

formulation of bioethanol

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Formulation of Bioethanol From Pineapple Skin Waste and Applied as Wax Inhibitors

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Abstract. The problem of paraffin wax deposition is one of the problems that occurs in the oil industry which causes blockages during the oil production process. To prevent this problem, you can use a chemical method is injecting wax inhibitors. one of the effective wax inhibitors is solvent. So this paper aims to produce bioethanol solvent through biomass engineering, namely pineapple skin waste. For the production of bioethanol through several methods, namely pretreatment, hydrolysis which aims to convert cellulose into glucose with HCl acid, fermentation which aims to convert glucose into bioethanol with the help of *saccharomyces cerevisiae* yeast and finally distillation. The result in the hydrolysis process with concentrations of HCl 1M, 2M, 3M, 4M and 5M produces glucose 7 Brix, 16 Brix, 20 Brix, 21 Brix and 24 Brix. And with variations in heating time of 1 hour to 5 hours, the optimum glucose was obtained at 3 hours of 24 Brix. The results of fermentation with a variation of 1 day to 6 days, the optimum bioethanol content in fermentation for 3 days is 18%. 18% bioethanol is mixed into waxy crude oil with a pour point value of 43°C, and the result is able to reduce the pour point is 3°C. To increase the decrease in the pour point of bioethanol, it is blend with a toluene, and the result is to reduce the pour point value by 8°C. So it can be concluded that mixing bioethanol and toluene as a solvent can inhibit paraffin wax deposition

Keywords : Wax, Solvent, Bioethanol, Pour point.

1. Introduction

Petroleum is a world energy source which is grouped into four compounds, namely Aliphatic, Aromatic, Naphthenic and Olefin [1]. Paraffin is part of an aliphatic compound with a carbon atom number of 18-75 [2]. Paraffin wax deposition and crystallization is one of the most common flow assurance problems in the oil and gas industry worldwide both offshore and onshore [3], [4]. The



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crystallization and deposition of paraffin wax occurs due to a decrease in temperature so that it can limit the flow of oil which causes blockages during fluid production [5]. Therefore, this problem must be overcome to prevent the oil and gas industry companies from disadvantage is caused the problem.

There are several solutions to prevent crystallization and wax deposition, including mechanically, thermally and chemically or a combination of these methods [6]. One chemical that can be used is a solvent. Solvent is a wax inhibitor that can change the rheology of paraffin so that oil and gas transportation is easier [7]. Bioethanol is one of the solvents that can reduce the pour point value of crude oil containing wax paraffin a[8]

Bioethanol can be made from various materials which are generally divided into three, namely ingredients containing sucrose (sugar cane, coconut juice, sweet sorghum), starch (corn, potatoes, wheat), and lignocellulose (wood, grass, fruit skin) [9]. Pineapple skin is a lignocellulose waste containing high cellulose and hemicellulose, based on these contents pineapple peel can be processed into bioethanol by pretreatment, hydrolysis, fermentation and distillation methods [10], [11]. In addition, pineapple is one type of fruit that is widely found in Indonesia. Based on the 2019 Central Bureau of Statistics, pineapple fruit production in Indonesia reached 1,805,506 tons. This production increased from the previous year, namely 2017. The increase in pineapple fruit production will produce more pineapple skin waste. One pineapple produces leather waste in the range of 34.61% by weight [12]. Therefore, the utilization of pineapple peel waste into useful products such as bioethanol has a good impact in cleaning the environment from waste. In addition, the availability of pineapple skin waste can be obtained at little or no cost [13]. To improve the function of the bioethanol solvent in reducing the pour point value or dissolving paraffin wax, this research will mix with one of the commercial solvents, namely toluene. Toluene is a commercial solvent that has the ability to dissolve a small amount of wax molecules and can reduce the pour point value [14].

This research has never been done before, intended to see the level of compatibility between solvents from agricultural materials and commercial solvents in overcoming the problem of paraffin wax. Previous research only discussed the effect of pineapple peel waste on wax inhibitors. Meanwhile, this research will product bioethanol using pineapple skin and blending solvent with the commercial solvent, which is toluene, which is expected to inhibit the wax formation process in terms of a significant reduction in pour points compared to previous studies..

2. Materials and Methods

The material used is biomass of long rimbo pineapple skin from Pekan Baru. Other chemicals used include HCl acid, NaOH, Aquadest, yeast *Saccharomyces cerevisiae* and urea. For application as a wax inhibitor, wax crude oil is used with a pour point value of 43 C from the SPR Langga company.

