

Quality Improvement of Crude Glycerol from Biodiesel Production Using Activated Carbon Derived from Krabok (*Irvingia malayana*) Seed Shells

Wuttichai Roschat^{1,2,3†}, Sarunya Donrussamee¹, Phatcharanan Smanmit¹, Samlit Jikjak¹,
Tappagorn Leelatam^{2,3,4}, Sunti Phewphong^{2,3}, Krittiyane Namwongsa⁵,
Preecha Moonsin⁶, and Vinich Promarak⁷

¹Program of Chemistry, Faculty of Science and Technology, Sakon Nakhon Rajabhat University,
Sakon Nakhon 47000, Thailand

²Biomass Energy Research Laboratory, Center of Excellence on Alternative Energy, Research and Development Institution,
Sakon Nakhon Rajabhat University, Sakon Nakhon 47000, Thailand

³Innovation in Chemistry for Community Research Unit, Faculty of Science and Technology,
Sakon Nakhon Rajabhat University, Sakon Nakhon 47000, Thailand

⁴Appropriated Technology Center, Faculty of Science and Technology, Sakon Nakhon Rajabhat University,
Sakon Nakhon 47000, Thailand

⁵Department of Biochemistry, Faculty of Medicine, Khon Kaen University, Khon Kaen 40002, Thailand

⁶Program of Chemistry, Faculty of Science, Ubon Ratchathani Rajabhat University, Ubon Ratchathani 34000, Thailand

⁷Department of Material Science and Engineering, School of Molecular Science & Engineering,
Vidyasirimedhi Institute of Science and Technology, Rayong 21210, Thailand

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Abstract This research investigated the preparation of activated carbon derived from Krabok (*Irvingia malayana*) seed shells to improve the quality of crude glycerol obtained during biodiesel production. The activated carbon was prepared using a dry chemical activation method with NaOH, utilizing an innovative biomass incinerator. The results revealed that the resulting KC/AC-two-step exhibited favorable physicochemical adsorption properties, with a high surface area of 758.72 m²/g and an iodine number of 611.10 mg/g. These values meet the criteria of the industrial product standard for activated carbon No. TIS 900-2004, as specified by the Ministry of Industry in Thailand. Additionally, the adsorption efficiency for methylene blue reached an impressive 99.35 %. This developed activated carbon was then used to improve the quality of crude glycerol obtained from biodiesel production. The experimental results showed that the KC/AC-two-step increased the purity of crude glycerol to 73.61 %. In comparison, commercially available activated carbon (C/AC) resulted in a higher crude glycerol purity of 81.19 %, as analyzed by the GC technique. Additionally, the metal content (Zn, Cu, Fe, Pb, Cd, and Na) in purified glycerol using KC/AC-two-step was below the standards for heavy metals permitted in food and cosmeceuticals by the Food and Drug Administration of Thailand and the European Committee for Food Contact Materials and Articles. As a result, it can be inferred that Krabok seed shells have favorable properties for producing activated carbon suitable as an adsorbent to enhance crude glycerol purity. Furthermore, the improved crude glycerol from this research has potential for various industrial applications.

Key words activated carbon, Krabok seed shells, *Irvingia malayana*, crude glycerol, purification.

1. Introduction

Nowadays, biodiesel oil, which can be in the form of fatty acid methyl ester (FAME) or fatty acid ethyl ester (FAEE), is

another type of liquid biomass fuel that is widely produced and used worldwide, including in Thailand. Biodiesel oil, a renewable fuel, is obtained from a range of sources, including both edible and non-edible plant oils, as well as animal

[†]Corresponding author

E-Mail : roschat1@gmail.com (W. Roschat, SNRU)

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fats, all of which contain triglyceride compounds. The biodiesel is produced through a chemical process that entails the reaction of these triglycerides with alcohol, utilizing alkaline, acid or enzyme. The main product of this process is an ester compound, with glycerol being produced as a by-product.¹⁻³⁾ Thailand is currently experiencing a growing trend in biodiesel production to replace diesel usage with annual increases. This rapid expansion in biodiesel production has led to the generation of glycerol, with glycerol from the biodiesel production process accounting for 10 % of the total product volume.^{4,5)} Glycerol also known as propane-1,2,3-triol with the chemical formula $C_3H_8O_3$, serves as a key ingredient in numerous everyday products. It is widely recognized that glycerol has a diverse range of applications in industries such as food, pharmaceuticals, cosmetics, perfumery, coatings, and more.⁴⁻⁷⁾

Due to their superior performance and accelerated reaction rates, the most commonly employed base catalysts are homogeneous, specifically sodium hydroxide (NaOH) and potassium hydroxide (KOH). Nevertheless, utilizing these catalysts often generates substantial effluent during the purification of biodiesel and glycerol.^{2,3)} The glycerol residue generated as a by-product of the transesterification reaction typically has low purity. Crude glycerol contains impurities like excess alcohol, spent catalysts, ash, water, and fatty acids, which are closely akin to crude glycerol in terms of their physical properties. The purity level can fluctuate depending on the conversion methods, the choice of alcohol, and the catalyst utilized.⁷⁻¹⁰⁾ Crude glycerol is purified using techniques like distillation, ion exchange, and sequential physicochemical methods such as saponification, acidification, phase separation, solvent extraction, neutralization, and adsorption using activated carbon. The adsorption method is widely acknowledged as an efficient and cost-effective process for removing organic compounds from aqueous solutions.⁹⁻¹²⁾ This is due to its low energy consumption, ability to operate at ambient temperature and pressure, the potential for regenerating spent adsorbents, and the abundance of available adsorbents. An example of an abundant and reasonably inexpensive adsorbent is activated charcoal, which can be prepared through physical or chemical activation methods or a combination of both.¹²⁻¹⁵⁾

Hence, this study aims to improve the purity of raw glycerol obtained from biodiesel production by employing

activated carbon derived from Krabok (*Irvingia malayana*) seed shells. This initiative is inspired by the plentiful Krabok trees in Sakon Nakhon Province, Thailand, whose seed flesh is employed for diverse purposes, including consuming and producing various goods. Nevertheless, the potential of Krabok seed shells remains untapped, and a large amount of them is left over. Krabok seed shells have a harder texture than coconut shells and resemble hardwood; thus, they are suitable for use as raw materials for the production of activated carbon. Therefore, utilizing Krabok seed shells can serve as a way to enhance their value and contribute to the preservation of local plant resources. Furthermore, it involves not only the utilization of the Krabok seed shell but also the enhancement of the quality of crude glycerol through suitable purification methods, thereby increasing its value.

