REMOVAL OF RESIDUAL KOH FROM PALM OIL BASED BIODIESEL USING DEEP EUTECTIC SOLVENTS

Kaveh Shahbaz[1]

[1]Author affiliation:
University of Malaya
Chemical engineering department, university of Malaya, Kuala Lumpur, Malaysia
Shahbaz.kaveh@gmail.com

Farouq Sabri Mjalli[2]

[2]Author affiliation:
Sultan Qaboos University
Petroleum and Chemical Engineering Department, Sultan Qaboos University, Oman
♬

farouqsm@yahoo.com

Mohd Ali Hashim[3]

[3]Author affiliation:
University of Malaya
Chemical engineering department, university of Malaya, Kuala Lumpur, Malaysia
alihashim@um.edu.my

Inas Moen AlNashef[4]

[4]Author affiliation:
King Saud University
Chemical Engineering Department, King Saud University, Riyadh, Saudi Arabia ♬

alnashef@ksu.edu.sa

ABSTRACT

The Produced biodiesel through the alkali-catalyzed transesterification must be treated before utilization as an alternative fuel. Low content of catalyst is one of the important pointers needed for passing the EN 14214 and ASTM D6751 international biodiesel standards. In this work, potassium hydroxide (KOH) was selected as catalyst for the transesterification reaction. Two different salts namely; methyl triphenyl phosphunium bromide (MTPB) and choline chloride (ChCl) in combination with four different hydrogen bond donors namely; glycerol, ethylene glycol, 2,2,2-trifluoroacetamide and triethylen glycol were selected to synthesis eighteen deep eutectic solvents (DESs). These DESs were employed as solvents for the removal of residual KOH from palm oil based biodiesel. The results revealed that all tested DESs were able to reduce the content of KOH. The [ChCl:glycerol] DESs and [MTPB:glycerol] DESs proved to be capable of high KOH removal from palm oil-based biodiesel with an average removal efficiency of 98.59% and 97.57% respectively.

INTRODUCTION

The growing energy demand, global warming, environmental pollution, decreasing of fossil fuels resources and the requisite of using clean fuels in the world have encouraged the researchers to investigate alternative renewable energy sources (Meher et al. 2006; Atadashi et al. 2010). Biodiesel is one of alternative fuels produced from all sorts of plant oils or animal fats. It is an attractive alternative fuel due to its advantages such as: being biodegradable; renewable; burning with no net addition to atmospheric CO_2 levels

and reduces the global warming problems; low sulfur and aromatic content; its cetane number and flash point are as high as fossil-based diesel; its agricultural; environmental benefits; being of domestic origin reduces the dependency on imported petroleum; good engine performance and facility to be blended with normal petroleum-based diesel (Ma and Hanna 1999; Munack 2006; Alptekin and Canakci 2008; Lapuerta et al. 2008).

Currently, there are four main methods to produce biodiesel namely; direct use and blending of raw oils (Adams et al. 1983; Engler et al. 1983; Strayer et al. 1983), micro-emulsions (Schwab et al. 1987), thermal cracking (Chang and Wan 1947; Crossley et al. 1962; Pioch et al. 1993), and transesterification (Leung et al. 2010). Amongst these methods, transesterification is the most common method for synthesizing the biodiesel. Generally, the transesterification reaction can be catalyzed by alkalis, acids, and enzymes (Ma and Hanna 1999). Recently, enzyme catalysts have attracted more scientists' attention because these catalysts prevent the soap formation and the purification process is effortless to complete. Nevertheless, they haven't been utilized commercially due to their long reaction times and high cost (Fukuda et al. 2001; Leung et al. 2010). The alkali and acid catalysts are more used in the production of biodiesel compared to enzyme catalysts because of their inexpensive raw material chemicals. The acid-catalyzed transesterification reaction requires a high molar ratio of methanol to oil; moreover, its reaction time is very long as compared with alkali catalysts and the acidic catalysts are corrosive and non-environmentally friendly (Marchetti and Errazu 2008; Rashtizadeh et al. 2010). The most common catalysts for the transesterification reaction are the alkali catalysts (homogeneous base catalysts) such as sodium hydroxide (NaOH) and potassium hydroxide (KOH) which are being used commercially in biodiesel production (Demirbas 2008; Dias et al. 2008; Rashid et al. 2008).

