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Sequential Stage Treatment of Crude Glycerol Towards Synthesis of Monoethylene Glycol for Gas Hydrate Inhibition

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Abstract

Gas hydrate is a critical flow assurance challenge in deepwater oil and gas production and natural gas transmission. It grows rapidly to form plugs that interrupt the free flow of fluid in flowlines. The prevention of gas hydrate has been effectively achieved using thermodynamic hydrate inhibitors such as methanol and monoethylene glycol (MEG). These chemicals are synthesized from petrochemical feedstock which is a non-renewable resource that is currently limited by environmental regulations. However, recent studies utilized biomass-derived glycerol as an alternative feedstock for the synthesis of a range of commercial chemicals. In this study, the quality of crude glycerol obtained by transesterification of triglyceride in vegetable oil was improved to technical grade glycerol in three process steps. The purified sample was characterized for its physicochemical properties following standard procedures, and the quality was analyzed using UV–Visible and FT–IR techniques. The absorption frequency obtained from the IR spectrum of the purified sample corresponds to the vibrational absorption bands of glycerol. The combined H₃PO₄ acidification to pH of 3, extraction with isopropanol at 66% (%v/v), and decolourization using adsorption onto activated charcoal at 180g/1000mL glycerol solution increased the purity of crude glycerol to 97%.

Keywords: Gas hydrate, Glycerol, Inhibitor, Monoethylene glycol, Transesterification.

1. Introduction

Gas hydrates are crystalline inclusion compounds that formed at thermodynamic conditions typical of deepwater oil and gas production systems. Its deposition and growth in these systems constitute a critical flow assurance challenge and a threat to operational safety, health, and the environment. The conventional method to overcome this challenge is by inhibiting using a certain class of chemicals. Thermodynamic inhibitors such as alcohols and glycols have been used for nearly as long as the hydrate flow assurance problem is known ^[1-2]. They are widely used due to their unique combination of properties, and they are most suitable at every stage of hydrate formation in production systems from the start-up to disperse and/or dissociate hydrate plugs to shut down for fluid displacement.

However, these chemicals are usually obtained from petrochemical feedstocks which are currently limited by health and environmental factors as well as costly and complex conversion processes. Biomass-derived materials have become alternative low-cost feedstocks that are employed in the green reaction medium and synthetic chemistry ^[3-6]. One such bio–feedstock is glycerol obtained from the homogenous catalytic transesterification of the triglyceride in vegetable oils or animal fats with excess alcohol according to Scheme 1.

Glycerol is a multifunctional organic compound and the most widely distributed polyhydric alcohol with three hydroxyl groups which are responsible for its hygroscopic nature and the formation of intra- and inter-molecular hydrogen bonds ^[7]. It possesses both hydrophilic and hydrophobic properties, is highly stable under most reaction conditions, and is capable of being reacted as alcohol to form an unusually large number of functional derivatives. It is a sweet, clear, colourless, odourless, highly viscous, and high boiling liquid. It is compatible with

many other chemicals (often used as a substitute for the ethylene glycols in most applications), virtually non-irritating, and has no negative environmental effects. Its versatility is a tribute to its renewable origin, and the unique combination of physicochemical properties makes it suitable for most industrial applications.



Scheme 1. Transesterification of triglycerides in vegetable oils

Glycerol has a ubiquitous use pattern, as shown in Figure 1. It was usually disposed of as a waste by-product of the transesterification of triglyceride at its first discovery by Scheele in 1779. However, the synthesis of trinitroglycerine by Alfred Nobel in 1866 used in explosive devices thrust glycerol into economic and military importance. Glycerol is primarily used as a constituent to maintain texture, prevent freezing, prolongs shelf life, and add humectancy to foods, drugs, oral and personal care, and several other industrial products.



Figure 1. Glycerol market (after ^[8])

The incorporation of glycerol as an oilfield chemical for petroleum production has been greatly achieved with such applications as down hole lubricants, antifreeze solutions, fuel additives, emulsifiers, polymers, and surfactant ^[9,10]. The most recent research focused on its use as a building block for the production of industrial chemicals. For example, glycerol is converted to hydrogen-rich syngas by different methods, including catalytic treatment ^[11], pyrolysis ^[12], and steam reforming ^[13-14]. Other liquid products include diols, acrolein, acetal, acetaldehyde, formaldehyde, hydroxyacetone, bio-oil and bio–lubricant additives ^[8,15-16].