2. Experimental Procedure

2.1. Synthesis of activated carbon

In this study, the synthesis of activated carbon from Krabok seed shells occurred in two main steps, as illustrated in Fig. 1, which presents a process flow diagram. In the first step, Krabok seed shells as precursors of the activated carbon were washed with distilled water several times and then air-dried in an oven at 100 °C for 6 h. In the second step, activated carbon can be prepared from Krabok seed shells using a one-step preparation method known as the KC/AC-one-step. This is done by soaking the Krabok seed shells in a 1:1 ratio with NaOH for approximately 24 h and then drying them until they were completely dry. After that, the carbonization process was conducted using an innovative biomass incinerator from the Appropriate Technology Center at Sakon Nakhon Rajabhat University at 600 °C for 2 h. Next, the carbonized carbon was cooled and kept for use in the next step.

Meanwhile, the preparation of activated carbon from Krabok seed shells using a two-step preparation method, referred to as the KC/AC-two-step, involves crushing the KC/AC-one-step product, sieving it into small particles, and then mixing it with NaOH at a char to NaOH weight ratio of 1:3. This procedure follows the guidelines outlined in reports by Mistar et al.¹⁶⁾ and Kosheleva et al.¹⁷⁾. In the next step, the mixture of char was heated in an innovative biomass

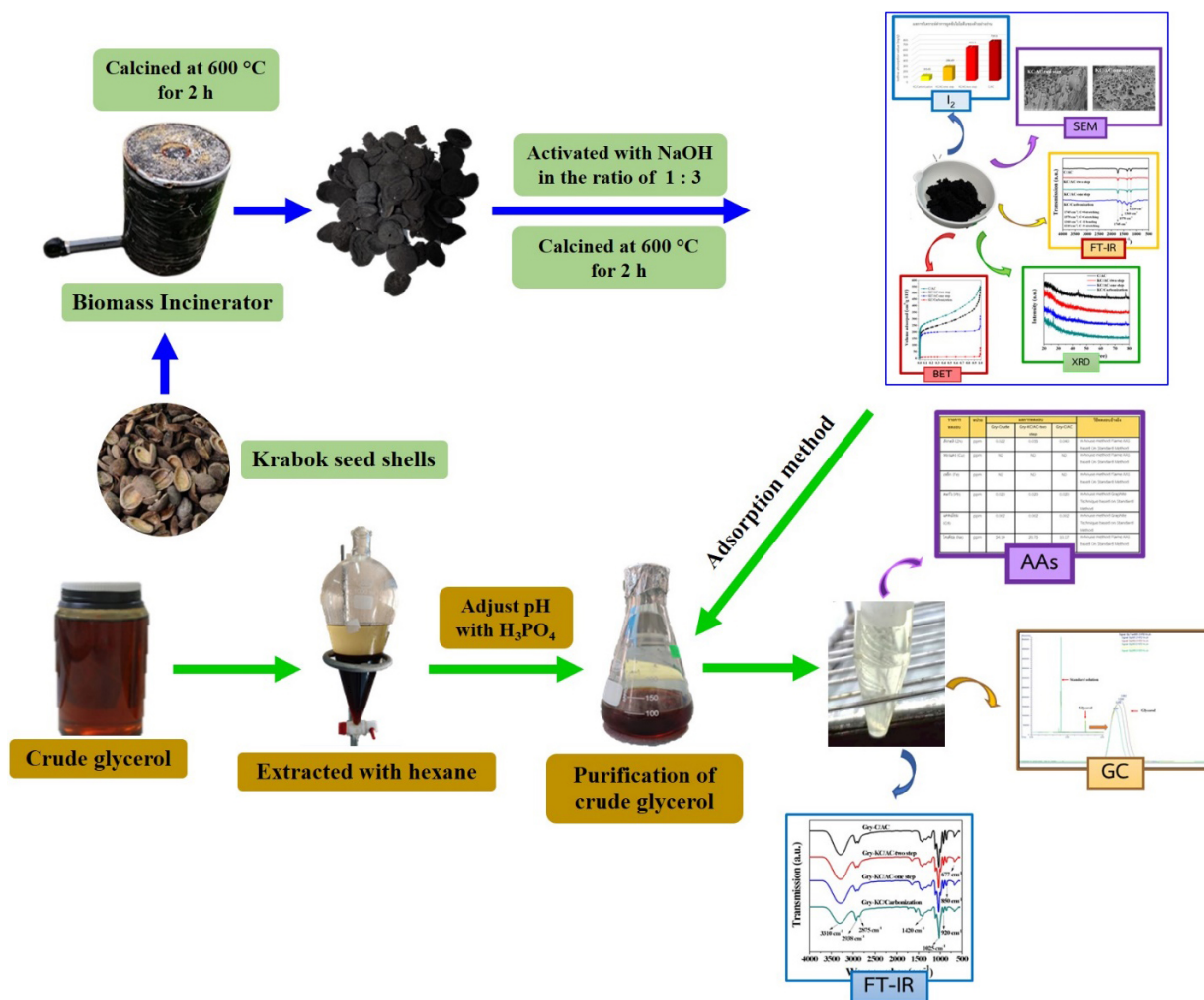


Fig. 1. The process diagram depicts the improvement of crude glycerol from biodiesel production using activated carbon derived from Krabok seed shells.