Hideki et al.(Fukuda et al. 2001) initiated that the best production of biodiesel is by using homogeneous alkaline catalysts; however, biodiesel includes unreacted components and unwanted products such as residual catalyst, water, un-reacted alcohol, free glycerol, and soaps that were generated during the transesterification reaction. Hence, several stages such as neutralization, evaporation of residual methanol and water washing are necessary to remove these impurities from biodiesel before using it as an alternative fuel.

The presence of residual alkali metals in biodiesel can form deposits in fuel injection system components (carbon residue) and poison emission control systems. In addition, it causes the blockage filter in engine and weakens the engine (Berrios and Skelton 2008). Moreover, its presence in biodiesel reduces the quality of the produced biodiesel. Therefore, removal of the catalyst at the end of reaction in alkali-catalyzed transesterification method is essential. As per the ASTM D6751 and EN 14214 international standards which control the quality and reliability of the finally produced biodiesel, the maximum allowable content of alkali metals must be 5 (mg/kg) (ASTM).

Generally, three most important approaches are adopted for purifying biodiesel: water washing, dry washing and membrane extraction (Dubé et al. 2007; Cooke 2009). The most traditional method which is being used in industry is water washing. Normally, a neutralization step to neutralize any residual catalyst and to split the soap is carried out before washing action (Gerpen 2005). The water washing method has many disadvantagessuch as: the increased cost and production time; polluting liquid effluent, considerable product loss due to retention in the water phase. Moreover, emulsion formation when processing used cooking oils or other feeds with high free fatty acid content may take place due to soap formation (Canakci et al. 2001). The dry washing method replaces the water with a magnesium silicate powder or an ion-exchange resin

to remove the impurities. Both the ion exchange and the magnesium silicate process have the advantage of being waterless. However, using these methods will increase the production cost because of the added materials expenses and in some cases overrun the limits specified in the EN Standard. Another method for the purification of biodiesel is by membrane extraction. Using this method, the final production cost is increased and the processing throughput decreases due to exiting contaminants (Leung et al. 2010).

Due to ionic liquids (ILs) unusual favorable properties, they have attracted more attention and became a focus point for many applications. However, there is still a challenge for the large-scale applications of ionic liquids in industry, because of complicated synthesis processes and the expensive raw material chemicals (Robin 2003). Hence, deep eutectic solvents (DESs) can be utilized as a low cost alternatives of ILs (Hou et al. 2008). DESs are formed from mixtures of organic halide salts with an organic compound which is a hydrogen bond donor (HBD) and able to form a hydrogen bond with the halide ion (Shahbaz et al. 2011). Deep eutectic solvents have properties similar to ionic liquids, especially, their potential as tuneable solvents that can be customized to a particular type of chemistry (Nkuku and LeSuer 2007). Currently, DESs are being used in research as well as industry because of their potential as an alternative environment friendly solvents and advantages over traditional ionic liquids such as ease of preparation in high purity at low cost, non-toxicity, non-reactivity with water and being biodegradable (Abbott et al. 2004). In addition, DESs are being studied comprehensively in many research areas as media for organic and inorganic reactions and separations. In addition, the advantages of the DESs have the best answer for the industrial requirements for large-scale applications.

Several of the studies reported that high-quality biodiesel which is economically viable can be achieved when suitable biodiesel separation process is employed. Abbott et al. 2004 showed that ionic liquids formed from the eutectic mixtures formed of quaternary ammonium salts and hydrogen bond donors can be utilized as effective inexpensive, non-toxic and environmentally benign solvents systems. They have also reported that a Lewis basic mixture of quaternary ammonium salt with glycerol was found to be successful as an extraction media for glycerol from biodiesel based on rapeseed and soy beans (Abbott et al. 2007). Furthermore, our research group investigated the application of a low cost quaternary ammonium salt-glycerol based ionic liquid as a solvent for extracting glycerol from palm oil-based biodiesel in a continuous separation process. The effect of DES to biodiesel ratio and the composition of DES on the efficiency of extraction process were investigated. The best molar ratio of DES:biodiesel was (1:1) and the DES composition was (1:1, Salt:HBD by mole) (Hayyan et al. 2009). Recently, we have presented two different successful non glycerol-based deep eutectic solvents to eliminate all free glycerol from palm oil-based biodiesel. The optimum molar ratios for non glycerol-based DESs were found to be 1:1 of DES to biodiesel. This study improved the previous findings and highlighted the importance of DES composition on the glycerol removal from palm oil-based biodiesel (Shahbaz et al. 2010). Recently, our research group introduced new deep eutectic solvents synthesized by the reaction of phosphunium-based salts with different hydrogen bond donors (Kareem et al. 2010).

