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Purification of Crude Glycerol Using Regenerated Spent Bleaching Earth as Palm Oil Refinery Process

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Abstract. Spent Bleaching Earth (SBE) is a type of solid waste generated during the palm oil refining process. Several studies have investigated the potential reuse of SBE as a valuable material, particularly as an adsorbent in glycerol purification. Crude glycerol, a by-product of biodiesel and oleochemical production from palm oil, is typically of low quality and contains numerous impurities, including free fatty acids (FFA) and pigmented compounds such as β-carotene and chlorophyll. In this study, SBE was regenerated using various solvents and subsequently calcined at 500 °C for 2 hours. Its surface was further modified with cetyltrimethylammonium bromide (CTAB) to enhance its adsorption capacity. The results showed that SBE regenerated with n-hexane and subjected to calcination exhibited the highest removal efficiency for β-carotene and chlorophyll. Further analysis of the physical and chemical properties of crude glycerol purified using uncalcined, calcined, and CTAB-modified SBE demonstrated that the modified SBE delivered the best adsorbent performance. The purified glycerol using modified SBE had the following characteristics: moisture content (10.3%), Density (1.25 g/cm³), FFA content (1.2%), salt content (4.6%), β-carotene content (0.843 ppm), chlorophyll content (0.11 ppm), and pH approximately 6.

Keywords: *SBE, palm oil, crude glycerol, adsorbent, oleochemical*

Abstrak. *Spent Bleaching Earth* (SBE) merupakan salah satu jenis limbah padat yang dihasilkan selama proses penyulingan minyak sawit. Beberapa penelitian telah mengkaji potensi pemanfaatan kembali SBE sebagai material bernilai, khususnya sebagai adsorben dalam pemurnian gliserol. Gliserol kasar merupakan produk samping dari produksi biodiesel dan oleokimia yang dihasilkan dari minyak sawit, umumnya memiliki kualitas rendah dan mengandung banyak pengotor, seperti asam lemak bebas (FFA) dan senyawa berpigmen seperti β-karoten dan klorofil. Dalam penelitian ini, SBE diregenerasi menggunakan berbagai pelarut yang selanjutnya dikalsinasi pada suhu 500°C selama 2 jam. Permukaannya dimodifikasi lebih lanjut dengan setiltrimetilamonium bromida (CTAB) untuk meningkatkan kapasitas adsorpsinya. Hasil penelitian menunjukkan bahwa SBE yang diregenerasi dengan n-heksana dan dikalsinasi menunjukkan efisiensi penghilangan β-karoten dan klorofil tertinggi. Analisis lebih lanjut terhadap sifat fisik dan kimia gliserol mentah yang dimurnikan menggunakan SBE yang tidak dikalsinasi, dikalsinasi, dan dimodifikasi CTAB menunjukkan bahwa SBE yang dimodifikasi memberikan kinerja adsorben terbaik. Gliserol yang dimurnikan menggunakan SBE yang dimodifikasi memiliki karakteristik sebagai berikut: kadar air (10,3%), densitas (1,25 g/cm³), FFA (1,2%), kadar garam (4,6%), kadar β-karoten (0,843 ppm), kadar klorofil (0,11 ppm), dan pH sekitar 6.

Kata kunci: *SBE, minyak sawit, gliserol kasar, adsorben, oleokimia*

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INTRODUCTION

Vegetable oils require processing and refining to remove unwanted impurities before further use. After extraction (oilseed milling), crude oil must undergo several steps: filtration to remove insoluble impurities, degumming (phosphatide removal), neutralization (free fatty acid removal), bleaching with adsorptive clay (to remove color), hydrogenation (hardening, if necessary), and finally, deodorization. One of the key stages in palm oil refining is the bleaching stage. Natural adsorbents such as clay and bentonite also called bleaching earth (BE) have a high absorption capacity for removing undesirable elements from oil (Fadliyani & Atun, 2016). SBE typically consists of bentonite, montmorillonite, sepiolite, and attapulgite (Julfi *et al.*, 2023).

