Enhanced efficiency of dissolved air flotation for biodiesel wastewater treatment by acidification and coagulation processes

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A B S T R A C T

A novel approach was developed to enhance the efficiency of the dissolved air flotation (DAF) for biodiesel wastewater by acidification and coagulation. Firstly, Grease & Oil and Chemical oxygen demand (COD) removal efficiencies of biodiesel wastewater using acidification with pure hydrochloric acid and pure sulfuric acid at pH = 3 and 1 day retention time were more than 80%, and 50%, respectively. Secondly, Grease & Oil and COD removal efficiencies of biodiesel wastewater using alum, polyaluminum chloride and ferric chloride coagulants without acidification at 1.0 g/L were more than 90% and 30%. Thirdly, DAF alone and DAF with acidification could not separate Grease & Oil from biodiesel wastewater. Thus, DAF with the acidification and coagulation is suggested for biodiesel wastewater treatment. Biodiesel wastewater treatment by acidification before alum coagulation allowed the alum concentration to be reduced by 60–90% compared to treating without acidification. In addition, the efficiency of Grease & Oil removal from biodiesel wastewater by DAF with alum and acidification was 85–95%. It can be concluded that the efficiency of Grease & Oil removal from biodiesel wastewater using DAF with acidification and coagulation was 10% greater compared to other processes.

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1. Introduction

Increased demand for energy, crude oil price increase, global warming due to green house gasses missions, environmental pollution, and the fast diminishing supply of fossil fuels are the major key factors leading to the search for alternative sources of energy [1]. Biodiesel has recently attached much attention, given that it has various advantages. It provides an alternative to petroleum-based fuel, it is renewable and non-toxic fuel, it allows a favorable energy balance, and it is fewer harmful emissions [2]. Furthermore, the Thai government has promoted the production and the use of biodiesel as a substitute diesel fuel and to promote the use of alternative energy made from domestic crops [3]. At present, biodiesel production capacity in Thailand is approximately 1.5 million liters per day with 43 biodiesel plants registered with the Department of Alternative Energy Development and Efficiency [4].

Biodiesel is produced from the transesterification reaction of triglycerides from vegetable oils or fats with alcohols like methanol and ethanol in the presence of a homogenous base catalyst like NaOH or KOH [5] to yield fatty acid alkyl esters (i.e., biodiesel) and glycerol [6]. Over 90% of biodiesel production in Thailand uses palm oil as the raw material by a transesterification reaction method using alkali catalysis [3]. The biodiesel production process with transesterification reaction uses a large amount of water, 20–120 L per 100 L of biodiesel [7], for product rinsing to remove undesirable substances like soap and other residual substances [8,9]. Thus, wastewater from the biodiesel production process is basic (alkaline), with a high content of oil and grease, and a low content of nitrogen and phosphorus [3]. Many researchers [10–12] have been reported the following wastewater characteristics that pH was 8.5–10.5, the suspended solid (SS) was 1,500–28,790 mg/L, the chemical oxygen demand (COD) was 60,000–545,000 mg/L, the biological oxygen demand (BOD5) was 105,000–300,000 mg/L and Grease & Oil was 7,000–44,300 mg/L. Accordingly, biological treatment of biodiesel wastewater is expected to be very difficult [11].

Presently, typical treatments of oily wastewater in Thailand employ a dissolved air floatation (DAF) technique, Grease & Oil trap unit or other Grease & Oil commercial removal units to separate oil and grease prior to biological treatment [3]. DAF is an effective method for removing low density particles from suspension and clarifying low turbidity [13]. In addition, DAF has been used for treating wastewater to separate oil from aqueous dispersion, chemically treated wastewater, and oil refinery wastewater [14]. However, these conventional flotation techniques are not satisfactory for removing emulsified oils without chemical pre-treatment [14,15]. Chemical pre-treatment of oil–water, by acidification with
coagulation [16], is based on the addition of chemicals that destroy the protective action of the emulsifying agent, overcoming the repulsive effects of the electrical double layers to allow finely-sized oil droplets to form larger droplets through coalescence [17]. Wenqi et al. [18] found that the acidification process of coal gasification wastewater using pure hydrochloric acid can reduce COD, total organic carbon (TOC), and oil by about 3.1%–11.3%, 6%–10.8% and 25.2%–57.4%, respectively, with pH value in the range of 4 to 7. El-Gohary et al. [19] found that ferric chloride, ferrous sulfate and alum were highly effective coagulants in reducing COD. Chemical treatment is also very important as a pre-treatment process for DAF and an essential requirement for efficient treatment [13]. It requires the optimum conditions of pre-treatment for reducing SS, Greases & Oil and separating Greases & Oil sludge from water [20]. Therefore, the enhanced efficiency of oily wastewater treatment using DAF needs optimum conditions for chemical pre-treatment.