In this work the potential of using DESs as appropriate solvents for the purification of biodiesel is explored by using two classes of deep eutectic solvents namely: DESs based on ammonium salt and phosphunium salt as solvents to extract the residual catalyst (KOH) from palm oil-based biodiesel

EXPERIMENTAL SECTION

Synthesis of biodiesel and DESs

In this work, the transesterification of palm oil with methanol to fatty acid methyl ester (FAME) using KOH as homogenous catalyst is considered. Palm oil (FFM Sdn Bhd) was supplied at local food store and Methanol (99.8%), potassium hydroxide (98.9%) were purchased from Sigma–Aldrich, Malaysia. 500 g palm oil was poured in the batch reactor to carry out the transesterification reaction. Homogeneous potassium methoxide was prepared by dissolving potassium hydroxide (1wt% of palm oil) in methanol and then added to the reactor at the reaction temperature of 60 ºC. The molar ratio of palm oil to methanol was 1:10 and agitation was set at a constant speed (400 rpm) during the experiment. After 2 hours, the mixture was poured to a separation funnel. After allowing overnight settling, the upper layer (biodiesel phase) was separated from the lower layer (glycerol-rich phase).

In this work, nine ammonium salt-based DESs and nine phosphunium-based DESs were synthesized and utilized as solvents in liquid-liquid extraction for the removal of residual catalyst (KOH). Choline chloride ($C_5H_{14}ClNO$) and methyl triphenyl phosphunium bromide ($C_{19}H_{18}PBr$) as salts and 2,2,2-trifluoroacetamide ($C_2H_2F_3NO$), glycerol ($C_3H_8O_3$), ethylene glycol ($C_2H_6O_2$) and triethylen glycol ($C_6H_{14}O_4$) as hydrogen bond donors were supplied from Merck Chemicals with high purity)> 99(% and utilized for the synthesis of DESs without further purification. The deep eutectic solvents were formed by stirring the salt with different HBD in different molar ratios under an argon atmosphere and at 353.15 K until a homogenous and colorless liquid appeared. Table 1 presents the compositions of the different DESs synthesized in this study (termed in this study as DES1 to DES18). The freezing point measurements of all DESs were made using Mettler Toledo Differential Scanning Calorimeter)DSC). Furthermore, the water content of all DESs was determined by Karl Fisher titration method. Table 2 shows the freezing temperature and water content of the studied DESs in this work.

Salt	HDB	Molar ratio (Salt:HBD)	Abbreviation
Choline chloride	Glycerol	1:1	DES1
	Glycerol	1:2	DES2
	Glycerol	1:3	DES3
	Ethylene glycol	1:1.75	DES4
	Ethylene glycol	1:2	DES5
	Ethylene glycol	1:2.5	DES6
	2,2,2-Trifluoroacetamide	1:1.75	DES7
	2,2,2-Trifluoroacetamide	1:2	DES8
	2,2,2-Trifluoroacetamide	1:2.5	DES9
Methyltriphenylphosphonium bromide	Glycerol	1:2	DES10
	Glycerol	1:3	DES11
	Glycerol	1:4	DES12
	Ethylene glycol	1:3	DES13
	Ethylene glycol	1:4	DES14
	Ethylene glycol	1:5	DES15
	Triethylene glycol	1:3	DES16
	Triethylene glycol	1:4	DES17
	Triethylene glycol	1:5	DES18

Table 1. Compositions and abbreviation of the synthesized deep eutectic solvents.

Table 2. Freezing Temperatures and water content for Studied DESs.

DES	Freezing temperature (K)	Water content (mg/kg)	DES	Freezing temperature (K)	Water content (mg/kg)
1	281.18	716	10	276.9	603
2	237	413.1	11	267.6	817
3	240.5	661	12	288.9	923
4	239.83	824	13	226.9	758
5	207.14	563	14	223.8	806
6	277.3	766	15	224.6	558
7	270.45	156	16	264.9	1063
8	243.14	178	17	254.3	1388
9	229.53	201	18	251.6	1223

Extraction Process

To perform the extraction of KOH from palm oil-based biodiesel, each DES was added to the biodiesel phase individually in different molar ratios (ranging 0.75-3 molar units DES per 1 molar unit biodiesel). The samples were then agitated for 1 hour at 200 rpm using an orbital shaker and at ambient temperature. Finally after two hours settling, the upper layer was separated and directly analyzed for its KOH content by measuring the potassium element using inductively coupled plasma-optical emission (ICP-OES).

