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Studies on Purification of Crude Glycerol using Ion Exchange Resin

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***Abstract*:-As the demand and production of biodiesel grows faster rate, the commercial utilization of the glycerol is increasing rapidly. This research work was carried out to analyze the crude glycerol to obtain purified glycerol using best suitable ion exchange resin and studied its alkalinity, ash content, water content and matter organic non glycerol present it also obtained good percentage of glycerol by utilising this resin. The conventional methods of glycerol purification such as neutralization, evaporation, purification and refining steps. Novel methods such as Ion exchange and membrane separation. The use of ion exchange resins for treatment of crude glycerol needs to be explored, but very few resins been used for purification of glycerol and need to undergo the purification study by using other ion exchange resins and check its purity level. By using this ion exchange resin method, which could directly perform the purification which would avoid the many other series steps of conventional methods. Crude glycerol was purified by using ion exchange resin bed column method and carried out the experiments for various bed height, by changing the residence time, by varying the temperature of the process, then following the UV and GC analysis crude glycerol sample contained 18% of glycerol is purified to 40%. The percentage purity level obtained was good about 40% purity level. So at 6cm bed height in cm and temperature about 40 ºC was the best suitable for the purification study. This is the very new research work carried out at experimental level and can be application to industrial level for better purification as direct method.**

Key words: Crude glycerol, ion exchange, purification, GC, column study

1. INTRODUCTION

Today the demand for fossil fuel are high, but the fuel required to meet the demand are very limited sources, so the alternative fuel utilization such biodiesel are becoming very attractive. Crude glycerol is one of important byproduct of biodiesel production process. The crude glycerol is the one of source, when it is purified and meet the suitable standard are well applicable as raw material for production of value added products such as 1-3 propandiol, ethanol, and animal feed etc. It is also as a raw material for bioenergy production. There are many series of methods are available to purify the biodiesel such as distillation, drying, saponification, acidification and neutralization, extraction, ion exchange and adsorption. Industry economics utilization of crude glycerol for value added products are critically important and the recovery and purification of glycerol is play an important role in biodiesel cost reduction. So in this study the purification performance of the crude glycerol was carried out and compared it with purified glycerol parameters by using ion exchange resins through the column study. Crude glycerol is 30-40% pure and is often concentrated and purified prior to commercial sale to 95.5–99% purity (1).

EXPERIMENTAL SECTION

2.1 Materials and Methods

Materials

The Crude Glycerol which was used for the research work was procured from University of Agricultural Science, Gandhi Krishi Vignyana Kendra, campus, Bangalore, ion exchange resin was procured from Thermochem Corp PVT. Ltd RT Nagar, Bangalore, NaOH Solution, Oxalic acid crystals, HCl acid, KOH pellets, Phenolphthalein indicator, Isopropyl Alcohol were of analytical grade.

2.2 Characterization of crude glycerol

The analysis of crude glycerol sample was carried out to study its alkalinity, water, ash, matter organic non glycerol and glycerol content. The analysis of alkalinity found that it was basic in nature, water and ash content are lowered after treated with ion exchange resin, the glycerol content percentage also increased after the purification of the crude glycerol. so that could be easy to identify it's total amount of the compositions such as glycerol, ash , water, and matter organic non glycerol content present in it and how best the purification level been achieved by following the ion exchange resin method.

2.3 Purification method

The purification of the crude glycerol was carried out to remove or lower its impurities which might cause the process of purification of glycerol to value added products. Column was fabricated to operate as per the experimental steps to be followed during the research work. The scale was provided in cm using graph sheet and sample glycerol inlet and outlet were provided hence to carry out the ion exchange process at predetermined time intermittent time duration and collected the purified glycerol for further analysis. The study carried out was the batch studies to purify glycerol using ion exchange resin column and followed the further analysis of crude glycerol and purified glycerol by using UV and GC techniques. These UV and GC analysis were carried out to check its absorbance level and the purity level in percentage and also to know it composition levels/purity as per peak areas were calculated to check its concentrations before purification and after purification.(2).

