

# Preparation of Biodiesel with Acid Oil in Fixed Bed Reactor by Immobilized Lipase\*

CHEN Ying-ming<sup>1,2</sup>, CHANG Jie<sup>1</sup>, LU Peng-mei<sup>1</sup>, FU Yan<sup>1</sup>, WANG Tie-jun<sup>1</sup>, LU Ji-dong<sup>2</sup>, XIAO Bo<sup>3</sup>,

(1. Guangzhou Institute of Energy Conversion, Chinese Academy of Science, Guangzhou 510640, China;

2. School of Energy & Power, Huazhong University of Science and Technology, Wuhan 430074, China;

3. School of Environmental Science and Engineering, Huazhong University of Science and Technology, Wuhan 430074, China)

**Abstract:** The transesterification of acid oil and methanol to biodiesel catalyzed by immobilized *Candida* lipase in fixed bed reactors was studied. The acid oil and methanol were pumped into the reactors in three-steps which were kept the molar ratio as 1:1. The result of orthogonality experiment indicated that: the optimal conditions for transesterification of acid oil were as following: 15% immobilized lipase, 10% hexane and 10% water of acid oil, reaction temperature 45 °C, flow velocity of reactant 0.8 g · min<sup>-1</sup>. The content of fatty acid methyl ester of 90.18% could be obtained under the optimal conditions.

**Key words:** immobilized lipase; acid oil; biodiesel; fixed bed reactor

**CLC number:** S216 **Document code:** A **Article ID:** 0529-6579 (2007) S1-0192-02

Biodiesel (fatty acid short-chain alkyl ester), which is derived from triglycerides by transesterification with short-chain alcohol, has attracted more and more attention now as a renewable, biodegradable, and nontoxic fuel<sup>[1-2]</sup>. Biodiesel is industrially produced through alkaline catalysis, but this process is highly energy consuming and the reaction product constituted by a mixture is difficult to separate. Enzymatic transesterification using lipase can overcome the drawbacks and is the suitable method for the oil with high acid value. In order to purify easily and reduce the cost of lipase, the lipase is usually immobilized<sup>[3-5]</sup>. In this work, acid oil was used to synthesize fatty acid methyl ester with methanol by immobilized *Candida* lipase in fixed bed reactor.

## 1 Experimental

### 1.1 Materials

Acid oil; *Candida* lipase; methanol, n-hexane, petroleum ether, 85% phosphoric acid; activated clay; methyl salicylate, methyl palmitate, methyl stearate, methyl oleate, methyl linoleate, methyl linolenate (chromatographic grade); fixed bed reactor, magnetic stirring apparatus, GC-2010 gas chromatography, thermostat water bath, peristaltic pump.

### 1.2 Methods

1.2.1 Pretreatment After being filtrated, degumming and bleaching, the saponification value of 178.73 mg · g<sup>-1</sup> and the acid value of 109.18 mg · g<sup>-1</sup> were determined.

1.2.2 Transesterification of acid oil The immobilized lipase of which enzyme activity was 222.5 IU, was placed in the reactor column and separated by glass beads with the piled density of 0.283 g · cm<sup>-3</sup>. After being mixed even with magnetic stirring apparatus, a specified weight of mixtures of acid oil, methanol, solvent hexane and water were added into the top of reactors. The temperature in the reactor was kept constant through water bath. Three reactors are connected to form a three-steps reaction system of which the acid oil and methanol were kept the molar ratio as 1:1 and glycerol was separated in every step.

1.2.3 Analysis The content of fatty acid methyl ester in the product liquid was analyzed by a gas chromatography, hydrogen flame being the detectors and methyl salicylate being the internal standard. The operating conditions are: DB-1HT capillary column, temperature at sampling inlet: 370 °C, detector temperature: 375 °C, split ratio: 50, pre-column pressure: 100 kPa, carrier gas: N<sub>2</sub>.

\* 收稿日期: 2007-01-10

基金项目: 广州市科技计划资助项目(2004J1-C0101), 中国科学院知识创新工程资助项目(KGCX2-YW-306)

作者简介: 陈英明(1977年生), 男, 博士研究生; 通讯联系人: 常杰; E-mail: changjie@ms.giec.ac.cn

## 2 Results and discussion

Considering synthetically the affecting factors of the content of methyl ester, orthogonality experiment was adopted to seek the relationship between the factors and the content of methyl ester.

The experimental result showed that the effect of each factor on the content of methyl ester followed the sequence: enzyme amount > solvent amount > water amount > flowing velocity of reactant > temperature. 90.18% content of methyl ester can be got under the optimum conditions as following:  $m$  (lipase):

$m$  (hexane) :  $m$  (water) :  $m$  (oil) = 15 : 10 : 100, temperature 45°C, flowing velocity of reactant 0.8 g · min<sup>-1</sup>.

## 3 Conclusions

Acid oil can be synthesized to biodiesel in three - steps fixed bed reactors catalyzed by immobilized lipase efficiently.

## References:

- [1] GERHARD K, DUNN R O, BAGBY M O. Technical aspects of biodiesel standards [J]. INFORM, 1996, 7 (8): 827 - 829.
- [2] MIN E Z, TANG Z, DU Z X. Perspective of biodiesel industry in China [J]. Eng Sci, 2005, 17 (4): 1 - 4.
- [3] WATANABE Y, SHIMADA Y J, SUGIHARA A. Continuous production of biodiesel fuel from vegetable oil using immobilized *Candida Antarctica* lipase [J]. JAOCS, 2000, 7 (4): 355 - 358.
- [4] SAMUKAWA T, KAIEDA M, MATSUMOTO T. Pretreatment of immobilized *Candida Antarctica* lipase for biodiesel fuel production from plant oil [J]. Biosci Bioeng, 2000, 90 (2): 180 - 183.
- [5] MAMORU I, CHEN B X, EGUCHI M. Production of biodiesel fuel from triglycerides and alcohol using immobilized lipase [J]. J Mole Catal B: Enzymatic, 2001, 16 (1): 53 - 58.

# 酸化油固定床酶法合成生物柴油研究

陈英明<sup>1,2</sup>, 常杰<sup>1</sup>, 吕鹏梅<sup>1</sup>, 付严<sup>1</sup>, 王铁军<sup>1</sup>, 陆继东<sup>2</sup>, 肖波<sup>3</sup>

(1 华中科技大学能源与动力工程学院, 湖北武汉 4300741;

2 中国科学院广州能源研究所, 广东广州 510640;

3 华中科技大学环境科学与工程学院, 湖北武汉 430074)

**摘要:** 以固定化的假丝酵母酶为催化剂, 在三段式固定床反应器内, 醇油摩尔比为 1:1, 采用分级流加甲醇的方式, 将高酸值的酸化油转化为生物柴油, 探讨了酶量、溶剂量、水量、温度、反应液流速等与产物中甲酯含量的关系。正交实验结果表明, 反应的最适条件为酶用量、溶剂量、水量分别为油重的 15%、10%、10%, 反应液流速为 0.8 g · min<sup>-1</sup>, 温度为 45 °C, 在此条件下, 产物中甲酯含量达到了 90.18%。

**关键词:** 定化脂肪酶; 酸化油; 生物柴油; 固定床

**中图分类号:** S216