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Substantial Purification of Waste Glycerol – A Byproduct of Biofuel Industry its Product Characterization (AbstractView.aspx?PID=2019-12-2-34)

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Substantial Purification of Waste Glycerol – A Byproduct of Biofuel Industry its Product Characterization

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ABSTRACT:

An increase in greenhouse gas effect and depletion of fossil fuel leads to work on alternate renewable source of energy. Biofuel, in which biodiesel has gained its importance over a decade due to its promising characteristics. Crude glycerol is produced as an inevitable byproduct during biodiesel production. The carbon content of the waste glycerol can be utilized by many microorganisms as a cheap carbon and energy source for the production of high value chemicals like ethanol, citric acid, propanediol etc. In this present work simple sequential procedures like distillation, acidification, activated charcoal adsorption, filtration and centrifugation were carried out made to remove major impurities economically and increases the glycerol concentration. The removal of impurities from crude glycerol and the increase in the concentration of glycerol were witnessed from GCMS and GC-FID reports. The procedure followed in this work provided a realistic and concrete target shows a momentous proliferation of increased glycerol concentration from 10.43% to 44 %. The other properties of glycerol were studied and compared with standard methods.

KEYWORDS: Biodiesel, crude glycerol, high-value chemicals, GC-MS and GC-FID.

1. INTRODUCTION:

The world's economy largely depends on the transportation of goods and its service. The anticipated energy requirement after a decade will be increased by 50%, which is witnessed by an increase in the number of vehicles[1]. On one hand, the energy crisis has been confronting the world due to the excessive exploitation of the world's oil reserves[2].

Increasing energy demand and a significant increase in unburned hydrocarbons, carbon monoxide and other emissions from fossil fuels forced the researchers to work on renewable energy[3]. The renewable energy sources lead to the production of biofuel has gained a sustainable source gained an important form of technological progress in diminishing pollution[4].

The increase in biodiesel production inevitably increases the production of waste stream glycerol with copious impurities. The byproduct from the biodiesel production was rich in glycerol content, generally in the ranges from 10 to 20% of the total volume[5, 6]. The composition of crude glycerol vary from one biodiesel production plant to another and is mainly determined by the feedstock oil composition and quality, the oil and methanol molar ratio used in transesterification, type of catalyst used, and the detailed procedure such as with or without methanol recovery[7,-9].

Furthermore, the expansion of biodiesel production and the p ınol, unreacted triglycerides and catalyst have made the price of glycerol to fa Act, the crude glycerol must be disposed of in a short period or to be sol isposal is also expensive[11]. Therefore, direct use or partial purification of cru ion of glycerol to biodiesel is an interesting way of utilizing the original or partially purified crude glycerol.

The composition and nature of impurities in the crude glycerol can have a positive or negative effect on the progress of biotechnological fermentation processes[15,16]. An assortme of glycerol as a sole or complementary carbon source and ene as dihydroxyacetone, succinic acid, organic acid, ethanol and pr e removed by different stages. In the first stage, the crude gl h methanol was removed as it shows an inhibitory effect on r ol content which can be achieved through precipitation which occurs during acidification, whereby free fatty acids and some salts are removed[19-21]. The final step is the purification and refining step which can be achieved with the e desired degree. Combination of methods includes adsorption, filtration and centrifugation which aids in removing the precipitated free fatty acid and other suspended solids[22].

In this paper, positive efforts are made for the communal removal of impurities are reconnoitered by three step purification

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In this paper, positive efforts are made for the continual removal of impurities are reconnoitered by three step purification processes. The concentration and characterization of glycerol at both the stages were carried out by GC- FID, GC-MS analysis and other standard methods.

2. MATERIAL AND METHODS:

2.1. Sample collection:

Increased production of biodiesel made easier for the availability of crude glycerol stream round the year, which encourages the researchers to work on it. The crude glycerol sample was obtained from biodiesel producing facilities located in Bannari Sugar Pvt. Ltd., Tamil Nadu. The pH of crude stream glycerol was found to be 9.6 and it appears highly viscous brown fluid. The crude glycerol obtained from the single batch was used throughout the study. Other chemicals used for this study are of commercial analytical grade.

2.2 Removal of methanol (Step 1):

Methanol the major impurity present in crude glycerol was separated initially. As crude glycerol was highly viscous and semi-solid condition, it was then heated in a simple distillation process at 65^{o}C to recover the unreacted methanol during transesterification process. The distillation was carried out with 200 ml of crude glycerol sample at various intervals of time from 5 to 20 min. The concentration of recovered methanol was estimated by GC-FID.

2.3 Acidification process (Step 2):

The concentrated crude glycerol sample was subsequently acidified with concentrated HCl and H_3PO_4 separately with various volumes to lower the pH of the sample. After acidification, samples were stirred for 10 min at room temperature and left undisturbed in a separatory funnel for the phase separation to occur. Three distinct phase like free fatty acid at the top layer, glycerol-rich phase in middle and salt at the bottom layer.