2.1 Pretreatment

The raw material in the form of pineapple skin as much as 300 grams is given physical treatment including washing, drying in the oven and reducing the size of the pineapple skin using a blender.

2.2 Hidrolisis

Hydrolysis was carried out in a 1:1 ratio between the catagorisers and the pineapple skin samples. The results of the pretreatment were samples of pineapple skin which had been added with aquadest. The pineapple peel sample was inputted into 150 mL at 500 mL beaker glass. Then add a catalyst in the form of hydrochloric acid (HCL) as much as 150 mL. The concentration of hydrochloric acid was 1 M, 2 M, 3 M, 4 M, and 5 M to see the condition of the resulting reducing sugar levels. The sample was then stirred using a stirrer until it was homogeneous. Furthermore, The hydrolysis process is carried out in an oven with a temperature of 100 C with a variation of 1 hour, 2 hours, 3 hours, 4 hours and 5 hours [12].

2.3 Fermentation

The hydrolysis sample was then cooled. Then measure the pH of the sample to keep the yeast alive. The pH is adjusted in the range 4-5 by adding a number of NaOH solutions. Then add 0.015 g / mL with a variable fermentation time of 1 day, 2 days, 3 days, 4 days, 5 days and 6 days at room temperature [15].

2.4 Distillation

The fermentation product is then purified using a distillation process. The fermented sample is put into a distillation flask which has been coupled with a condenser. The distillation temperature must be maintained at 70,4°C to produce bioethanol [16]. The bioethanol obtained is then measured for the ethanol content produced. Measurement of ethanol levels using an alcoholmeter and Gas Chromatography Mass Spectrometry (GC-MS).

2.5 Pour point testing of waxy crude oil

Pour point testing is carried out based on ASTM D5853-17a to see the drop in pour points on waxy crude oil mixed with bioethanol products that have been made and adding *toluene* additives.

3. Result and Discussion

3.1 The effect of variations in the concentration of hydrochloric acid on reducing sugar levels

In this hydrolysis process, it will produce reducing sugar (glucose) levels which are measured using a portable refractometer HT113ATC [17]. In this study, researchers used HCl catalyst with 5 variations in the concentration of 1 M, 2 M, 3 M, 4 M and 5 M which were heated at 100 C for 3 hours. Following are the results of reducing sugar (glucose) levels against acid concentration

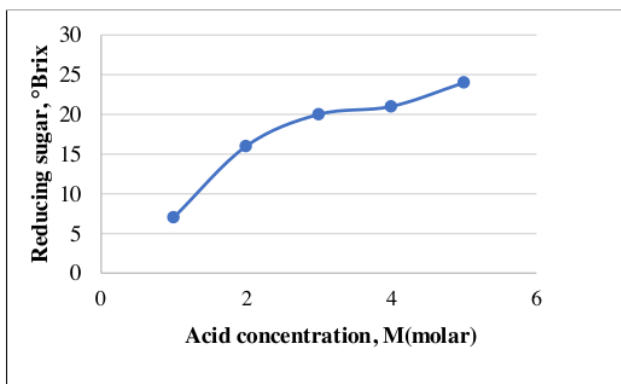


Figure 1. The results of reducing sugar for variation in acid concentration.

Based on the results of reducing sugars in Figure 1, it is explained that at 1M concentration the sugar content is 7°Brix. Whereas at concentrations of 2M, 3M, 4M and 5M, the reducing sugar content was 16°Brix, 20°Brix, 21°Brix and 24°Brix. These results can be concluded that the higher the acid concentration, the higher the reduced sugar content is produced, this is in accordance with the statement of Dewi et al., (2015) that increasing the concentration of acids will increase reducing sugar [18]. The increase in reducing sugar is due to the increase in acid concentration which will accelerate the rate of reaction which causes the decomposition of cellulose into glucose or reducing

sugar [19]. The resulting glucose concentration or reducing sugar content continues to increase and has not yet reached the optimum point.