incinerator at 600 °C for 2 h, and after calcination, the char mixture was cooled to room temperature. Both the resulting KC/AC-one-step and KC/AC-two-step were washed several times with a 0.1 M HCl solution at 80 °C and stirred for 30 minutes until the solution pH measured ~7 to remove NaOH residues and other inorganic matter generated during the activation process. Finally, the obtained activated carbon was dried at 110 °C for 24 h, milled, sieved to achieve a particle size between 125 µm and 250 µm, and stored in a tightly sealed glass bottle.

2.2. Characterization of activated carbon

The obtained activated carbon physical and chemical characterization was investigated using XRD, FT-IR, BET surface area analysis, SEM, iodine number measurement, and methylene blue adsorption. X-ray diffraction analysis

(XRD) was conducted using a Brüker D5005 X-ray diffractometer from Germany. The investigation followed the parameters of Cu Ka radiation ($k = 1.5418 \text{ \AA}$) at 1,600 W, 40 kV, and 40 mA, with a 0.02° per step, and a range of 2θ from 20° to 80° . This analysis aimed to determine the crystallinity or amorphous nature of the obtained activated carbon. The functional groups in all materials were analyzed using a Perkin-Elmer FT-IR spectrometer, specifically the RXI model. The surface area and morphology were investigated using BET with a Bel-sorp-mini II (Bel-Japan) and SEM with a JEOL JSM 5410LV. The analysis of the adsorption efficiency of activated carbon was conducted to study the iodine adsorption value following the Thai Industrial Standard Institute, 2004 (TIS 900 - 2004) of Thailand. Furthermore, the activated carbon obtained was assessed by analyzing the percentage of methylene blue (MB) adsorption to

evaluate its MB adsorption capacity, following the guidelines provided in the report by Dao and Luu¹⁸⁾.

2.3. Purification of the crude glycerol and analysis of the purified glycerol sample

After the biodiesel production process was completed, the separation between biodiesel oil and glycerol was carried out. The ion exchange process was performed using commercially available cation-exchange resins following the reports of Roschat et al.^{4,5)}, Nasir et al.⁹⁾, and Ardi et al.¹⁹⁾. Next, measure 200 mL of crude glycerol, extract it with 400 mL of hexane to separate the non-polar substances, separate the glycerol layer underneath, and then evaporate the hexane. Then, the crude glycerol was neutralized by dissolving 100 g of glycerol in 100 mL of ethanol and adding 270 mL of phosphoric acid (H_3PO_4) to the glycerol. Stir the mixture thoroughly and measure the pH value until it reaches ~ 7 . Set the mixture aside for 30 minutes to allow the solution to precipitate, filter the phosphate salt using a vacuum filter, and then transfer it to the rotary evaporator for the ethanol removal process. The obtained glycerol will be absorbed in the next step.

The adsorptive purification of the pretreated crude glycerol with the synthesis of activated carbon from Krabok seed shells was carried out at room temperature. The experimental procedure began by weighing 5.00 g of the activated carbon sample into a 250 mL Erlenmeyer flask. Then, 5.00 g of the pretreated crude glycerol was weighed into the same Erlenmeyer flask containing the activated carbon sample, and 100 mL of ethanol was added. The mixture was stirred with a stirrer for 3 h at a speed of 300 rpm to disperse the activated carbon and facilitate the adsorption process. Then, the activated carbon powder was separated from the glycerol solution using a vacuum filter, and the filtered glycerol solution was subjected to ethanol removal via a vacuum evaporator. The purified glycerol sample was weighed and collected in a closed container to prevent moisture absorption. Subsequently, the obtained purified glycerol sample was further analyzed using various techniques.

The obtained purified glycerol sample was analyzed through various methods, including pH measurement, FT-IR spectroscopy, and Gas Chromatography (GC). In addition, the metal elements in the obtained purified glycerol sample

were analyzed using the Atomic Absorption Spectroscopy (AAs) technique. This analysis included Zn, Cu, Fe, and Na, using the In-house Flame AAs method based on the standard procedure, while Pb and Cd were analyzed using the In-house Graphite Technique based on the standard method.

3. Results and Discussion

3.1. Characterization of prepared and commercial activated carbon

From the results of the experiment on the synthesis of activated carbon from Krabok seed shells, three types of charcoal samples were obtained. These include one sample of Krabok seed shells charcoal (KC/Carbonization) and activated carbon that was chemically activated with NaOH using the dry method, two samples, namely KC/AC-one-step and KC/AC-two-step, and one sample of commercial activated carbon, known as C/AC. The analysis results to determine the iodine adsorption value, according to the TIS 900-2004 standard as displayed in Fig. 2(a), found that KC/Carbonization charcoal has the lowest iodine absorption value. Meanwhile, the KC/AC-one-step has an increased iodine absorption value but still needs to meet the standard criteria for determining the required iodine number, which should be at least 600 mg/g of charcoal sample.^{20,21)} However, the results of the experiment also revealed that the KC/AC-two-step had the highest iodine adsorption value at 611.10 mg/g of charcoal sample, meeting the standard criteria. When compared with commercial activated carbon, it was observed that C/AC had an iodine absorption value of 739.30 mg/g of charcoal sample.

