

Characterisation and Purification of Crude Glycerol Recovered from Transesterification of *Jatropha curcus* Oil

P. CHITRA, P. VENKATACHALAM AND A. SAMPATHRAJAN

Department of Bio-Energy, AEC&RI, Tamil Nadu Agricultural University, Coimbatore-3, India.

Abstract : Glycerol, a by-product obtained from transesterification of *Jatropha curcus* oil was analysed for different characteristics, using standard test methods and a preliminary study was undertaken to purify the crude glycerol. The characteristic analysis of crude glycerol, namely, pH, soap content, ash content had the average values of 10.25, 3.97 per cent and 2.03 per cent, respectively. On average, 41.8 per cent distilled glycerol was recovered during refining process. The average methanol and water contents distilled over were found to be 5 and 11.4 per cent, respectively. The mean pH of the distilled glycerol was found to be 1/3rd of the pH of the crude glycerol. The soap content was nil and ash content was found to be 0.051 per cent in the distilled glycerol.

Key words: Characterisation, Purification, Crude glycerol and Distilled glycerol.

Introduction

Glycerol (C₃H₈O₃) is an alcohol with three hydroxyl groups (OH), which is obtained as a by-product of biodiesel production through transesterification of *Jatropha curcus* oil with methanol. The glycerol obtained is crude which poses a disposal problem and (Yong *et al.* 2001) needs purification. The crude glycerol obtained as a byproduct, from transesterification is only about 50 per cent glycerol. It contains some excess methanol and most of the catalyst (NaOH) and soap (Jon Van Gerpen, 2004). A process to recover the distilled glycerol from crude glycerol and characterization of the crude and distilled glycerol is described.

Materials and Methods

The lab scale glycerol distillation apparatus was developed for the purification of glycerol and it involved different procedural steps. The steps followed for the purification of glycerol obtained as a by-product from alkali-catalysed transesterification of *Jatropha curcus* oil are acidulation and separation (neutralisation) (Jon Van Gerpen, 2004), distillation of methanol

(Foidl *et al.* 1996), evaporation of water (Zhang *et al.* 2003) and vacuum distillation (Yong *et al.* 2001).

Three samples of crude glycerol (CG1 to CG3) and distilled glycerol samples (DG1 to DG3) were analysed for the following characteristics by using standard test methods viz. pH (Mettler Toledo, MP 220), NaOH / Soap content Product Safety Bureau Ref. Manual (Canada) method C14-1, 1987, Ash content IS: 1448-1992 method and Free fatty acid and acid value A.O.C.S Official Method Ca5a - 40.

Results and Discussion

After having analysed the characteristics of crude glycerine, the samples were acidified and pH was brought down below the neutralization point and taken for refining. The table 1 shows the mass balances and appearances of distilled fractions recovered from crude glycerol upon refining. The average amount of distilled glycerol obtained from 10 g of crude glycerol was determined as 4.18 g, which was slightly yellowish

Table 1. Mass balances and appearances of distilled fractions recovered from crude glycerol

Crude glycerol (g)	Distilled glycerol (g)	Distilled bottom/residue (g)	Distilled methanol (g)	Distilled water (g)	Losses (g)
10	4.48	3.48	0.4	1.33	0.31
10	3.84	4.39	0.6	0.98	0.19
10	4.12	4.08	0.5	0.10	0.20
Average	4.18(41.8%)	4.18(41.8%)	0.5 (5 %)	1.14(11.4%)	0.24 (2.4%)

Table 2. Characteristic analysis of crude glycerol and distilled glycerol obtained from transesterification of *Jatropha curcus* oil

Sl. No.	Characters	Crude glycerol samples				Distilled glycerol samples			
		CG1	CG2	CG3	Avg.	DG1	DG2	DG3	Avg.
1	PH	10.04	10.30	10.40	10.25	3.80	3.90	4.20	3.97
2	Soap (%)	4.50	3.56	3.84	3.97	Nil	Nil	Nil	Nil
3	Ash (%)	1.80	2.30	2.00	2.03	0.048	0.054	0.051	0.051
4	Free fatty acids (%)	0.137	0.149	0.143	0.143				
5	Acid value (mgKOH/g of sample)	0.273	0.296	0.285	0.285				

in colour. The residue left after distillation was dark brown in colour.

The crude glycerol samples of 10 g were subjected to vacuum distillation and 7.7 g of distillates were obtained at 110 to 120°C (Hazimah *et al.*, 2003). On average 41.8 per cent was recovered from the glycerol content of 50.4 per cent in the crude glycerine (Yong *et al.* 2001) as some of the glycerol was lost in the side reactions described and remained in the distilled bottom.

The average methanol and water recovered were found to be 0.5 g and 1.14 g respectively

as shown in table 1. There were also some losses, which accounted to be 0.24 g on average. The moisture content of crude glycerol varied from 1.5 to 6.5 percent (Hazimah *et al.*, 2003).

The characteristic analysis of crude glycerol (CG) obtained from transesterification of *Jatropha curcus* oil and distilled glycerol (DG) collected upon refining of crude glycerol is shown in table 2.

The mean pH of the crude glycerol (CG) was found to be 10.25 as shown in table 2. The average soap content and ash content