However, the glycerol obtained by homogenous catalytic transesterification is crude and not pure enough for direct use in the aforementioned value-added applications. The purity ranges between 15 – 80% depending on the type and amount of catalyst used, the amount of alcohol in excess, and the conversion achieved ^[17]. A typical composition (w/w) of crude glycerol is 30% glycerol, 50% methanol, 13% free fatty acids (FFAs), 2% moisture, 2–3% inorganic salt (catalyst residue), and 2–3% Matter Organic Non-Glycerol (MONG, such as esters, glycerides and vegetable colour) ^[18]. Crude glycerol is usually purified into three major grades; technical grade (95.5 wt%), USP grade (>96 wt%), and CP grade (99 wt%). The different purification methods include crystallization ^[19], vacuum distillation ^[20-21], ion-exchange chromatography ^[22-23], electrodialysis ^[24], membrane filtration ^[25], physical and chemical treatment such as evaporation, neutralization, solvent extraction, and adsorption ^[26-27]. Nevertheless, the crystallization method could not be commercialized due to the slow rate of crystallization, difficulty in obtaining seed crystals and separating them from the mother liquor. The use of vacuum distillation is a complex and energy-intensive process. Both vacuum distillation and ion–exchange resins produce a low yield of USP grade glycerol, while membrane

filtration yields USP grade glycerol provided the crude glycerol sample undergo prior purification to reduce the salt and MONG content.

The global production of glycerol is growing at a compounded annual rate of 6.3% due to industrialization and an increase in the production of fatty acid methyl esters for biofuel formulations ^[28]. Also, the global demand is expected to increase geometrically as new applications of glycerol continuously emerge and a wide variety of organic reactions and synthetic methodologies successfully developed. A similar production increase is expected in Nigeria following the number of notable proprietary researches towards the substitution of petro–fluids with bio–fluids in different applications. The utilization of glycerol as a building block for the production of value-added products is highly significant, and the purification of crude glycerol is critically important. Therefore, this study aimed to improve the quality of crude glycerol to technical grade glycerol suitable for the synthesis of monoethylene glycol as a gas hydrate inhibitor.

2. Materials and methods

Pure vegetable oil extracted from palm seeds, NaOH (\geq 95%, pellet, Merck), KOH (\geq 85%, pellet, Merck), phenolphthalein (powder, Merck), NaCl (\geq 95%, powder, Merck), commercial glycerol (98%, BDH), H₂SO₄ (98%, Merck), H₃PO₄ (85%, Merck), HCl (37%, Merck), methanol, ethanol and isopropanol (99.9%, BDH), activated charcoal (powder, BDH), deionized water.

2.1. Esterification

Fresh vegetable oil was refined to reduce the free fatty acid (FFA) content by neutralization with 0.5N NaOH at 70°C (equation 1), and NaCl (10 wt%) was added to enhance soap separation. The refined oil was separated, washed repeatedly with warm water, dried at 110°C, and stored appropriately.

 $RCOOH + NaOH \xrightarrow{70^{\circ}C} RCOONa + H_2O$

(1)

2.2. Transesterification

The transesterification of the refined oil with excess methanol using NaOH as catalyst was conducted batch-wise in a three-necked round bottom flask equipped with a condenser, thermometer and stir bar. The reaction products were kept sufficiently long enough to separate by gravity, and the crude glycerol bottom layer was recovered for purification.

2.3. Purification of crude glycerol

2.3.1. Evaporation

Excess methanol in the crude glycerol sample was completely evaporated under vacuum at 40°C and recovered using a rotary evaporator.

2.3.2. Acidification and neutralization

The crude glycerol sample was titrated with sulphuric acid, phosphoric acid, and hydrochloric acid to the desired pH. A total of nine 300g molten crude glycerol samples contained in 500mL beakers were prepared and repeatedly titrated with each acid to pH 1, 3, and 5. The mixtures were kept sufficiently long enough to separate by gravity into two or three distinct layers. The top layer was gently decanted, while the glycerol-rich middle layer was separated from the bottom inorganic salt layer (if present) by filtration. Each glycerol-rich filtrate was neutralized to pH 7.0 using 5M NaOH and kept sufficiently long enough for salt precipitation. The samples were heated to 100°C to evaporate water and to further enhance salt precipitation and recovery.

2.3.3. Solvent extraction

The purity of the glycerol sample was further enhanced by extraction in low molecular weight polar solvents (methanol, ethanol, and isopropanol) at varying proportions ranging from 75%(v/v) to 25%(v/v). The glycerol solution was separated from the salt by filtration.