The results of the analysis of the exposed surface area determined using nitrogen gas adsorption with the Brunauer-Emmett-Teller technique (BET) are shown in Fig. 2(b). The BET analysis results, including surface area, pore volume, and average pore diameter, were presented in Table 1. The results showed that the nitrogen gas adsorption isotherm of the KC/Carbonization tends to have the lowest nitrogen gas absorption value. In contrast, the KC/AC-one-step and KC/AC-two-step tend to show an increase in the nitrogen gas adsorption isotherm. When considering the analysis results of BET surface area, pore volume, and average pore diameter, it was found that the KC/AC-two-step had the highest

BET surface area value of 758.72 m²/g, followed by KC/AC-one-step with a value of 360.97 m²/g, and KC/Carbonization with a value of 34.22 m²/g, respectively. In contrast, C/AC had a BET surface area as high as 990.05 m²/g, surpassing all activated carbon samples synthesized in this research. Nevertheless, according to the definition of activated carbon, it must have a highly porous structure or a relatively high surface area per mass ratio, typically ranging from 600 to 2,400 m²/g compared to regular charcoal.^{10,16,22)} As a result, the experiment demonstrated that the KC/AC-two-step activated carbon synthesized using this research method met the standard criteria mentioned earlier.

From Fig. 2(b), it is evident that the nitrogen gas adsorption isotherms of the synthesized KC/Carbonization and KC/AC-one-step exhibit characteristics consistent with Type I isotherms as defined by the IUPAC system. These isotherms are typical of adsorbents with small pore sizes, approximately 2 nm or even less, and are characterized by the adsorption of molecules or atoms of the adsorbed substance, forming a single, complete monolayer. In comparison, the KC/AC-two-step and C/AC isotherms conform to Type II, which are typical for large porous adsorbents (microporous). In this type of adsorption, molecules initially form a monolayer before gradually adsorbing in multiple

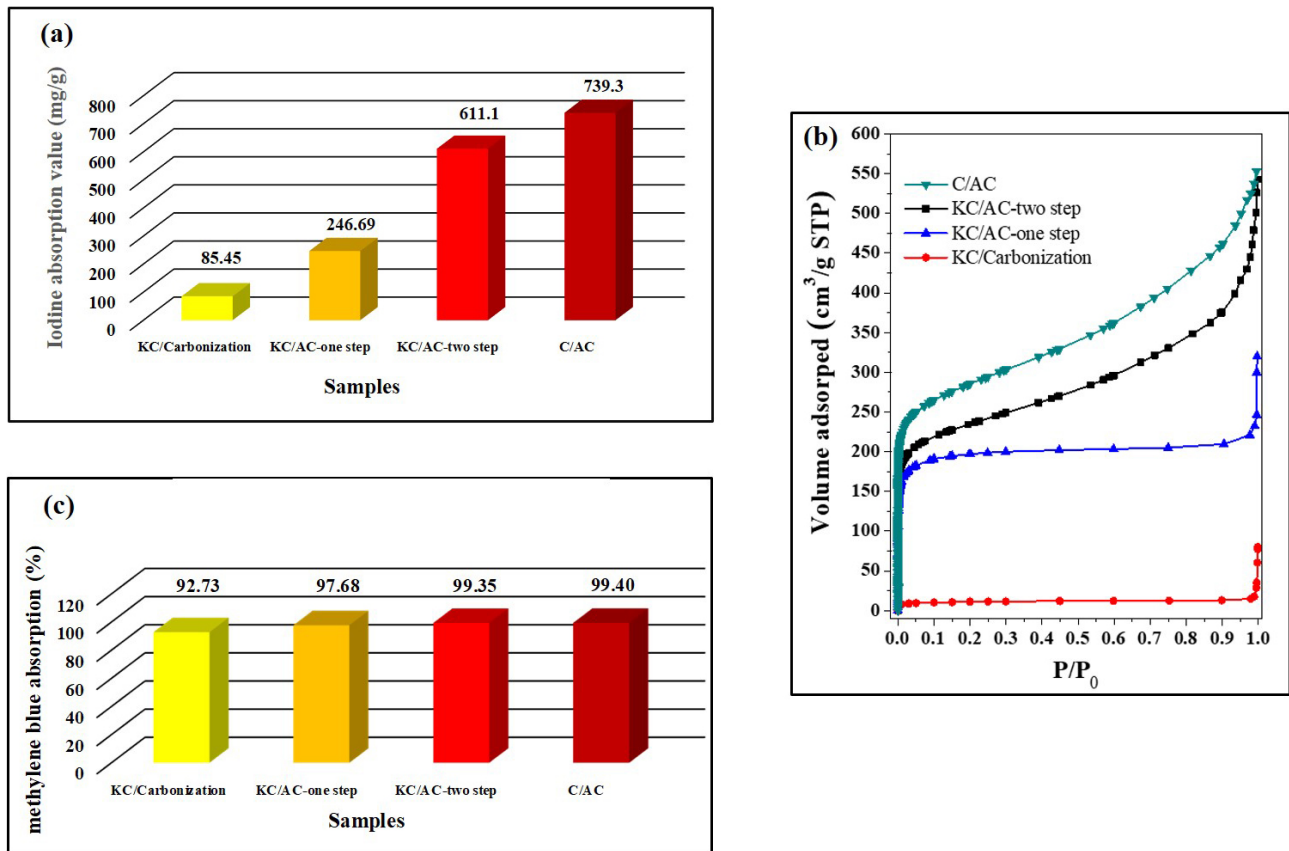


Fig. 2. (a) the iodine adsorption value, (b) the BET surface area, and (c) methylene blue (MB) adsorption capacity of the KC/Carbonization, KC/AC-one-step, KC/AC-two-step, and C/AC.

Table 1. Comparison of the BET surface area, pore volume and average pore diameter of the KC/Carbonization, KC/AC-one-step, KC/AC-two-step, and C/AC.

