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Purification of Crude Glycerol Recovered From Fish Processing Waste Biodiesel Process

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Abstract: Biodiesel and crude glycerol are byproducts of the esterification and transesterification reactions that occur between triglyceride molecules and alcohol in the presence of acid and base catalysts. Purification of crude glycerol is necessary to increase the economic sustainability of the biodiesel industry. This study was carried out for pre-treatment of crude glycerol sourced from fish processing waste during biodiesel manufacturing, treated through an acidification process using phosphoric acid (H_3PO_4), and then using further purification treatment with Jatropha curcas bio-adsorbent. Based on the measurement, the quantity of 0.1% phosphoric acid (H_3PO_4) solution was required to treat the crude glycerin. The reaction conditions include heating solution to 60°C and stirring it at 40 rpm for varying treatment periods such as 15, 30, 45, and 60 min. The crude glycerol's pH was adjusted to begin the acidification process, and soap was then converted into fatty acids and salts using phosphoric acid. The maximum purified glycerol yield of 76.6% by weight was obtained with treatment time of 15min using vacuum distillation at 120°C. The crude and purified glycerol's density was measured as 1.013 g/mL and 1.001 g/mL, respectively. However, after bio-adsorbent treatment at temperature of 50°C and 10% loading weight of the Jatropha curcas bio-adsorbent, the best impurity elimination was accomplished in 15 min. The research findings demonstrated that, following acidification and bio-adsorbent treatments, the FFA% decreased from 2.82% to 2.24%, while its density increased from 1.002 g/mL to 1.032 g/mL, respectively. The purified glycerol properties were found in accordance with BS 2621-1979 standard, showing that the acidification method for purifying of crude glycerol, in conjunction with bio-adsorbent treatment is efficient in enhancing the glycerol purity. It also improves glycerol's application in generating high-value products that enhance revenue streams for the biodiesel production.

Keywords: Biodiesel, crude glycerol, purification, acidification, Jatropha curcas, bio-adsorbent.

Introduction

In today's world, the depletion of fossil fuels has become a growing concern, prompting a pressing need for renewable energy sources. This urgency arises from several factors, including escalating energy demands, decreasing global oil reserves, and the undeniable impacts of climate change. In response to these challenges, biodiesel has emerged as a promising renewable fuel option with distinct advantages, particularly its lower emission levels of toxic gases. By offering a potential solution to mitigate climate change and enhance energy security, biodiesel has drawn significant attention (Saifuddin and Boyce 2017). Researchers have been exploring various avenues to produce biodiesel, for example, using oils derived from inedible plants, microorganisms, and used cooking oil that has been recycled. Biodiesel offers a safer alternative to petroleum diesel due to its non-toxic and non-flammable properties, minimizing environmental damage in case of spills or releases (Liu et al., 2022). With a flash point exceeding 130°C, biodiesel surpasses the approximately 52°C flash point of petroleum diesel. The oils and fats are transformed into long-chain mono-alkyl esters, or biodiesel, via a chemical process called transesterification. The increasing biodiesel production worldwide underscores its recognized environmental benefits. Moreover, the utilization of raw glycerol, a residual product of biodiesel manufacturing, adds to its commercial viability (Liu et al., 2022). From the biodiesel production process, crude glycerol recovered as a primary by-product. Research indicates that for every 10 kilograms of biodiesel produced, approximately 1 kilogram of crude glycerol is generated (Pitt et al., 2019). Consequently, significant expansion in biodiesel manufacturing will inevitably lead to the accumulation of substantial volumes of waste glycerol. The distinctive composition of glycerol contributes to its attributes, making it highly promising for various applications. Glycerol's inherent hygroscopic and water-soluble characteristics stem from its molecular structure, which comprises of three carbon atoms and three hydrophilic hydroxyl groups. Glycerol recovered without undergoing any chemical treatment or

