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POTENTIAL OF USING ACTIVATED SLUDGE AS FEEDSTOCK FOR BIODIESEL PRODUCTION IN TAIWAN

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ABSTRACT

Commercial production of biodiesel mainly uses refined edible oil as the feedstock which constitutes about 70% to 85% of the overall cost of biodiesel. Finding cheap and abundant new feedstock for producing biodiesel is necessary. The use of non-edible oil such as waste cooking oil and algae oil may help in reducing the production cost of biodiesel. In this study, 5 wet sludge samples were collected from a wide spectrum of industries in Taiwan and which were treated by subcritical water to enhance the amount of their extractable lipids. Reaction temperature was found to be the most important factor in increasing the extractable neutral lipids (2 to 3.5 times) which can be esterified with methanol to produce biodiesel. The economic potential of using activated sludge as feedstock for biodiesel production in Taiwan was discussed.

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1 INTRODUCTION

Subcritical water (SCW) can be applied to treat biomass from agriculture and wastes such as activated sludge (AS) and micro-algae which have high water content. Water contained in feedstock can be utilized as reactant in the in situ hydrolysis reaction which means drying of feedstock prior to pretreatment is not required.

The use of transesterification and esterification reactions in subcritical/supercritical methanol to produce biodiesel were first investigated by Saka and Kusdiana (2001). The esterification is a combination of two steps: free fatty acid (FFA) obtained by the hydrolysis of oil and the esterification of FFA and methanol in supercritical state to produce fatty acid methyl ester (FAME) with high yield (90%) at 300°C in short time (12 min) (Kusdiana and Saka, 2001; Kusdiana and Saka, 2004). Approximately 95% FFA yield can be obtained at a hydrolysis temperature of 330 to 340°C and a water to oil ratio of 2.5 to 5.0 in SCW condition (King *et al*., 1999).

Taiwan imports about 98% of its energy demand, the government proclaimed to look for new and clean energy sources and biodiesel is one of the preferred choices (Tsai *et al*., 2007; Huang and Wu, 2008). Different feedstock, not only edible/non-edible oil from the seeds but also the other sources such as used cooking oil, algae and sludge were investigated for their potential to produce biodiesel (Alhassan *et al*., 2014). In Taiwan, the annual production of dry sludge is about 60,000 tons, with a biodiesel potential of over 12,000 tons (Lee, 2007). The utilization of sludge from various sources by converting it into valuable resources was summarized by Tyagi and Lo (2013). Four fatty acids (C16:0, C18:0, C18:1 (n-9) and C18:2

(n-6)) were found to be dominated the lipid composition of raw sewage.

Boocock *et al*. (1992) obtained 18.0 wt.% lipids by solvent extraction from sludge. FFA and glyceride contents in the extracted lipids were 65.0 wt.% and 7.0 wt.%, respectively. Jardé *et al*. (2005) studied the extracted lipid of 48 sludge samples from different wastewater treatment plants and concluded that the dominated fatty acids were between C10 and C18; fatty acid and steroid made up 36.8 wt.% of the dry sludge. Dufreche *et al*. (2007) estimated that the biodiesel cost per gallon produced from municipal sludge would be around $3.11, however, the total biodiesel production cost from soybean oil is $4.60 per gallon while feedstock cost occupies from 80 to 85 % of total production cost (Yeboah *et al*., 2013). Mondala *et al*. (2009) did an economic analysis of producing biodiesel from primary and secondary AS using in situ transesterification and concluded that biodiesel could be produced from sludge at $3.23 per gallon which is cheaper than the diesel price in United States (US) hitting $4.00/gallon in February 2013 (U.S. Energy Information Administration, 2013). Of course, more thorough economic evaluation by taking into account other costs such as distribution, marketing and taxation is required.

The objective of this study was to investigate the feasibility of producing biodiesel using AS as raw material. AS samples collected from various industries in Taiwan, their lipid contents analyzed both before and after SCW treatment. Finally, the economic feasibility of biodiesel production from AS was assessed.

2 MATERIALS AND METHODS

2.1 Chemicals

All chemicals used were either gas chromatography (GC) or analytical reagent grade and obtained from commercial sources. FAME standard mixture (47885-U, 37 components FAME mixture) purchased from Supelco (Bellfonte, PA). Standards of FFA and alkane standard mixture (C_{10}– C_{40}) obtained from Sigma Aldrich Corp. (St. Louis, MO). All chemicals, standards, and gases were used as received.

