Bioresource Technology 185 (2015) 185–193

Contents lists available at ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

# Process development for scum to biodiesel conversion

Chong-hao Bi [a,b], Min Min [b,*], Yong Nie [c], Qing-long Xie [b], Qian Lu [b], Xiang-yuan Deng [b], Erik Anderson [b], Dong Li [a], Paul Chen [b], Roger Ruan [b]

[a] College of Engineering, China Agricultural University, Beijing 100083, China
[b] Center for Biorefining, and Department of Bioproducts and Biosystems Engineering, University of Minnesota, 1390 Eckles Avenue, St. Paul, MN 55108, USA
[c] College of Chemical Engineering, Zhejiang University of Technology, Hangzhou 310014, China

## HIGHLIGHTS

- It is the first time that scum was utilized and converted to produce liquid fuel.
- Technical aporia have been solved including impurity and layer difficulty.
- 70% dried and filter scum could be converted to biodiesel.
- Designed process is highly scalable and has great commercialization potential.
- Not only bring revenue to the wastewater plant but reduce waste and pollution.

## ARTICLE INFO

*Article history:*
Received 29 November 2014
Received in revised form 17 January 2015
Accepted 19 January 2015
Available online 18 February 2015

*Keywords:*
Biodiesel
Acid washing
Glycerol washing
Scum
Wastewater treatment plant

## ABSTRACT

A novel process was developed for converting scum, a waste material from wastewater treatment facilities, to biodiesel. Scum is an oily waste that was skimmed from the surface of primary and secondary settling tanks in wastewater treatment plants. Currently scum is treated either by anaerobic digestion or landfilling which raised several environmental issues. The newly developed process used a six-step method to convert scum to biodiesel, a higher value product. A combination of acid washing and acid catalyzed esterification was developed to remove soap and impurities while converting free fatty acids to methyl esters. A glycerol washing was used to facilitate the separation of biodiesel and glycerin after base catalyzed transesterification. As a result, 70% of dried and filtered scum was converted to biodiesel which is equivalent to about 134,000 gallon biodiesel per year for the Saint Paul waste water treatment plant in Minnesota.



## 1. Introduction

As a renewable fuel, biodiesel has become an attractive alternative for diesel fuel. Vegetable oil has been the major source for global biodiesel production since 1990s (Krawczyk, 1996; EIA, 2014). It is still the largest biodiesel feedstock in U.S. now. For instance, in 2013, 5507 million pounds of soybean oil in the U.S. were processed into biodiesel (48.8% of all kinds of feedstock); meanwhile, a total of 10,302 million pounds of soybean oil worldwide were used to produce biodiesel (U.S. Department of Energy, 2014). However, the main problem with making biodiesel a competitive fuel on the world market is its high cost. Currently, for vegetable based biodiesel, the feedstock alone accounts for 70–80% of the total production cost (Cooperation, 2010). Many efforts have been focused on using spent oil or waste oil, such as waste cooking oils, gutter oil, and wastes from animal or vegetable oil processing operations, to reduce the cost (Haas et al., 2006; Canakci and Sanli, 2008).

Scum is a floatable material skimmed from the surface of primary and secondary settling tanks in wastewater treatment plants. It contains animal fat, vegetable oil, food wastes, plastic material, soaps, waxes and many other impurities discharged from restaurants, households and other facilities. Based on the report of the U.S. EPA's Office of Solid Waste, approximate 1–3 billion gallons of waste grease, oil and fats are produced every year in the 30 metropolitan areas in the United States (Wiltsee, 1998). Some of these wastes (yellow grease) are collected in restaurants and converted to biodiesel. However, approximately 60% of waste oils enter the sewer systems and end up in wastewater treatment plants. Due to a lower density than water, most of the oil floats on the surface of treatment facilities and conglomerates with other

* Corresponding author. Tel.: +1 612 625 1710; fax: +1 612 624 3005.
*E-mail addresses:* minxx039@umn.edu (M. Min), ruanx001@umn.edu (R. Ruan).

http://dx.doi.org/10.1016/j.biortech.2015.01.081
0960-8524/

wastes to form scum, which makes scum a rich source of energy for recovery. The oil content of scum could be as high as 60%.

The conventional technology for energy recovery is to co-process scum with sludge in anaerobic digestion where the produced biogas can generate electricity for plant use (Outwater and Tansel, 1994). However, this technology raises many problems in operation. For example, the scum floats on the top of the digester and forms a thick layer that impedes digester performance. As a result, many wastewater treatment plants choose to directly dispose scum in landfills. The scum disposal not only increases the cost of treatment facilities, but also causes many environmental problems. For instance, the Metropolitan wastewater treatment plant at St. Paul. MN (Metro Plant) spends $100,000 a year just for landfilling the scum. The landfill leachate could be a potential source of underground water pollution. In order to solve these problems, an alternative process/technology needs to be developed to recover energy from the scum and at the same time reduce the environmental impact due to landfilling scum.

In this study, a new scum-to-biodiesel conversion process was developed so that the fat, free fatty acids and soap content in the scum can be successfully converted to ASTM-grade biodiesel. Since the final product is a liquid transportation fuel-biodiesel, it could gain a much higher economic value than biogas generated through anaerobic digestion. Due to the abundance of natural gas, biogas has the selling price close to $0.80/therm; while the biodiesel has the selling price about $3.2/therm which is three times higher than biogas (CenterPoint Energy, 2014). In addition, the byproducts such as glycerol and solid residues can be used as a heating source that not only decreases the energy input but also reduces the cost of the process. In a word, the current developed process has obvious economic and environmental advantages over other treatments and disposal processes.