purification after transesterification reaction is referred as crude glycerol, which typically ranges in purity from 40% to 88% content. The purity level of crude glycerol falls within the range of 60% to 80%, whereas commercially synthesized or pure glycerol purity ranges from 99.5% to 100%. Glycerol of higher purity can be utilized to transform or synthesize various products and chemical intermediates (Borówka et al., 2023). Alternative, environmentally friendly solutions must be developed for the use of glycerol in order to prevent problems with its usage and disposal from impeding the production of biodiesel. Glycerol's versatility allows it to be utilized as a raw material in a number of industrial sectors to create chemical intermediates and products. The significant potential markets for the supply of glycerol made from biodiesel production includes co-gasification, co-liquefaction, and co-digestion of organic residues, as well as for the chemical, food, polymer, fuel additive, hydrogen generation, and energy generating sectors (Monteiro et al., 2018).

Scientifically glycerin is known as 1,2,3-propanetriol, and is classified as an alcohol compound due to its three hydroxyl groups (Pitt et al., 2019). This colorless liquid substance boasts a boiling point of 290°C, despite its relatively low molecular weight of 92.09 g/mol (Duell and Pankow 2018), its remarkably high boiling point can be attributed to the formation of robust hydrogen bonds between adjacent glycerin molecules. In practical scenarios, glycerol obtained from transesterification typically contains a mix of impurities including triglycerides, methyl ester, free fatty acids, methanol, water content, inorganic salts, and various organic residues. The characteristics raw glycerol varies depending largely according to the specific processing method employed and the quality of the raw materials (Lima et al., 2022). Therefore, depending on both the method employed and the quality of feedstock used, raw glycerol recovered from transesterification of triglycerides, typically has a glycerol content yield ranging from 30% to 60% (Hudha and Dimas, 2018).

The surplus glycerol produced in recent decades as a result of the global expansion in manufacturing biodiesel has emerged with a novel type of challenge in the purification process of crude glycerol. The development of technologies for purifying crude glycerol through a variety of approaches and appropriate methods involves vacuum distillation, ion exchange, activated carbon, chemical pre-treatment, methanol removal and membrane separation technique. The technologies discussed here offer strategies for the long-term growth and sustainability of the biodiesel production process. Currently, vacuum distillation is the most popular technique for purifying pure glycerol, despite being expensive and energy-intensive. However, it is crucial to investigate more affordable crude glycerol purification methods in order to sustain the long-term, sustainable growth of biodiesel production.

The use of substitute approaches, for example membrane separation method, is thought to be a growing field with a bright future. However, for many of these technologies to become operationally and economically viable, further research and development is still needed. A more energy-efficient technique of glycerol purification that produces 100% glycerol purity will probably be integrated into a biodiesel plant as part of a long-term design. Neutralization is the most commonly used pre-treatment method for crude glycerol purification. It involves a chemical reaction with a strong acid to remove catalyst and soaps (Ardi et al., 2015).

Crude glycerin is subjected to an acidification procedure that involves reacting it with up to 36 mmol of sulphuric, phosphoric, and nitric acids. This process separates the glycerin from contaminants such as salt and fatty acids (Hudha and Laksmana, 2018).

The purpose of this research project was to examine the purification of crude glycerol, obtained from the production of biodiesel from fish processing waste. The research focuses on analyzing the effect of different parameters on purification efficiency using acid treatment method. Additionally, it explores novel technique to transform purified glycerol into a value-added product, aiming to achieve high purity using bio-adsorbent activated carbon obtained from *Jatropha curcas* de-oiled seed cake.

Materials and Methods

Fish Processing Waste Biodiesel Production

The biodiesel from fish processing waste of *Cyprinus Carpio*, was produced by outlined set of procedures. Initially, fish fats obtained from various discarded fish species underwent preparatory steps like heating and filtration to extract crude fish oil. This oil, identifiable by its golden-yellow colour and containing impurities such as water, fish residue, and blood formed the basis for biodiesel production. The biodiesel production involved transesterification of the filtered fish oil using methanol and sodium hydroxide (NaOH) as catalyst. In this process, the molar ratio of oil to alcohol was 1:6 with a catalyst, sodium hydroxide (NaOH) was employed and 1% of the weight of the oil used is required for the base catalyst. The catalyst and methanol mixture is heated on a hot plate magnetic stirrer set at 250 rpm for 2 hrs at 55 to 60ºC (Saifuddin and Boyce 2017). Afterward, the resulting solution underwent separation into crude biodiesel and glycerol layers,

initiating subsequent purification steps encompassing separation, washing, and drying. To remove impurities and ensuring the quality of the final product, the biodiesel produced is washed with warm distilled water at 50 to 60ºC and dried in oven at 90 to 100 ºC for 2 hrs.