ICP-OES analysis

The measurement of potassium was conducted using the PerkinElmer Optima 7000 DV ICP-OES instrument (PerkinElmer, Inc., Shelton, CT, USA) equipped with WinLab32 and Aragon (99.99%) was used as the carrier gas. The ICP-OES analytical conditions are given in Table 3. The wavelength for the analysis of potassium was set at 766.486 nm. The oil standard for potassium (500 mg/kg) and oil based were supplied from Conostan Company and diluted in xylene solution (Merck, > 99%) at six different concentrations (0.5 ppm, 1ppm, 2 ppm, 5ppm, 7 ppm and 15ppm). The resulting solutions were directly injected into the ICP-OES to perform the calibration. The linear correlation coefficient (R^2) was found to be 0.998 which indicated reasonable linearity. Instrument Detection limit (IDL) was determined by three times the standard deviation of the blank (ten replicates) which was found to be 0.03 mg/kg. For the calculation method detection limit (MDL), seven portions of calibration standard of 1ppm were prepared and analyzed then thet standard deviation of the results was calculated and multiplied by 3.14. The obtained MDL value was 0.1 mg/kg. All biodiesel samples were injected to the ICP-OES and after determination of potassium peak area, the potassium content in each biodiesel sample was calculated with reference to calibration.

Table 3. ICP analytical conditions.

Plasma gas flow	15 L/min
Auxiliary gas flow	1.5 L/min
Nebulizer gas flow	0.45 L/min
RF power	1450 watts
Pump flow rate	1 mL/min
Read delay	90 second
Processing	Peak area
Replicates	3
Torch	Quartz for axial view

RESULT AND DISCUSSION

The biodiesel was synthesized by the alkali-catalysed transesterification of palm oil and without any purification or washing step was directly analysed by the ICP-OES. A KOH content of 79.588 mg/kg was determined which is exceeds the ASTM D6751 and EN 14214 international standards (5mg/kg) acceptable limits.

Choline chloride (ChCl) was mixed separately with three different Hydrogen bond donors namely; glycerol, ethylene glycol and 2,2,2-trifluoroacetamide at three different molar ratios to form the nine DESs based on ChCl (DES1 to DES9). Moreover, methyl triphenyl phosphunium bromide (MTPB) was mixed with three different HBD (glycerol, ethylene glycol and triethylen glycol) at three different molar ratios to synthesis the nine DESs based on MTPB (DES10 to DES18). In this work, all molar ratios of salt: HBD were selected based on the minimum freezing temperature. As indicated by Table 2, all the synthesized DESs gave low freezing points as compared to their constituting components and below ambient temperature. This is consistent with the general behaviour of deep eutectic solvents. In addition, water content of all ChCl-based DESs was less than 0.1 wt%. Also for the all MTPB-based DESs, the water content was less than 0.1 wt% except DES16, DES17 and DES18 which were 0.106 wt%, 0.138 wt% and 0.122 wt% respectively (Table 2). Nonetheless, all DESs water content were consistent with physical properties of deep eutectic solvents.

For the removal of residual KOH from palm oil-based biodiesel, the synthesized DESs were mixed with the produced biodiesel for one hour at six different molar ratios of DES to biodiesel (0.75:1, 1:1, 1.5:1, 2:1, 2.5:1 and 3:1) to determine maximum removal efficiency. The extraction process was carried out at ambient temperature. After two hours settling, The DES layer was separated wholly and no emulsion was formed. The biodiesel layer was analysed for its KOH content by measuring potassium element using ICP-OES. Intermolecular attractions of solvent (DES) with solute (KOH) exist in ion pairing, hydrogen bonding, dipole and Van der Waals interactions. However, Due to the polarity of DESs, existence of the hydroxyl group in both DES and KOH and the solvation forces for KOH in biodiesel, DESs have high affinity for the attraction of KOH through the hydrogen bonding and dipole-dipole attraction.