The removal of unwanted elements, including dyes and heavy metals, from vegetable oil using virgin bleaching earth (VBE) produces spent bleaching earth (SBE), a combustible waste that still contains 20–40% oil. SBE is the largest contributor of solid waste in the vegetable oil refining process. Its properties vary depending on the source and type of oil used. SBE waste has an energy content ranging from 11 to 16 MJ/kg. Disposing of SBE without proper treatment poses environmental risks (Tsai *et al.*, 2002), prompting factories to adopt recycling practices to reduce these impacts. Therefore, developing cost-effective SBE disposal and utilization methods is essential to ensure long-term ecosystem sustainability (Julfi *et al.*, 2023).

SBE has been reported to have various industrial and environmental applications. Residual oil in SBE has been utilized for the production of biodiesel, biofertilizers, and briquette fuels, helping to reduce waste from the vegetable oil refining industry and enhance its sustainability. Regenerated SBE is widely used in adsorption processes, including applications in vegetable oil refining, wastewater treatment, CO_2 capture, animal feed, fertilizers, and more (Merikhy *et al.*, 2019)

The vegetable oil industry, particularly palm oil, is not limited to oil refining. One growing segment is the biodiesel industry. Biodiesel is typically produced through the transesterification of vegetable oil with methanol, resulting in triglycerides and glycerol as a by-product. Glycerol accounts for about 10% of the triglyceride volume. Also known as 1,2,3-propanetriol or glycerine, glycerol is widely used in the oleochemical industry. It can also be produced via saponification (soap production) and fatty acid hydrolysis. Currently, 10% of glycerol is produced by hydrolysis, 12% by saponification, and 50–80% by transesterification. Global glycerol production is estimated at around 41.9 billion Liters (Prasetyo *et al.*, 2012)

However, glycerol produced by these processes is usually of low purity. Crude glycerol typically contains 60–80% glycerol, while pure or synthetic glycerol has a purity of 100%. Crude glycerol contains various impurities such as alcohol, organic and inorganic salts, heavy metals, water, soap, non-glycerol organic matter (MONG), fatty acid methyl esters (FAME), fatty acid esters (FAE), free fatty acids (FFA), polyols, and ash. commercially available. Analytical-grade glycerol has the highest purity (>98%) with no contaminants. The United States Pharmacopeia (USP) standard for glycerol purity is 96–99.5%, commonly used in food, pharmaceuticals, personal care products, and cosmetics.

However, the purification of crude glycerol can be costly, especially for small-sized and medium-sized producers. Therefore, cheaper and more efficient purification methods are still urgently needed (Rezayat & Ghaziaskar, 2011). This study investigates the potential of using SBE, a waste product from crude palm oil (CPO) refining, to increase the purity of crude glycerol. SBE obtained from the oleochemical industry will be regenerated and its surface chemically and physically modified to enhance its performance as an adsorbent. The novelty of this study lies in modifying the SBE surface using a hydrothermal method and cetyltrimethylammonium bromide (CTAB), aiming to improve SBE's adsorptive properties. This study is expected to contribute to environmentally friendly and sustainable technological solutions for the palm oil industry.

MATERIAL AND METHODS

Materials

SBE and crude glycerol were obtained from oleochemical plants in the North Sumatra region. The chemicals used in this study included CTAB (sigma-Aldrich), n-hexane (Merck), oxalic acid (Merck), sodium hydroxide (Merck), sulfuric acid (Merck), phenolphthalein (Merck), methanol (Merck), ethanol (Merck), acetone (Merck), ethyl acetate (Merck), silver nitrate (Merck), sodium dichromate (Merck), and distilled water (aquadest).

Instrumentation

The instrumentation were used in this research are furnace, centrifuge, Fourier Transform Infra Red (Bruker Alpha II) and Spectrophotometer UV-Vis (Thermo Scientific Genesys 10S) .

Procedure

SBE regeneration

Prior to modification, oil residues in the SBE were removed by maceration. SBE and n-hexane were mixed in a beaker at a molar ratio of 15:1 and stirred constantly at 300 rpm at 40 °C for 30 minutes. After stirring, the mixture was centrifuged at 4500 rpm for 30 minutes to separate the SBE from the supernatant. The recovered SBE solid was then dried in an oven at 100 °C for 60 minutes to remove any remaining n-hexane. Calcination was subsequently carried out in a furnace at 550 °C for 120 minutes to obtain calcined SBE.