2.4 Chemical treatment and Centrifugation (Step 3):

The glycerol-rich phase recovered individually from HCl, H_3PO_4 andtreated with glycerol. It was then placed in water bath at 80^{o}C for 30 minutes with the addition of sodium oxalate, which helps to remove the minor impurities and whose action was not clearly known[23]. As the glycerol sample was the clear and straw yellow colour it was further, treated with activated charcoal. The adsorption process was carried out with 1 % activated charcoal to remove odour, colour and filtered using Whatmann filter paper No. 1. Finally, the filtered samples were centrifuged at 8000 rpm for 10 min to remove suspended solids and free floating fatty acids.

2.5 Characterization of glycerol samples:

The physico-chemical properties like density, pH, ash content, flash point, fir point, cloud point, and pour point of step 2 and step 3 purified glycerol samples were compared with crude and commercially available pure glycerol in standard methods. The concentration of glycerol samples at the end of stage 2 and stage 3 were analyzed in GC-FID and impurity removal rate and increase in the concentration of glycerol was studied through GCMS.

3. RESULT AND DISCUSSIONS:

The nature and purity of glycerol produced from biodiesel industries mainly depend on the feed stock used. Many microorganisms have high affinity towards glycerol as it is a good carbon and energy source it can be used as the substrate for the industrial fermentation process.

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3.1 Crude glycerol analysis:

Crude glycerol obtained from the supplier was darn brown and highly viscous the pH 0f 9.6. The GC-FID analysis revealed that initial concentration of glycerol about 10.43%. GCMS studies in Fig 1 reveals that crude glycerol sample consists of unknown impurities like Acetophenone, benzoic acid, methyl ecanoic acid, cyclopentadecanone etc., apart from the common impurities like

Fig 1 GC-MS analysis of crude glycerol

Step 1:

The presence of methanol in crude glycerol stream may b n process not only helps to remove the methanol but also ano m methanol is recovered at 15th min and as time proceeds as redistilled further to increase methanol concentration and e excess non-reactive methanol for reuse is an economic wa of methanol is cost-effective when compared with commercially available one and also reported that recovery of methanol from crude glycerol was necessary, before using it in bio-conversion process[24, 25]. The concentration of recovered methanol after distillation was found to be 54.7%. The redistilled methanol shows 59.7% which was 5% higher than the first distillation and samples were analyzed in GC-FID.

Step 2:

After distillation, 200 ml of residual glycerol was added to phosphoric acid and hydrochloric acid separately to alter pH. The initial pH of crude glycerol was 9.6 and it is reduced to acidic condition. This is because, the H^+ ions of the mineral acids can convert the soluble soap into insoluble free fatty acids, which floats at the top layer. Then the remaining ions coupled with a catalyst (sodium or potassium) used for biodiesel production which precipitates at the bottom layer[26]. The middle layer constitutes glycerol rich phase, which was removed by simple decantation method using separating funnel. 10 ml of phosphoric acid showed significant result in separation time and maximum liberation of glycerol. Further, increasing the volume of acid decreases the pH, which concomitantly decreases glycerol phase separation and increases the volume of free fatty acids. The separation of glycerol-rich phase was well in the range of pH 4-6 which was achieved by adding acid in glycerol[27]. At the end of step 2 the glycerol samples were collected carefully from the two different separating funnel. Phosphoric acid showed better phase separation time, high glycerol concentration and high volume of glycerol rich phase when compared with hydrochloric acid. GC-MS analysis of distilled glycerol treated with hydrochloric acid and phosphoric acid reveals the reduction in impurities in fig 2 and fig 3.

Fig 2 GCMS analysis of glycerol treated with HCl in step 1 purification process

Fig 3 GCMS analysis of glycerol treated with H_3PO_4 in step 1 purification process

Step 3:

The resultant step 2 glycerol treated with two mineral acids and it was subjected to stage 3purification process. Step 2 glycerol sample was treated separately with sodium oxalate to remove impurities in stage 2 purification process. The reaction temperature of sodium oxalate with glycerol had a great impact on removing impurities and noticed that 80°C for 30 min showed acceptable impurity removal rate than 60 or 70°C temperature whereas change in colour of glycerol was observed at 90°C. Other work on sodium oxalate also reported that 19.8% of impurities were removed at 80°C for 30 min than other temperatures[23]. Adsorption process with 1% charcoal reduces the color of glycerol from dull brown to straw yellow. The filtered sample after adsorption process was centrifuged to remove suspended solids based on its density difference and traces of free fatty acids which floats on the top surface. The single glycerol peak was achieved in stage 3 purification process and displayed in fig 4 and 5. GC-FID analysis of glycerol concentration at the end of step 2 and step 3 purification process was estimated for two mineral acids and compared with crude glycerol as displayed in table 1. Phosphoric acid shows a better separation of glycerol rich phase and removes the maximum impurities in crude glycerol sample.