Figure 1 represents the ICP results for KOH content after extraction by ChCl to glycerol DES1 and DES2. It is clear from figure that DES1 at all molar ratios of DES1:biodiesel and DES2 at ratios of 0.75:1 and 1:1 (DES2: biodiesel) were able to reduce the KOH content. The ICP results unveiled that the potassium element after extraction by DES2 at ratios 1.5-3:1 (DES2: biodiesel) and DES3 at all molar ratios (DES3: biodiesel) was

not detected by ICP-OES due to the method detection limit (MDL) of 0.1 mg/kg. This indicates that the KOH content was below the MDL. The removal efficiency of KOH content by using these DESs was above 99.87%. It can be concluded that all ChCl to glycerol DESs at all ratios of DES:biodiesel excluding DES1 at 0.75:1 (DES1: biodiesel) ratio were successful to decrease the KOH content lower than the value required by ASTM D6751 and EN 14214 international standards. Moreover, the content of KOH was reduced with the increase in mole fraction of glycerol in eutectic mixture of ChCl: glycerol.
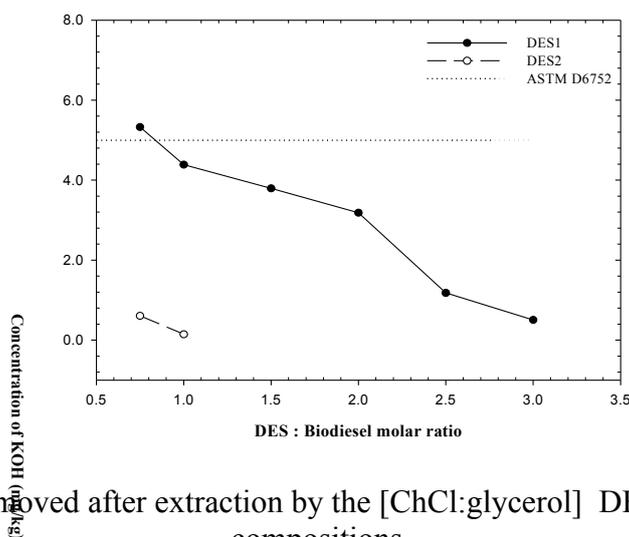
Fig. 1. KOH removed after extraction by the [ChCl:glycerol] DES with different compositions.

The ICP results for the removal of KOH by ChCl to ethylene glycol DESs (DES4 to DES6) and [ChCl:2,2,2-trifluoroacetamide] DESs (DES7 to DES9) are shown in Figure 2 and Figure 3 respectively. Clearly, the KOH removal profiles show that the removal efficiency for KOH increases with the increase in DES:biodiesel molar ratio. As indicated by Figure 2, the removal efficiency of KOH was increased by increasing ethylene glycol mole fraction and only DES5 at molar ratios of 2.5:1 and 3:1 (DES5: biodiesel) and DES6 at molar ratios 1.5-3:1 (DES6: biodiesel) could fulfil the ASTM D6751 and EN 14214 specification for KOH content. The maximum removal efficiency for KOH content by ethylene glycol DESs occurred at 3:1 (DES6: biodiesel) molar ratio with 95.47% removal. As can be seen from Figure 3, the removal efficiency of KOH declined with the increase in mole fraction of 2,2,2-trifluoroacetamide. The DES7 at molar ratios of 1.5-3:1 (DES7: biodiesel) and DES8 at 2-3:1 (DES8: biodiesel) were successful to remove the KOH content below the required standards. Furthermore, a maximum removal efficiency of 97.07% by the [ChCl:2,2,2-trifluoroacetamide] DESs took place at 3:1 ratio using DES7.
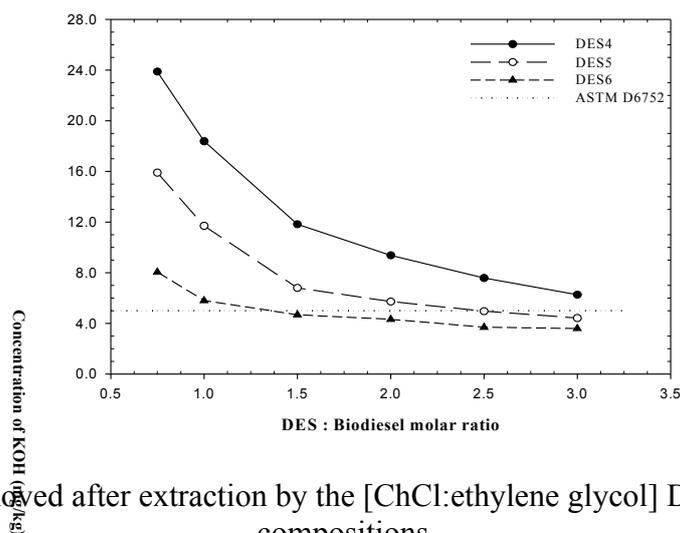
Fig. 2. KOH removed after extraction by the [ChCl:ethylene glycol] DES with different compositions.
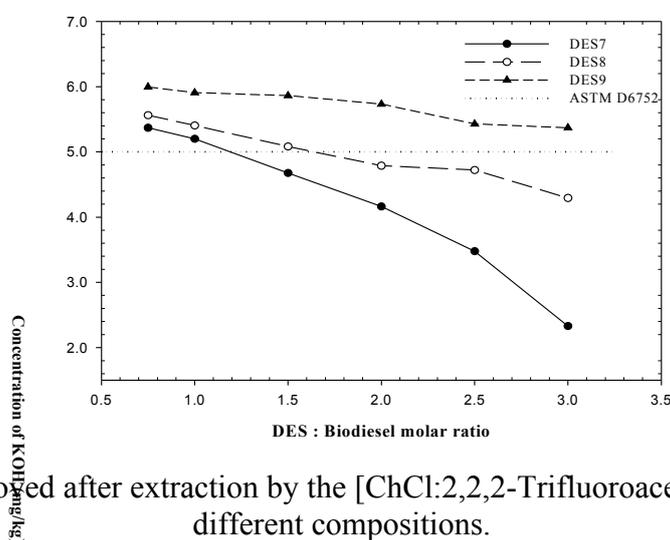