Purification of Crude Glycerol

For the process of purifying crude glycerol, initially the quantity of phosphoric acid (H_3PO_4) required to neutralize each gram of crude glycerol was determined. The initial step is preparing the crude glycerol solution, accomplished by mixing 1 g of raw glycerol with 50 mL of distilled water. The solution was then treated with four to five drops of bromophenol blue indicator. Subsequently, a titration was conducted using 0.1% phosphoric acid (H_3PO_4) solution, prepared by combining 5 mL of 85% phosphoric acid with 45 mL of distilled water. The titration proceeded until the solution changed colour from blue to yellow (Rahman et al., 2019).

Four different samples, each consisting of 40 g of crude glycerol were prepared in the laboratory for experimentation at varying time intervals. The next step involved adding the required amount of acid to the glycerol sample i.e., 0.3 mL of acid needed to neutralize 1 g of glycerol. The acid is introduced slowly into crude glycerol, with continuously stirring at a constant 40 rpm and at 60ºC using a heating and magnetic stirrer (C-MAG HS 7, Carl-Roth, Germany) at varying treatment time. Prepared crude glycerol samples analyzed and were subjected to heating at varying time intervals, for 15 min (sample A), 30 min (sample B), 45 min (sample C) and 60 min (sample D), respectively (Pitt et al., 2019). To obtain the average of the purified glycerol results, each experiment including varying heating time for the treatment was done in duplicate.

The residual glycerol post-neutralization underwent the process of vacuum filtration system attached with a high-capacity vacuum pump (Gast, Cole-Parmer, UK). This was done while maintaining a temperature of around 60°C, ensuring the lower viscosity, which made it easier to filter and remove the potassium phosphate salt that was produced. A vacuum filtration system using a Buchner funnel and filter paper was set up, utilizing a vacuum pump set at a pressure of 350 mmHg. Subsequently, the solution was filtered to facilitate the separation of glycerol from the impurities. The filtrate was then poured into a round-bottom flask and given a settling period of 24 hrs to allow the layer of glycerol to be separated from the layer containing free fatty acids. The layer containing free fatty acids was extracted using a pipette, while the subsequent layers containing glycerol and salt underwent further filtration. To prevent the salt from dissolving in the glycerol, the solution was processed through vacuum filtration at ambient temperature. The process of distillation was conducted under vacuum conditions with a maximum temperature of 120°C (i.e. boiling point of glycerol) to separate the fraction of water and alcohol from the solution using heating mantle (HM-200 Series Heating Mantle, Cole-Parmer, UK). This ensured that the glycerol was retained within the filtrate, preventing the crude glycerol's degradation. (Pitt et al., 2019). The purified glycerol was then subjected to adsorption to further enhance its purification (Fig. 1).

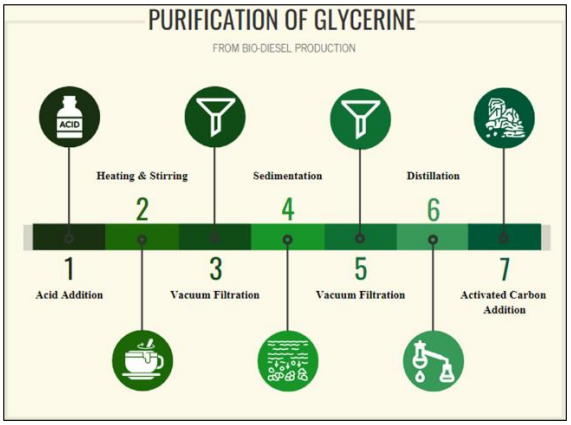

Fig. 1 Glycerol purification steps adopted in this study.

