*Jurnal Teknologi Industri Pertanian 34 (3): 205-210, Desember 2024*
*DOI: https://doi.org/10.24961/j.tek.ind.pert.2024.34.3.205*
*ISSN: 0216-3160 EISSN: 2252-3901*

*Terakreditasi Peringkat 2*
*SK Dirjen Pendidikan Tinggi, Riset, dan Teknologi No. 158/E/KPT/2021*
*Tersedia online http://journal.ipb.ac.id/index.php/jurnaltin*

# UTILIZATION OF NIPAH FRUIT WASTE ACTIVATED CARBON AS ADSORBENT IN THE PURIFICATION PROCESS OF GLYCEROL BY-PRODUCTS OF PALM OIL BIODIESEL PRODUCTION

## *PEMANFAATAN KARBON AKTIF LIMBAH BUAH NIPAH SEBAGAI ADSORBEN PADA PROSES PEMURNIAN GLISEROL HASIL SAMPING PRODUKSI BIODIESEL KELAPA SAWIT*

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*Paper: Accepted June 10, 2024; Corrected September 23, 2024; Approved October 25, 2024*

## *ABSTRAK*

*Residu gliserol hasil produksi biodiesel mengandung banyak residu yang menjadikannya produk bernilai komersial rendah dengan aplikasi yang terbatas, sehingga residu gliserol biasanya dibuang sebagai limbah di tempat pembuangan akhir. Oleh karena itu, penelitian ini bertujuan memurnikan gliserol dari hasil samping produksi biodiesel kelapa sawit dengan menggunakan metode ekstraksi-adsorpsi. Penelitian tahap pertama adalah pemurnian menggunakan metode ekstraksi petroleum eter dan toluena yang kemudian dilanjutkan dengan tahap adsorpsi menggunakan adsorben berupa karbon aktif dari limbah buah nipah yang memiliki kapasitas adsorbsi iod sebesar 966,978 ± 31,9433 mg/g dan luas permukaan karbon 1066,3853 ± 35,2271 $m^2$/g. Berdasarkan hasil pengujian FTIR, gliserol dengan adsorben berupa karbon aktif limbah buah nipah memiliki karakteristik yang sesuai dengan gliserol komersial dan hasil penelitian sebelumnya, dimana puncak gugus -OH terbentuk dengan jelas pada 3433,271 $cm^{-1}$ dan puncak gugus -CH pada 2934,672 $cm^{-1}$ . Berdasarkan hasil pengujian diperoleh nilai densitas sampel gliserol dengan adsorben karbon aktif limbah buah nipah dengan nilai rata-rata sebesar 1,255 ± 0,007 g/$cm^3$ yang telah sesuai dengan British Standard (BS) 2621:1979 yaitu 1,2671 g/$cm^3$. Kemudian, hasil pengujian diperoleh nilai kadar air sampel gliserol dengan adsorben karbon aktif limbah buah nipah dengan nilai rata-rata 8,563 ± 0,768% yang telah sesuai dengan British Standard (BS) 2621:1979 yaitu < 10%. Hasil pengujian diperoleh nilai kadar gliserol sampel gliserol dengan adsorben karbon aktif limbah buah nipah dengan nilai rata-rata 93,922 ± 2,523%.*

*Kata kunci: adsorpsi, ekstraksi, gliserol, karbon aktif, limbah nipah*

## ABSTRACT

The glycerol residue from biodiesel production contains a lot of residue that makes it a low commercial value product with limited applications, so the glycerol residue is usually disposed of as waste in landfills. Therefore, this study aims to purify glycerol from palm biodiesel production by-products using an extraction-adsorption method. The first stage of the research was purification using petroleum ether and toluene extraction methods, followed by the adsorption stage using adsorbent in the form of activated carbon from nipah fruit waste which has an iodine adsorbing capacity of 966.978 ± 31.9433 mg/g and carbon surface area of 1066.3853 ± 35.2271 $m^2$/g. Based on the results of FTIR testing, glycerol with adsorbent in the form of nipah fruit waste activated carbon characteristics are in accordance with commercial glycerol and the results of previous research, where the peak of O-H group is clearly formed at 3433.271 $cm^{-1}$ and the peak of C-H group at 2934.672 $cm^{-1}$. Based on the test results, the density value of the glycerol sample with nipah fruit waste activated carbon adsorbent was obtained with an average value of 1.255 ± 0.007 g/$cm^3$ which was in accordance with British Standard (BS) 2621: 1979 of 1.2671 g/$cm^3$. The test results obtained the value of the water content of the glycerol sample with nipah fruit waste activated carbon adsorbent with an average value of 8.563 ± 0.768% which is in accordance with British Standard (BS) 2621: 1979 of < 10%. The test results obtained the value of glycerol content of glycerol samples with activated carbon adsorbent of nipah fruit waste with an average value of 93.922 ± 2.523E0%.