Fig. 3. KOH removed after extraction by the [ChCl:2,2,2-Trifluoroacetamide] DES with different compositions.

The KOH removal results by [MTPB:glycerol] DESs (DES10 to DES12), [MTPB:ethylene glycol] DESs (DES13 to DES15) and [MTPB:triethylen glycol] DESs (DES16 to DES18) are demonstrated in Figure 4, figure 5 and figure 6, respectively. It is clear from the figures that removal efficiency of KOH increases gradually by increasing DES:biodiesel molar ratios. In addition, with more mole fraction of HBD in the mixture of [MTPB:HBD], the KOH removal efficiency increases incrementally. Figure 4 shows that all the [MTPB:glycerol] DESs at all DES:biodiesel ratios removed the KOH content under both international standards specification. However, the [MTPB:ethylene glycol] DES13 at the molar ratios of 0.75:1 and 1:1 and DES14 at ratio of 0.75:1 (DES: biodiesel) could not satisfy the standards specification for KOH content (Figure 5). Moreover, [MTPB:triethylen glycol] DES16 and DES17 at 0.75:1 and 1:1 and DES18 at 0.75:1 DES:biodiesel molar ratios were also unsuccessful to meet the standards specification (Figure 6). Maximum removal efficiency of KOH content by [MTPB: glycerol] DESs, [MTPB:ethylene glycol] DESs and [MTPB:triethylen glycol] DESs occurred at 3:1 DES:biodiesel molar ratio using DES12, DES15 and DES18 with 99.83%, 98.54% and 98.94% removal, respectively.
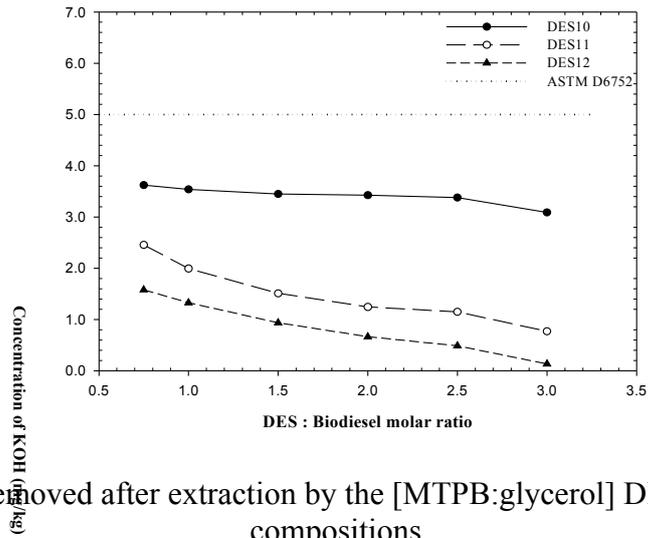
Fig. 4. KOH removed after extraction by the [MTPB:glycerol] DES with different compositions.
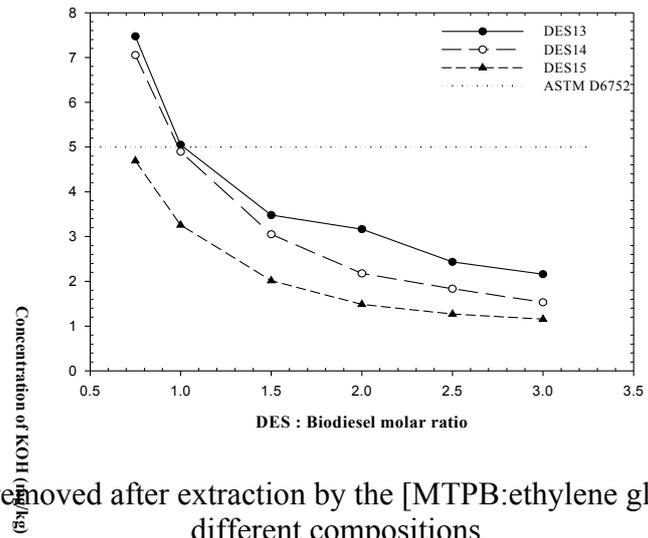