There has been no research on using DAF combined with acidification and coagulation for biodiesel wastewater treatment. Thus, the aim of this research was to investigate the optimization of operational parameters to enhance the efficiency of DAF for biodiesel wastewater treatment. In this study, biodiesel wastewater was pre-treated by acidification and coagulation.

2. Material and method

2.1. Wastewater

Biodiesel wastewater was obtained from Specialized R&D Center for Alternative Energy from Palm Oil and Oil crop, Faculty of Engineering, Prince of Songkla University, Thailand. This plant uses palm oil and oil crop as feedstock and an alkali-catalyzed transesterification process. The characteristics of this biodiesel wastewater were analyzed according to the standard methods for examination of water and wastewater [21] and are shown in Table 1. The biodiesel wastewater contained a high concentration of white muddy particles, and wastewater [21] and are shown in Table 1. The biodiesel wastewater was analyzed according to the standard methods for examination of water and wastewater [21] and are shown in Table 1. The biodiesel wastewater contained a high concentration of white muddy particles, which looked like milk due to the oil emulsified in water, a high concentration of COD (60,000–150,000 mg/L) and Grease & Oil (7,000–15,000 mg/L). The ratio of BOD5/COD averaged 0.4–0.5.

2.2. Treatment procedure

The experiment runs are presented in Fig. 1. Firstly, 1 N of pure HCl and H2SO4 was used for acidification from 1 L of biodiesel wastewater. The experiments were conducted using Phipps & Bird standard jar test unit comprising six paddle rotors (24.5 mm × 63.5 mm), equipped with six beakers of 1000 mL each. Each sample was conducted under condition of stirring at 30 rpm for 20 min [22]. Treated effluent of biodiesel wastewater was collected after 1 h and at various retention times settling for determining the chemical characteristics. Secondly, alum (Al2SO43; 18H2O), polyaluminium chloride [(AlCl3(OH)2)7]+ and ferric chloride (FeCl3) from Ajeax CO., Ltd were used as inorganic chemical coagulants. The optimum coagulant concentration was determined for each coagulant. For coagulation, the experiments were conducted using a Phipps & Bird standard jar test unit. Each sample was coagulated under conditions of rapid and slow stirring at 100 rpm for 1 min and 30 rpm for 20 min, respectively [22]. Treated effluent of biodiesel wastewater was collected after 1 h and at various retention times settling for determining the chemical characteristics. Thirdly, the treated biodiesel wastewater objective under optimum condition was subjective to acidification only and acidification with coagulation, followed to DAF. The experiments were conducted using a bench scale of Capital Controls, Oxfordshire DAF unit. It consisted of a 9 L stainless steel unpacked saturator vessel of oil-free air compressor and a 1.5 L flotation cell. DAF was designed for rapid mixing, flocculation and flotation can be operated in the same vessel. The cell was installed with a needle valve through which air saturated water was injected; besides there were two sampling points and a drain. The conditions of the DAF operation including the pressure, retention time and pressurization type were investigated. The optimum of the three steps was applied to treating the biodiesel wastewater. The wastewater samples were collected at the middle depth of the flotation cell after 20 min settling for determining the chemical characteristics. The characteristic parameters of raw and treated wastewaters analysis were Grease & Oil, COD, BOD5 and oily sludge (SS) concentrations according to the standard methods for examination of water and wastewater [21].

