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Influence of Solvent Temperature and Type on Naphthalene Solubility for Tar Removal in a Dual Fluidized Bed Biomass Gasification Process

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Abstract

Tar condensation is a cause of blockage in downstream application of the gasification process. An oil scrubber is considered as an effective method for tar removal. In this research, the naphthalene solubility in different local Thai oils and water was investigated in a laboratory-scale test-rig. The solubility value was conducted at 30, 50, 70, and 80°C. Biodiesels investigated were rapeseed methyl ester (RME) and two different palm methyl esters (PME 1 and PME 2). Furthermore, vegetable oils including sunflower oil, rice bran oil, crude palm oil, and refined palm oil were examined. The results showed that higher temperature enhanced naphthalene solubility in all types of investigated oils. Biodiesel has the highest value of naphthalene solubility. All scrubbing oils have similar naphthalene solubility trends at the temperature range of 50-80°C in the order of RME > PME 1 > PME 2 > diesel > sunflower oil > refined palm oil > rice bran oil > crude palm oil. Based on these experimental investigations, PME 1 has a naphthalene solubility value similar to RME. Therefore, PME 1 has been selected to be tested as scrubbing solvent in the 1 MW_{el} prototype dual fluidized gasifier located in Nong Bua district, Nakhon Sawan province, Thailand.

Keywords: tar removal; oil scrubber; solvent scrubbing; naphthalene; dual fluidized bed biomass gasification
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1. Introduction

Biomass gasification transforms biomass and solid carbonaceous fuel into a gaseous secondary energy carrier. When using steam as gasifying agent, reforming reactions occur inside the gasification reactor. The so-called product gas from the gasification process contains primarily hydrogen, carbon monoxide, carbon dioxide, methane, and C_2-C_4 hydrocarbons [1]. The product gas has a high heating value of 10-18 MJ/Nm^3 compared to air gasification (4-7 MJ/Nm^3) and thus can be further used for efficient generation of electricity, chemicals, and liquid fuels [2, 3]. However, there are undesired components in the product gas, mainly a mixture of high molecular weight hydrocarbons, defined as tar, and traces of ammonia, hydrogen sulfide, and hydrogen chloride [4-6].

Tar is an organic complex mixture of hydrocarbons that can be condensed, such as aromatics and polyaromatic hydrocarbons [7]. The tar composition and concentration depend on the type of biomass and operating conditions [7, 8]. “Tar is divided into five classes based on molecular weight, which are (1) GC-undetectable, (2) heterocyclic aromatics, (3) light aromatic (1 ring), (4) light polycyclic aromatic hydrocarbon (PAH) compounds (2 - 3 rings), and (5) heavy PAH compounds (4 - 7 rings)” [7, 8]. Tars in the product gas condense when the temperature of the product gas decreases. The class 5 tar components condense at higher temperatures even at low concentrations. The tar classes 2 and 4, heterocyclic aromatics and light PAHs, condense at around 0-125°C even at very low concentrations [8]. Tar condensation causes blocking in the downstream equipment. This is a major problem in biomass gasification and therefore tars must be removed to obtained a reliable operation [9].

There are many tar removal techniques. Yet, physical tar removal is widely used in the downstream cleaning process because it is easy to control and requires low energy [10-13]. Physical tar removal processes can be classified into two systems, which are dry systems such as cyclones and filters, and wet systems such as spray towers and packed column scrubbers. For scrubbers, the scrubbing solvent plays an important role for the design and tar removal efficiency. Water scrubbing has been reported as a common technique [14]. Nevertheless, the main disadvantage of the water scrubber is the low solubility of tar compounds. This is because water belongs to the hydrophilic (polar) group, but the main tar components (PAHs) belong to the hydrophobic (non-polar) group. So, only a few kinds of tar components can dissolve in water [15, 16]. This means that water is not a suitable solvent to remove tar in the product gas [17]. Besides water scrubbing, oil scrubbing is an interesting option because oil belongs to the hydrophobic group, which is the same as tar [18-20].

It was reported that the tar solubility in oil was much higher than in water. Phuphuakrat *et al.* [16] found that a water scrubber could remove only 31.8% of gravimetric tar. The removal efficiencies of tar compounds in each type of oil absorbents were observed to be in the following descending order: diesel fuel > vegetable oil > biodiesel fuel > engine oil > water [16]. The authors recommended that vegetable oil and biodiesel were interesting and were suitable solvents for tar removal [16]. Ahmad and Zainal [21] showed 22% and 75% total tar removal efficiency when water and waste palm cooking oil collected from restaurants were used as scrubbing solvent, respectively. Moreover, temperature is a primary parameter that affects tar solubility in the solvent [21]. In the work of Nakamura *et al.* [22], the tar removal performance of bio-oil as a scrubbing solvent at different temperatures was investigated. The result showed that the tar removal efficiency was 63.8, 73.3, and 54.3% at 40, 50, and 60°C, respectively.

