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Preliminary study at lipids extraction technology from municipal sludge by organic solvent

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Abstract

With the development of our society, waste water treatment ratio is going higher and as a result there are more and more sewage sludge. Because of its high water content, containing heavy metal and some virus, sewage sludge has been a serious environmental problem. Considering the energy crisis and serious environmental pollution, and because of the fact that there are considerable lipids in sewage sludge, which can be turned into biodiesel by esterification or transesterification, we proposed a new solution for sewage sludge problem which is to make biodiesel from sewage sludge. The whole process is divided into three steps: heat-drying, extraction of lipids and transesterification. In this research, we are going to discuss the extraction step. We extracted lipids from sewage sludge by referring to the national standard for food. We used many organic solvents such as ether, hexane, acetone, chloroform – methanol. Then we will discuss that which solvent will be more favorable to extract the lipids from the sludge by measuring the quantity of the lipids we have obtained. After the experiment, we have found that as to the single solvent extraction, bromo-propane solvent has the highest extraction effect, the other sort of the solvents are ether, toluene, N-hexane, petroleum ether. While, as to mixed solvent extract, the hexane – ethanolthe solvent has higher efficiency than other mixed solvent. The results show that mixed solvent has higher efficiency than the single solvent and the polar solvents are easier to extract more lipids than the non-polar solvents from the sludge.

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Key words: sewage sludge, organic solvent, biodiesel, lipids extraction

1. Introduction

With the rapid development of the society, the over-consumption of the energy has lead to energy crisis. “Sustainable development” has been the leading concept for our country and even the world, which

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also asked us to find renewable and environment-friendly energy [1]. Biodiesel could be one of the solutions. Biodiesel is environmental-friendly and renewable, whose raw materials are from plants and animals. Compared with traditional fossil fuels, biodiesel not only has the ability to circulate, at the same time, it also has high-quality combustion performance and safety performance [2]. But researches show that the cost of traditional raw materials for biodiesel amount to almost 80% of the total cost of biodiesel production, which strongly prevent biodiesel to be used widely. So cheaper raw material is demanded [3]. Sewage sludge turned to be suitable substitute. In China the average concentration of organic matter is 38.4% in China, among the total organic, carbohydrates account for 55%, protein account for 20%, and lipids account about 20% [4]. Though the average content of organic compounds in the sludge are lower than the developed countries, in some big cities like Beijing the it has been almost at the same level about 60%. Another attractive point is that sewage sludge is zero or even minus cost as raw material. If we can extract all of the lipids from the sewage sludge to make biodiesel, it can bring us huge economic benefits.

Besides the big amount of sewage sludge, it contains lots of hazardous substances such as pathogens, heavy metals, and even dioxins, which will be harmful to our health and environment [5-7]. This situation has made disposal problem of sludge becoming the focus of the environmental issues. Though there are lots of ways to dispose sludge such as sanitary landfill, incineration, anaerobic fermentation, compost and so on. But as to each method, there are shortcomings, for example, high operating costs of incineration, possible serious secondary pollution of landfill and incineration, abundant land use of landfill, anaerobic fermentation and compost, and others [5]. Because of some these reasons, recycling technology which is consistent to “sustainable development” strategy has been a welcomed trend to treat all waste [8]. Recycling sewage sludge to make biodiesel is one of the recycling technologies and extraction of lipids from sludge is one of the important steps to make biodiesel. We will discuss this research here.

According to the complex sources of sewage and the complicated waste water treatment technology, the lipids in sewage sludge should exist in several forms which would make the extraction not easy to operate. However lipids have a common characteristic that they are generally soluble in organic solvents but don't dissolve in water. And certainly more similar the polarity is, more easily soluble they are [9]. According to this feature, we can use organic solvents to extract lipids from the sludge. However, the organic solvents might not be simple or the extraction step might be a little complicated. Because lipids extraction technology from sludge has not been taken seriously, there is no national standard in lipids assessment for sewage sludge, and we decided to refer to the national standard of the food industry. In the literature review, we have found that some solvents such as ethanol, ethyl ether, petroleum ether, hexane, chloroform – methanol, bromo-propane, hexane – ethanol have been used for extraction lipids [10, 11]. Besides these solvent we will try more solvents such as dichloromethane, ether-petroleum ether and dichloromethane-methanol to extract lipids from sludge. We want to find out which solvent (single or mixed) will be better to extract the lipids from the sludge by measuring the quantity of the lipids.

2. Experiment Materials and Methods

2.1. Experimental materials:

Dewatered sludge from one Sewage Treatment Plant in Beijing. The moisture content of the sludge sample is $79\% \pm 0.5\%$. The experimental instruments we used to check the sludge component were listed in Table 1. And the typical element component of this kind of sewage sludge was shown in Table 2.