Surface activation of the SBE was performed by mixing it with a 0.75% CTAB solution. The mixture was stirred for 3 hours at 40 °C and then subjected to hydrothermal treatment in an autoclave at 180 °C. The resulting solid was filtered using Whatman filter paper and washed repeatedly with distilled water. The modified SBE was then dried at 120 °C for 1 hour. The dry solid was ground using a mortar and pestle to obtain particles in the size range of 100–150 μm. In this study, different solvents including n-hexane, acetone, methanol, ethanol, ethyl acetate, and sulfuric acid were tested for their effectiveness in removing oil residues from SBE (Yuliana *et al.*, 2021).

Crude glycerol purification with regenerated SBE

To evaluate the activity of regenerated and surface-modified SBE in glycerol purification, SBE was mixed with crude glycerol at a ratio of 3% and stirred at 300 rpm at 30 °C for 60 minutes. After adsorption, the SBE was separated using filter paper. The purified glycerol was then analyzed for carotenoid, FFA, and chlorophyll content. These impurities were quantified using standard methods: AOCS Ca 50-40, MPOB 2005, and AOCS Cc13d-55, respectively. The impurity removal rate (%) was calculated using the following equation (1).

$$\text{Impurity removal (\%) (\%)} = \frac{Ci - Cf}{Ci} \, x \, 100 \ldots(1)$$

where *Ci* is the initial impurity concentration and *Cf* is the concentration after purification.

Free fatty acid determination

FFA were determined using a titrimetric method based on AOCS Ca 5a-40, 1997 (AOCS, 2003b). Approximately 3–5 g of liquid oil was added to 50 mL of hot, neutral 95% ethyl alcohol and 2 mL of phenolphthalein indicator solution, then mixed until homogeneous in 250 mL Erlenmeyer flask. The mixture was titrated with 0.1 N standard sodium hydroxide solution while shaking, until a pink color appeared matching the color of the neutral alcohol before sample addition and persisted for 30 seconds, indicating the endpoint.

$$\text{FFA content as oleate (\%)} = \frac{mL \; NaOH \; x \; N \; x \; 28{,}2}{sample \; weight} \ldots(2)$$

Carotenoid determination

The β-carotene content was analyzed using a spectrophotometer, following the MPOB test method (Dauqan *et al.*, 2011). A total of 0.1 g of the filtrate was weighed into a 25 mL volumetric flask and diluted to the mark with *n*-hexane. The absorbance was then measured at a wavelength of 446 nm using a UV-Vis spectrophotometer.

$$\text{Total carotenoids} = \frac{25 \, x \, 383 \, x \, Abs}{100 \, x \, W} \ldots(3)$$

Note: 25 = dilution volume; 383 = extinction coefficient for carotenoids; *Abs* = sample absorbance; *W* = sample weight (g)

Chlrophyll determination

Liquid samples were homogenized, and if cloudy, immediately filtered using medium-pore-size filter paper before analysis. The samples were analyzed using a UV-Vis spectrophotometer at wavelengths of 630 nm, 670 nm, and 710 nm.

RESULT AND DISCUSSION

SBE Regeneration

The samples used in this study were SBE and crude glycerol, both collected from oleochemical plants in the North Sumatra region. These materials are waste products from oil refining that still contain residual oil. Previous research conducted in the bioprocess laboratory had analyzed SBE from the same plant, reporting an average oil content of 15–19.5%. In comparison, Naser *et al.*, (2021) reported that the average oil content in SBE from oil refining waste was around 35%. The presence of oil residue in SBE must be minimized, as it can inhibit the adsorption capacity of SBE when used as an adsorbent (Muslich *et al.*, 2020). Therefore, optimizing oil residue removal is essential.

In this study, oil residue removal was carried out by maceration using a magnetic stirrer at 300 rpm and 40 °C (Arninda *et al.*, 2022). Several solvents with varying polarity levels were tested to identify the most effective solvent for oil residue removal which be shown in Table 1.

Table 1. Oil Residue Removal from SBE Using Various Solvents

No	Solvent	Filtrate colour	Oil residue content (%)
1	n-Hexane	Pale Yellow	15,6
2	Ethyl acetate	Pale Yellow	14,3
3	Acetone	Clear	19,5
4	Ethanol	Clear	18,8
5	Methanol	Clear	17,2

Although oil is generally non-polar, the use of non-polar solvents such as n-hexane does not necessarily result in the most effective removal of oil residue from SBE. Naser *et al.* (2021) reported that methanol showed higher efficiency in removing oil residue from SBE compared to n-hexane. Specifically, methanol achieved a removal efficiency of 67% and reduced oil residue to 23.5%, while n-hexane achieved 45% efficiency with a final oil residue of 15.7%. The filtrate obtained using methanol was also more concentrated in color than that from n-hexane, indicating better extraction performance (Fajar *et al.*, 2019).

