husk, six experiments were conducted within their size in the laboratory. In addition to varying their size, through the time of hydrolysis, concentration of dilute acid varied to each of the sample (i.e. 1%or 4%) to break the lignocelluloses to simple sugar. But the one which was hydrolyzed with 1% of dilute sulfuric acid with size of 0.5mm of coffee husk powder was optimized with relative to others. From 100g of coffee husk powder 69% of yield (bioethanol) obtained from the sample two (S2) of size 0.5mm.Bio-ethanol which produced in the laboratory was characterized by different methods like, PH meter, UV spectroscopy, and size and dilute acid concentration. When UV spectroscope was used for characterization from absorption versus wavelength, absorbition is directly proportional to concentration of bioethanol.

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