Fig.5. KOH removed after extraction by the [MTPB:ethylene glycol] DES with different compositions.
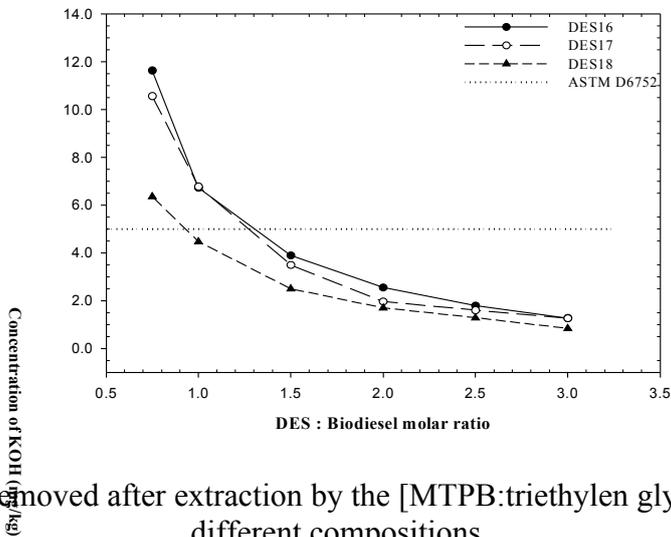

Fig. 6. KOH removed after extraction by the [MTPB:triethylen glycol] DES with different compositions.

The KOH average removal efficiency of 98.59% and 97.57% by the [ChCl:glycerol]

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DESs and [MTPB:glycerol] DESs proved that glycerol-based DESs are efficient to remove the KOH content from palm oil-base biodiesel. Consequently, glycerol as a HBD in the eutectic mixture of salt:HBD is more suitable than the others for the current application. One reason can be the high solubility of KOH in glycerol.

CONCLUSION

In the present work, we examined a new purification technique for removal residual KOH from palm oil-based biodiesel using ammonium-based DESs and phosphonium-based DESs. For this purpose, nine ChCl-based DESs and nine MTPB-based DESs at different compositions were tested as solvent in for the removal of KOH content. All tested DESs (DES1 to DES18) were able to remove the residual KOH. The removal efficiency of KOH by all used DESs increased with increase in DES:biodiesel molar ratio. Moreover, KOH removal efficiency using all tested DESs increased by increasing the mole fraction of HBD in the mixture of salt:HBD apart from 2,2,2-trifluoroacetamide as HBD. The KOH average removal efficiency of 98.59% and 97.57% by ChCl: glycerol DESs (DES1 to DES3) and MTPB to glycerol DESs (DES10 to DES12) proves that these DESs were more efficient than other DESs to remove the KOH from palm oil-base biodiesel. These DESs at all DES:biodiesel molar ratios excluding DES1 at 0.75:1 (DES1: biodiesel) ratio were successful to reduce the KOH content below the value required by ASTM D6751 and EN 14214 international standards.

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BRIEF BIOGRAPHY OF PRESENTER

First Author is PhD candidate at the Department of chemical Engineering, Faculty of engineering, university of Malaya, Malaysia. He can be reached at <shahbaz.kaveh@gmail.com>.