Keywords: adsorption, extraction, glycerol, activated carbon, nipah waste

## INTRODUCTION

Biodiesel is an alternative fuel that has undergone various developments both from raw materials and production processes. Biodiesel is derived from renewable feedstock sources such as vegetable oils or animal fats which then react with alcohol and catalysts (Colombo *et al.*, 2017). Biodiesel is produced through the conversion of triglycerides formed in oils and fats in the presence of a short-chain alcohol (methanol or ethanol) and a catalyst (base, acid or enzyme), where biodiesel production also produces by-products of glycerol and

**Coressponding Author*

excess alcohol (Colombo *et al*., 2019). The glycerol residue produced from transesterification reactions is generally of relatively low purity, which varies according to the conversion method and the type of alcohol and catalyst used (Contreras-Andrade *et al*., 2015a). This glycerol residue also contains excess alcohol, catalyst and soap residue, making it a product of low commercial value with limited applications making it usually disposed of as waste in landfills (Nasir *et al*., 2017). Research by Xiao et al. (2013) stated that crude glycerol usually contains less than 65% glycerol by weight, so purification is the first step towards its utilization. This becomes even more important given the value of commercial glycerol products have a minimum purity level of 95%. Furthermore, for the food and drug industry, USP (United States Pharmacopeia) sets the purity level of glycerol at 96-99%, while for halal food types, USP/FCC (Food Chemical Codex) sets the purity level of glycerol at 99.7% (Ciriminna *et al*., 2014; Guerrero-Ruíz *et al*., 2022).

Glycerol purification has a wide variety of methods that can be applied according to the composition of the crude glycerol present. Research by Chol *et al*. (2018), combined physicochemical treatment and semi-continuous membrane filtration using ultrafiltration tubular membranes in the process of purifying glycerol from biodiesel production by-products to produce glycerol with a purity level of 93.7%. Research by Dhabhai *et al*. (2016), combined physicochemical treatment, membrane filtration and activated charcoal adsorption to obtain 97.5% glycerol purity. Then, the research of Contreras-Andrade *et al*. (2013), used a fractional vacuum distillation method at the final stage of the biodiesel production process to produce glycerol with a glycerol purity level between 97.4-98.9%, where the process was monitored by 1H NMR. The method in this study was also carried out by Oliveira *et al*. (2022), namely by fractional vacuum distillation, followed by the selection of the methanol recovery route in the deacidification process and the removal of methanol from the reaction medium to obtain glycerol purity of 99.77%. Another case with the research of Contreras-Andrade *et al*. (2015b), who used an extraction method with organic solvents, followed by adsorption with activated coal and monitoring by 1H NMR spectroscopy. Through this study, 99.2% pure glycerol was obtained. This technique was shown to allow the purification of glycerol using fewer drastic or hazardous conditions than those commonly applied in vacuum distillation.

In this research, a crude glycerol purification procedure will be developed using a combination of extraction methods with organic solvents such as petroleum ether and toluene (Contreras-Andrade *et al*., 2015a) and adsorption with an adsorbent, namely nipah fruit waste activated carbon (Kartikaningrum *et al*., 2022). Activated carbon of nipah fruit waste that has been made by (Kartikaningrum *et al*., 2022) has an average value of characteristics in accordance with SNI 06-3730-1995 standards with the value of water content 15.0767 ± 1.09137%; ash content 8.7733 ± 0.96671%; fly substance content 8.51 ± 3.09875%; carbon content 67.6367 ± 1.63882%; iod adsorption 966.978 ± 31.9433 mg/g; yield 95.485 ± 2.4936%; and carbon surface area 1066.3853 ± 35.2271 $m^2/g$. Based on research (Contreras-Andrade *et al*., 2015b), combination of this method can produce high levels of glycerol purity with lower levels of risk in the operational process.

