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Submission date: 16-Jun-2023 11:36AM (UTC+0700)

Submission ID: 2117090525

File name: 15.0.._MAKALAH_LENGKAP_WOLFRAM_IJCST_UPNJAKARTA.pdf (1.64M)

Word count: 1877

Character count: 10072

Solketal from Glycerol, Aceton, and Ethanol Solution using Woifram Acid Phosphotungistic Catalyst

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Abstract. Energy is a need of society and industry which is increasing every year. However, the world's oil reserves continue to run low, so the development of renewable alternative energy is needed. One of the new renewable energies that can be developed is vegetable fuel or biofuel. Biodiesel which is a type of biofuel is being widely produced recently. The cetane number determines the quality of biodiesel. Solketal is an additive added to biodiesel to increase the cetane number. The ketonation reaction was used in this study to obtain the solketal. In the manufacture of solketal a catalyst is needed to speed up the reaction and increase conversion. Phosphotungstic Woifram Acid (PWA) is a heterogeneous catalyst chosen because it can be separated from solution. Based on the results of the study, the optimal time for making solketal was 4 hours and the highest solketal conversion was at a reaction temperature of 60°C with a constant reactant mole ratio of 1:3:1 and a percentage of 2% catalyst, which was 66.7%.

Keywords. Solketal, Glycerol, Aceton, Ethanol, Phosphotungstic Woifram Acid, Ketalization

1 Introduction

One of the crucial problems faced by the world today is energy. The energy needs of society and industry are increasing every year. Given the depletion of primary oil reserves in the community, it is necessary to develop new renewable energy. Renewable energy is a source of energy produced from energy resources that naturally will not run out and can be sustainable if appropriately managed, one of which is biofuels [1]. The development of biofuels is expected to reduce the demand for petroleum [2].

Biofuel is energy made from living matter. One type of biofuel that has been widely produced lately is biodiesel [3]. Generally, additives are added to biodiesel to increase the cetane number. The cetane number is a measure that indicates the quality of the fuel for diesel [4, 5]. The higher the cetane number in the fuel, the minor knock from the diesel engine, so the engine runs smoother, smoother, and quieter. Meanwhile, if the cetane value is low, there will be a delay, adding to the knock on the combustion process [4].

The additive added in biofuel solketal [6]. In general, solketal is carried out using homogeneous base catalysts such as NaOH and KOH because it has a higher catalytic ability than other catalysts. However, using this catalyst has the disadvantage that it is difficult to separate from the reaction mixture. It cannot be reused

and will eventually be wasted as waste that can pollute the environment [1].

Heterogeneous base catalysts such as PWA can be used as an alternative to manufacture biodiesel. In this research, solketal of glycerol and acetone was carried out with PWA catalyst. The highest conversion is obtained at optimum operating conditions, which is expected to increase the cetane number in alcohol.

2 Material and Methods

2.1 Materials

Glycerol (96%) and Ethanol (96%) were used as reactants for ketalization reaction with Acetone (98%) as a solvent and tungstic phosphoric acid (PWA; 100 gr) as a catalyst.

2.2 Methods

The catalytic reaction occurred in a three-neck flask in the presence of heating and cooling back (Fig. 1). Pure solketal is obtained using a distillation process whose apparatus circuit is shown in Fig. 2. GCMS analysis was used to prove the presence of solketal resulting from the ketalization reaction.

Ketalization. The ketalization process was started by adding Glycerol, Acetone, and Ethanol in a ratio of 1:3:1 (% mol) into a 500 ml three-neck flask equipped

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with a stirrer. Then add Tungstic phosphoric acid (PWA) catalyst with a catalyst presentation of 2% wt. The ketalization process was carried out two times. The first at 4, 5, 6, 7, and 8 hours at a boiling temperature and the second at the optimum time at 45, 50, 60, and 65oC.

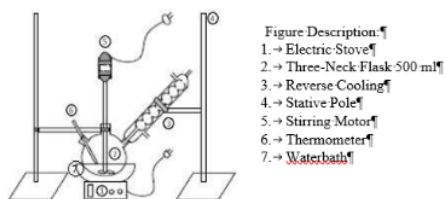


Figure-Description:
 1. → Electric Stove
 2. → Three-Neck Flask 500 ml
 3. → Reverse Cooling
 4. → Stative Pole
 5. → Stirring Motor
 6. → Thermometer
 7. → Water bath

Fig. 1. Experimental setup for ketalization process.

Distillation. Distillation is carried out to separate the reacted solketal from those that do not react based entirely on the boiling point. After ketalizing, the solution is cooled and distilled at a time and temperature according to the boiling point of the solution. The distillate was collected and analyzed using GCMS while the residue, namely glycerol in the distillate flask, was taken to calculate the conversion. The conversion can be calculated using:

$$X = \frac{\text{intial mode} - \text{mol bereaksi}}{\text{mol awal}} \quad (1)$$

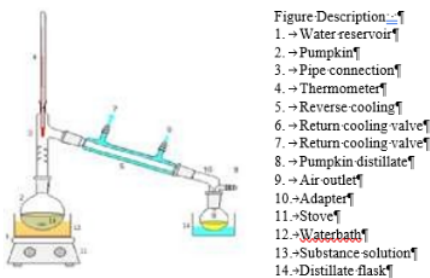


Figure-Description:
 1. → Water reservoir
 2. → Pumpkin
 3. → Pipe connection
 4. → Thermometer
 5. → Reverse-cooling
 6. → Return cooling valve
 7. → Return-cooling valve
 8. → Pumpkin distillate
 9. → Air outlet
 10. → Adapter
 11. → Stove
 12. → Water bath
 13. → Substance solution
 14. → Distillate flask

Fig. 2. Experimental setup for Distillation process.