Sample	BET surface area (m ² /g)	Pore volume (cm ³ /g)	Average pore diameter (nm)
KC/Carbonization	34.22	0.0241	2.8139
KC/AC-one-step	360.97	0.1750	2.6398
KC/AC-two-step	758.72	0.3981	2.9331
C/AC	990.05	0.8277	3.3588

layers (multilayer).^{16,23)}

In an experiment to determine the methylene blue (MB) adsorption values of all samples in this research, as shown in Fig. 2(c), the results demonstrated that C/AC had the highest MB adsorption efficiency at 99.40 %. In comparison, the KC/AC two-step activated carbon showed a similar MB adsorption efficiency to C/AC with an adsorption value of 99.35 %. The KC/AC-one-step exhibited an MB adsorption value of 97.68 %, slightly lower than that of the C/AC and KC/AC-two-step. In contrast, KC/Carbonization showed an MB adsorption efficiency of approximately 92.73 %. This MB efficiency value aligns with the experimental results for the surface area analysis per unit mass of the sample.¹⁸⁻²⁴⁾ Fig. 3 provides an explicit surface morphology characterization of all samples using the SEM technique. The results showed excellent agreement between the SEM micrographs and those obtained from the analysis of BET surface area and pore structure via N₂ adsorption isotherms, iodine adsorption value, and MB adsorption. The C/AC and KC/AC-two-step samples exhibited evident pore characteristics, relatively large pore volume, uniform pore features, and orderly arrangement. Conversely, the KC/AC-one-step displayed noticeable pore formation but with uneven and irregularly arranged pores, whereas KC/Carbonization exhibited a rough surface with minimal pores.

The results of the identification study were analyzed through functional group analysis using FT-IR, and the X-ray diffraction (XRD) techniques for all samples are illustrated in Fig. 4(a, b). The results demonstrated that the functional group vibrations were similar in each sample of FT-IR spectra. The primary component that formed bonds between the C=O at a wavenumber of 1,745 cm⁻¹, the C=C of the aromatic compound at 1,570 cm⁻¹, the group of C-H at 1,360 cm⁻¹, and the group of C-O at 1,210 cm⁻¹ was shown in the main spectra of all samples. These bond vibrations could be functional groups of carbon. The results of this experiment could explain that the identified functional groups may be attributed to organic compounds such as cellulose, hemicellulose and lignin, which are the primary components of Krabok seed shells. Additionally, some residues may occur during the combustion process in the synthesis stage of charcoal, albeit in small amounts.^{16,18,23)}

Meanwhile, XRD patterns of the C/AC, KC/AC-two-step, KC/AC-one-step, and KC/Carbonization samples were determined, and the experimental results indicated that all samples exhibited XRD patterns characteristic of the main chemical structure of an amorphous solid. However, a characteristic peak indicating a slightly crystalline chemical structure was also observed in all samples. This occurrence may be attributed to the carbonization and chemical activa-

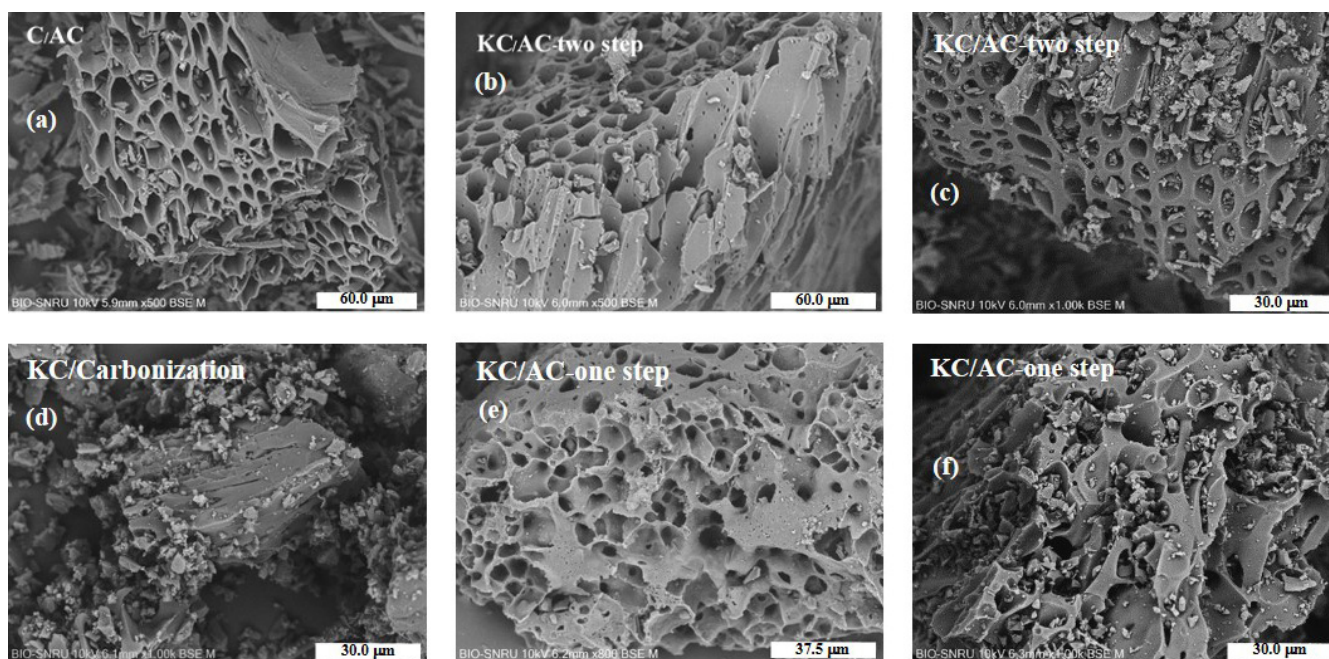


Fig. 3. SEM image of the surface of the (a) C/AC, (b) and (c) KC/AC-two-step, (d) KC/Carbonization, and (e) and (f) KC/AC-one-step.

