

Production of Magnesium Oxides from Raw Salt Solution Using Electrochemical Precipitation Method as a Heterogeneous Catalyst for Transesterification of Coconut Oil

KAMISAH D. PANDIANGAN*, WASINTON SIMANJUNTAK, RADEN SUPRIYANTO, ILIM ILIM, PONCO PRASETYO, SUTOPO HADI

Department of Chemistry, University of Lampung, Bandar Lampung, 35145, Indonesia

Abstract: *This study was conducted to explore the production of magnesium oxide from raw salt solution using electrochemical precipitation, followed by calcination. Electrochemical precipitation was conducted by electrolysis of the salt solution using nickel rods as cathodes and graphite as anodes. Two sets of salt solutions were prepared: one set without pretreatment and another with BaCl₂ pretreatment. The solutions were used to study the effect of salt concentration, potential, and electrolysis time. The representatives of the MgO produced were tested as catalysts for transesterification of coconut oil. The results indicate that optimum mass of precipitate was produced from 400 mg/L salt solution electrolyzed using 8 V for 60 min. Elemental analysis using X-Ray Fluorescence (XRF) revealed the presence of Mg as the main component of the precipitate, confirming the electrochemical conversion of Mg²⁺ into solid Mg(OH)₂. The MgO with the purity of 74.23% and 88.87% was produced from non-pretreated and pretreated salt solution, respectively. The transesterification experiments indicate that the yield of 90% and 98% was achieved using the MgO produced from non-pretreated and pretreated salt solution, respectively.*

Keywords: *coconut oil, electrolysis, MgO, raw salt, transesterification*

1. Introduction

Vegetable oils are highly important natural products obtained from many types of plants. Typically, vegetable oils are used in cooking. However, currently, they are known as functional compounds as they can be used in the production of petrochemicals and clean hydrocarbon fuels [1] and as a lubricant [2], agglomerant [3], nanocarrier for bioactive compounds in health supplements [4], and biodiesel [5-8]. Biodiesel is a renewable energy source produced through transesterification reaction, in which simple alcohol, mostly methanol, acts on vegetable oils, resulting in the synthesis of fatty acid methyl esters (FAMES). Biodiesel is often blended with petrochemical diesel and used as a liquid fuel in transportation and other industries [6-10]. As such, biodiesel is highly essential to reduce energy dependence on fossil fuels, considering the undeniable depletion of fossil energy reserves and accelerating increase in energy demand [6-8, 10].

Being a non-spontaneous and reversible reaction, transesterification proceeds only in the presence of a catalyst. In practice, chemical catalysts, either acids or bases, and enzymes [6, 11, 12] are used for this purpose, but chemical catalysts are highly preferred due to their higher activity and lower cost compared to enzymes. To manipulate transesterification, the reaction is commonly run in a non-stoichiometric ratio of the reactants, in which alcohol is used in excess to push the reaction toward the product.

Considering the crucial role of a catalyst in biodiesel production, most studies are focusing on developing heterogeneous catalysts as substitutes for homogeneous catalysts. Strong mineral acids, such as nitric acid and sulfuric acid, and strong bases soluble in alcohol, such as sodium hydroxide and potassium hydroxide, are examples of homogeneous catalysts. These catalysts have high catalytic activity, allowing for the reaction to proceed at a high rate and produce high yield, causing complete conversion of the treated oils or fats [9, 13].

*email: kamisah.delilawati@fmipa.unila.ac.id

However, the resulting biodiesel needs to be washed with a large quantity of water to remove the dissolved catalyst. This additional step contributes to not only treatment cost but also environmental pollution due to the corrosive nature of the strong acid or base. In addition, reuse of homogeneous catalyst is typically not possible.

Heterogeneous catalysts, on the contrary, are solid catalysts that remain unaltered during the reaction and, therefore, can be separated from the biodiesel by using a simple filtration process. Moreover, heterogeneous catalysts can be reactivated and reused, thus reducing treatment cost associated with catalyst and reducing or even eliminating environmental concerns originating from catalyst disposal [5, 9, 14]. Considering these advantages, many types of heterogeneous catalysts have been developed. Among them, but not limited to, are alkaline earth oxides and several alkaline metal compounds supported on alumina, silica, or zeolite [14-18]. MgO is a potential heterogeneous catalyst belonging to alkaline earth metal oxides. This oxide possesses strong alkaline (basic) characteristic, making it a suitable replacement of a homogeneous base catalyst. Moreover, it can be used as a single catalyst or as an active dopant in various supported catalyst systems, without losing its catalytic activity [14, 19].