Biodiesel, namely rapeseed methyl ester (RME), has been used for tar removal in commercial dual fluidized bed (DFB) steam gasification processes in Europe [23]. In Thailand, the 1 MW$_{el}$ prototype DFB steam gasification process, engineered and constructed by Gussing Renewable Energy Company, is the first plant in Asia [24]. It is a pioneering and innovative DFB gasifier that can be fuelled with various types of biomass and waste [24]. This DFB gasifier power plant is located at Nong Bua district in Nakhon Sawan province.

One of major operating costs of this DFB gasifier is from the use of imported RME in the tar removal process. Substituting the imported RME with a locally supplied solvent as the tar scrubbing solvent was an objective. Therefore, it was decided to investigate the tar solubility of different local oils in Thailand in a laboratory scale test-rig. Based on previous gas chromatography-mass spectrometry (GC-MS) analysis of used RME after scrubber and other studies, it was observed that naphthalene was present as a component of high content in the product gas from the DFB gasifier [25, 26].

The aim of this research work was to find a feasible local Thai oil to substitute for the imported biodiesel that is currently used in the tar scrubbing system of the prototype commercial-scale DFB gasification process located in Thailand. Naphthalene was used as a tar model compound. Its solubility in various types of local Thai oils and water at various temperatures was studied.

2. Materials and experimental setup

2.1 Materials

2.1.1 Tar model compound

Naphthalene was employed in this study as a representative of total tar due to its high content in the product gas from the DFB steam biomass gasifier. Naphthalene is classified as a class 4 type of tar, and it is condensable at low temperature and at very low concentration [8]. The purity of naphthalene used was 99%, and it was purchased from Sigma-Aldrich Inc.

2.1.2 Scrubbing solvents

The solvents used in this research are divided into two types, namely the hydrophilic (polar) group and hydrophobic (non-polar) group. Water is the only representative solvent of the hydrophilic group. For hydrophobic group, there are three kinds of oil, i.e. (1) methyl ester or biodiesel, (2) vegetable oils (sunflower oil, rice bran oil, crude palm oil, and refined palm oil), and (3) diesel. All of the scrubbing solvents, except RME, were purchased locally in Thailand.

The RME used in this current research was imported from Germany and had the same specification as that used in the prototype commercial-scale DFB gasification process in Thailand and the commercial DFB gasifier power plant in Austria. Two palm methyl esters (PME), namely PME 1 and PME 2, were purchased locally in Thailand.

Solvent properties were also experimentally determined in this research. The densities of the oils were determined using the Archimedes' principle. The viscosities of oils were measured by Brookfield DVIII Ultra. A Gas Chromatograph-Mass Spectrometer (GC-MS) was used to analyze the components of oil. In the GC-MS analysis, oils were diluted at 100:1 in hexane except for diesel, which was diluted at 100:1 in acetone. Sample volume of 0.2 µl was injected into HP-5 Column. Helium was used as carrier gas at the flow rate of 0.9 ml/min. The oven temperature was started from 40°C until the temperature reached 250°C.

2.2 Experimental set up and analysis

Figure 1 displays the experimental set up for the investigation of the naphthalene solubility in various solvents. The effect of the temperature was studied by varying solvent temperature at 30, 50, 70, and 80°C. These temperatures are in the operating ranges of the scrubber in the commercial DFB biomass gasification process.

In the experiment, scrubbing solvent of 20 ml was heated in a water bath to the desired temperature. The temperature of the solvent (T1) was controlled steadily by a thermostat of the water bath temperature (T2). Naphthalene was slowly added to the solvent at the same rate for all experiments. The temperatures of the solvents were kept constant through the controlling of the water bath temperature. The mixing of naphthalene in solvent was performed by a magnetic stirrer with the same constant speed of 750 rpm for all scrubbing solvents. The total time consumed for a complete test of a single solvent at a particular temperature was between 3 and 5 h, and tests were done in triplicate.