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2.4 Ion exchange resin method

The ion exchange resins were studies by using resin column methods to check their ability effect methanol, Glycerine, soap, acid value and oxidative stability parameters of the biodiesel. The crude glycerol was used for the study. From this method it was found that ion exchange resin has little effect on methanol concentration after the bed has reached equilibrium. Purification with the ion exchange resin caused the acid value of the purified glycerol to increase slightly. Oxidative stability did not seem to be affected. Ion exchanging was a special type of separation process where ions were adsorbed from the fluid surrounding the ion exchange material. This adsorption releases ions of the same charge from the ion exchange material into the fluid soap and Glycerine were reduced significantly. Macroporous resin was used for the ion exchange mechanism, which involves an exchange of one or more ionic components of impurities of the glycerol sample, so major impurities components will be removed by this method in a given set of time intermittent. The purified glycerol then been analysed to check its concentrations and purity levels by UV and GC techniques. It gave a good purification at higher level when its temperature been increased during the ion exchange process(3).

RESULTS AND DISCUSSION

3.1 Alkalinity test

The alkalinity for varying resin bed height in cm was studied. Alkalinity is nothing but amount of glycerol alkalinity or acidity

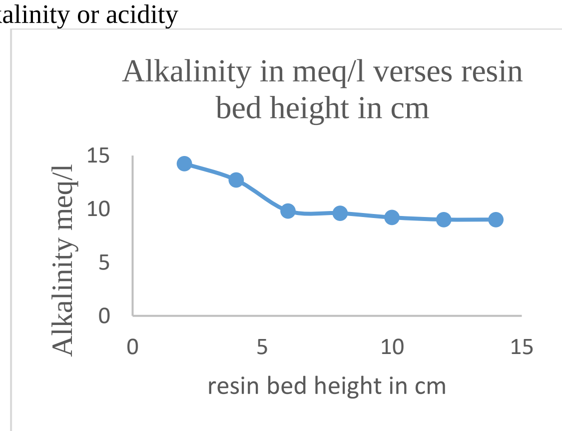

Fig 1: Graph of Alkalinity in meq/l verses resin bed height in cm

is the number of milli alkali or acid equivalent contained in glycerol 100gram of sample. The alkalinity in meq/l was calculated for each different resin bed height such 2cm,4cm,6cm,8cm,10cm,12cm etc, and compared the values. Alkalinity is the extent of base present in glycerol sample as the increase resin bed height, the alkalinity value found to be decreasing, showed that it was basic in nature as shown in fig.1. When compare it with alkalinity of crude glycerol sample, there is more variation towards decreasing the alkalinity of the purified sample.

3.2 UV analysis for purified glycerol

By observing the results obtained for absorbance verses resin bed height in cm, the absorbance was increased as there is increase in the resin bed height in cm, so UV-VIS analysis shows that the absorption at characteristic of the OH bond, it was

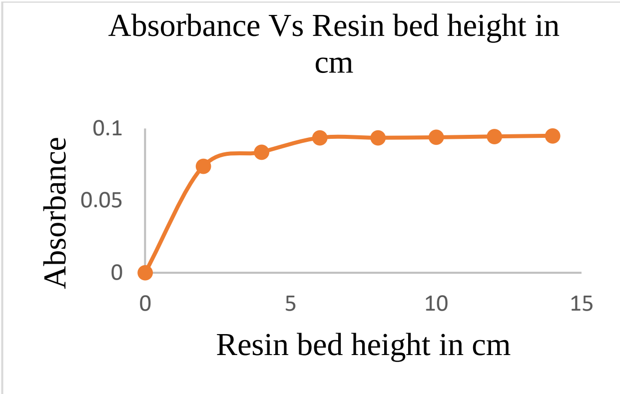

Fig 2 : Graph of absorbance verses resin bed height in cm

observed that at 6cm resin bed the absorbance remains constant as shown in fig.2. So the purified samples for 6cm bed height would be the optimum bed height for the glycerol purification study. The 6cm resin bed height parameter been suitable for the purification study.