Purification of Glycerol with Activated Bio-adsorbents and its Measurement of Purity

Sample of 10 g of purified glycerin underwent further purification using two different adsorbents involving activated charcoal and bio-adsorbent derived from *Jatropha curcas* de-oiled seed cake. The glycerin was treated with 1:1 (by weight ratio) of activated charcoal and bio-adsorbent by heating for 15 min at 50°C using heating and magnetic stirrer (C-MAG HS 7, Carl-Roth, Germany) and filtered by filter paper. Then the quality of purified glycerol after acidification method was compared with crude glycerol.

Determination of Purity of Glycerol

The purified glycerol obtained after treatment was confirmed using a standard glycerol on a TLC (Thin Layer Chromatography) following the ASTM E2227-02 standard method mentioned in literature (Bansal et al., 2008; Lee et al., 2013).

Free Fatty Acids Analysis

Free Fatty Acid (FFA) test was performed on the glycerol sample having highest yield achieved, according to (ISO-660:1996) standard. For the FFA test, glycerol sample (1g) was dissolved in ethyl alcohol (50 mL) and mixed with 3 to 4 drops of

phenolphthalein indicator and stirred vigorously for 1 min. Then the solution was heated on a bunsen burner for 1 min to its boiling point (78°C) the solution was titrated against a 0.5 N KOH solution until neutralization, as indicated by a distinct reddish-brown colour change. Percentage of free fatty acid and acid value were computed using following formulas

$$Acid\ value = \frac{56.1\ V \times N}{w} \quad (1)$$

$$\%\ FFA = Acid\ value \times 0.503 \quad (2)$$

Where v represents the volume of KOH used, N denotes normality of KOH and w is the weight of sample used. Using the standard method (ISO 2464-1973), as shown in the equation below, the organic non-glycerol (MONG) content of glycerol was calculated by subtracting the total of glycerol, ash, and water content (Kongjao et al., 2010):

$$Mong\ \% = 100 - (Glycerol\ \% + Water\ Content\% + Ash\ content\%) \quad (3)$$

Statistical Analysis between Independent and Dependent Variables

The one-way ANOVA was used to compare the means of four experimental conditions i.e., one independent variable heating for 15 min (sample A), 30 min (sample B), 45 min (sample C) and 60 min (sample D) respectively, where there was one dependent variable (purified glycerol) using Minitab version 17 (USA) at a significance level of α = 0.05.

Characterization of Glycerol

After purification the glycerol was further subjected to various tests to assess its quality. The density and the kinematic viscosity of the purified glycerol was determined using viscosity meter (VDM-300, Lemis, USA). The pH values were determined using pH indicator strips (Merck, Germany) in accordance with ASTM D86 guidelines, while moisture content was analyzed following the procedure mentioned in ASTM D5348.

Results and Discussion

The glycerol samples' density was analyzed showing variations that correlate together with the presence of unwanted contaminants in the samples. These impurities comprise of fatty acids, water content, alcohol, catalyst residues, and additional compounds originating from the feedstock oil used in biodiesel manufacturing influence the density measurements. The findings tabulated in Table 1 outline the density values of various samples obtained throughout the purification process. Notably, the unpurified raw glycerol exhibited a value of 1.013 g/mL for density, whereas the purified samples demonstrated values ranging from 1.002 to 1.006 g/mL. Distillation, a pivotal stage in purification, typically leads to an increase in this parameter by facilitating the removal of residual alcohol and water. Moisture content is a crucial parameter which was evaluated following ASTM D5348 standard, which presents the amount of water content in glycerol, serving as an impurity (Table 1) The results showed amount of moisture content present in glycerol before and after purification treatment method. From the results, it can be observed that initially, waste crude contained moisture content of 2.63% which was significantly reduced to 0.29% through purification. Concentration of free fatty acids is being detected by pH, which plays a role in assessing the effectiveness of purification procedure having influence on treatment method. The results from this analysis showed pH of crude of glycerol was found to be 8 (in basic range), whereas pH of all four samples A, B, C and D was found between 3 to 4 (i.e., in the acidic range). The percentage yield of the assessed samples was determined by comparing the measured weight and volume before and after purification processes. Due to the assessment of samples at different time intervals, there was variability in the percentage yield achieved. For instance, sample A exhibited the highest yield at 76.56%, while other assessed samples gave a yield of 26.13%, 18.38%, and 23.90%, respectively (Table 1).