3. Results and discussions

3.1. Treatment of biodiesel wastewater using acidification

3.1.1. Effect of pH value

The results from microscope photograph presented in Fig. 2 show oil drop coalescence in the biodiesel wastewater after adjusting various pH level with HCl and H2SO4. Oil drop coalescence in the biodiesel wastewater at pH = 5 was bigger and larger in amount than pH = 7. This result showed that oil drop coalescence reaction was high rate when the pH value of biodiesel wastewater was low. Regarding the efficiency of Grease & Oil removal, the result showed that both

**Table 1**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Unit</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Color</td>
<td>-</td>
<td>White (emulsion)</td>
</tr>
<tr>
<td>pH</td>
<td>-</td>
<td>8.5–10.5</td>
</tr>
<tr>
<td>Oily sludge</td>
<td>mg/L</td>
<td>1,500–5,000</td>
</tr>
<tr>
<td>Chemical oxygen demand (COD)</td>
<td>mg/L</td>
<td>60,000–150,000</td>
</tr>
<tr>
<td>Biological oxygen demand (BOD5)</td>
<td>mg/L</td>
<td>30,000–60,000</td>
</tr>
<tr>
<td>Grease &amp; Oil</td>
<td>mg/L</td>
<td>7,000–15,000</td>
</tr>
<tr>
<td>Saponification number</td>
<td>mg KOH/g</td>
<td>4.77–18.79</td>
</tr>
<tr>
<td>Free fatty acid (FA)</td>
<td>% Palmitic acid</td>
<td>0.14</td>
</tr>
</tbody>
</table>

Note: the analysis method according to the standard methods for examination of water and wastewater [21].
acids were similarly effective (Fig. 3). The decreased pH from 7 to 5 showed that Grease & Oil sludge concentration (C/C₀) increased and was higher than in raw wastewater (C/C₀<1) because pH = 5 of wastewater affected the carboxyl function (−COO⁻) on the surface of oil droplets, become a natural charge [23]. Then, oil droplets could be located to close and flocculated to each other. The big oil droplets from flocculation rose to the surface, which was the mechanism for removing Grease & Oil from wastewater. Fujii et al. [20] claimed that this phenomenal is demulsification. Emulsification–demulsification cycle is reversible and related to pH values. Stable emulsion was found at pH>7.7, the emulsification–demulsification process or unstable emulsion was found at pH 5.0–6.1 and the big oil droplets rose up to the surface, at pH<4. Moreover, Grease & Oil concentration of wastewater after adjusting to pH ≥ 3 decreased more than 80% and was the most efficient for COD removal (more than 45%). Therefore, adjusting pH = 3 was suitable for treating biodiesel wastewater by acidification because of the high efficiency removal.

3.1.2. Effect of retention time
Grease & Oil removal efficiency increased with increasing retention time (Fig. 4). Grease & Oil removal ranged from 81.65% at one day retention time to 95.4% at five days of retention time. This result shows that the long retention time affected the demulsion effectiveness [18]. Therefore, a retention time of 1 to 3 days was suitable for treating biodiesel wastewater by acidification because of the high removal efficiency and short retention time.

3.1.3. Characteristics of oil sludge
Grease & Oil sludge in the upper stratum of the biodiesel wastewater after acidification at pH = 3 and 3 days retention time was analyzed. Grease & Oil concentration increased by value about 1–3% as volume with increasing retention time. Further analysis showed that the palmitic acid concentration of this oil was 18%. Then, this oil could be recovered to produce biodiesel by two-step processes [24,25] for reducing the biodiesel waste. Therefore, oil recovery was the one
alternative method for enhancing the efficiency of biodiesel wastewater treatment.

3.1.4. The selected acid for biodiesel wastewater treatment by cost evaluation

The use of optimum acid concentration and retention time for acidification resulted in the demulsification of the wastewater. Then, big oil droplets of water rose to the surface and the bottom water became clear. The Grease & Oil and COD removal efficiency using both acids at pH = 3 and 1 day retention was more than 80%, and 50%, respectively and both acids were equally effective. Therefore, acid selection should be based on the operating cost of the acid. In this case, the cost of HCl was 25% higher than H2SO4. HCl is also more harmful to activate sludge systems than H2SO4 [26]. Hence, H2SO4 was found to be the more suitable acid for biodiesel wastewater treatment with acidification due to its cheaper operating cost and its usefulness for biological wastewater treatment and biodiesel production. However, even using H2SO4 for acidifying the concentration of COD (20,000–36,000 mg/L) remained higher than the wastewater quality standard for industry in Thailand [27]. Thus, this process would require further wastewater treatment.