The purpose of this study was to examine the utilization of activated carbon from nipah fruit waste as an adsorbent in the purification process of glycerol by-products of palm biodiesel production. The hypothesis of this research is that nipah fruit waste activated carbon has an effective ability to improve the purity of crude glycerol. This research is expected to provide an explanation and illustration of the utilization of nipah fruit waste as activated carbon can be a solution in glycerol purification.

## MATERIAL AND METHODS

### Tools and Materials

The tools used in this research are funnel separator, polytetrafluoroethylene (PTFE) stopcock, digital scale, pH meter, pycnometer, glass beaker, measuring flask, dropper pipette, funnel, stirrer, magnetic stirrer, oven, test tube, centrifugation tube, centrifuge, water bath shaker. The materials used in this study were crude glycerol, $H_2SO_4$, periodic acid, potassium iodide, sodium thiosulfate, amylum solution, dichloromethanes, glacial acetic acid, zeolites, bleaching earth, and activated carbon from nipah fruit waste. KOH, petroleum ether, toluene, distilled water, and fine filter paper were also used

### Research Procedure

The research flow diagram is presented in Figure 1.

#### *Organic Solvent Extraction*

In this study, crude glycerol undergoes liquid-liquid extraction using petroleum ether followed by toluene. At first, the crude glycerol will be extracted using a mixture of petroleum ether and water as the liquid phase. The extracted glycerol resulting from the previous step is directly used for the next extraction step, which uses the organic solvent toluene and the liquid phase is water. Extraction using non-polar organic solvents, namely petroleum ether and toluene, aims to remove residual biodiesel, free fatty acids, FAME, diglycerides, monoglycerides and other minor impurities (Contreras-Andrade *et al.*, 2015a). The extraction was performed with a non-polar organic solvent to liquid phase ratio of 1:1 (50 mL : 50 mL) in a 250 mL funnel separator and shaken continuously for 10-15 minutes.