3.2 Effect of time variations on reducing sugar levels

In this study, the researchers also saw the effect of heating time on the resulting reducing sugar levels. Heating time used is 1 hour, 2 hours, 3 hours, 4 hours and 5 hours at a temperature of 100 °C

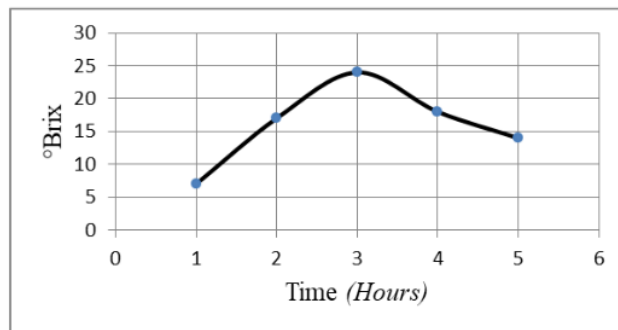


Figure 2. The results of reducing sugars based on temperature variations use concentration acid 5M.

From Figure 2 it is explained that when heating 1 hour to 3 hours the resulting reduction sugar levels increase, namely 7 Brix 16 °Brix and 24 °Brix, this is because the longer the hydrolysis heating time will cause the contact time between cellulose and acid increases so that more the amount of glucose formed [20]. But at 4 hours and 5 hours the reduced sugar levels decreased, this is because the additional heating time would cause a decrease in the resulting reduction in reducing sugar levels due to the conversion of glucose into other compounds and glucose was damaged because it took too long in the heating process [21]

3.3 Effect of pineapple skin fermentation on bioethanol levels

In this process, the observed variable is the fermentation time to the ethanol content produced. To determine the level of ethanol produced, measurements were made using an alcoholmeter. The following is the result of the resulting ethanol content

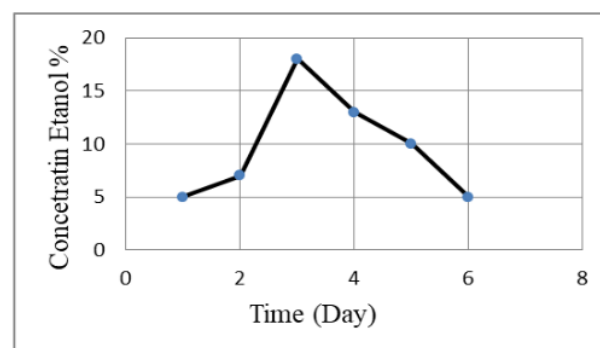


Figure 3. The results of bioethanol levels are based on variations Of the time.

It can be seen from Figure 3 that the difference in fermentation time has an effect on bioethanol levels. In fermentation for 1 day, 2 days and 3 days the ethanol content produced tended to increase with a value of 5%, 7% and 18%. However, at 4 days, 5 days and 6 days the ethanol content tended to decrease with a value of 13%, 10% and 5%. It can be concluded that the longer the fermentation time will lead to an increase in bioethanol levels up to a certain time, after which the bioethanol levels will decrease, this is in accordance with the statements contained in the study siti Miskah et al that the longer the fermentation time will increase the bioethanol content until the optimum condition the bioethanol content will decrease [22].

3.4 Density and specific gravity (SG) bioethanol

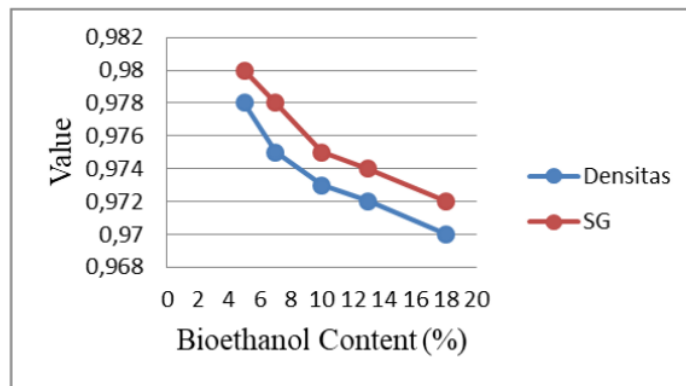


Figure 4. Graph of bioethanol content on density and SG.