3.2. Treatment of biodiesel wastewater using coagulation

3.2.1. Effect of coagulant concentration

Alum (Al2(SO4)3·18H2O), polyaluminum chloride [[Al13O4( OH)26]3+7 and ferric chloride (FeCl3) at 0.5, 1.0, 1.5, 2.0 and 2.5 g/L were used to determine the effect of coagulant concentration. Firstly, alum, polyaluminum chloride and ferric chloride were used in the range of 0.5–10.0 g/L to coagulate the biodiesel wastewater by Jar test without pH control. They were the ineffective to remove Grease & Oils because they required the desired pH for a hydrolysis reaction [28]. pH control is also a very important process of the coagulation mechanism for generating flocculation [5,30]. Thus, the effect of coagulant concentration with various pH controls at 5, 6 and 7 was investigated by fixing the COD concentration of biodiesel wastewater at 7120 mg/L. The results are shown in Figs. 5, 6 and 7. It was found that using alum and ferric chloride at 0.5 g/L and various pH controls at 5, 6 and 7 less than 20% and 90%, respectively of Grease & Oil removed. The principle removal mechanism at low concentration of alum and ferric chloride is adsorption [19,31]. However, using alum and ferric chloride at 1.0 g/L, more than 95% of Grease & Oil was removed. Thus, 1.0 g/L of alum and ferric chloride used without acidification was sufficient to flocculate Grease & Oil from biodiesel wastewater because the optimum concentration of a coagulant is defined as the value above which there is no significant difference in the increase of removal efficiency with a future addition of coagulant [32]. Furthermore, pH control affected the concentration of polyaluminum chloride used. The efficiency of Grease & Oil removal using polyaluminum chloride at 1.0 g/L and pH = 6–7 was more than 90%. If pH = 5, the concentration of polyaluminum chloride should be increased to 2.0 g/L and would then be highly effective in removing more than 90% Grease & Oil. The decreasing in removal efficiency of polyaluminum chloride occurred when the pH tends towards acidic or basic values and is in accordance with the amphoteric character of aluminum hydroxide that precipitates at pH 6–7; its solubility increases as the solution becomes either more acidic or alkaline [33].

With regard to the COD removal from biodiesel wastewater using coagulation, the concentrations of alum, polyaluminum chloride and ferric chloride affected the efficiency of COD removal. The efficiency of COD removal (more than 30%) varied with the concentration of alum and ferric chloride in the range 0.5 to 1.5 g/L, and polyaluminum chloride in the range 0.5–2.0 g/L because coagulant concentrations were sufficient to demand reaction and optimum pH control. When concentrations of alum (>1.5 g/L), polyaluminum chloride (>2.0 g/L) and ferric chloride (>1.5 g/L) were more than necessary, the efficiency of COD removal decreased because the flocs formed by coagulation were smaller and less compact and were not favorable for sludge. The addition of coagulation aids could overcome this disadvantage [34].

3.2.2. Characteristics of coagulated wastewater

Grease & Oil sludge concentration in biodiesel wastewater after coagulation using alum, polyaluminum chloride and ferric chloride and pH = 5, 6 and 7 was analyzed. It was found that Grease & Oil sludge concentration in biodiesel wastewater after coagulation was still high (Table 2), which was flocculation between aluminum or ferric substances and Grease & Oil from biodiesel wastewater. The positive charge of soap molecules in biodiesel wastewater was absorbed with aluminum or ferric molecules because of the different charge [35,36].

Aluminum and ferric concentrations in the wastewater were analyzed before and after coagulation. Before coagulation, the use of 1.0 g/L of alum and polyaluminum chloride concentration produced 81 and 75 mg/L wastewater, respectively, of initial the aluminum concentration, and the use of 1.0 g/L of ferric chloride concentration produced 334 mg/L wastewater of initial ferric concentration. After coagulation, 0.2 and 2.9 mg/L wastewater of aluminum concentration was found in the treated wastewater sample for alum and polyaluminum chloride used, respectively, and 0.05 g/L wastewater of ferric concentration was found in the treated wastewater for ferric chloride used. This result showed that most of aluminum and ferric was contaminates with Grease & Oil sludge coming from the adsorption and the sweep coagulation of the coagulation process [28,29,37,38]. However, the contamination of Grease & Oil sludge suspended in biodiesel wastewater was more than 10,000 mg/L of COD concentration. Therefore, biodiesel wastewater after coagulation
with alum, polyaluminum chloride and ferric chloride required further wastewater treatment.