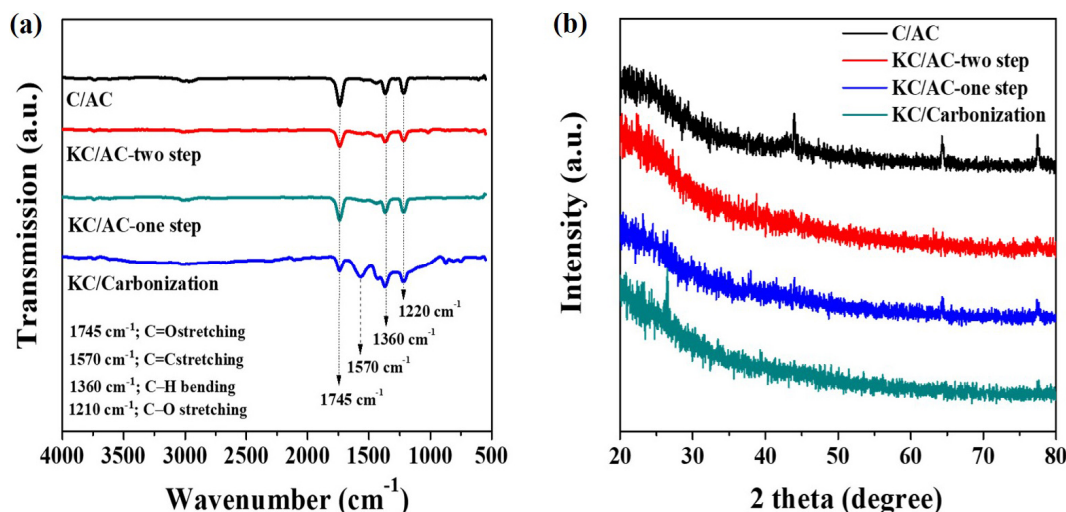


Fig. 4. (a) FT-IR spectra and (b) XRD of the KC/Carbonization, KC/AC-one-step, KC/AC-two-step, and C/AC.

tion processes involving heating in an innovative biomass incinerator at temperatures ranging from 600 to 800 °C for 2 h, potentially causing a physical transformation in the amorphous carbon structure. When the chemistry is organized, crystals of graphite carbon can form and the peak is typically observed at a 2θ position of approximately 26.42°. Additionally, peaks corresponding to the alkaline metal group and the silica (Si) group were detected. These peaks observed at 2θ positions of approximately 43.92°, 64.40°, and 77.40° may indicate contamination during the preparation of activated carbon or could be inherent components of the raw materials used for its preparation. Notably, these peaks were present in only small amounts.^{15,18,25,26}

3.2. Purification of crude glycerol with activated carbon derived from Krabok seed shells

This section presents and discusses the results of pH values, FTIR, GC, and AAs analyses of the obtained purified glycerol sample. The study of pH values in glycerol samples before and after the purification process was conducted at room temperature, and the obtained results are presented for comparison in Table 2. The results indicated that the crude glycerol obtained from the biodiesel production process exhibited a pH value of 10.20, attributed to the use of NaOH as a catalyst in the reaction process. This circumstance influenced the pH value of the obtained crude glycerol, as Na⁺ and OH⁻ could dissolve and mix thoroughly with the glycerol component. After pretreatment, the pH value of the crude glycerol decreased to 8.08. This reduction was achieved

Table 2. The results of pH value analysis of samples before and after the purification process using activated carbon derived from Krabok seed shells.

Sample	pH value
Crude glycerol	10.20
Pretreated crude glycerol	8.08
Purified glycerol with KC/Carbonization	7.86
Purified glycerol with KC/AC-one-step	7.33
Purified glycerol with KC/AC-two-step	7.20
Purified glycerol with C/AC	7.23
Glycerol AR. Grade (99.5 %)	7.02

through an ion exchange process and neutralizing it with H₃PO₄. During these steps, a significant elimination of Na⁺ and OH⁻ ions occurred.^{5,9,13} Furthermore, the results also indicated that purified glycerol with KC/Carbonization showed a slight reduction in pH value to 7.86 compared to pretreated crude glycerol. The pH value of Glycerol AR Grade (99.5 %) was 7.02, reflecting a neutral nature. Similarly, purified glycerol samples obtained through KC/AC-one-step, KC/AC-two-step, and C/AC processes exhibited pH values of 7.33, 7.20, and 7.23, respectively. This is attributed to the adsorbent nature of KC/AC-one-step, KC/AC-two-step, and C/AC, which can adsorb Na⁺ and OH⁻ ions. The results of the pH value analysis for all samples were consistent with the surface area and adsorption efficiency of KC/Carbonization, KC/AC-one-step, KC/AC-two-step, and C/AC, which were used as adsorbents in the purification process of crude glycerol.

Nevertheless, C/AC exhibited superior performance compared to the other cases of KC/AC-two-step, although the pH value of glycerol after adsorption was similar. A possible reason in this case may be the similar efficiency in the adsorption process of Na^+ and OH^- ions for both C/AC and KC/AC-two-step. Because, in this experiment, the purification of crude glycerol was studied under the same conditions —i.e., the amount of the activated carbon sample was 5 grams, the pretreated crude glycerol was 5 grams, and 100 mL of ethanol was used as a solvent with stirring for 3 h at a speed of 300 rpm. For this reason, the adsorption processes of Na^+ and OH^- ions for both C/AC and KC/AC-two-step may reach equilibrium or saturate, resulting in the pH values of glycerol being closely aligned.