Earlier, it has been proved that phosphoric acid is the most effective purifying agent among other acids (Nanda et al., 2014). Phosphoric acid acidified the crude glycerol, and the reaction between the acid and the soap molecules produced less soluble sodium/potassium salts and free fatty acids: RCOOK → RCOOH + KH2PO4 + H3PO4. The employed purification process significantly reduced the concentration of free fatty acids (%) in crude glycerol from 21.164 % to 2.82%, enhancing its purity (Table 1). Almost all of the alkali content present in the crude glycerol are neutralized by the acid in highly acidic environment, which causes them to precipitate out as solid residue (salt) at the bottom and react with the soap to produce free fatty acids as the top phase (Nanda et al., 2014). The employed purification process significantly reduced the % free fatty acids present in crude glycerol from 21.164 % to 2.82%, enhancing its purity (Table 1). The moisture content was found having values comparable with previous literature (Rizky et al., 2023), with 94% and 87%, respectively, while purified glycerol yield were 42.9% and 65.88%, respectively. It is observed that the MONG content is the primary contaminant in crude glycerol. The MONG is made up of contaminants found in the glycerol from the biodiesel production stages, such as soap, alcohol, and methyl esters.

Table 1. Physical and chemical properties of crude and treated glycerol.

Sample	Treatment time (min)	Volume		Purified glycerol obtained by weight (g)	Density (g/mL)	pH	Glycerol content % (w/w)	Ash content % (w/w)	Moisture content % (w/w)	Mong % (w/w)	Purified glycerol yield % by weight	Acid Value (mg KOH/g of glycerol)	FFA %
		Glycerol (mL)	Waste fatty acid layer (mL)										
Crude glycerol	-	-	-	-	1.013	8.5	9.72	4.44	2.63	68.74	-	42.075	21.164
A	15	13	30	22.627	1.002	6.5	69.40	0.646	0.32	29.63	76.60	5.61	2.822
B	30	11	32	10.45	1.006	6.4	-	0.733	0.35	-	26.13	-	-
C	60	8	29	7.35	1.003	6.7	-	0.745	0.33	-	18.38	-	-
D	45	10	30	9.56	1.003	6.3	-	0.694	0.36	-	13.90	-	-

Table 2. Characteristics of purified glycerol after post-treatment with adsorbents.

Name of adsorbent	Density (g/mL)	pH	Acid Value (mg KOH/g of glycerol)	FFA %
Activated charcoal	1.017	7.8	4.12	2.073
Jatropha curcas	1.032	6.5	5.22	2.643

Table 3. Statistical relationship between means of glycerol purification yields.

Source	DF	Sum of squares	Mean squares	F-stat	p-value
Between groups	3	4963.43	1654.48	9508.49	0
Within groups	4	0.7	0.17		
Total	7	4964.13			

At different values of pH utilized in the phase separation stage facilitated by acidification, the MONG (%) proportion in the crude glycerol that was purified was less (29.63%) than that in the crude glycerol (68.74%) prior to treatment (Table 1). Strong acidic conditions caused the crude glycerol to remove large amount of free fatty acid, which raised pH in the purification process and reduced the amount of MONG in the purified crude glycerol (Kongjao et al., 2010).