Based on Figure 4 it can be explained that the ethanol density values with levels of 5%, 7%, 10%, 13% and 18% are 0.978 g / mL, 0.975 g/mL, 0.976 g / mL, 0.972 g / mL and 0.970. The increase in ethanol content causes the density value to decrease, so that the relationship between ethanol content and density is inversely related. The average density value of bioethanol is 0.975, this figure is greater than the absolute density value of bioethanol, which is 0.789 gr / ml. This is because in making the distillation used in making bioethanol is simple distillation, so there is a human error factor or accuracy in maintaining temperature stability in the distillation process so that the steam produced is not ethanol but mixed with water [23]

In addition to the density value, the value of specific gravity is also observed in this study, the value of specific gravity is obtained from a comparison of the density value of ethanol and the value of water density. From Figure 4 it can be seen that the results of SG are 0.98, 0.978, 0.977, 0.974 and 0.972. This result is directly proportional to the density value, this is in accordance with the theory presented Udegbumam & Mike-Anosike, (2018), that the density relationship with SG is directly proportional [24]

3.5 Bioethanol viscosity

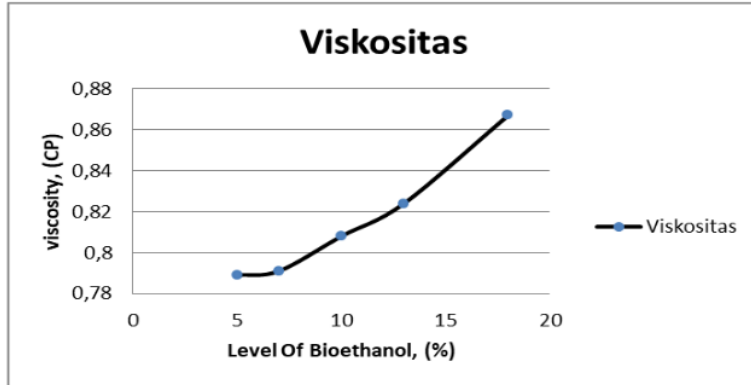


Figure 5. Graph of bioethanol content againts viscosity.

Based on Figure 5 which is the result of the graph of the viscosity value for ethanol content of 5%, 7%, 10%, 13% and 18%, it is 0.694, 0.714, 0.719, 0.734 and 0.744. Based on these results, it is stated that the increasing levels of bioethanol will lead to an increase in the value of viscosity. This is in accordance with Wahyuni's (2010) theory that an increase in the concentration of bioethanol causes an increase in the viscosity value [25]. he increase in the viscosity value can occur because of the difference in the SG value of each ethanol where the ethanol which has the highest content contains the least water content [26].

3.6 Characterization of gas chromatography mass spectrometry (GC-MS) results

3.6.1 Result of bioethanol gas chromatography

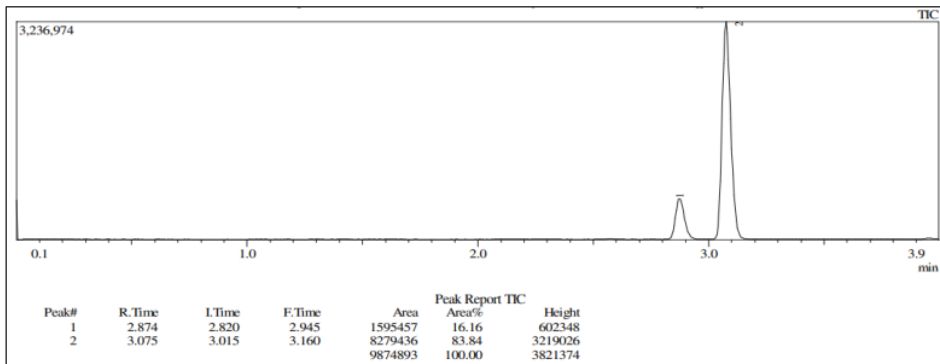


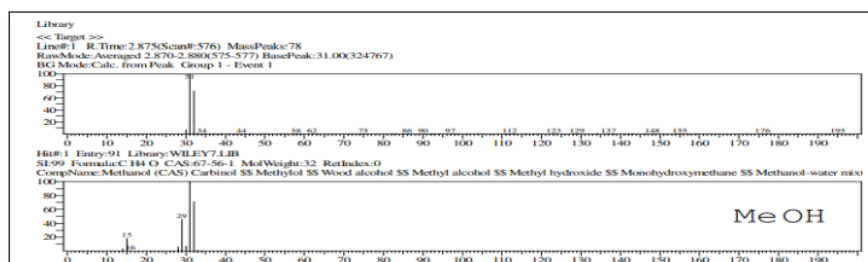
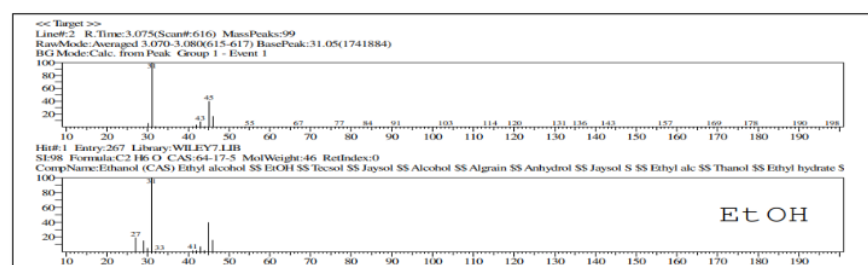
Figure 6. Results of pinapple skin bioethanol gas chromatography.