The FT-IR tests were applied to determine the functional groups in the glycerol samples that underwent an absorption process using KC/Carbonization, KC/AC-one-step, KC/AC-two-step, and C/AC, as presented in Fig. 5(a). The results showed that the FT-IR spectra of all samples exhibited similar vibrational characteristics of functional groups. The dominant spectra of all samples consist of O-H stretching vibrations peak at approximately $3,310\text{ cm}^{-1}$, C-H stretching at $2,938$ and $2,875\text{ cm}^{-1}$, C-H, $-\text{CH}_2-$, $-\text{CH}_3$, and O-H bending at $1,420\text{ cm}^{-1}$, C-OH stretching at $1,025\text{ cm}^{-1}$, O-H bending at 920 cm^{-1} , and C-H, $-\text{CH}_2-$, $-\text{CH}_3$ rocking and bending out of plane at 850 cm^{-1} and 677 cm^{-1} , respectively. The results of this study are consistent with those reported by Roschat et al.^{4,5}, Nasir et al.⁹, and Rahul et al.²⁷, who analyzed glycerol

using the FT-IR technique.

The results of the study on the analysis of the purity of glycerol, which underwent purification using four types of charcoal powder samples (Gry-KC/Carbonization, Gry-KC/AC-one-step, Gry-KC/AC-two-step, Gry-C/AC), were obtained through gas chromatography (GC) technique. The comparison was made with standard glycerol, and a standard solution of 2-propanol, as illustrated in Fig. 5(b). The results of the experiment found that Gry-KC/Carbonization, Gry-KC/AC-one-step, Gry-KC/AC-two-step, and Gry-C/AC have purity values of 43.45 %, 61.41 %, 73.61 %, and 81.19 %, respectively, compared to the standard glycerol sample with a purity of 99.50 %. In addition, it was observed that crude glycerol (Figure c) had a dark brown and opaque appearance, while pretreated crude glycerol (Figure p) exhibited a clear dark brown appearance. In contrast, pretreated crude glycerol undergoing KC/Carbonization, KC/AC-one-step, KC/AC-two-step, and C/AC adsorption processes exhibited a clear yellow to colorless tint, respectively. This experimental result aligns with the surface area, porosity, and adsorption efficiency of C/AC, with higher values observed for the KC/AC-two-step, KC/AC-one-step, and KC/Carbonization, respectively. The percentage purity of glycerol depends on the techniques, methods, and processes employed to refine raw glycerol. It is also influenced by the main factor, such as glycerol as a starting material obtained from various biodiesel oil synthesis processes, which can introduce contaminants. The physical characteristics, including color, odor,

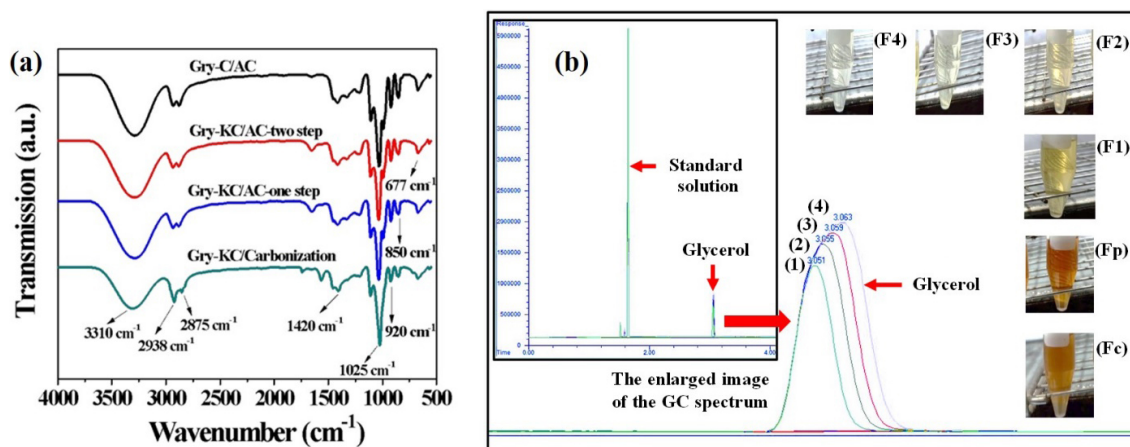


Fig. 5. (a) FT-IR spectra and (b) GC-chromatogram of the glycerol samples that underwent an absorption process using KC/Carbonization (peak 1), KC/AC-one-step (peak 2), KC/AC-two-step (peak 3), and C/AC (peak 4). Noted: Fp instead of pretreated crude glycerol, Fc instead of crude glycerol, and F1, F2, F3, F4 instead of glycerol underwent an absorption process using KC/Carbonization, KC/AC-one-step, KC/AC-two-step, and C/AC, respectively.

and viscosity, vary in the obtained raw glycerol.^{5,10,28)} Furthermore, it was observed that the KC/AC two-step process was approximately 8 % less effective in enhancing glycerol compared to the C/AC method. In comparison to the study by Pitt et al.²²⁾ which improved glycerol using commercial activated carbon powder and achieved a glycerol purity value of 78.72 %, our KC/AC-two-step process exhibited a slightly lower efficacy.