2.2 Sample preparation

Five dewatered AS samples were collected from a wide spectrum of waste water treatment plants in Taiwan: Chung Hwa Picture Tubes LTD Taoyuan Factory (S1), Jhunan Brewery (S2), Hsin-Tung-Yang LTD Da-Yuan Factory (S3), China Petroleum Corporation Kaohsiung Refinery (S4) and Chun-Hwa Pulp (S5). More details information about the five sludge samples are given in Table 1. The fresh collected samples were kept in a freezer at - 84°C before use.

2.3 SCW treatment

SCW treatment of AS was carried out in a high pressure reactor. Details of the reactor can be found elsewhere (Tran-Nguyen *et al*., 2013). Wet AS sample (30 g) was loaded in the reactor. Before heating was started, an initial pressure of 2 MPa was introduced into the reactor by using nitrogen gas to ensure that water was at subcritical state at temperatures (150 to 225°C) carried out in this study. After reaching the desired temperature, the treatment was run for 15 – 30 min. The reactor was then cooled down to room temperature, the treated sample was collected and dried. Crude lipid in the pretreated sample as well as in untreated samples was extracted by n-hexane. Three replicates were carried out for SCW treatment of each sample.

2.4 Fatty acid profile

AOCS official method Ca6b-53 (AOCS, 1998-2002) was employed for determining the unsaponifiable fraction of the crude lipid. The saponifiable fraction was collected and acidified to pH 2 using sulfuric acid and stirred at 60°C in a water bath until the reaction was completed. The organic phase containing fatty acids was separated by hexane. The fatty acids were then converted into their corresponding FAMEs by adding boron trifluoride (BF_3) in methanol at 60°C.

2.5 GC analysis

Fatty acid profile analysis was carried out in a Shimadzu GC-2010 (Kyoto, Japan), equipped with a split injector and a flame ionization detector. Separation was run on a ZB-5HT (5% phenyl) - methylpolysiloxane nonpolar column (15 m x 0.32 mm i.d., 0.1 µm film thickness, Zebron, Phenomenex, Torrence, CA, USA). Twenty-milligram sample was dissolved in 1 mL ethyl acetate and 1 µL sample was injected into the GC. The carrier gas was nitrogen with a linear velocity of 30 cm/s at 80°C. The operating conditions were as follows. Both injector and detector temperatures were set at 370°C. The initial oven temperature began at 80°C, increased to 365°C at 15°C/min and held for 8 min. The total analysis time was 29 min. The hydrogen flow, air flow and make up flow were set at 50 mL/min, 500 mL/min and 30 mL/min, respectively

while the linear velocity and purge flow were 8.0 cm/s and 3.0 mL/min, respectively.

3 RESULTS AND DISCUSSION

Table 1 shows that water contents of the sludge samples vary from ~ 80% (S2) to ~ 90% (for S1, S3 and S4), except for S5 which is about 48%. Table 2 presents the amounts of crude lipid extracted from five sludge samples. The amount of neutral lipid extracted from the SCW treated AS is about 1.5 to 4 times that of the untreated one, except for sample S5 which changed insignificantly before and after SCW treatment. One of the possible reasons is that S5 is a primary sludge. Primary sludge is obtained in the first stage of the process and collected by sedimentation in primary clarifier, however, the secondary sludge is taken from secondary clarifier after a biological process (Tsigie *et al*., 2012). Lipid in AS originates from accumulated lipid inside waste as well as lipid during growth of microorganisms (Boocock *et al*., 1992; Jarde *et al*., 2005; Tyagi and Lo, 2013).

Table 1: Sources of sludge samples and their moisture content

Category	Sources	Moisture Content (%)
Chung-Hwa Picture Tubes LTD, Taoyuan Factory (S1)	Waste contained water from washing glass substrate, waste oil and waste solution (waste acids and waste alkaline). Sludge was produced by secondary biological treatment process in the electronic industry.	91.39 ± 0.20
Jhunan brewery sludge (S2)	Brewery wastewater contains organics such as sugar, soluble starch, ethanol and volatile fatty acids. Sludge was generated by secondary biological treatment processes in the brewing and beer industry.	80.86 ± 0.66
Hsin-Tung-Yang LTD Da-Yuan Factory, Food processing sludge (S3)	Product includes meat and bakery products, snacks, noodles, tea, etc. Sludge was produced by secondary biological treatment processes in the food/drink industry.	89.01 ± 0.14
China Petroleum Corporation Kaohsiung Refinery (S4)	Various petroleum products and petrochemical raw materials from refined crude oil. Sludge was generated by secondary biological treatment processes in the petroleum industry.	86.30 ± 0.51
Chung-Hwa Pulp, pulp sludge (S5)	Produces pulp products, cultural paper, etc. Sludge was produced by wastewater treatment in the paper and pulp and paper product industries.	47.17 ± 1.46