3.2.3. The selected coagulant for biodiesel wastewater treatment by Cost evaluation

The use of optimum pH control, coagulant concentration and type in the coagulation process generated the flocculation of Grease & Oil in wastewater. The flocculation rise to the surface and the bottom water became clear. The Grease & Oil and COD removal efficiencies using three coagulants at 1.0 g/L without acidification were more than 90%, and 30% and three coagulants were equally effective. Therefore, the coagulant selection should be based on the operating cost of the coagulant and acid used. It was found that alum was cheaper than polyaluminum chloride by 10% and was cheaper than ferric chloride by 263%. Consistent with El-Gohary et al. [19], the annual cost estimation using alum, including chemical cost and sludge treatment and disposal cost, was lower than the cost of using ferric chloride and sulfate for pre-treatment of personal care product wastewater. Hence, alum was found to be a suitable coagulant for treating biodiesel wastewater. However, biodiesel wastewater treatment using coagulants showed a high concentration of COD and that floc was still suspended in the water. Thus, this process would be required further wastewater treatment.

3.3. Treatment of biodiesel wastewater using DAF with acidification and coagulation process

3.3.1. Treatment of biodiesel wastewater using DAF

Many reports [14,16,39] suggest that DAF is suitable for treating contaminated Grease & Oil wastewater because of its ability to separate
oil from aqueous dispersion. Therefore, this research was conducted using DAF test apparatus to treat the biodiesel wastewater sample. Firstly, the comparison of dissolved air value in terms of dissolve oxygen (DO) form at 3, 4 and 5 bar gage and room temperature was determined. DO concentration was found to increase at the initial pressure (0–2 min of retention time) and increased from 6.3 mg/L to 10.0, 11.6 and 12.3 mg/L at 3, 4 and 5 bar gage, respectively, because air saturation value depended on the dissolved capacity of oxygen and nitrogen in water, which varied with pressure according to Henry’s Law [16].

In addition, DO concentration slightly increased when the retention time increased to 4 min and was 11.3, 12.2 and 12.6 at 3, 4 and 5 bar gage, respectively. When the retention time increased by more than 4 min, it was found that dissolved air in water was stable. Many reports [16,39] suggested that the pressure operation design of the DAF system should be the range of 3–5 bar gage and 1–4 minutes retention time. Thus, 4 bar gage and 4 min of pressure tank were suitable condition for biodiesel wastewater treatment with DAF.

Furthermore, the pressurization operation of the DAF system consisted of two systems: full-stream pressurization and recycle-stream pressurization [16,39]. With full-stream pressurization, the influent is pressurized and then released in the flotation tank where the bubbles are formed. With recycle-stream pressurization, a portion of treated wastewater is pressurized and recycled to the flotation tank [14]. Thus, biodiesel wastewater treatment using full-stream pressurization and recycle-stream was investigated. The result showed that the use of DAF with either of two pressurization operations did not separate Grease & Oil or reduce the emulsion from the wastewater. Therefore, this process required a pre-treatment process for biodiesel wastewater treatment. The combination of pre-treatment and DAF showed the effective performance for treating and reducing the emulsion of oily wastewater [14,36,40,41].

### 3.3.2. Treatment of biodiesel wastewater using DAF with acidification and coagulation

The treatment procedure of the novel approach for biodiesel wastewater treatment consisted of third step. Firstly, the biodiesel wastewater was treated by acidification at pH = 3 with 3 days retention time. Secondly, the treated wastewater after acidification was flocculated by alum as the selected coagulant. Thirdly, the treated wastewater after acidification and coagulation process was treated by recycle-stream pressurization of DAF system because Grease & Oil sludge clogged up the flotation cell of the DAF system if the full-stream pressurization was used.