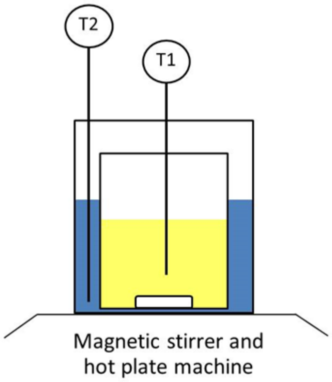

Figure 1. Experimental set up for tar solubility study

The solubility value of naphthalene was calculated using equation (1). The amount of naphthalene was visually determined when first crystals appeared at constant solvent temperature.

$$\text{Solubility value} = \frac{\text{Amount of tar (g)}}{\text{Volume of solvent (l)}} \quad (1)$$

3. Results and Discussion

3.1 Effect of solvent temperature on naphthalene solubility

The average value of naphthalene solubility was calculated from three repetitive test values for each set of conditions, i.e. solvent type and temperature. The average value of naphthalene solubility (g naphthalene/l solvent) is presented in Figure 2. The percentage of naphthalene solubility standard deviation for all conditions does not exceed ± 5%.

Figure 2 shows the dependency of naphthalene solubility with temperature and type of the scrubbing solvent. All scrubbing solvents showed a similar trend with increasing the temperature. When the temperature of the scrubbing solvent was increased, the naphthalene solubility increased. The solubility of naphthalene slightly increased at low temperatures from 30°C to 50°C but significantly increased at high temperatures from 50°C to 80°C.

When naphthalene was added into the scrubbing solvent, the temperature of scrubbing solvent decreased, as observed during the experiments. According to thermodynamics principles known as Le Chatelier’s principle, the solubility of naphthalene is an endothermic dissolution process. Therefore, increasing the temperature of a solvent leads to an increase in naphthalene solubility. The increasing rate of the naphthalene solubility is related to its melting point. The melting point of naphthalene influences the solubility. The naphthalene solubility was significantly increased from 50°C to 80°C because the temperature was getting closer to the melting point of naphthalene, which was at 80-82°C [27].

3.2 Effect of solvent type on naphthalene solubility

As shown in Figure 2, the solubility of naphthalene in the solvents can be ranked in the order of biodiesel > diesel > vegetable oil > water. The biodiesel group showed the highest naphthalene solubility and among the three biodiesels tested, it was found that RME and PME 1 had competitive solubility values of 271.23±6.65and 271.00±8.05g/l at 30°C, 582.55±23.60 and 586.90±26.45 g/l at 50°C, 2070.77±9.08 and 1906.30±3.15 g/l at 70°C, and 4499.00±349.33 and 4468.18±166.17 g/l at 80°C, respectively. At 70°C, RME has slightly more tar solubility than PME 1. For PME 2, it showed competitive solubility values at the solvent temperatures of 30°C to 50°C, but showed a lower solubility than RME and PME 1 at the solvent temperatures of 50°C to 80°C.

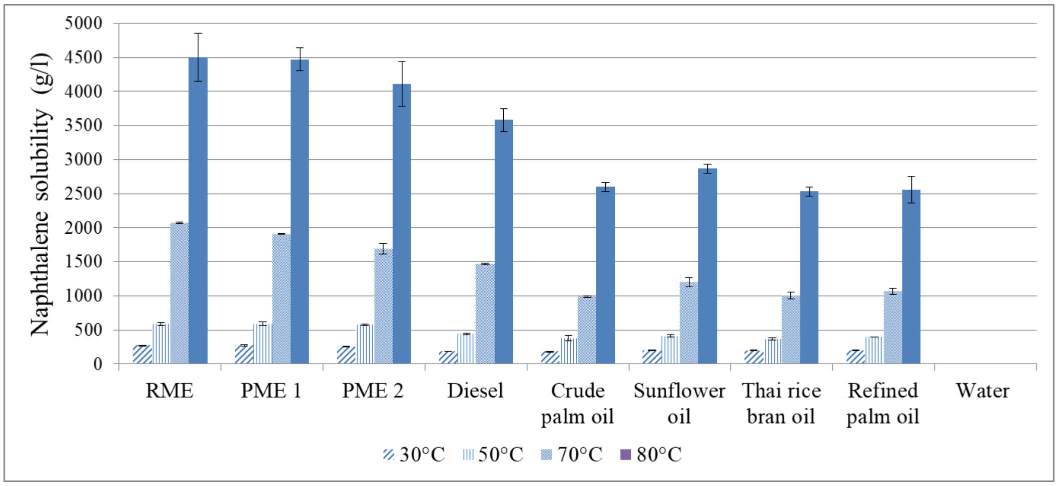

Figure 2. Naphthalene solubility in various scrubbing solvents at different temperatures

All scrubbing solvents had similar naphthalene solubility trends for 50-80°C as RME > PME 1 > PME 2 > diesel > sunflower oil > refined palm oil. The naphthalene solubility of Thai rice bran oil and crude palm oil were slightly different. Except at 30°C, diesel showed higher naphthalene solubility than sunflower oil, refined palm oil, and rice bran oil.