Table 2: Extractable lipid contents (g lipid/g dried sludge) of sludge samples

Sludge	Before SCW treatment		After SCW treatment[a]		Increase (%)[b]
	Crude lipid	Neutral lipid	Crude lipid	Neutral lipid	
S1	1.67 ± 0.19	1.24	7.08 ± 0.11	4.34	350.00
S2	5.35 ± 0.03	3.88	9.83 ± 0.25	6.37	164.18
S3	17.99 ± 0.78	17.49	45.42 ± 0.45	37.96	217.04
S4	26.91 ± 0.95	2.77	26.55 ± 1.32	7.91	285.56
S5	0.53 ± 0.03	0.15	0.52 ± 0.02	0.18	120.00

[a]SCW treatment at 175 °C for 15 min *[b]Increase of neutral lipid (%) after SCW treatment*

3.1 Effects of SCW treatment on extractable crude lipid

3.1.1 Effect of temperature on SCW treatment

Tsigie *et al*. (2012) reported that temperature was the most influential factor on extractable lipid in the SCW treatment of biomass. In this study, temperature was varied from 150°C to 225°C in the SCW treatment of sludge samples with a fixed treatment time of 15 min. Only the data of S2 was shown in this section. As shown in Figure 1, after SCW treatment, the amount of extractable lipid increased about 1.9 times which agree with the study of Tsigie *et al*. (2012).

A similar trend was observed in the increase of FFA content in the extracted lipid after SCW treatment (an increase of 2.74 times). The increase of FFA can be explained because SCW provides an acidic reaction environment and water can act as a

neutral acid-base catalyst for extraction and reactions such as hydrolysis and biodegradation (Fujii *et al*., 2006; Huynh *et al*., 2012; Tsigie *et al*., 2012;). Another reason could be that the presence of FFA in sludge can help as a catalyst to produce more FFA during SCW treatment. Additionally, under SCW treatment, TG and phospholipids were released and hydrolyzed resulting in an increase in FFAs. Both crude lipid and FFA obtained slightly decreased at 200°C and 225°C, may be due to degradation or breakdown of unsaturated lipids. As can be seen in Table 2 and Figure 1, a significant portion of the increase in lipid extracted after SCW treatment came from the increase of FFA. Tran Nguyen *et al*. (2013) proposed a mechanism based on the SCW hydrolysis of cell phospholipid bilayer to explain this increase in FFA extracted after SCW treatment of biomass. Sample S4 was collected from a petroleum refinery and its extractable crude lipid increased insignificantly after SCW treatment, however, the neutral lipid of treated sample rose to 2.86 times.

3.1.2 Effect of time on SCW treatment

The amount of extractable lipid did not change significantly as SCW treatment time was increased from 15 min to 30 min; however, a slight decrease in FFA content in the extracted lipid was observed (data not shown). The slight decrease in FFA content may be due to the degradation of FFA at high temperature and prolonged reaction time as suggested by Fujii *et al*. (2006). In this study, SCW treatments were carried out at 175°C for 15 min to minimize undesired reactions.