3.3 UV analysis for purified glycerol for varying temperature

From the results obtained from above data, it was observed that the as increase in the temperature of the resin bed column, there was decrease in the absorbance and it remained constant from 40 °C as shown in

fig.3, and the percentage purity of glycerol was 40%, so obtained more purity by using this temperature, so 40 °C was the suitable temperature for the glycerol purification study.

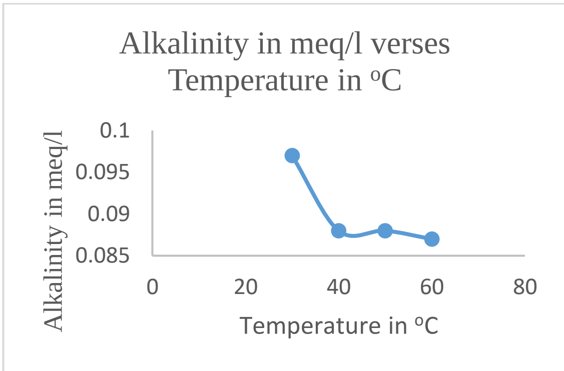

Fig 3 : Absorbance verses temperature in °C

3.4 GC analysis of crude glycerol, purified glycerol and commercial grade glycerol.

This was the GC analysis report for the crude glycerine. Hence the crude which contains impurities, it shows the percentage purity of glycerol was 18% as shown in fig.4. The peak area represents the amount

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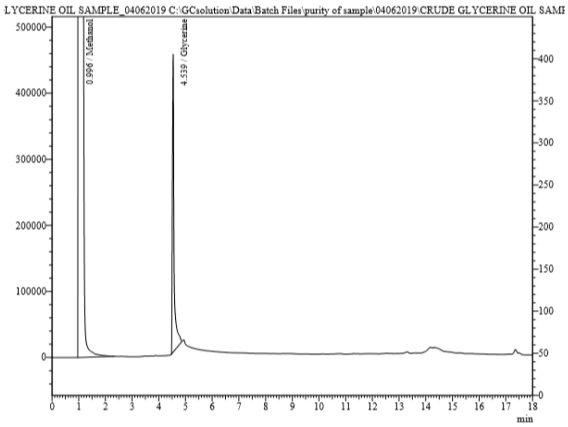
of compound that has passed through the detector. Peak area tells how much of glycerol sample was present and retention time tells what was the sample are present. This GC analysis was carried out methanol solvent to give the accurate GC chromatogram. The results presented as a percentage of the area/height ratio (4).

3.5 GC analysis for purified glycerol with resin

This was the GC analysis report for the standard glycerine. Hence the crude which contains impurities. The peak area represents the amount of compound that has



Peak#	Name	Ret.Time	Area	Area%	Height	Height%
1	Methanol	0.996	673394739.218	99.7059	27957168	99.6499
2	Glycerine	4.539	1986468.354	0.2941	449611	0.3501
Total			675381207.572			

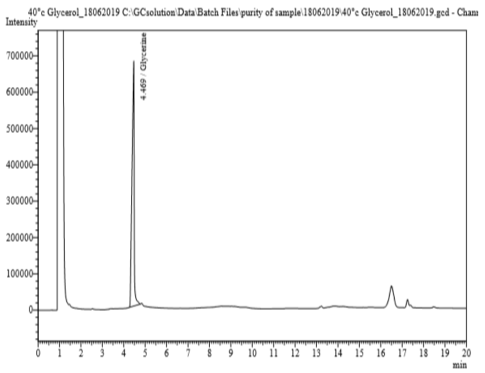
Fig 4: GC chromatogram of crude glycerol

passed through the detector. Peak area tells how much of glycerol sample is present and

retention time tells what was the sample are present. It showed the percentage purity of glycerol was 27% as shown in fig.5. This GC analysis was carried out methanol solvent to give the accurate GC chromatogram. The results presented as a percentage of the area/height ratio (5).