Table 1. Experimental instruments

Items	Instruments
Metallic element	Atomic Absorption Spectroscopy, HITACHI, Z-2000
TC	JENA multi N/C 2100S and YENA HT1300 Solid module
Cl-	Ion Chromatography, DIONEX, ICS-900

Table2. Sludge composition

Items	VSS(%)	TC(mg/g)	Cl(mg/g)	K(mg/g)	Fe(mg/g)	Cu(mg/g)	Zn(mg/g)	Mn(mg/g)	Ca(mg/g)	Pb(mg/g)
Contents	52.5	385.4	9.75	1.71	28.26	0.137	1.26	0.255	28.4	0.032

Reagents: Hydrochloric acid (36%~38%), Ethanol (95%), Ether, petroleum ether, acetone, hexane and other organic solvents

2.2. Experimental methods [12]:

We adopted the method of GB/T 5009.6-2003, the acid hydrolysis method for determination of fat in foods, but we tried different extraction solution.

Important steps in GB/T 5009.6-2003: (1). Make the solid sample grind it to a granular can, add 8ml water and 10ml hydrochloric acid (36%~38%). Put them into the tube and heat it in the water bath of 70~80°C for about 40~50 minutes; (2). Take out the tube, add 10ml ethanol (95%) into it, Transfer the solution to a 100ml beaker after cooling the mixture, and then filter the solution in the beaker to the erlenmeyer flask. Pour prepared 25ml ether and 5ml ether - petroleum ether into the tube to wash the lipids in it, then remove them to the flask together. Keep standing for 10 or 15 min, and then remove the supernatant to the flask after the upper liquid is clear. (3). Put the flask (constant weight) on thermostatic electric hot plate to evaporate to dryness for about 8 hours, the temperature will be kept at 105°C. At last, we put the flask on the electronic balance to measure the result.

However, we made two modifications of the above steps when we did this experiment. The first is that in the second step we transfer the mixture solution into a separating funnel than got the supernatant not by sucking the supernatant out. The second and the most important is that we replaced the original extraction solvent, ether, by other solvents such as petroleum ether, toluene, hexane, isopropyl, chloroform - methanol, ether - petroleum ether, hexane – ethanol.

3. Experimental Results and Discussion:

According to this method, we have tried to extract lipids from the sewage sludge by using various organic solvents with three kinds of sludge sampled in three different times. All experimental results are shown in Figure 1:

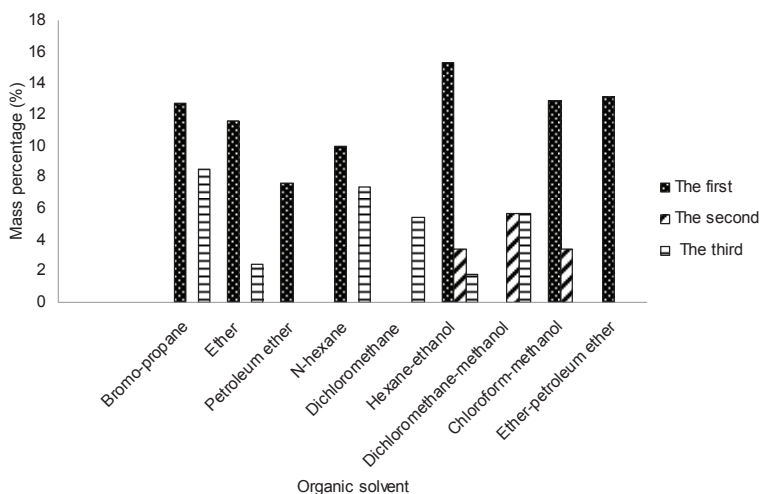


Fig.1. lipid extraction results

From the experiment results, it can be seen that the amount of extracted lipids depends on the solvent used to extract as to the same sludge sample. The amount of lipids we extracted from the sludge sampled in the first time are significantly higher than those samples in the second and the third, which can be possibly explained that the sludge taken for the first time has higher organic matter such as lipids than the other two times. But to each kind of sludge, the tendency is roughly the same: mixed solvent has higher efficiency than the single solvent and the polar solvents are easier to extract lipids than the non-polar solvents. We can explain it like that: there are many kinds of lipids (both polar and non-polar) in the sludge. In primary sludge, the lipids are mainly non-polar because they derived from glyceride in the water. As to the sludge from the secondary sedimentation tank, they have lots of microbial cells, the lipids in whose cell wall are main polar phospholipids. The polarity order of the solvent is that: Bromo-propane > Dichloromethane > Ether > N-hexane > Petroleum ether. In other words, polar lipids are very possibly more than non-polar in the sewage sludge.

4. Conclusions

Recycling sewage sludge to be raw material for making biodiesel is a relatively possible choice for big cities to solve the problems of sewage sludge, energy crisis and limited area. Referring to the national standard for food industry, we have done experiments to test the extraction of lipids from sewage sludge, which is the very important step for biodiesel making. We got the following conclusions.

As to the single solvent extraction, bromo-propane solvent has the highest extraction effect, then followed with ether, toluene, N-hexane, petroleum ether. While, as to the mixed solvent extraction, the hexane – ethanol solvent has higher efficiency than other mixed solvent such as ether – ethanol and chloroform – methanol. Compared with single solvent extraction, the mixed solvent has higher efficiency.

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