Glycerol Purification with Bio-adsorbent

The adsorption procedure employed two materials i.e., activated carbon of Jatropha curcas and activated charcoal. The purpose was to investigate which substance could best purify the sample by eliminating color pigments, which served as impurities. Sample A showed the highest degree of purification was achieved when the adsorption process was conducted at a temperature of 50°C, lasting for 15 min, with adsorbent loading at 10 wt% (Anandan and Janakiram, 2022). The characteristics of glycerol obtained after bio-adsorbent and activated coal treatment are presented in Table 2. During this process, impurities within the glycerin were adsorbed onto the surface of the activated carbon (Fig. 2). Activated carbon has its high affinity for organic compounds, thus effectively eliminated impurities including methanol, salts, soap, and colorants from the glycerin. Subsequently, the solution was filtered using filter paper to obtain the final purified product (Yuliana et al., 2021).

FTIR Analysis

FTIR analysis of the four samples indicated the presence of various functional groups. The detection of C-O, C=O, and OH groups suggest possibility of ethanoic acid present. This likely occurs due to the reaction not reaching completion, leaving unreacted reactants or intermediates in the crude glycerol. Commercial glycerol study demonstrated a broadband with a corresponding wave number between 2852.84 cm-1 to 3326.81 cm-1, revealing an OH (acid) group. Aliphatic CH group, absorption band was found at wave numbers 2922.45 cm-1 and 2852.84 cm-1. There is a likelihood that in the fingerprint region, C-O absorption appears at wave number 1653 cm-1 while the absorption of the C=O functional group is observed around 1741.53 cm-1.

Fig. 2 Showing (L to R): (a) Raw glycerol obtained from biodiesel production, (b) pre-treated glycerol and (c) purified glycerol after bio-adsorbent treatment.

The results indicate the presence of ethanoic acid. In some cases, the transesterification reaction may not proceed to completion, resulting in unreacted reactants or intermediates in the crude glycerol. If ethyl esters (biodiesel) are produced alongside glycerol, ethanoic acid could be present due to the hydrolysis of ethyl esters under acidic conditions. FTIR analysis of glycerol after vacuum distillation was caried out. A peak at 3278.68 cm-1 can be observed which indicates the presence of OH (acid) group. Another peak can be analyzed at 1636.6 cm-1 which shows the presence of a C=O functional group. The presence of C=O (carbonyl) and OH (hydroxyl) groups in the Fourier Transform Infrared (FTIR) analysis of glycerol after vacuum distillation suggests the formation of an aldehyde or ketone compound.

Glycerol, a trihydroxy alcohol, typically does not contain a carbonyl group in its structure. However, during vacuum distillation, glycerol may undergo thermal decomposition or dehydration reactions, leading to the formation of intermediate compounds. These compounds could include aldehydes or ketones, which possess a carbonyl group (C=O) and may also retain a hydroxyl group (OH). The presence of the C=O and OH (acid) groups indicates the formation of an organic compound that contains both functional groups. This compound can be a ketone if the carbonyl group is inside the carbon chain or an aldehyde or it is positioned at the carbon chain's terminal carbon. The profile of adsorption of glycerol using Jatropha Activated Carbon and Adsorption of Glycerol using Activated Charcoal represents more or less the same results as glycerol after vacuum distillation (Fig. 3). It can be concluded that after the distillation process, the purified glycerol contains the C=O and OH (acid) functional groups. The specific compounds or functional groups present in the purified glycerol, can be further analyzed using additional characterization techniques such as chromatography, spectroscopy, or mass spectrometry.

Comparison of Purity of Glycerol Obtained with Standard Glycerol

Thin Layer Chromatography (TLC) is a feasible and effective method for detecting the glycerol's purity which was obtained as a residual product of biodiesel manufacturing, by comparing it with standard glycerol. The results show that the purified glycerol by-product is as pure as the standard glycerol, as evident by the same Rf (Retardation factor) values of the first and second spots on the TLC sheet (Fig. 4).