3.6 GC analysis for purified glycerol with resin at 40 °C

This was the GC analysis report for the purified glycerol at 40 °C. Hence the crude which contains impurities. The peak area represented the amount of compound that has passed through the detector. Peak area tells how much of glycerol sample was present and retention time tells what was the sample were present. It showed the percentage purity of glycerol was 40% as shown in fig.6. This GC analysis was



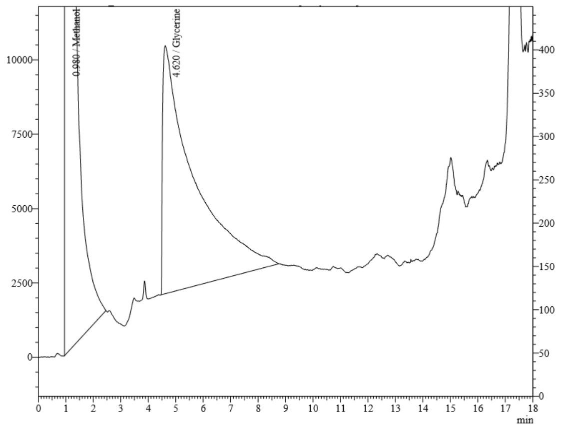
Peak#	Name	Ret.Time	Area	Height	Area%	Conc.
1	Glycerine	4.47	4669951	673347	100.00	100.000

Fig 6: GC Chromatogram of purified glycerol at 40 °C

carried out methanol solvent to give the accurate GC chromatogram. The results presented as a percentage of the area/height ratio (6).

3.7 GC Analysis for commercial grade glycerin

This is the GC analysis report for the standard glycerine, in this GC chromatogram we observe that, hence the crude glycerol which contains impurities. The peak area represents the amount of compound that has passed through the detector. Peak area tells how much of glycerol sample is present and retention time tells what is the sample are present. It shows the percentage purity of glycerol is about 41.7% as shown in fig.7. This GC analysis was carried out methanol solvent to give the accurate GC chromatogram. The



Peak#	Name	Ret.Time	Area	Area%	Height	Height%
1	Methanol	0.980	717089835.945	99.9076	36794057	99.9939
2	Glycerine	4.620	663176.330	0.0924	8341	0.0061
Total			717753012.275			

Fig 7: GC Chromatogram of pure glycerol results presented as a percentage of the area/height ratio (7).

CONCLUSION

The various parameters were investigated to study its purity levels. The percentage amount of glycerol content in crude obtained was 18%, and by carrying out the purification with the resin with bed height 6 cm was 27%, then by studying the purification by variation of temperature about 40 °C was

40% which was almost close to the commercial grade glycerine purity level. By comparing the data obtained from alkalinity and UV analysis for various bed height, observed that the alkalinity which represents the extent of base present in the sample, from 6 cm onwards the alkalinity remains constant , by comparing the UV analysis data at 40 ºC the absorbance almost remains constant. The percentage purity level obtained was good about 40% purity level. So at 6cm bed height in cm and temperature about 40 ºC is the best suitable for the purification study. As have carried out the analysis of alkalinity for different time period, such as 8, 16 and 24 hours , so 8 hours considered as suitable time. Because more than 8 hours time alkalinity of the sample doesn't vary much. And as per the various temperature parameter changes 40 ºC is the suitable range to run the column studies, and obtained good percentage of glycerol content at 40 ºC temperature. Hence this temperature was optimum parameter to carry out the column studies for glycerol purification. From the GC analysis for crude glycerol and the purified glycerol it was observed that , the percentage amount of glycerol increased by performing the ion exchange process at 6cm optimum resin bed height. So the results presented as a percentage of the area/height ratio. This ion exchange method could be most suitable direct method for the study of purification level of glycerol. When compared the results of variations in temperature parameter, percentage purity level obtained here of less compared with 40 ºC temperature. Hence 40 ºC was considered as the suitable temperature for the best suitable purification study(8).

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