After the filtration process, the SBE with reduced oil content was dried in an oven at 105 °C for 2 hours. The dried SBE was then calcined at 500 °C for 2 hours. The purpose of calcination is to physically activate the SBE by removing insoluble organic compounds that block its surface pores. This process is a key step in SBE regeneration and can significantly enhance its adsorption capacity, particularly for use in glycerol purification (Paramitha & Sitompul, 2020).

Figure1. Uncalcined and calcined SBE

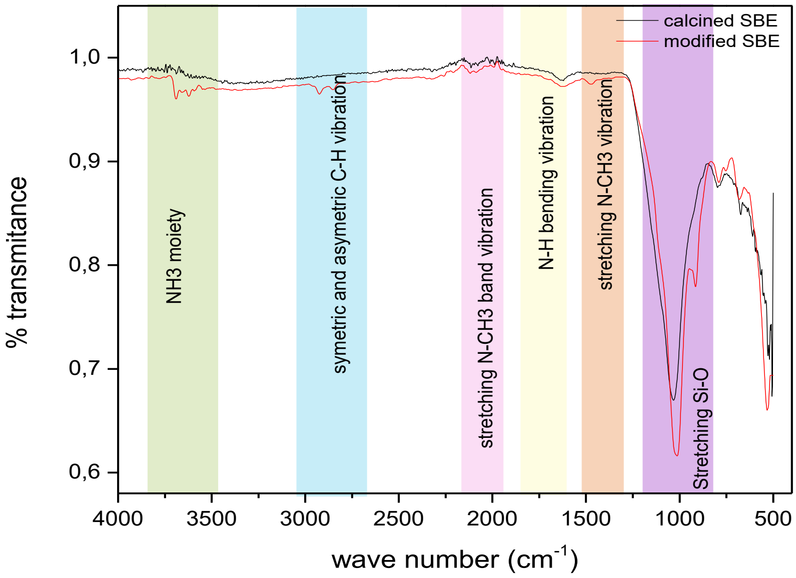

Figure 2. FTIR spectra for modified and unmodified SBE with CTAB

A noticeable change in the color of SBE was observed before and after calcination in Figure 1. Uncalcined SBE appeared darker (black), whereas calcined SBE turned gray. This color change indicates that some organic compounds remaining in the SBE were removed during the heating process. After calcination, the SBE was chemically modified using CTAB. It is expected to modify the surface of SBE by introducing hydroxyl (-OH) groups into the SiO_2 structure, forming silanol (Si–OH) groups. The presence of silanol groups is anticipated to enhance the interaction between SBE and the compounds in glycerol such as FFA, chlorophyll, and beta-carotene hereby improving the adsorption capacity and efficiency of the SBE.

The surface modification was conducted using a hydrothermal method at 180 °C for 4 hours. The product of this hydrothermal treatment was labeled as modified SBE and was tested for its adsorption performance in comparison with calcined (but unmodified) SBE.

The nitrogen adsorption results aligned with the FTIR spectrum shown in Figure 2, which identified several characteristic CTAB functional groups : an ammonia band at 3586 cm^{-1}, symmetric and asymmetric C–H stretching bands from $–CH_2$ at 2930–2852 cm^{-1}, $N^+–CH_3$ stretching bands at 1652 cm^{-1} and 1467 cm^{-1}, an N–H bending vibration band at 1570 cm^{-1}, and a Br^- band at 732 cm^{-1} (Wang *et al.*, 2024) . These peaks confirm that CTAB was successfully incorporated into the lamellar structure of SBE.

Crude glycerol purification with regenerated SBE

The crude glycerol sample used in this study was obtained from PT VVF Indonesia and is a by-product of the fatty acid splitting process. The crude glycerol had a grayish-brown color. Before purification, it was analyzed for FFA content, water content, density, beta-carotene content, chlorophyll content, and salt content. The FFA content was determined using acid-base titration, water content by the gravimetric method, density using a pycnometer, salt content by argentometric titration, and beta-carotene and chlorophyll content by UV-Vis spectrophotometry.