2.3.4. Adsorption with activated charcoal

The coloured impurities, salt, and MONG in the glycerol solution were further reduced by adsorption unto activated charcoal. The process was evaluated at the different weights of activated charcoal (50g, 80g, 120g, 150g, 180g, and 200g) in 1000mL glycerol solution. The mixture was stirred at 100°C and refluxed for 60 minutes in a three-necked round bottom flask (equipped with a condenser, thermometer, and stir bar). The mixture was separated by vacuum filtration, and the solvent was recovered by simple distillation to obtain pure glycerol, dried at 110°C and stored appropriately for further analysis.

2.4. Analytical procedures

2.4.1. Quantitative analysis

The %FFA of fresh vegetable oil was determined according to equation 2 by titrating the oil sample against KOH in hot isopropanol solution using phenolphthalein as an indicator ^[29]. The water content of the glycerol sample was determined using the volumetric Karl Fisher titration method, while the ash content was determined by heating 1g of the sample at 750°C for 3 hours in a muffle furnace ^[30].

 $\% FFA = \frac{28.2(NV)}{W}$

(2)

where: N-normality of KOH solution (mL); V-volume of KOH soultion (mL); W-weight of oil (g).

The glycerol concentration was determined by matching its refractive index measured at 20°C with the standard table of glycerol–water solution ^[31–33]. The MONG content was determined according to equation 3 ^[34].

 $MONG\left(\%\frac{w}{w}\right) = 100 - \left(\%\frac{w}{w}\right)[Glycerol\ content + Water\ content\ + Ash\ content\]$ (3)

2.4.2. Qualitative analysis

The physical properties of the glycerol samples were measured at 25°C according to standard methods. The density was determined by measuring the volume and weight of the samples using a pycnometer ^[35], while the pH of the aqueous solution of glycerol (1g/50mL deionized water) was measured using a digital pH meter. Two chemical analysis methods were used to determine the quality of the glycerol samples. The functional groups of organic compound(s) in each sample were determined from the FT-IR spectrum generated within the range of 4000cm⁻¹ to 400cm⁻¹ at 1cm⁻¹ resolution using an infrared spectrometer. The vibration frequencies of functional groups of organic compound(s) identified on each spectrum were interpreted according to Coates ^[36]. The colour of the glycerol sample was determined under visible light (800nm to 400nm) using a UV–Vis spectrophotometer.

3. Results and discussion

The FFA content of the fresh vegetable oil was reduced after the refining process (Table 1). Free fatty acid exists in the varying amount in vegetable oils and constitutes a major impurity in the products derived from this feedstock if it is not properly refined. In transesterification, a high concentration of FFAs reduces the conversion of vegetable oil and increases the decomposition of the reaction products to form soap and water. However, these effects are negligible in oils with less than 3% FFA, but oil containing more than 4% FFA is refined by esterification using an aqueous solution of NaOH. Transesterification was carried out in excess methanol to favour the forward reaction (Table 2).

Table 1	L. Pro	perties	of the	refined	oil
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Properties	Fresh Oil	Refined Oil
Density @ 25°C (g/mL)	0.9488	0.9270
Viscosity @ 25°C (centistoke)	47.40	43.86
FFA (%)	5.00	1.15

Variables	Values		
Temperature (°C)	70		
Time (minutes)	60		
Reactant molar ratio (oil:methanol)	1:6		
NaOH (g)	1.5% w/v oil		
% Yield (crude glycerol)	14		
% Yield of product = $\frac{mass \ of \ product}{mass \ of \ reactant} \times 100$			

Table 2. Transesterification reaction conditions

The purification process was evaluated, and the optimum conditions were determined. The properties of the glycerol samples are listed in Table 3. The ash content of crude glycerol is high, and it is largely composed of sodium salts that originated from NaOH catalyst, while the water content of the purified sample is due to the absorption of moisture.

Table 3.	Properties	of	alycerol	samples
			5.7	

Properties	Crude glycerol	Purified glycerol	Control
Density (g/mL) @27°C	1.09	1.24	1.25
Viscosity (cP) @27°C	-	136.44	136.79
Refractive index	1.39	1.469	1.470
pH @25°C	10.60	7.55	6.87
Water content (wt%)	10.02	0.09	0.02 a
Glycerol content (wt%)	45	97	98ª
Ash content (wt%)	4.1	0.05	0.04ª
MONG (wt%)	43.28	2.86	1.94

^a Manufacturer specified values

3.1. Effect of acidification

Acidification separated the crude glycerol mixture into two or three layers depending on the type of acid used. The separation into three layers was only achieved after acidification with phosphoric acid at pH of 3 and 5, while only two layers were achieved for other acids and for phosphoric acid at pH of 1 (Table 4).