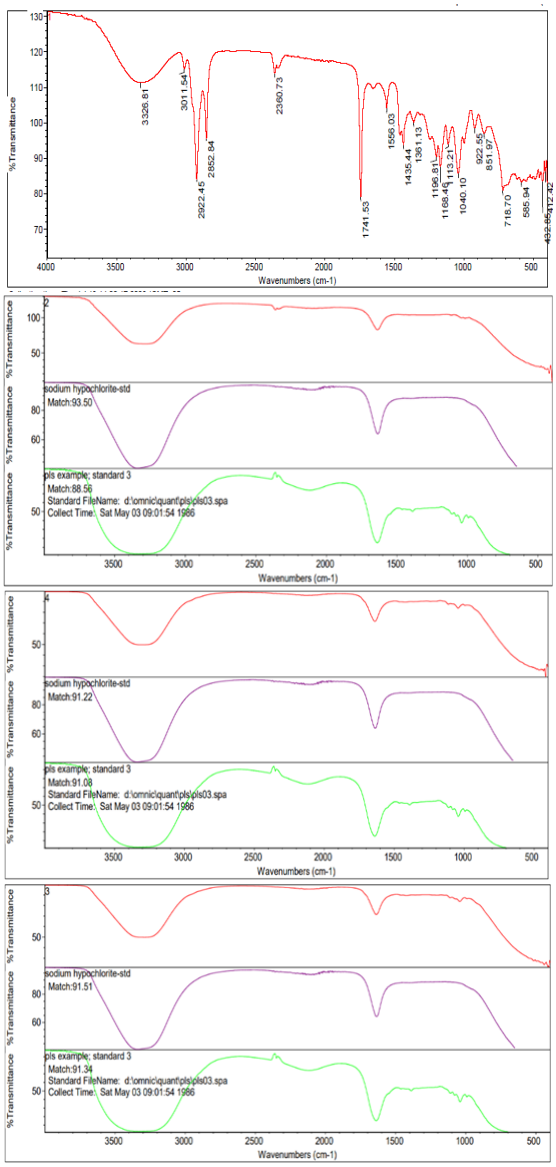

Fig. 3 Clockwise: Raw glycerol, glycerol after vacuum distillation, adsorption of glycerol using Jatropha activated carbon and adsorption of glycerol using activated charcoal.

(a) **(b)**

Fig. 4 (a) TLC of standard glycerol and purified glycerol immediately after removal from $KMnO_4$ solution, (b) TLC of standard glycerol and purified glycerol after drying treatment.

Statistical Analysis of Purified Glycerol

The statistical analysis of one-way ANOVA, showed the means of four experimental conditions of varying heating time were found significantly dependent on purified glycerol yield (Table 3).

Conclusion

Incorporating energy-efficient methods and technologies into glycerol purification can contribute to lowering greenhouse gas emissions, aligning with the sustainable development goals to mitigate climate change. The purity of crude glycerol was significantly improved through thermochemical treatment of acidification using H_3PO_4, which effectively reduced impurities such as free fatty acids, water, residual catalysts, and color pigments. This enhancement process involved transforming crude glycerol into a higher-value product through a purification sequence that included neutralization followed by acidification. The maximum yield achieved was 76.6% under ideal parameters of pH 6.5, temperature 60°C and a reaction time of 15 min, with reduction in the fatty acid content from 21.16% to 2.82%. After acidification purification method, commercial activated carbon and Jatropha curcas bio-adsorbent were used to achieve further purification. During adsorption, both the temperature and the mass of the adsorbent used exhibit notable effects on the elimination of pigmented compounds. The optimal removal of impurities was achieved at an adsorption temperature of 50°C, and with a 10 % by loading weight of the bio-adsorbent (Jatropha curcas) for a duration of 15 min. The results showed reduction in FFA% from 21.16% to 2.24% and density was increased from 1.002 g/mL to 1.032 g/mL after acidification and bio-adsorbent treatments, respectively.

It is concluded that glycerol, a byproduct of the production of biodiesel, can be refined by the acidification process and then treated with a bio-adsorbent to enhance its quality as a product with added value. The Jatropha curcas de-oiled seed cake (bio-adsorbent) obtained from biodiesel processing unit as a feedstock, is used to comply with waste management policy and reduce the glycerol purification cost. It is recommended to use renewable energy sources such as a solar parabolic concentrator to supply heat treatment during glycerol purification process, thus reducing carbon footprint associated with the production and purification of glycerol.

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