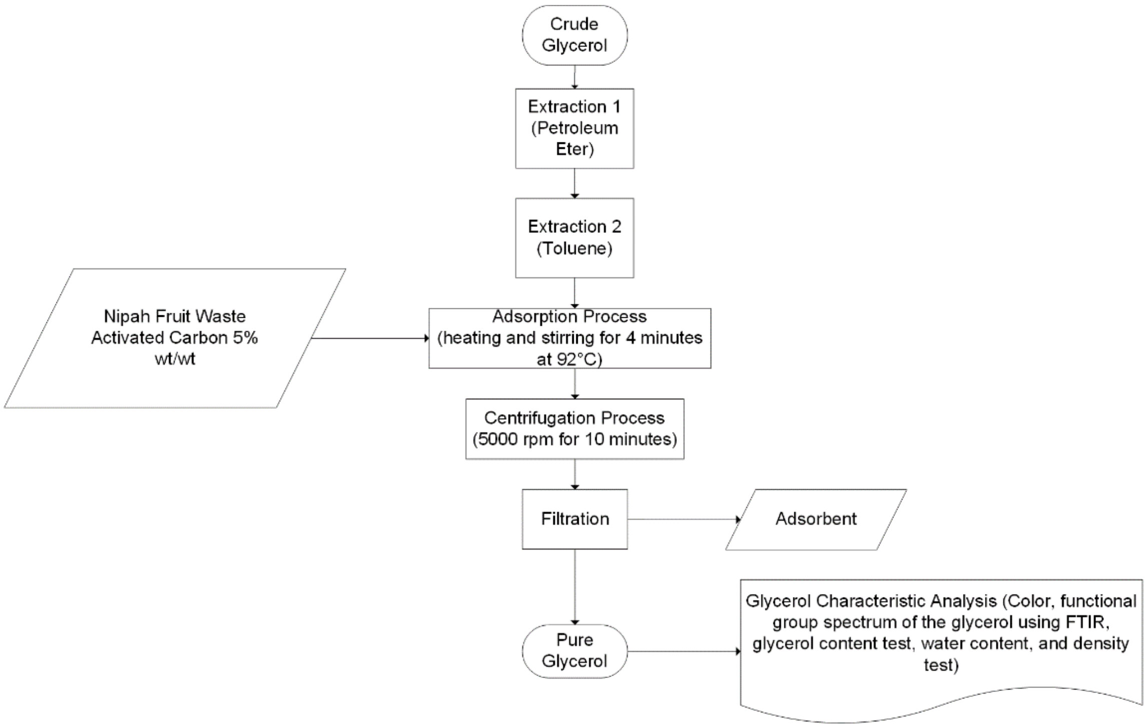


Figure 1. Research flow chart

Then, let the above mixture stand for balance and separation distribution for 10 minutes. Next, the liquid phase was separated from the organic phase. One volume of feed in the funnel separator with polytetrafluoroethylene (PTFE) stopcock was extracted three times for both organic solvents.

***Adsorption***

*Preparation of Activated Carbon from Nipah Fruit Waste*

The manufacture of activated carbon is based on research conducted by Shabrina (2020) and (Kartikaningrum *et al.*, 2022), the process begins with the preparation of waste materials from nipah fruit in the form of shells and nipah fibers which are then chopped using a knife. The next stage is the dehydration stage, namely drying the material using a tunnel dryer for 4 hours at a temperature of 50ºC. Next, the carbonization stage is carried out using a electrical pyrolyzer which lasts for 2 hours at 400ºC to produce charcoal from nipah. The charcoal that has been formed is then pulverized using a blender, then filtered using mesh 100 and carried out the activation stage.

The activation stage is carried out by mixing fine charcoal with chemical activation ingredients in the form of 1M HCl and stirring until homogeneous and allowed to stand at room temperature for 24 hours. After standing for 24 hours, the charcoal sediment was filtered using filter paper and neutralized with 0.1 N KOH until a neutral pH of 6-7 was obtained. The neutralized charcoal was then washed with distilled water and dried using an oven for 5 hours at 110ºC. The last step in making activated carbon is to cool the charcoal that has been dried using a desiccator until a stable temperature is obtained.

Activated carbon from nipah fruit waste that has been made has an average value of characteristics in accordance with SNI 06-3730-1995 standards with a moisture content of 15.0767 ± 1.09137%; ash content of 8.7733 ± 0.96671%; fly matter content 8.51 ± 3.09875%; carbon content 67.6367 ± 1.63882%; iod adsorption 966.978 ± 31.9433 mg/g; yield 95.485 ± 2.4936%; and carbon surface area 1066.3853 ± 35.2271 $m^2/g$

*Adsorption Process*

Adsorption is carried out on the last sample of sequential extraction results, where adsorption aims to change the color of the sample to be lighter by absorbing impurities contained in the sample. In this study, adsorption was carried out using activated carbon made from nipah fruit waste. The usage ratio of each adsorbent was 5% wt/wt. The adsorption process is in accordance with the research of (Kartikaningrum *et al.*, 2022), which is carried out by heating and stirring for 4 minutes at a temperature of 92 °C using a water bath shaker and continued with a centrifugation process of 5000 rpm for 10 minutes and filtration at 18 mm Hg qualitative filter paper so as to collect liquid and remove residue from filtration.

*Sample and Data Analysis*

Characterization of crude glycerol, purified glycerol and commercial glycerol was carried out in two ways, namely qualitatively and quantitatively. Qualitatively glycerol was observed from the spectrum of functional groups using FTIR. The resulting spectrum from FTIR was then analyzed for the absorption area of the bond between atoms. Quantitative analysis included glycerol content test, water content test, and density test using a pycnometer. The purity levels of crude glycerol, refined glycerol and commercial glycerol were tested using the American Oil Chemist Society method commonly called the iodometric-acidimetric method (AOCS Ca 14-56, 2017 or ASTM D 6584).

