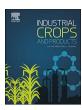
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Magnetic and reusable MgO/MgFe₂O₄ nanocatalyst for biodiesel production from sunflower oil: Influence of fuel ratio in combustion synthesis on catalytic properties and performance



Shervin Alaei^{a,b}, Mohammad Haghighi^{a,b,*}, Javad Toghiani^{a,b}, Behgam Rahmani Vahid^{a,b}

- ^a Chemical Engineering Faculty, Sahand University of Technology, P.O. Box 51335-1996, Sahand New Town, Tabriz, Iran
- ^b Reactor and Catalysis Research Center (RCRC), Sahand University of Technology, P.O. Box 51335-1996, Sahand New Town, Tabriz, Iran¹

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ABSTRACT

The successful synthesis of magnetic MgO/MgFe₂O₄ nanocatalyst via combustion method was achieved that has far less time and cost than the other methods. This catalyst was used in biodiesel production reaction from vegetable oil. By changing the fuel to nitrates ratio, proper structure of catalyst was obtained to produce biodiesel. Physiochemical analysis including XRD, FESEM, EDX dot-mapping, BET-BJH, FTIR and Surface Particle Size Distribution (SPSD) were used to define the characteristics of synthesized catalysts and optimum fuel ratio in combustion synthesis method. To evaluate the performance of the synthesized nanoceramics, all samples were used in the biodiesel production reaction. The results of XRD analyses showed the successful synthesis of MgFe₂O₄ crystals and also determined that other materials peaks (iron oxide phases) does not exist in the catalyst structure. BET-BJH analyses reveal the structures with large pore (more than 10 nm) and relatively good surface area (97.8 m²/g) for synthesized catalysts by combustion method. By biodiesel production reaction in the conditions of temperature = 110 °C, methanol-to-oil molar ratio = 12, catalyst concentration = 4 wt.% and reaction time = 4 h, it was found that the catalyst has great potential to produce biodiesel and also maximum conversion of 91.2% was obtained. For the stability test of catalyst, after convenient catalyst separation by magnet, it was used for five consecutive transesterification reactions that the results were very acceptable and finally the lowest conversion was achieved to be 82.4%. By the simultaneous consideration of analyses and reactor tests results, it was found that synthesized catalyst with fuel ratio of 1.5 has the best performance and it is very suitable for biodiesel production reaction.

1. Introduction

Due to global concerns about the increase of pollutants in the atmosphere and limitations of fossil resources, the search for other fuels has gained much assiduity (Erenturk and Korkut, 2018; Korkut and Bayramoglu, 2018; Salinas et al., 2018). Fatty Acid Methyl Esters (FAME) which is also called biodiesel is one of the renewable, nontoxic and biodegradable fuels (Guldhe et al., 2017a; Kaur et al., 2018; Mardhiah et al., 2017). Biodiesel can be obtained by a transesterification reaction of triglycerides with methanol by using a catalyst (Dehghani and Haghighi, 2017; Guldhe et al., 2017b; Kirubakaran et al., 2018). Due to some problems such as soap formation, waste water treatment and difficulty of recycle catalysts; researchers have focused on heterogeneous catalysts instead of homogeneous catalysts for transesterification reaction of triglycerides (Abdullah et al., 2017;

Kumar et al., 2015; Mansir et al., 2018).

Heterogeneous acid catalysts such as WO_3/ZrO_2 (Sania et al., 2014) and ZnO/SiO_2 (Corro et al., 2014) have lower catalytic activity than solid base catalysts in transesterification reaction, thus they can be used for feedstocks with large amount of FFAs in esterification reaction (Peng et al., 2008). Alternatively, solid base catalysts such as $CaO-ZrO_2$ (Xia et al., 2014), $MgO/MgAl_2O_4$ (Rahmani Vahid and Haghighi, 2016, 2017) and MgCaAl (Guzmán-Vargas et al., 2015) can be used in transesterification reaction due to its high activity, low cost and reutilization (Sandesh et al., 2016; Xian-fa et al., 2013).

Spinel ferrites of the form XFe_2O_4 , where X is the divalent metal cation imbibe researchers attention due to their unique and tunable magnetic, electronic and structural properties (Azam et al., 2015; Manikandan et al., 2014). Among various spinel ferrites, $MgFe_2O_4$ nanoparticles have drawn much attention because of its chemical,

^{*} Corresponding author at: Reactor and Catalysis Research Center (RCRC), Sahand University of Technology, P.O. Box 51335-1996, Sahand New Town, Tabriz, Iran. E-mail address: haghighi@sut.ac.ir (M. Haghighi).

¹ http://rcrc.sut.ac.ir.

reproducible (Manikandan et al., 2014) and magnetic properties (Meng et al., 2004). Due to these special properties, it has miscellaneous applications such as magnetic technologies (Manikandan et al., 2014) and heterogeneous catalysts (Ilhan et al., 2015).

In recent years, self-propagating high-temperature synthesis (SHS) or combustion synthesis (CS) method has appeared as an effective, low-cost and less time consuming method for production of various materials (Aruna and Mukasyan, 2008; Baneshi et al., 2014; Naik et al., 2007). This methodology is composed of metal nitrate and fuel (e.g. glycine, urea, etc.) which are dissolving in water and heated over a hot plate to evaporate the water and combustion synthesis reaction take place and finally the powder formed (Groven et al., 2013; Jeong et al., 2016; Kumar et al., 2015).

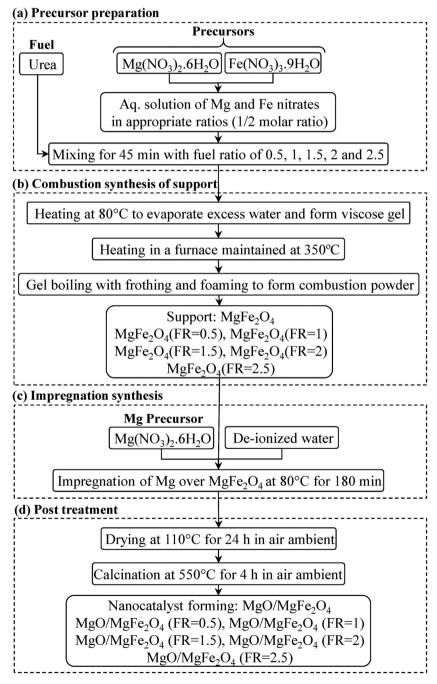
In this study, MgFe₂O₄, which is a magnetic spinel, is synthesized by combustion method with the various fuel ratios and MgO was loaded on

 $MgFe_2O_4$ as active phase by impregnation method. The synthesized $MgO/MgFe_2O_4$ as heterogeneous catalysts were further tested in transesterification reaction. Effects of the fuel ratio on combustion method were investigated to select best catalyst for transesterification reaction.

2. Materials and methods

2.1. Materials

For the synthesis of nanostructured MgO/MgFe $_2$ O $_4$ catalysts using combustion method, magnesium nitrate (Mg(NO $_3$) $_2$:6H $_2$ O; Merck, 99%), iron (III) nitrate nonahydrate (Fe(NO $_3$) $_3$:9H2O; Merck, 99%) and urea (NH $_2$ CONH $_2$; Romil, 99.5%) were used without any purification. The sunflower oil which was purchased from market (With acid



 $\textbf{Fig. 1.} \ Combustion \ synthesis \ of \ magnetic \ MgO/MgFe_2O_4 \ nanocatalyst \ with \ various \ fuel \ ratios.$

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