The properties of solvent affect the solubility value. The diffusion process is related to the viscosity of solvent. A lower viscosity solvent has a higher diffusion coefficient, and hence a higher dissolution rate than a higher viscosity solvent [15, 16]. A high dissolution rate often means high solubility. The viscosities of the solvents are presented in Tables 1 and 2. Biodiesel showed approximately 10 times lower viscosity than vegetable oil, and thus naphthalene solubility in biodiesel was noticeably higher than in vegetable oil. It means that naphthalene is absorbed in biodiesel more than in other oils at the same temperature. Comparing diesel with biodiesel, although diesel shows approximately 1.47 cP lower viscosity than biodiesel, naphthalene solubility in diesel is lower than biodiesel.

Table 1. Properties of biodiesel

Properties	Unit	RME	PME 1	PME 2
Density at 30°C	g/cm^3	0.8817	0.8758	0.8755
Viscosity at 30°C	cP	4.93	4.97	4.94
Ester content	wt %	98.00	98.80	98.80
- Saturated ester	wt %	7.19	50.59	64.39
- Unsaturated ester	wt %	90.81	48.20	34.37
Methanol	wt %	0.02	0.017	0.041
Water content	% (m/m)	0.0185	0.023	0.029
Cloud point	°C	-4	14	19
Flash point	°C	-	>170	155

Table 2. Properties of diesel and vegetable oil

Properties	Unit	Diesel	Sunflower oil	Refined palm oil	Thai rice bran oil	Crude palm oil
Density at 30°C	g/cm^3	0.8380	0.9259	0.9273	0.9362	0.8373
Viscosity at 30°C	cP	3.46	40.06	52.90	53.87	1072.67
Ester content	wt %	21.895	97.762	92.259	91.399	96.892
Alkanes	wt %	68.673	-	-	-	-
Aromatic	wt %	9.435	-	-	-	-
Alcohol	wt %	-	2.238	7.130	2.682	3.107
Organic acid	wt %	-	-	-	5.920	-
Aldehyde	wt %	-	-	0.611	-	-

In addition, interactions between solute and solvent also influence the naphthalene solubility. These interactions are; solute-solute interaction, solvent-solvent interaction, and solute-solvent interaction. In this research, solute-solvent interaction is focused since this study tested only one solute in various solvent types. Diesel and biodiesel have straight-chain alkanes but the alkane chain length of diesel is shorter than that of biodiesel. With the increase in alkane chain length, the solubility of naphthalene also increases. Naphthalene dissolves in diesel through the bonding of London dispersion forces, which are the weakest intermolecular forces [28]. On the contrary, naphthalene dissolves in biodiesel through the bonding of the dipole-induced dipole forces, which are stronger than London dispersion forces [29]. Strong intermolecular bonding interaction of solute-solvent leads to high dissolution of solute in a solvent. Therefore, naphthalene can be dissolved in biodiesel more than diesel [28-30].

3.2.1 Naphthalene solubility in biodiesel

According to Table 1, RME, PME1 and PME2 show insignificantly different viscosities and ester contents. Methanol belongs to the alcohol group, which is classified as polar substance. Thus, naphthalene can better dissolve in RME, PME1 and PME2, respectively. In addition, RME, PME1 and PME2 have different amounts of saturated esters and unsaturated esters. The degree of unsaturated ester content also influences the naphthalene solubility. Saturated ester such as palmitic acid (C16:0) has no carbon double bonds whereas unsaturated esters such as oleic acid (C18:1) and linoleic acid (C18:2) have double carbon bonds. The aromatic tar solubility increases with increasing unsaturated ester content. Saturated esters are straighter-chained hydrocarbon molecules than unsaturated esters. This means that saturated esters have a solvent structure that is more closely packed [29]. An increment of unsaturated ester attributes to an increase of freedom of movement in solvent structure [29]. This allows aromatic tar molecules to bond with unsaturated esters more easily than saturated esters [29]. As illustrated in Figure 3, RME has the highest unsaturated ester (mainly oleic acid and linoleic acid) followed by PME1 and PME2. Therefore, RME has the highest naphthalene solubility while PME2 has the lowest naphthalene solubility.