		Composition (% v/v)			
Acid	рΗ	Top layer	Middle layer	Bottom layer	
	1	72.6	25.7	0	
H_2SO_4	3	45.2	52.5	0	
	5	6.3	93.1	0	
	1	35.4	65.3	0	
H_3PO_4	3	38.9	46.6	13.2	
	5	59.2	35.1	3.7	
	1	7.26	92.1	0	
HCL	3	18.9	79	0	
	5	22.2	77.2	0	

Table 4. Acidification of crude glycerol

The formation of two layers mostly depends on the water content of crude glycerol. High water content in the crude glycerol sample enhances the dissolution of the inorganic salts formed. This is consistent with the salt solubility in aqueous solutions where Na_3PO_4 is less soluble in organic solutions and water (12.1g/100mLH₂O at 20°C), while Na_2SO_4 and NaCl readily dissolve in water at 19.5g/100mLH₂O (20°C) and 35.89g/100mLH₂O (20°C) respectively. Based on the distinct layer separation and precipitation of salt, the glycerol-rich middle layer of H_3PO_4 acidification to pH 3 was selected for the next purification step.

3.2. Effect of solvent extraction

The glycerol purity was further enhanced by solvent extraction due to its solubility in polar solvents. Three different polar solvents were evaluated at varying proportions ranging between 75%(v/v) to 25%(v/v). The precipitation of salt was highest with isopropanol at 66%(v/v), followed by ethanol at 75%(v/v), while no salt was recovered from methanol extraction. Solvent extraction was best achieved by isopropanol extraction at 66%(%v/v).

3.3. Effect of adsorption with activated charcoal



The removal of coloured impurities in the glycerol sample (after H_3PO_4 acidification to pH 3 and extraction with isopropanol) was achieved by adsorption onto the activated charcoal powder. The glycerol solution becomes clearer with the increasing weight of charcoal, and the maximum colour reduction was achieved at 180g/1000mL glycerol solution, as shown in Figure 2.

Figure 2. Glycerol colour reduction determined under visible light at 800nm

3.4. Qualitative analysis of glycerol samples

3.4.1. UV-Vis spectral analysis

The analysis of the glycerol samples in visible light was used as a measure of the degree of contamination by coloured impurities. Crude glycerol is almost opaque and has a very high absorbance under visible light, which is consistent with its dark-brown colour shown in Figure 3. The wavelength of maximum absorption of visible light compared favourably for the purified and commercial glycerol samples, as shown in Figure 4. This indicates the effectiveness of activated charcoal powder in removing the coloured impurities.



Figure 3. Sample pictures of (a) Crude glycerol (b) Purified glycerol (c) Commercial glycerol





3.4.2. FT-IR analysis

With reference to Coates ^[36], the functional groups of organic compounds in the glycerol samples were identified using the frequency of the absorbance peaks on the FT–IR spectra in Figure 5. Additional peaks were identified in the spectrum of crude glycerol, which clearly indicates the presence of impurities. For example, the peak at $1580cm^{-1}$ correlates with the C=O of carboxylate ion contained in the fatty acid salt (soap) formed by the reaction of the catalyst and FFA. The sharp peak at $1740cm^{-1}$ corresponds to C=O of esters, while the broad peak above $3000cm^{-1}$ signalled the presence of O–H due to contamination by water. These absorption bands are completely absent in the purified sample, where the spectrum showed all the vibrational frequency of bonds found in the commercial glycerol sample, such as the narrow non–bonded O–H stretch of alcohol ($3200 - 3400cm^{-1}$), C–H stretch of alkane ($2880 - 2900cm^{-1}$), O–H bend of tertiary alcohol ($1410 - 1310cm^{-1}$), and the C–O stretch of tertiary alcohol ($1050cm^{-1}$).





4. Conclusions

Crude glycerol was produced and treated to technical grade glycerol suitable for the synthesis of monoethylene glycol as a gas hydrate inhibitor. This was respectively achieved by transesterification, followed by the sequential application of evaporation, acidification and neutralization, polar solvent extraction, and adsorption techniques. The FFA content of the fresh vegetable oil was effectively reduced by esterification. Acidification with H_3PO_4 at a pH of 3 separated the crude glycerol sample into three distinct layers, while neutralization and solvent extraction enhanced the recovery of salt from the glycerol-rich middle layer. The colour of the

glycerol solution becomes clearer with the increasing amount of activated charcoal powder up to 180g/Litre glycerol solution. The quality of the FT–IR and UV–Vis spectra indicate that the purified glycerol sample is pure, and the properties are compared favourably with the commercial glycerol. The combined chemical and physical purification method is sustainable and promising and can be scaled–up for commercialization without much modification.

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