The results of the bioethanol content test using GCMS obtained bioethanol content of 16.45%. The bioethanol level obtained comes from the bioethanol gas chromatography reading in Figure 6. Based on the comparison between the GC spectrum in Figure 6 and the MS unknown spectrum with the library data in Figure 7 and Figure 8, it can be seen that the compounds from pineapple skin bioethanol are as shown in the table.1 following:

Table 1. Content of bioethanol compounds

No. Peak	Retention time	Compound Name	Molecular Formulas	Area %
1	2.874	Metanol	CH ₃ OH	16.16
2	3.075	Etanol	C ₂ H ₅ OH	83.84

3.6.2 Mass spectrometry results

**Figure 7.** The results of the methanol compound bioethanol mass spectrometry.**Figure 8.** The results of the ethanol compound bioethanol mass spectrometry.

From the results of mass spectrometry testing on bioethanol samples, it can be seen in Figure 7 that methanol compounds are at peak 1 with a retention time of 2,874 of 16.16%. The results of mass spectrometry give molecular ion peaks at molecular weights of 32. The fragmentation peaks with molecular weights of 31 (main peaks), 29, 16 and 15 are followed.

From the results of mass spectrometry testing on bioethanol samples, it can be seen in Figure 8 that the ethanol compound is at peak 2 with a retention time of 3.075 of 83.84%. The results of mass spectrometry give molecular ion peaks at molecular weight 46. The fragmentation peaks with molecular weights of 41, 33, 31 (main peak), and 27 are followed.

The results of the GC-MS test showed that the ethanol content was 16,45%, the bioethanol content was much different in the alcoholmeter test. This is because the GC-MS test has greater accuracy than the alcoholmeter. In the GC-MS test, it measures all components in a sample and the results are identified peak compounds [27].

3.7 Effect of mixing bioethanol and toluene additive with waxy crude oil



Figure 9. An example of reducing pour points.

The drop in the pour point is an indicator to see the effect of mixing solvent and waxy crude oil. The pour point is the temperature at which crude oil can flow from a non-flowable condition [28]. The bioethanol used in this research is bioethanol which has an optimum level of 16% which is mixed with a sample of waxy crude oil from the PT SPR Langgak field which has a pour point value of 43 ° C. The first test is mixing bioethanol with waxy crude oil at a concentration of 10%, 25%, 50%, 75%, 100%, 150%, 200% and 250%.

3.7.1 Decrease the pour point using bioethanol and toluene

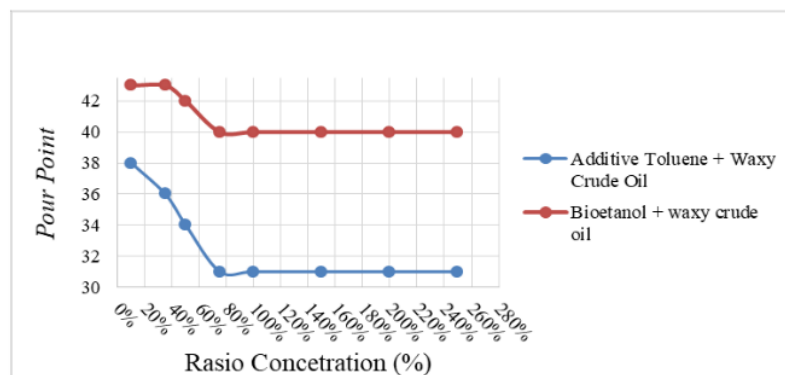


Figure 10. Graph of pour point test results.