## RESULTS AND DISCUSSION

### Qualitative Analysis of Purified Glycerol

Qualitative analysis is reviewed from the analysis of functional groups using. In Figure 2, we can see the comparison of IR spectra between crude glycerol, refined glycerol and commercial glycerol. FTIR results of crude glycerol samples show that there are many impurities so that many peaks appear unclear. It can be seen that the wavelength of 3448.648 $cm^{-1}$ is very close or almost intersects with the wavelength of 3223.028 $cm^{-1}$, and the wavelength of 2942.458 $cm^{-1}$ is connected to the peak at 2885.656 $cm^{-1}$ which is an alkane C-H group. After purification, it can be seen that almost all impurities have disappeared in the adsorption process, this can be seen from the IR spectra which do not seem to overlap with each other, where the peak of the O-H group is clearly formed at 3433.271 $cm^{-1}$ and the peak of the C-H group at 2934.672 $cm^{-1}$. Commercial glycerol is the glycerol used as a comparison for crude glycerol and refined glycerol. Glycerol has two typical peaks, namely the O-H group which will be read at a wavelength of (3500-3200) $cm^{-1}$ (Hájek and Skopal, 2010). Commercial glycerol has an O-H group at 3422,232 $cm^{-1}$. The C-H group is read on a curve that coincides at 2940.672 $cm^{-1}$. Based on Figure 2, it can be seen that the characteristics of glycerol resulting from the purification process through a combination process of extraction and adsorption using an adsorbent in the form of nipah fruit waste activated carbon are almost the same as commercial glycerol. The results of this study are also in accordance with research (Suseno *et al.*, 2019), which states that technical glycerol has O-H functional groups in the range of 3323.67 $cm^{-1}$ and 3337.48 $cm^{-1}$ with C-H groups at 2922.63 $cm^{-1}$ and 2922.62 $cm^{-1}$, thus indicating that the glycerol purification results with nipah fruit waste activated carbon adsorbent are in accordance with commercial glycerol and previous research results.

### Quantitative Analysis of Purified Glycerol

***Density***

Glycerol density is one of the benchmarks that can be used as a reference in assessing the quality of glycerol. The density of a material becomes a determinant of the description of the population of materials in the product (Hazra and Septiawan, 2014). Density testing of purified glycerol uses a measuring instrument in the form of a pycnometer. Based on the test results, the density value of the glycerol sample with nipah fruit waste activated carbon adsorbent was obtained with an average value of 1.255 ± 0.007. In addition, the density test of glycerol has also been carried out testing the density value of crude glycerol and commercial glycerol. The test results show the density value of crude glycerol is 1.161 and the density value of commercial glycerol is 1.282. The results of glycerol density analysis in this study are in accordance with British Standard (BS) 2621: 1979 of 1.2671 g/cm3 (British Standard, 1979). Based on (Ardi *et al.*, 2015); (Nadeak and Sinaga, 2019), the density of glycerol is influenced by the water content contained in the product, where the higher the water content, the lower the density value of glycerol.

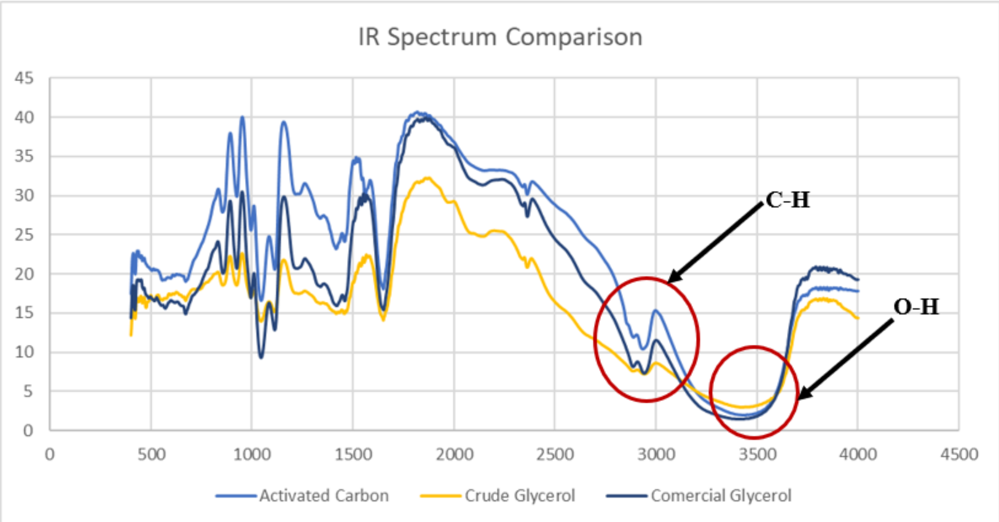


Figure 2. FTIR spectrum comparison of glycerol adsorbent of nipah fruit waste activated carbon, crude glycerol, and commercial glycerol