There was a noticeable difference in the appearance of the glycerol (CG) before and after the adsorption process using SBE. The glycerol became visibly clearer after purification, with the clearest result obtained using modified SBE. In contrast, uncalcined SBE did not show a significant color change, suggesting that its pores remained blocked, limiting its adsorption capacity for pigments and other impurities.

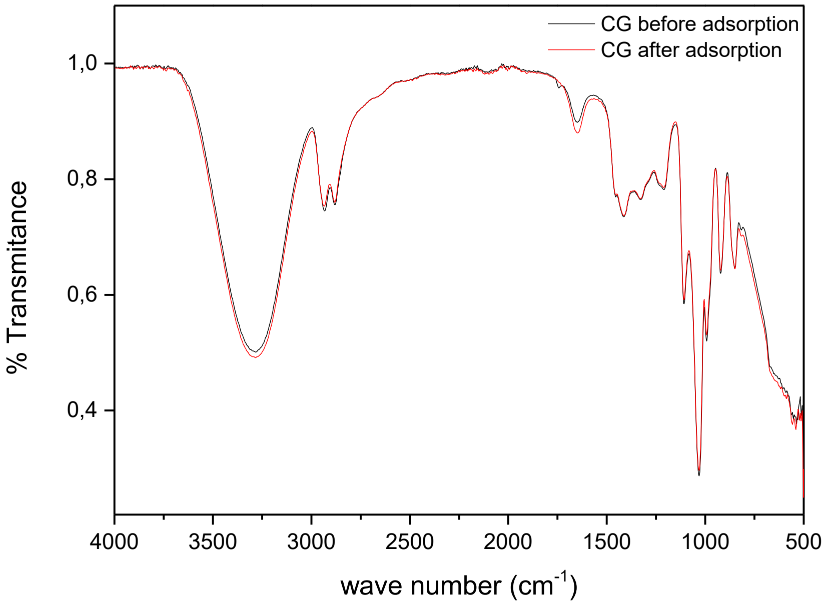

Figure 3. FTIR spectra for CG before and after adsorption with modified SBE

Based on the FTIR results in Figure 3, there were no significant changes in glycerol composition before and after adsorption. Several quality parameters were measured to evaluate glycerol purification, including water content, salt content, and ALB content. The salt content in crude glycerol typically varies depending on the production process. In the oleochemical industry, crude glycerol often contains salts formed from reactions involving sodium hydroxide or potassium hydroxide used in the process. These can lead to compounds such as sodium chloride, sodium methoxide, or others. Acid neutralization during the refining process may also produce additional salts. As a result, crude glycerol may contain 2–5% or more salt, depending on the separation efficiency of the glycerol phase. High salt content can limit the applicability of crude glycerol in industries such as pharmaceuticals or cosmetics, where high purity is required.

FFA is another key organic impurity in crude glycerol, originating from incomplete reactions or unreacted fat and oil residues during processing. FFA levels typically range from 2–10%, but can be higher if the raw material had high initial FFA content.

Table 2. Physical and chemical properties of CG before and after purification

No	Properties	Results			
		Before	Uncalcined	Calcined	Modified
1	Moisture contents (%)	13.5	10.9	10.5	10.3
2	Density (g/cm^3)	1.24	1.27	1.27	1.25
3	Free fatty acid (%)	4.5	1.5	1.1	1.2
4	Salt (%)	7.5	4.9	4.3	4.6
5	β-carotene (ppm)	9.34	4.21	1.15	0.84
6	Chlorophyll (ppm)	1.69	0.72	0,14	0,11
7	pH	4.5	5.5	6	6