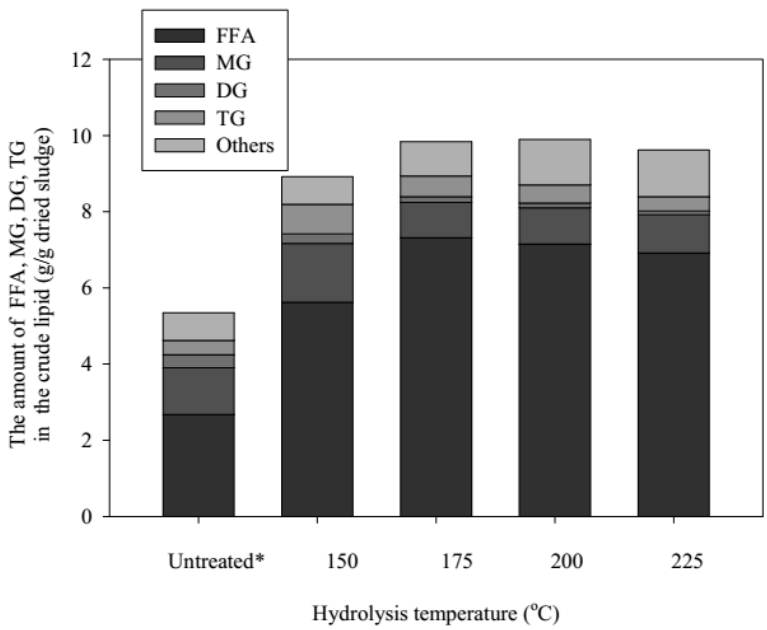

Fig. 1: Effects of temperature on crude lipid yield and FFA, MG, DG, TG content in the SCW treatment of wet S2

**untreated is the sludge without SCW treatment*

3.2 Fatty acid profile of lipid

Fatty acid profiles of lipids from untreated and SCW treated AS samples were determined. Various fatty acids were detected which may be the result of the large, uncontrollable diversity of microorganisms living in sludge. Table 3 shows the fatty acid profiles of 4 AS samples. S5 is not included because it contains too little lipid (0.52 %) so its fatty acid profile was not determined. In general, albeit increase in extractable lipid after SCW treatment, fatty acid profiles of untreated and SCW treated samples are very similar. Fatty acid profile varies from sample to sample due to different environments in which microorganisms were cultivated. It is clear from Table 3 that there is significant difference between the fatty acid profiles of S3 and the others. S3 was collected from a food processing plant with pastry, meat and drink as the main products. Almost half of fatty acid is palmitic acid which reflects the fact that palm oil is widely used in confectionary industry in Taiwan. As men-

tioned previously, after SCW treatment, the amount of extractable lipid for AS increased from one and a half to four times. This increase was the result of breakdown of structure lipid during SCW treatment. For example, C16:1 in sample S3 increased about 1.5 times after SCW treatment. More than 90% of fatty acids in the SCW treated S3 sample (from food processing plant) are saturated and mono-unsaturated. Moreover, S3 sample contains high amount of neutral lipid (17.49%) which increased significantly to 37.96% after SCW treatment. Hence, sludge from food processing industry seems to be a good feedstock of biodiesel production.

Table 3: Fatty acid profiles of sludge sample S2, S3, and S4

Fatty acids	S2[a]		S3[b]		S4	
	Untreated	SCW	Untreated	SCW	Untreated	SCW
C10:0	0.30	1.78	ND	ND	2.6	ND
C11:0	0.28	ND	ND	ND	ND	1.5
C12:0	1.76	2.59	0.23	0.24	4.2	3.1
C12:1	ND[c]	ND	ND	ND	2.6	ND
C13:0	0.80	1.03	0.07	0.07	ND	ND
C14:0	2.94	3.21	1.87	2.03	1.4	3.4
C15:0	2.78	3.21	0.37	0.36	ND	0.9
C16:0	29.26	24.79	46.24	50.36	13.5	22.5
C16:1	5.32	7.92	ND	ND	6.3	5.3
C17:0	1.89	2.17	0.56	0.58	2.7	1.0
C17:1	ND	ND	ND	ND	2.9	1.4
C18:0	15.97	11.75	30.51	31.61	5.0	6.1
C18:1	14.29	17.15	2.88	4.29	14.9	13.4
C18:2n6c	5.79	5.83	2.99	1.49	ND	ND
C18:3n3	ND	ND	ND	ND	ND	ND
C20:0	0.95	0.85	0.94	0.96	ND	ND
C22:0	0.92	0.56	ND	ND	ND	ND
C22:n	2.06	0.99	1.22	0.55	ND	ND
C24:0	ND	ND	ND	ND	17.1	20.1
Others	14.68	16.17	12.11	7.45	29.4	21.3

[a,b]Data reported in Tran Nguyen et al. (2013); [c]ND: not detected

4 FEASIBILITY OF ACTIVATED SLUDGE AS FEEDSTOCK FOR BIODIESEL PRODUCTION

Biodiesel is a renewable energy. It is biodegradable, environmental friendly and sustainable. The main obstacle for widespread use of biodiesel is its higher cost than petro-diesel due to the high cost of feedstock oil, as feedstock may drastically lower the total biodiesel production cost (Siddiquee and Rohani, 2011).