GC-MS Analysis. This GCMS analysis proves that solketal is a product of the reaction between acetone and glycerol with ethanol as a solvent using a PWA catalyst.

In the GC-MS method, mass spectrometry is combined with gas chromatography methods. Distillate compounds were first analyzed using gas chromatography, and then each component was analyzed using mass spectrometry [7]. The chromatogram of the distillate compound analyzed produces a graph. Each peak of the graph results from GC (Gas Chromatography) was analyzed using MS (Mass Spectrometry), which was then compared with the existing database (library) [7, 8]. The specifications of the GCMS used are Brand and Type: Shimadzu GCMS-QP2010 SE; Temperature: 50 – 300oC; Mass Analyzer: Quadrupole; Scan speed: 10,000 u/sec; Mass range: 1.5 – 1000 m/z; Ionization method: Electron Ionization (EI).

3 Result and Discussion

3.1 Time Variables

The study of the reaction equilibrium for the Solketal was greatly influenced by the reaction time between Acetone and Glycerol. Experiments were carried out to determine the optimum time for solketal formation. In this experiment, a constant mole ratio of Glycerol: Acetone: Ethanol is 1:3:1, the amount of catalyst is constant, 2% wt, and the reaction temperature follows the boiling point of the solution. While the experimental time used is 4, 5, 6, 7, and 8 hours. The components detected at each reaction time are shown in Table 1. Based on the results of GC-MS analysis on various time variables, at 4 hours, the solketal was formed, but the conversion and concentration were unknown. To know the quantitative results, a standard solution of solketal itself is needed, but it is not easy to find it. Because this research is qualitative, 4 hours is set as the optimum time for solketal formation. In addition, at a reaction time of 4 hours, no other by-products were formed apart from the solketal itself.

Table 1. Components detected in time variables based on GC-MS analysis

No.	Time (hour)	Total Peak	Component
1	4	1	Solketal (C16H12O3)
2	5	5	Solketal (C16H12O3), 4 Carene (C10H16), p-Cymene (C10H14) y-Terpinene (C10H16) a-Terpinolene (C10H16)
3	6	3	Solketal (C16H12O3), Glycerol (C3H8O3) a-Terpinolene (C10H16)
4	7	6	Solketal (C16H12O3), Diacetone Alcohol ((CH3)2C(OH)CH2COCH3) Decane (C12H26) M-Xylene (C6H4(CH3)2) Nonane (C9H20) Cyclohexane Methyl Ketone (C8H14O)
5	8	2	Solketal (C16H12O3) a-Terpinolene (C10H16)

3.2 Temperature Variables

The temperature variables were carried out in this experiment at 45, 50, 55, 60, and 65°C. While the conditions maintained were constant Glycerol: Acetone: Ethanol mole ratio of 1:3:1, the amount of catalyst was 2% wt, and the optimum time was 4 hours. Based on the

temperature variable data we analyzed using GC-MS analysis, as shown in Table 2, we get the same peak since the first temperature variable and no other peaks. This indicates that the Solketal has been formed since the beginning, but its conversion and concentration are unknown. Knowing the quantity's result required the standard solution of Solketal itself, but it is challenging to find it.

Table 2. Components detected in time variables based on GC-MS analysis

No.	Temperature (°C)	Total Peak	Component
1	45	1	Solketal (C ₁₆ H ₁₂ O ₃)
2	50	1	Solketal (C ₁₆ H ₁₂ O ₃)
3	55	1	Solketal (C ₁₆ H ₁₂ O ₃)
4	60	1	Solketal (C ₁₆ H ₁₂ O ₃)
5	65	1	Solketal (C ₁₆ H ₁₂ O ₃)

3.3 Effect of Operating Temperature on Glycerol Conversion

Temperature is one parameter that directly affects the biosorption process in the same direction as temperature. Conversion will increase with increasing temperature. It is because the value of the reaction rate constant is high in high-temperature conditions. This experiment was conducted to determine the optimum operating temperature for converting Solketal formation. The reaction conditions in this experiment are: pH = 5, catalyst mass = 2%, pressure = 1 atm, mole ratio of Glycerol: Acetone: Ethanol = 1:3:1, reaction time = 4 hours, first distillation temperature = 56°C, and second distillation temperature = 78°C. The effect of operating temperature on the conversion of glycerol formation is shown in Table 3. Based on the experimental results, it was found that the highest conversion was achieved at a temperature of 60°C. The difference in conversion from each temperature is not large, around 0.1 – 0.5%. Therefore, this glycerol conversion has reached its constant temperature of 66%.

Table 3. Components detected in time variables based on GC-MS analysis

No.	Temperature (°C)	X Glycerol (%)
1	45	66.3
2	50	66.2
3	55	66.6
4	60	6.75
5	65	66.6

4 Conclusion

Experimental data were obtained to determine the optimal operating conditions for the formation of Solketal. From the experimental study the following

conclusions were observed. The reaction time was observed for 4, 5, 6, and 7 hours. Based on the results of the GCMS analysis, the optimum reaction time was 4 hours. This reaction time is used for the temperature variable and the analysis of the effect of temperature on the conversion of solketal formation. The optimum conversion of solketal formation was achieved at a temperature of 60°C, which was 66.7%. The GC-MS analysis shows that the reaction conditions carried out can form a Solketal.

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