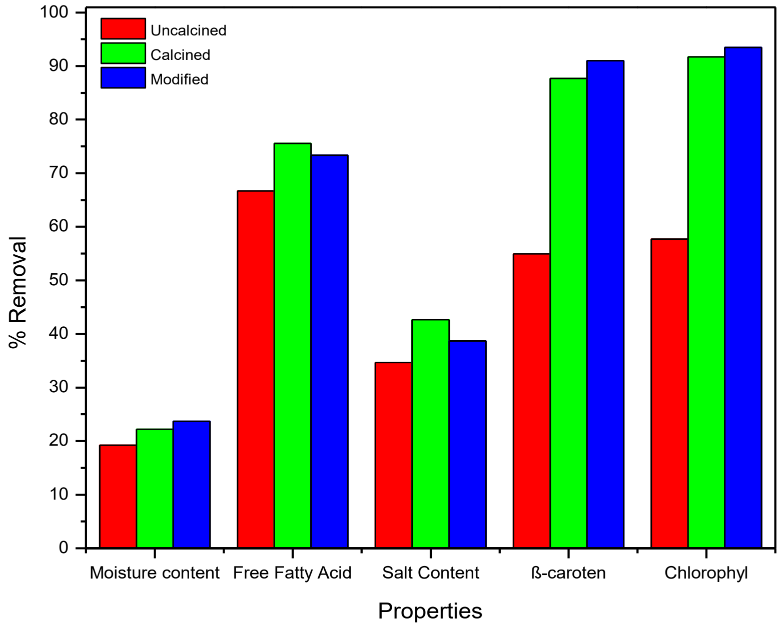

Figure 4. Properties CG after adsorbtion with uncalcined, calcined and modified SBE

Based on the data obtained in the Table 2, the quality of glycerol improved after purification using SBE. Modified SBE demonstrated relatively higher adsorption activity than unmodified SBE, as reflected in improved values for decreasing of water content, beta-carotene, and chlorophyll in crude glycerol. However, for salt content and ALB, unmodified SBE showed better performance. These results also were exhibited in Figure 4. Overall, several tested parameters indicate that the purified glycerol meets industrial-grade glycerol standards.

In addition to evaluating the effect of SBE modification on its adsorption capacity in glycerol purification, this study also investigated the impact of various solvents used for oil residue removal from SBE prior to its application as an adsorbent.

Based on the data obtained regarding the removal of organic pigments in crude glycerol in Figure 5 and 6, it was found that solvents such as *n*-hexane, ethanol, methanol, and sulfuric acid are relatively more effective for oil residue removal. These solvents resulted in improved SBE performance compared to others. The pigments in crude glycerol originate from a variety of organic and inorganic impurities that are typical by-products of oleochemical and biodiesel production processes. The color of crude glycerol varies from light yellow to brown or even black, depending on the raw materials and processing methods used.

Crude glycerol derived from palm oil typically has a very low beta-carotene content, usually below 10 ppm. A darker color often indicates a high concentration of organic impurities such as soap, FFA, and oxidized compounds. Therefore, dark-colored crude glycerol requires further purification before it can be used in sensitive industries such as pharmaceuticals and cosmetics.

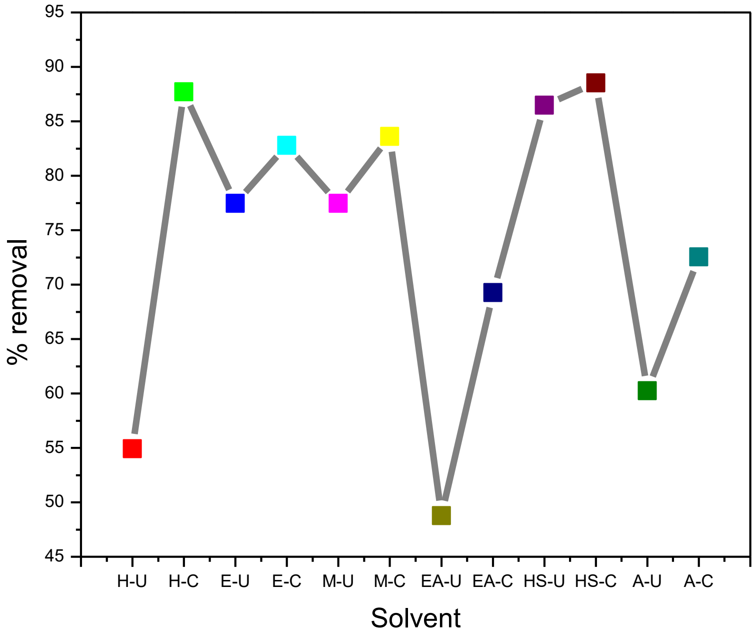

Figure 5. Effect of different solvents on the removal of oil residues and the resulting SBE activity in absorbing carotenoids from crude glycerol.