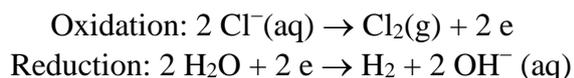
In addition to its use as a catalyst, many other applications of MgO are well known. Therefore, it is produced using various processes, such as thermal decomposition of different magnesium salts or magnesium hydroxide [19], hydrothermal process using magnesium nitrate solution [20], wet chemical method using magnesium nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent [21], aerogel procedure using a mixture of toluene and magnesium methoxide solution in methanol [22], sol-gel method using a magnesium alkoxide $Mg(OR)_2$ in an alcohol to produce hydroxide, which is followed by hydrolysis, condensation, and polymerization reactions and thermal dehydration for conversion of hydroxide to oxide (MgO) [23], and electrochemical deposition processes of magnesium oxide/hydroxide on glassy carbon (GC) electrode from magnesium nitrate hexahydrate melt [24].

In addition to the use of pure chemicals as a precursor, the use of seawater and bittern has been reported by others. In the context of MgO production, seawater is an attractive raw material as this unlimited natural resource contains Mg^{2+} at 1265 [25], 1290 [26], or 1600 ppm [27] and is the second most abundant cation after Na^+ . A previous study reported the production of MgO from seawater and bittern by using an electrochemical method [28]. The purity of MgO produced from seawater and bittern demonstrated purity in the range of 77.33%–92.65% and 85.72%–91.21%, respectively. In another study, recovery of MgO from bittern by decarboxylation/precipitation method using NaOH and $Ca(OH)_2$ as a precipitant was conducted [29]. They reported that the MgO with the content in the range of 77.9%–99.4% was achieved, depending on the experimental conditions applied.

During salt production, seawater is sun dried to evaporate the water and eventually produce the solid product that is commonly known as raw salt. In addition to NaCl as a main component of raw salt, other non-volatile components contained in seawater simultaneously accumulate in the salt. Therefore, raw salt is also enriched in magnesium, making it a more promising raw material for MgO production compared to seawater. However, the feasibility of using raw salt as an alternative raw material remains unexplored. In this regard, the present study is still a new initiative in exploring a prospective raw material for MgO production.

In this study, we explored the production of MgO from raw salt through a two-step process. In the first step, raw salt was dissolved in distilled water, then the solution was subjected to electrolysis using two nickel rods as cathodes and two graphite rods as anodes. Two types of solutions were used, i.e., raw salt solution without $BaCl_2$ pretreatment and that with $BaCl_2$ pretreatment. Pretreatment with $BaCl_2$ was intended to precipitate SO_4^{2-} , which is not easily oxidized electrochemically and, therefore, adsorbed in the electrolysis product. This feature of SO_4^{2-} ionic species is in contrast to Cl^- , which is converted to Cl_2 gas by electrochemical oxidation, which evaporates from the solution.

Precipitation of $Mg(OH)_2$ during the electrolysis process of Mg^{2+} -containing solution is based on the following main redox reactions:



The OH^{-} ions produced by electrochemical reduction of water, as can be seen in the aforementioned redox reactions, increases the alkalinity of solution and leads to the formation of $\text{Mg}(\text{OH})_2$ precipitate as a result of the reaction between Mg^{2+} and OH^{-} in the solution. Electrochemical precipitation applied in this study is an alternative to most commonly used methods for recovery of metal ions from water, such as ion exchange [30], membrane filtration [31], electrochemical process [32], and chemisorption using soybean waste [33]. Compared to other methods, the main advantages of electrochemical methods are ease of application, greater purity of the substance precipitated, and cost effectiveness, although the limitation of mass transfer should also be recognized.

Concerning electrochemical precipitation of $\text{Mg}(\text{OH})_2$, the main goal of this investigation was to study the effect of concentration of salt solution, potential, and electrolysis time on the mass and elemental composition of the precipitate, which reflects the purity of MgO , produced from untreated and BaCl_2 -pretreated salt solution. We focused on the above electrochemical variables for their supposed significant roles in electrolysis [24, 28]. The catalytic activity of the MgO produced was then tested in transesterification of coconut oil to produce biodiesel. Coconut oil was chosen because it is largely available in Indonesia and its use as cooking oil has been mostly replaced by palm oil. Therefore, the use of this oil for energy will not compete with food and help preserve the crop as a multifunctional natural resource. Lauric acid contained in coconut oil is converted into methyl laurate, which has a distinctive aroma, which can be used as a qualitative indication of the reaction and to know if the catalyst used possesses sufficient activity to drive the reaction.

2. Materials and methods

2.1. Materials and instruments

$\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ was purchased from Aldrich, and methanol from Merck. Raw salt, nickel rods, graphite rods, and coconut oil were obtained from several suppliers in Indonesia. Nabertherm electrical furnace (Lilienthal, Germany) was used for calcination treatment, *X-Ray Fluorescence* (XRF) PAN Analytical Epsilon 3 was used for elemental analysis. The transesterification experiment was conducted on a laboratory scale transesterification unit, and GC-MS-QP2010 SE from Shimadzu was used for biodiesel analysis.