From Figure 9 it can be seen that the pour point value for mixing bioethanol with a concentration of 10% and 25% does not experience a decrease, namely the pour point value remains 43°C. At a ratio of 50% the pour point value has decreased for the first time, namely 42 ° C. At a ratio of 75% it decreases again to 40 ° C. However, at a concentration ratio of 100% to 250%, the pour point value remains constant and does not experience any further decline at a temperature of 42 ° C. It can be concluded that the addition of bioethanol with a content of 16% in wax crude oil has an effect on reducing the pour point value. This is in accordance with the theory in research Sinsakulroj & Pengprecha, (2012) that the use of bioethanol solvent can affect the decline in the pour point value [28], [29].

To further increase the reduction in pour points, this study mixed with a commercial solvent, namely toluene. Based on the results of the graph above, it can be observed that the synthetic solvent toluene has an effect on reducing the pour point. From figure 10 which is a graph of the results of pour point reduction, it can be seen that at a concentration of 10% the pour point value decreases at a temperature of 38 ° C, at a concentration of 35% decreases again at a temperature of 36 ° C and likewise at a concentration of 50% and 75% of the pour value point back down to 34 ° C and 31 ° C. However, at a concentration of 100%, 150%, 200% and 250% the pour point value did not decrease, it remained constant at a temperature of 31 ° C. This is in accordance with the statement of Meighani et al., (2018) that after the optimum concentration the pour point value cannot decrease again [30]. The pour point value has decreased because toluene has the ability to dissolve very well against waxy crude oil so that waxy crude oil is more soluble [31].

3.7.2 The effect of mixing bioethanol + toluene additive with waxy crude oil

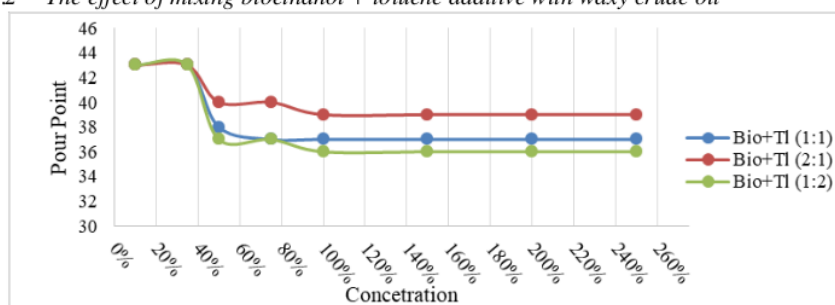


Figure 11. Graph of pour point test results.

Based on the graph from Figure 10 above, mixing toluene with 16% bioethanol gives an increase in pour point reduction. The mixing ratio of 16% bioethanol and toluene (1: 2) reduces the optimum pour point value at 100% mixing concentration by 7 ° C, from 43 ° C to 36 ° C. Meanwhile, the mixing ratio of 16% bioethanol and toluene (1: 1) can reduce the optimum pour point at a concentration of 75%, from 43 ° C to 37 ° C. Likewise, the mixing ratio of 16% bioethanol and toluene (2: 1) also has an effect on reducing the pour point, the optimum pour point reduction lies in mixing at a concentration of 100%, which decreases to 43 ° C to 39 ° C. It can be concluded that the addition of synthetic solvent toluene causes bioethanol to dissolve more with wax crude oil so that it is easier to eutectic with wax crude oil and consequently can reduce the pour point value which is more significant [32].

The non-polar characteristics of bioethanol and toluene have an effect on reducing the pour point and solubility of paraffin wax. Due to the van der Waals force, the long alkyl rental of bioethanol and toluene (non polar) will interact with paraffin wax (non polar) which is called like dissolve like reaction. So the van der Waals style interaction between paraffin wax with bioethanol and toluene will cause an increase in the solubility of paraffin wax in oil and consequently can reduce the pour point value [33].

4. Conclusions

From the results of the research that has been done, the researcher can conclude that, the pineapple skin can be converted into a solvent in the form of bioethanol with a level of 16.45%. The bioethanol properties that have been made are obtained with a density value of 0.97, specific gravity of 0.9758, viscosity of 0.815 cP. The use of bioethanol against waxy crude oil with a level of 16.45% can reduce

3 ° C at a concentration of 75%. The toluene additive with a concentration of 10% can reduce the pour point by 5 ° C. Whereas in the blending between bioethanol and toluene additive, the best scenario was obtained in the ratio variable bioethanol: toluene (1:2) with an optimum reduction at a concentration of 100% at 7 ° C. it can be concluded mixing bioethanol with commercial solvents to improve the performance of bioethanol solvents in dealing with wax problems

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