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Integrated flow reactor that combines high-shear mixing and microwave irradiation for biodiesel production

I. Choedkiatsakul ^{a,d}, K. Ngaosuwan ^b, S. Assabumrungrat ^a, S. Tabasso ^c, G. Cravotto ^{d,*}

^a Center of Excellence in Catalysis and Catalytic Reaction Engineering, Department of Chemical Engineering, Faculty of Engineering, Chulalongkorn University, Bangkok, 10330, Thailand

^b Department of Chemical Engineering, Faculty of Engineering, Rajamangala University of Technology Krungthep, Bangkok, 10120, Thailand

^c Department of Chemistry, University of Turin, Via P. Giuria 7, 10125, Turin, Italy

^d Dipartimento di Scienza e Tecnologia del Farmaco, and NIS – Centre for Nanostructured Interfaces and Surfaces, University of Turin, Via P. Giuria 9, 10125, Turin, Italy

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ABSTRACT

A new simple flow system which is made up of a multi-rotor high-shear mixer connected to a multimode microwave reactor has been assembled. This simple loop reactor has been successfully used in the NaOH-catalyzed transesterification of refined palm oil in methanol. Thanks to optimal mass/heat transfer, full conversion was achieved within 5 min (biodiesel yield of 99.80%). High-quality biodiesel was obtained that is in accordance with international specifications and analytical ASTM standards. The procedure's high efficiency and low energy consumption should pave the way for process scale up.

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1. Introduction

The world energy crisis has recently become a more crucial issue because energy demand outstrips energy supply. Alternative energy sources and fuels have therefore been investigated as a means to indemnify the higher demand for natural fuels, which require many years of formation. Over the last 40 years, several renewable energy sources have been developed. Biodiesel can play an important role here because its high

* Corresponding author. Fax: +39 011 6707687.

heating values are nearly equivalent to diesel fuels and it presents low environmental impact. It can be produced from many types of feedstock including waste cooking oil [1-3].

A concerned effort has been made to search out new highly efficient reactors that produce biodiesel as well as saving time and energy. Inefficient mass transfer is one of the main limitations in biphasic heterogeneous reactions, such as transesterification. Although many types of vigorous mixing have been investigated to address this requirement, most of them suffer from high energy demands [4–6]. Optimal mass/heat







E-mail address: giancarlo.cravotto@unito.it (G. Cravotto).

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transfer is doubtless the key to enhancing biodiesel production. Dielectric heating has been proved to dramatically enhance transesterification reaction because it provides the improved heat transfer over the conventional methods [7]. Microwave (MW) as a volumetric heating and the selective overheating of polar catalysts/reagents has been widely exploited [8,9]. Besides avoiding the wall effect, flow-MW reactors also reduce energy consumption because energy is directly transferred through the reaction mixture [10].

In conventional heating, heat transfer occurs by conduction and/or radiation. Moreover, the efficiency of heat transfer depends on material properties such as thermal conductivity, specific heat capacity and density, resulting in non-uniform heating in a reactor. However, MW irradiation can instantly interact with a sample matrix and does so via two mechanisms, dipolar rotation and ionic conduction. Dipolar rotation generates heat when sample dipoles try to align themselves after an oscillation in the electric field, while ionic conduction generates heat when the electric field direction is changed via friction at the molecular level and larger ions slow down. Both mechanisms lead to the localized superheating of material in a very short time [11,12].

As mentioned above, industry level scale up and energy consumption are necessary issues in economical biodiesel production. Moreover, most procedures use the conventional mechanical stirring which requires high energy consumption, before applied the MW irradiation.

Therefore, we herein report a more efficient set up which uses the combination of two commercial reactors: a High-Shear Mixer (HSM, Magic-Lab – IKA) and a multimode MW reactor (MicroSynth, Milestone). Such a combination dramatically enhances both mass and heat transfer during the NaOH-catalyzed transesterification of refined palm oil and methanol. Energy consumption for biodiesel production and the analytical properties of biodiesel produced by each single system and their combinations are treated as the main criteria in process optimization.

2. Experimental method

2.1. Equipment

The HSM (Magic Lab, by IKA Germany) is a new modular laboratory system which is especially designed for mixing, dispersing, wet milling and incorporation of powders into liquids. It can produce homogeneity and stability in emulsions and suspensions using its three rotors, high-shear dispersing module. The system configuration is illustrated in Fig. 1(a). There are three rotors that differ according to orifice shape and slot width, as shown in Fig. 1(b), which are used to provide varying dispersion levels. The rotors are arranged in series including coarse, medium and fine, respectively for optimization of performance and functionality. The configuration also includes a cooling system which disperses heat, generated by the rotors and the operating unit, and controls the operating parameters such as temperature, rotor speed and rotation time.

MW irradiation was performed in a MicroSynth MW oven (by Milestone, Italy), with a maximum power setting of 800 W

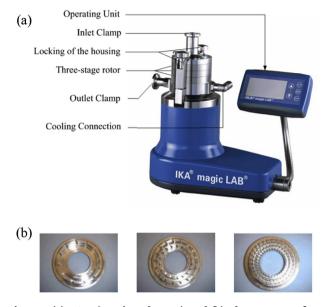


Fig. 1 - (a) HSM (Magic-Lab, IKA) and (b) Three types of rotor; coarse, medium and fine rotor, respectively.

and equipped with an IR pyrometer. The MW cavity is $35 \times 37 \times 35$ cm³. The precise reaction monitoring and parameters have been controlled by the software interface.

2.2. Chemicals

Commercial refined palm oil was kindly provided by Embouteille Company, Italy. Methanol reactant, of 99.9% purity, and methyl heptadecanoate, used as an internal standard for GC analysis, were purchased from Sigma–Aldrich. The NaOH catalyst, of 97.5% purity, and the heptane solvent, of 99.7% purity, were obtained from Carlo Erba Reagenti.

2.3. Experimental setup

The experimental set up was divided into 3 systems as follows:

<u>System 1:</u> A combination of the HSM (Magic Lab) and the MW reactor (HSM + MW). This combination is shown in Fig. 2. The catalyst was first mixed with methanol and then, all

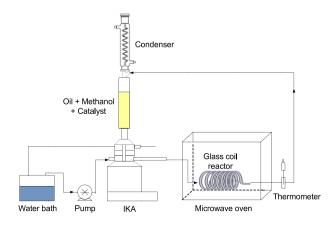


Fig. 2 – System 1: a combination of HSM and MW.

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