The efficiency of sludge removal by DAF depended on the ratio of air to sludge (A/S ratio) and the suggested range of the A/S Ratio was 0.005–0.06 (kg/kg) [16,39]. The result in Fig. 8 shows that the A/S ratio decreased with the increasing concentration of Grease & Oil sludge because of using an increasing alum concentration. The A/S ratio of the tap water used for the first time was similar to the A/S ratio of the treated wastewater used the second time. The A/S ratio for 20% of the recycle rate was in the range of 0.01–0.05, but the A/S ratio for 40% of the recycle rate was in the range of 0.03–0.09, which was relatively high value because Grease & Oil sludge from coagulation using alum was low when the alum concentration was less than or equal to 150 mg/L. This result shows that coagulation process with 20% of the recycle rate is sufficient to separate Grease & Oil sludge from treated biodiesel wastewater using alum.

Moreover, the retention time for flotation cell of DAF at 20 and 40% of the recycle rate was 7 and 9 min, respectively. These two values were lower than the suggested value for the DAF design, which was 20–30 min [16] because Grease & Oil sludge from coagulating the biodiesel wastewater with alum was low density. In the part of the rise rate value, the solid–air mixture [16], from calculation found that the rise rate values at 20% and 40% of recycle rate were 36.7–40.8 L m² min⁻¹ (0.901–1.001 GPM/ft²) and 20.4–28.5 L m² min⁻¹ (0.500–0.701 GPM/ft²), respectively. These two values were similar to the value for DAF design by suggested Mactcalf & Eddy [39], which was 8.0–160 L m² min⁻¹ (0.2–0.4 GPM/ft²). Therefore, DAF could be operated with this condition for this transesterification process of biodiesel wastewater treatment.

Fig. 9 shows that the efficiencies of Grease & Oil removal from biodiesel wastewater at 100–250 mg/L of alum concentrations with DAF was 98–99.6%, compared with Grease & Oil concentration after acidification which was 85–95%. The remaining Grease & Oil concentration in the effluent was 30–100 mg/L. The efficiency of Oil and Grease removal from biodiesel wastewater using DAF with acidification and coagulation was 10% more effective than the coagulation-dissolved air flotation process for treating vegetable oil refining industry wastewater [42] and personal care product wastewater [19]. In addition, the efficiencies of COD removal by DAF from treated biodiesel wastewater with 20 and 40% of the recycle rate were 20–30% and 40–50%, respectively and there was no remaining SS after treatment with DAF and ≥150 mg/L of alum dose. This result

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### Table 2

<table>
<thead>
<tr>
<th>Coagulant</th>
<th>Initial Grease &amp; Oil (mg/L)</th>
<th>Coagulant dosage (g/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alum</td>
<td>7120</td>
<td>2822 24 7 3 7</td>
</tr>
<tr>
<td>Polyaluminum chloride</td>
<td>7120</td>
<td>1134 219 23 13 72</td>
</tr>
<tr>
<td>Ferric chloride</td>
<td>7120</td>
<td>216 26 26 19 42</td>
</tr>
</tbody>
</table>

Note: Conditions: pH = 6; 1 hour retention time.
shows that the remaining COD concentration without SS came from the contamination of solution. Thus, the effluent (Table 3) of biodiesel wastewater treatment by DAF with acidification and coagulation required additional and further wastewater treatment processes to remove the remaining organic matters from wastewater. The occurrence of Grease & Oil sludge from biodiesel wastewater treatment by DAF with acidification and coagulation was also only 5–20 mL sludge/L wastewater, which was less than using only coagulation. Finally, the primary operating cost of biodiesel wastewater treatment using DAF with acidification and coagulation was evaluated. The operating cost of this process was lower than using only coagulation. Therefore, the pre-treatment of biodiesel wastewater by acidification and coagulation could enhance the efficiency of DAF and be more economical for biodiesel wastewater treatment.

4. Conclusion

The efficiency of Grease & Oil removal by DAF with alum from treated biodiesel wastewater with acidification was 85–95%. In addition, the efficiencies of COD removal by alum and DAF with 20% and 40% of the recycle rate were 20–30% and 40–50%, respectively and there was no remaining SS after treatment by DAF with ≥ 150 mg/L of alum dose.

The pre-treatment of biodiesel wastewater treatment by acidification and coagulation could be enhanced the efficiency of DAF and more economical for biodiesel wastewater treatment.

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