Table 3 presents the metal content in purified glycerol using activated carbon derived from Krabok seed shells (Gry-KC/AC-two-step), comparing it with purified glycerol using commercial activated carbon (Gry-C/AC) and the raw material crude glycerol (Gry-Crude). The results indicated that Gry-Crude, Gry-KC/AC-two-step, and Gry-C/AC contained trace amounts of the heavy metals Zn, Pb, and Cd, all of which were below the standards for heavy metals permitted in food and cosmeceuticals. This aligns with the regulations set by the Food and Drug Administration (FDA) of Thailand and the European Committee for Food Contact Materials and Articles. Additionally, the heavy metals Cu and Fe levels were so minimal that they were undetectable (non-detection; ND).²⁹⁻³¹⁾ Furthermore, it was observed that the Na metal content in raw glycerol (Gry-Crude) was 54.19 ppm, indicating contamination from the biodiesel production process using NaOH as a catalyst. However, employing the enhanced process with KC/AC-two-step for adsorption resulted in a roughly 50 % reduction in Na content compared to the raw glycerol. In contrast, using C/AC achieved an even greater reduction of approximately 80 %. The results of this experiment align with the surface area and porosity values of C/AC being higher than those of KC/AC-two-step, which was prepared from Krabok seed shells.

Based on the results of the experiment, which involved

enhancing glycerol purity with activated charcoal from Krabok seed shells, although the purity percentage was less than 80 %, the analysis of heavy metals and the sodium ion content in the improved glycerol met the criteria for food and cosmeceutical standards. However, glycerol improved using activated carbon from Krabok seed shells can be further processed into cosmeceutical products for external use, such as soap, cream, and lotion. Alternatively, if a higher purity is desired, the modified glycerol could undergo a distillation process.

4. Conclusion

This study aimed to purify glycerol obtained from biodiesel production using the adsorption method with activated carbon derived from Krabok (*Irvingia malayana*) seed shells. This study has demonstrated that activated carbon produced from Krabok seed shells using a two-step preparation method (KC/AC-two-step) exhibited a higher surface area (758.72 m²/g) compared to the one-step preparation method (KC/AC-one-step; 360.97 m²/g) and the carbonization process method (KC/Carbonization; 34.22 m²/g). This corresponded to the highest values of iodine number and methylene blue adsorption efficiency observed in the KC/AC-two-step. This result data was consistent with the pore distribution characteristics analyzed using the SEM technique. Based on the analysis results following TIS 900-2004 standards for iodine number and BET surface area values of the KC/AC-two-step, it is evident that the KC/AC-two-step meets the specified criteria for activated carbon. In the application of using the obtained activated carbon to improve the quality of crude glycerol from biodiesel production, the results revealed that the KC/AC-two-step activated carbon increased the purity of

Table 3. The content of the metals in the purified glycerol used activated carbon derived from Krabok seed shells (Gry-KC/AC-two-step) compared with the purified glycerol used commercial activated carbon (Gry-C/AC) and raw material crude glycerol (Gry-Crude).

Metal	Test results (ppm)			Reference test method
	Gry-Crude	Gry-KC/AC-two-step	Gry-C/AC	
Zn	0.022	0.035	0.040	In-house method Flame AAs based on standard method
Cu	ND	ND	ND	In-house method Flame AAs based on standard method
Fe	ND	ND	ND	In-house method Flame AAs based on standard method
Pb	0.020	0.020	0.020	In-house method Graphite technique based on standard method
Cd	0.002	0.002	0.002	In-house method Graphite technique based on standard method
Na	54.19	20.75	10.17	In-house method Flame AAs based on standard method

crude glycerol to 73.61 %. In comparison, utilizing commercially available activated carbon (C/AC) resulted in a higher crude glycerol purity of 81.19 % under the same conditioning process. Furthermore, the purified glycerol obtained through the KC/AC-two-step process exhibited metal content (Zn, Cu, Fe, Pb, Cd, and Na) below the permissible standards for heavy metals in food and cosmeceuticals, as outlined by both the Food and Drug Administration (FDA) of Thailand and the European Committee for Food Contact Materials and Articles. As a result, glycerol enhanced with activated carbon from Krabok seed shells can be further utilized in producing cosmeceutical products like soap, cream, and lotion for external use. Alternatively, the purified glycerol can undergo a distillation process for those seeking higher purity.

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Author Information

Wuttichai Roschat

Lecturer, Program of Chemistry, Faculty of Science and Technology, Sakon Nakhon Rajabhat University
Researcher, Biomass Energy Research Laboratory, Center of Excellence on Alternative Energy, Research and Development Institution, Sakon Nakhon Rajabhat University
Researcher, Innovation in Chemistry for Community Research Unit, Faculty of Science and Technology, Sakon Nakhon Rajabhat University

Sarunya Donrussamee

Bachelor Student, Program of Chemistry, Faculty of Science and Technology, Sakon Nakhon Rajabhat University

Phatcharanan Smanmit

Bachelor Student, Program of Chemistry, Faculty of Science and Technology, Sakon Nakhon Rajabhat University

Samlit Jikjak

Bachelor Student, Program of Chemistry, Faculty of Science and Technology, Sakon Nakhon Rajabhat University

Tappagorn Leelatam

Researcher, Biomass Energy Research Laboratory, Center of Excellence on Alternative Energy, Research and Development Institution, Sakon Nakhon Rajabhat University
Researcher, Innovation in Chemistry for Community Research Unit, Faculty of Science and Technology, Sakon Nakhon Rajabhat University
Educator, Practitioner Level, Appropriated Technology Center, Faculty of Science and Technology, Sakon Nakhon Rajabhat University

Sunti Phewphong

Researcher, Biomass Energy Research Laboratory, Center of Excellence on Alternative Energy, Research and Development Institution, Sakon Nakhon Rajabhat University
Researcher, Innovation in Chemistry for Community Research Unit, Faculty of Science and Technology, Sakon Nakhon Rajabhat University

Krittiyanee Namwongsa

Master Student, Department of Biochemistry, Faculty of Medicine, Khon Kaen University

Preecha Moonsin

Lecturer, Program of Chemistry, Faculty of Science, Ubon Ratchathani Rajabhat University

Vinich Promarak

Professor, Department of Material Science and Engineering, School of Molecular Science & Engineering, Vidyasirimedhi Institute of Science and Technology