***Water Content***

Testing the water content of glycerol purification results using a moisture analyzer. Based on the test results, the water content value of the glycerol sample with nipah fruit waste activated carbon adsorbent was obtained with an average value of 8.563 ± 0.768%. In addition, in testing the water content of glycerol, the water content value of crude glycerol and commercial glycerol has also been tested. The test results showed the water content value of crude glycerol was 15.273% and the density value of commercial glycerol was 1.283%. This shows that the crude glycerol purification process carried out in this study has succeeded in reducing the water content value quite significantly. The results of the analysis of the water content of the refined glycerol in this study are in accordance with British Standard (BS) 2621: 1979 of <10% (British Standard, 1979). Based on (Nadeak and Sinaga, 2019), raw glycerol has a fairly high water content, where through the purification process, especially with the adsorption method, the water content can decrease. The water content in glycerol purified using adsorption decreased due to the adsorption of some of the water content by the adsorbent. This is in accordance with the results of the study, where after the purification process with the extraction method followed by the adsorption method using activated carbon nipah fruit waste, the water content has decreased from the water content in crude glycerol.

***Glycerol Content***

Glycerol content is currently the main benchmark in assessing the quality of glycerol (Suseno *et al.*, 2019). Testing the glycerol content of the purification results using the iodometric-acidimetric method (AOCS Ca 14-56, 2017 or ASTM D 6584). Based on the test results, the glycerol content value of the glycerol sample with nipah fruit waste activated carbon adsorbent was obtained with an average value of 93.922 ± 2.523%. In addition, the glycerol content test has also been carried out testing the value of glycerol content in crude glycerol and commercial glycerol. The test results show the value of glycerol content in crude glycerol is 77.347% and the value of commercial glycerol content is 99.9%. The results of the analysis of glycerol content of purification results in this study are in accordance with British Standard (BS) 2621: 1979 which is > 80% (British Standard, 1979). Based on (Ardi *et al.*, 2015), glycerol levels increase as the purification process runs through various methods. In this study, the glycerol adsorption process was carried out with an adsorbent of 5% of the total amount of glycerol, where the amount of adsorbent also affects the glycerol content (Muhammad *et al.*, 2020). Based on the results obtained, the concentration of nipah fruit waste activated carbon adsorbent of 5% is quite effective in increasing the purity of glycerol. Based on the results obtained, 5% concentration of nipah fruit waste activated carbon adsorbent is quite effective in increasing the purity of glycerol. This is also evidenced by comparing the results of this study with research (Suseno *et al.*, 2019), using technical commercial activated carbon and p.a which produced a maximum glycerol content of 92.81%.

## CONCLUSIONS AND RECOMMENDATIONS

**Conclusions**

Nipah fruit waste activated carbon has an effective ability to improve the purity of crude glycerin, which based on the results of FTIR testing, glycerol with adsorbent in the form of nipah fruit waste activated carbon characteristics are in accordance with commercial glycerol and the results of previous research, where the peak of O-H group is clearly formed at 3433.271 $cm^{-1}$ and the peak of C-H group at 2934.672 $cm^{-1}$. Based on the test results, the density value, water content, and glycerol content of glycerol samples with activated carbon adsorbent of nipah fruit waste is in accordance with British Standard (BS) 2621: 1979.

**Recommendations**

Based on the research that has been done, research can be developed to examine the effect of variations in the number of adsorbents that are higher on the level of purity of glycerol by-products of palm biodiesel production. Furthermore, it can be be studied further regarding the utilization of nipah fruit waste activated carbon as an adsorbent in various purification or manufacturing processes as an adsorbent in various purification processes or product manufacturing.

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