Mondala *et al.* (2009) estimated that biodiesel cost is $3.23 per gallon at an assumed FAME yield of 10% per dry weight of sludge. They obtained 14.5% and 2.5% FAME yield at 75°C, 24 h, 5% (v/v) H_2SO_4 and a methanol to dried sludge mass ratio of 12 for primary sludge and secondary sludge, respectively. Revellame *et al.* (2011) mentioned that the price of biodiesel produced from dried sludge at the break-even point is $7.42/gallon at a FAME yield of 4.79%. For wet sludge, the price for biodiesel would be $14.48/gallon at 3.93% FAME yield. Those biodiesel break-even prices were considerably more expensive than that of petro diesel ($2.95/gallon) at the time of their study. They estimated that price of biodiesel from sludge can be cheaper than diesel if the capacity of biodiesel per year is higher than $3x10^5$ gallons (at 10% of FAME yield). Dufreche *et al.* (2007) presented a cost analysis of producing biodiesel from wet sludge and concluded that the total cost is $3.11/gallon at 7.0% FAME yield. This cost would reduce to $2.01/gallon if the FAME yield is increased to 15%. To compete with biodiesel from soybean oil, biodiesel produced from sludge should cost no more than $2.50/gallon with a FAME yield of 10.0%. In fact, the cost of drying and oil extraction constitutes more than 50% of the total cost in Dufreche's analysis. If drying and oil extraction can be avoided by using wet sludge in in-situ transesterification, the price of biodiesel from AS can be decreased significantly, making it competitive to

biodiesel from vegetable oils or even to petro diesel. In fact, Huynh *et al*. (2012) demonstrated that under subcritical condition wet AS (85% water) can be directly used to produce BD using the water it contains as the hydrolysis reagent to enhance the extraction of lipid in AS, and as a catalyst for the reaction of neutral lipids and methanol to form FAME. They were able to achieve a FAME conversion of 81% in 8 h at 175°C, 3.5 MPa and a methanol to sludge ratio of 30 ($mL.g^{-1}$).

In the process employed by Huynh *et al*. (2012), the time required is shorter, drying, oil extraction and the use of conventional acid/base catalyst are not required. The analysis of Dufreche *et al*. (2007) based on the assumption of 10.0% overall transesterification yield. If more than 50% of the total cost can be saved by avoiding drying (41.5% of the total cost) and oil extraction (11% of the total cost), then it is economic feasible to produce biodiesel from wet AS with an extractable lipid content of more than 6% which will give ~5% FAME yield assuming a FAME conversion of 81% (Huynh *et al*., 2012).

In 2012, the amount of AS declared in Taiwan is 693,165 tons (Industrial waste report and management system, 2012) out of this (in tons) 11,395 is from brewery industry, 226,661 from pulp and paper industry, 41,491 from food processing industry, 21,775 from livestock farming, 376,090 from municipal waste water treatments and 15,753 non-toxic sludge from petro industry. In this study, five AS samples were collected, representing a wide spectrum of industries in Taiwan. Out of the five samples collected in this study, S5 (from pulp and paper industry) and S1 (from Chung-Hwa Picture Tubes LTD, Taoyuan Factory) cannot meet the 6% extractable lipid criterion. The average yield of FAME from samples representing brewery industry, food processing industry and petroleum industry would be 5.41%, 30.11% (Tran-Nguyen *et al*., 2015) and 6.72%, respectively. Except AS from paper and pulp industry, which contains too little lipids (< 1%), about two thirds of annual AS produced in Taiwan can be used as the feedstock for biodiesel production. Due to the Personal Information Protection Act that blocks the obtaining of information about the amount of AS produced annually by each factory/industry, we were unable to estimate the amount of biodiesel that can be produced annually from AS in Taiwan.

5 CONCLUSION

This study shows that reaction temperature was the key parameters in enhancing the extractable neutral lipid (2 to 3.5 times) from AS in SCW treatment while time reaction has no effect. Wet sludge can be directly used as feedstock for producing biodiesel. The evaluation of potential biodiesel production from AS in Taiwan per annum was reported. Except for sludge from paper and pulp industry, about two thirds of the AS produced in Taiwan can be used as feedstock for biodiesel with price competitive to that of petro diesel.

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