Fig. 4 GC-MS analysis of step 3 glycerol treated with HCL

Fig. 5 GC-MS analysis of step 3 glycerol treated with H_3PO_4

Table 1. Concentration of crude and step 1 and step 2 glycerol samples

Sample/ Acid	Crude glycerol concentration	Glycerol concentration at the end of step 2	Glycerol concentration at the end of step 3
	10.43%		
HCL		22%	32.30%
H_3PO_4		25.10%	**44%**

3.3 Characterization of crude glycerol:

The properties of crude glycerol, purified glycerol and pure gly ed by standard methods and discussed in table 2. The characterization of crude with two acids were analyzed separately by GC-MS and found phosphoric ac values of flash point, fire point, cloud point etc., were very close and similar The H^+ ion is more concentrated in phosphoric acid when compared with hydrochloric acid which obviously results in low pH value of phosphoric acid treated glycerol.

Table. 2 Comparison table for the property values of HCL and H_3PO_4 a

S.No	Properties	Crude glycerol	Pure glycerol	Step 2 glycer treated with			
1	Cloud point	-11°C	-53.5°C	-60°C			
2	Pour point	-16°C	-58.5°C	-65°C	-67°C	-47.7°C	-56.7°C
3	Flash point	120°C	177°C	148°C	135°C	196°C	185°C
4	Fire point	211°C	204°C	169°C	157°C	230°C	220°C
5	Vapour pressure	0.04 kg/cm^2	0.16 kg/cm^2	0.08 kg/cm^2	0.083 kg/cm^2	0.09 kg/cm^2	0.14 kg/cm^2
6	Carbon residue	12%	11.25%	7.50%	7.50%	13%	12%
7	Ash content	11.25%	<0.2%	0.17%	0.17%	0.13%	0.13%

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8	Solubility with water	Miscible	Miscible	Miscible	Miscible	Miscible	Miscible
9	pH	9.6	6.9	5.6	5.4	4.8	4.5

4. CONCLUSION:

If the purified glycerol was utilized by biodiesel industries itself for the production of high-value chemicals such as ethanol, DHA and other platform chemical products. The biodiesel industries enjoy numerous benefits like self-disposal of crude glycerol, zero liquid discharge and eliminate the risk contamination followed by legal sanctions. Thus, phosphoric acid was found to be the best acidifying agent when compared with hydrochloric acid when tested for crude glycerol purification process. The phosphoric acid showed a significant result in phase separation time and volume of glycerol phase liberation. Phosphoric acid and sodium oxalate combination almost removed all major and minor impurities present in the crude glycerol and increases the glycerol content from 10% to 44%. Further purification process with high-end equipment, will accelerate the concentration of glycerol meanwhile it also increased the purification cost drastically. The other properties of step 3 phosphoric acid treated glycerol were also found closer to commercial pure glycerol. The purity of the purified products was confirmed by GC- MS/FID. Thus 40- 45 % of a partially purified glycerol was enough for the microbes to utilize it as sole carbon or energy source for the fermentative production of value-added products.

5. CONFLICT OF INTEREST:

The authors have no conflicts of interest to declare.

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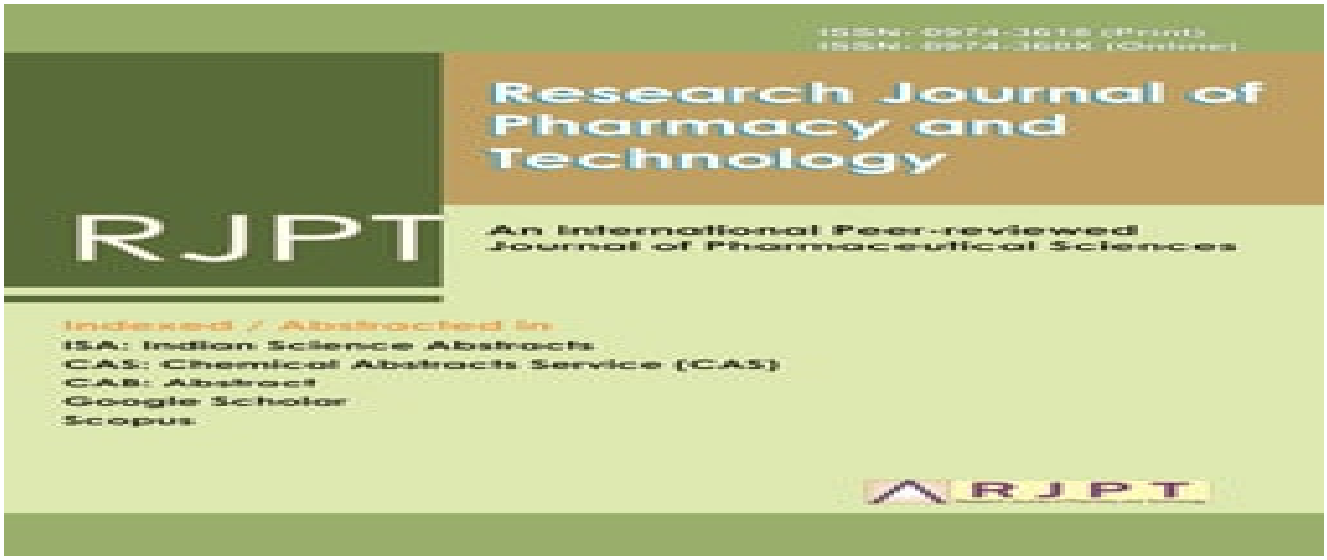
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