Currently, the most common process for biodiesel conversion is through base-catalyzed transesterification where triglyceride reacts with methanol to form fatty acid methyl ester (FAME). Potassium hydroxide (KOH) and sodium methoxide ($CH_3ONa$) are the most common catalysts. However in the base-catalyzed transesterification process, feedstock must be free of water and free fatty acid (FFA), otherwise soap formation will reduce the biodiesel conversion yield. When using low grade feedstock such as yellow grease or brown grease, the FFA content can be as high as 30%. A pretreatment method which reduces acid value below 2 mg KOH $g^{-1}$ of oil, must be used before proceeding with any base-catalyzed transesterification process. Scum as a complex material contains solids, water, soap, fatty acids, oil and many other impurities. To maximize the conversion yield, we explored several pretreatment methods in this project to clean up the scum oil. The objective of this project was to develop an effective pretreatment and conversion process to convert the scum to biodiesel.

## 2. Method

### 2.1. Material and reagent

The scum samples were collected from the Metro Plant at St. Paul, MN. Sulfuric acid (96.4%, AR) and hydrochloric acid (36.5–38.0%, AR) were obtained from Mallinckrodt Baker, Inc., Paris, Kentucky. Phosphoric acid (85.0%, GR) was obtained from EM Industries, Inc., Potassium methoxide (>90.0%) was purchased from Alfa Aesar. Butylated hydroxytoluene (BHT), methanol (anhydrous, 99.8%), chloroform (99.8%) and diethyl ether (99.7%) were obtained from Sigma–Aldrich, Inc. $BF_3$-methanol reagent (14% borontrifluoride, 86% methanol), sodium hydroxide 0.5 normal in methanol, heptane (HPLC grade) and glycerol (99.9%) were obtained from Thermo Fisher Scientific, Inc., potassium hydroxide concentrate (1.0 mol/L) was obtained from Fluka Analytical Sigma Aldrich Co., ethanol (200 proof) was from Decon Laboratories, Inc., distilled water was obtained from Premium Waters, Inc., MN, USA.

### 2.2. Process design

The general process for the scum to biodiesel conversion is illustrated in Fig. 1 and the main processes can be summarized according to the following six steps:

Filtering → Acid washing → Acid catalyzed esterification → Base catalyzed transesterification → Glycerol washing → Oil refining

Firstly large particles in scum were separated by filtration. Then acid washing followed by gravitational settling was used to convert soap (mainly $(RCOO)_2Ca$ and $(RCOO)_3Al$) to free fatty acid (FFA), separate oil from water and water soluble/insoluble electrolytes, and further remove fine particles. The upper layer oil was collected and subjected to acid catalyzed esterification and then base catalyzed transesterification processes to form FAME. The basic reactions were described in Eqs. (1)–(3). A glycerol washing was then performed to aid separating the glycerol and other impurities from the FAME. After separating the glycerol and methanol from the FAME, the crude FAME was then refined by fractional distillation to produce high grade biodiesel that can be directly used in diesel engines:

$$\text{Soap} + \text{Acid} \longrightarrow \text{Salt} + \text{FFA} \quad (1)$$

$$\text{FFA} + \text{Methanol} \xrightarrow{\text{Acid Catalyst}} \text{Water} + \text{FAME} \quad (2)$$

$$\text{Triglyceride} + \text{Methanol} \xrightarrow{\text{Base Catalyst}} \text{Glycerol} + \text{FAME} \quad (3)$$

#### 2.2.1. Filtering process

Scum oil was obtained in solid form at room temperature. It melted at about 40 °C (depending on its components) and the viscosity of scum was negatively related with the temperature. For easing the separation of the scum oil from solid particles and at the same time drying the oil, a filtration process was conducted under a relatively high temperature to reduce the viscosity of the scum oil. In a drying oven of 105 °C, 157 g of scum was loaded into a polyester mesh filter bag with pore size of 100 micron and a beaker was placed beneath the filter bag to receive the oil melted from the scum sample. The filtering process lasted for 24 h. Weights of filtered oil and remaining solids were recorded every 15 min during the first 8 h and at the end of the 24 h. The moisture content was calculated by subtracting the oil and solid weights from the total scum weight.

#### 2.2.2. Acid washing

Purpose of the acid washing was to convert any soap in scum to free fatty acids, maximize the biodiesel production yield, break emulsion for better water/oil separation, and further remove impurity from the scum oil. It also made the acid catalyzed esterification easier due to less impurity interference with the reactants. $H_2SO_4$, HCl, and $H_3PO_4$, with two $H^+$ strength 0.2 N and 1.2 N in water were compared for the scum oil pretreatment. The ratio of the scum oil-to-acid solution was 1:1 by weight. Raw scum oil was mixed with acid solution in a set of flask with condenser on top; a magnetic stirring water bath was applied to offer stirring power and keep the temperature of the reaction system at 60 °C (Leung et al., 2010). After acid washing for 1 h, the mixture was allowed to settle for about half an hour to collect the oil in the upper phase and sediment/water in the lower phase. The upper oil was then separated for the next process.