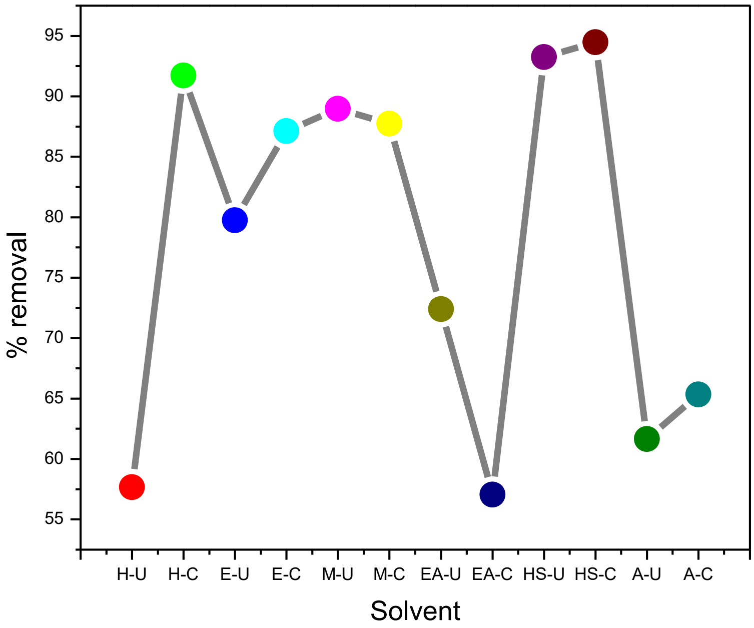

Figure 6. Effect of different solvents on the removal of oil residues and the resulting SBE activity in absorbing chlorophyll from crude glycerol.

The density of pure glycerol (99.5% purity) at room temperature (25 °C) is approximately 1.261 g/cm³. In contrast, crude glycerol being a biodiesel by-product usually has a lower density due to impurities such as water, salts, FFA, and catalyst residues. The density of crude glycerol typically ranges from 1.15 to 1.22 g/cm³, depending on the level of contamination.

Table 3. Effect of different solvents on crude glycerol density after oil residue removal from SBE.

No	Sample	Density (g/cm^3)
1	Glycerol	1.240
2	n-hexane calcined	1.272
3	n-hexane modified	1.253
4	n-hexane uncalcined	1.284
5	etanol uncalcined	1.274
6	etanol calcined	1.274
7	metanol uncalcined	1.275
8	metanol calcined	1.259
9	etil asetat uncalcined	1.279
10	etil asetat calcined	1.265
11	H_2SO_4 uncalcined	1.225
12	H_2SO_4 calcined	1.268

The density is a key parameter in determining glycerol purity. A density close to 1.261 g/cm³ in Table 3 generally indicates high purity. Additionally, density is used for calculations involving volume, mass, formulation, transportation, and chemical reactions.

The interaction between glycerol impurities particularly pigmented compounds and the modified SBE surface can be explained by three main factors: Surface properties of the adsorbent, Structural characteristics of the adsorbate, and Results from kinetic, isotherm, and thermodynamic studies.

SBE particles are modified by the intrinsic negative charges of bentonite, attapulgite, and sepiolite, with their surfaces coated by the cationic head group of CTAB. This modification creates a hydrophobic adsorbent with increased lamellar spacing (Yuliana *et al.*, 2021). Carotenoids and chlorophyll, being fat-soluble pigments, exhibit high hydrophobicity. Chlorophyll also contains a basic structure composed of a negatively charged porphyrin ring coordinated with a central magnesium ion.

Research findings suggest that carotenoid adsorption is primarily governed by hydrophobic interactions, whereas chlorophyll adsorption involves both electrostatic attraction (between negatively charged chlorophyll and positively modified SBE) and hydrophobic binding.

CONCLUSION

Based on the results of this study, it can be concluded that SBE, a waste product from the palm oil processing industry, can be reactivated and reused as an adsorbent in the purification of crude glycerol, a by-product of the oleochemical and biodiesel industries. The reactivation and surface modification of SBE using CTAB significantly enhance its adsorption capacity for crude glycerol purification. The binding mechanisms for organic pigments such as beta-carotene and chlorophyll in crude glycerol involve electrostatic interactions and hydrophobic attraction with both unmodified and modified SBE.

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