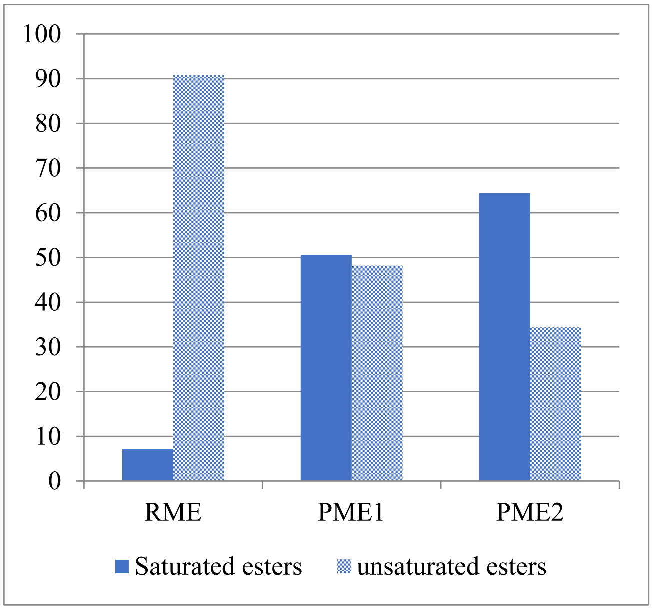

Figure 3. Percentage of saturated esters and unsaturated esters of biodiesel

3.2.2 Naphthalene solubility in vegetable oil

The properties of vegetable oils that affect naphthalene solubility are summarized in Table 2. The vegetable oils with higher viscosity have lower dissolution rate, and hence lower naphthalene solubility [15, 16]. Sunflower oil has the lowest viscosity. Thus, sunflower oil has the highest naphthalene solubility. Thai rice bran oil and crude palm oil have the lowest naphthalene solubility.

The composition of each vegetable oil also influences the naphthalene solubility. Among the main composition of vegetable oil are ester groups. When rating the polarity of functional groups from highest to lowest, they are in the following order: amide > organic acid > alcohol > aldehyde > ketone > amine > ester > ether > alkane. According to the solubility principle, a non-polar solute (naphthalene) is likely to dissolve in non-polar solvents. Sunflower oil shows the lowest polar substance (alcohol content). Thus, naphthalene solubility in sunflower oil presents the highest value. Furthermore, organic acid is a strong polar compound that is present in Thai rice bran oil. Thus, naphthalene solubility in refined palm oil is higher than in Thai rice bran oil. Naphthalene solubility in Thai rice bran oil and crude palm oil is lowest due to the high viscosity of crude palm oil and highly polar substances of Thai rice bran oil.

3.2.3 Naphthalene solubility in water

When using water as an absorbing solvent, it was found that naphthalene was insoluble in water at all temperatures because naphthalene is a cyclic aromatic compound and is non-polar whereas water belongs to a hydrophilic polar group [8]. So, a water scrubber is not an appropriate device for the removal of naphthalene from the product gas, but it can be used for some classes of tar such as phenol [16].

3.3 Implementation of naphthalene solubility in a tar removal process

From the results discussed above, PME1 was chosen as a substitute of RME for tar removal in the 1 MW_{el} prototype dual fluidized gasification in Nong Bua district, Nakhon Sawan province, Thailand. The tar concentrations in the producer gas when using the imported RME and PME1 were 93±59 and 86±28 mg/Nm^3, respectively. The tar concentration with the substituted local PME1 scrubbing solvent was considered to be comparable with the imported RME. The cost of the local PME as the scrubbing solvent for the tar removal process was approximately 25% lower that that of the imported RME.

4. Conclusions

The solubility of naphthalene in different solvents, locally available in Thailand, was investigated. For all the temperatures between 30 and 80°C, naphthalene solubility in biodiesel was the highest; hence it was predicted to be the best among all other tested solvents for tar removal. Based on these experimental results, PME1 showed a similar naphthalene solubility to RME. Thus, PME1 was selected to be tested as a scrubbing solvent in the Thailand 1 MW_{el} prototype DFB gasifier at Nong Bua district in Nakhon Sawan province, Thailand. The tar concentration was found to be comparable between RME and PME1, but the cost of PME1 was lower by approximately 25%.

In addition, more research work in the laboratory will be conducted to investigate other tar compounds mostly generated in the biomass DFB gasifier process such as anthracene, pyrene, fluoranthene, and biphenyl.

5. Acknowledgements

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