2.2. Preparation of salt solution

Raw salt that was previously oven dried at 115°C for 6 h was used to prepare a series of raw salt solutions in distilled water with concentrations ranging from 200 to 500 g/L. We tried to make a salt solution of 600 g/L, but the salt did not completely dissolve. Therefore, this solution was not considered further. Each of the solutions was filtered using a filter paper to remove insoluble impurities in the salt, and then divided into two parts. The first part was used without pretreatment and the second part was pretreated by dropwise addition of saturated BaCl_2 solution (g/L) until there was no precipitate, implying the formation of BaSO_4 . The precipitate was then separated by filtration.

2.3. Electrolysis

The electrochemical process was performed in a two-compartment electrochemical cell made of glass, using two pairs of electrodes with two nickel rods as the cathode and two graphite rods as the anode, schematically as shown in Figure 1. The two compartments were connected using a salt bridge constructed from a glass u-tube filled with NaCl suspended in agar gel. For the experiment, each compartment of the electrochemical cell was filled with 250 mL of salt solution, and the electrolysis was conducted at specified conditions, to study the effect of salt concentration, potential, and electrolysis time.

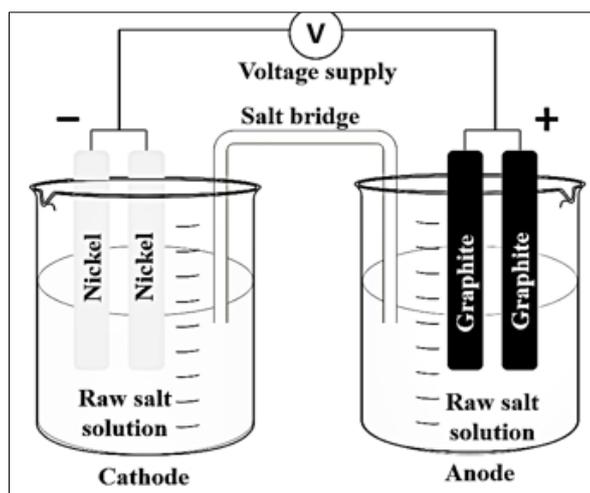


Figure 1. Schematic arrangement of electrochemical set-up used in the study

2.4. Transesterification

Two transesterification experiments were conducted to study the catalytic activity of two MgO samples, one sample produced from salt solution without pretreatment and another one from BaCl₂-pretreated salt solution. The reaction mixture comprised 20 mL coconut oil, 80 mL methanol, and 2 g MgO. The mixture was transferred into 200 mL two-neck round-bottom flask for connecting the flask with water condenser and placing a thermometer to measure the reaction temperature. A magnetic bar was also placed inside the flask, and then the assembly was placed inside a water bath placed on top of a heater with a magnetic stirrer. The reaction was run at 70°C for 3 h, and then the mixture was transferred into a separatory funnel for separation of biodiesel from unreacted oil. The biodiesel part was heated slowly to evaporate the unused methanol. The volume of biodiesel and the volume of unreacted oil were measured to determine the percentage of conversion of the oil. Finally, biodiesel was analyzed using GC-MS.

3. Results and discussions

3.1. Elemental composition of raw salt

Before use, the raw salt was analyzed using the XRF technique to determine the elemental composition of the salt. In addition to obtaining information on the chemical composition of the sample, the analysis was performed with a particular interest in Mg content. The results presented five of the most abundant elements found in the sample, which are listed in Table 1.

Table 1. Elemental composition of raw salt investigated in the present study

Element	Content (%)
Na	17.211
Mg	4.197
S	2.277
Ca	1.481
K	1.247
Cl	71.548
Br	0.127
Fe	0.102
Ba	1.065
Si	0.369
P	0.324
Others	0.052

Note: others refer to elements (components) with the quantity in ppm level

As can be seen in Table 1, the raw salt used in this study contains 4.197% mass of Mg on a dry base, or 4.197 g/100 g of raw salt. If it is related to the concentration of Mg in seawater, this value is around 1378 ppm, which is an average value of the findings reported in the literature [25-27]. The Mg content in 100 g of raw salt is equivalent to that in 3.045 L of seawater. In the context of MgO production, this is a highly interesting comparison as 100 g of raw salt can be dissolved in 1 L of water to give a solution with about 4.2 g of Mg, which is more than 30 times than the Mg content in 1 L of seawater.

3.2. Electrochemical precipitation

The first electrochemical experiments were conducted to study the effect of potential. The raw salt solution (500 g/L) was subjected to electrolysis for 60 min at different potentials of 4, 5, 6, 7, and 8 V. After the process was complete, the precipitate in cathodic compartment was collected by filtration and rinsed slowly with 200 mL of distilled water. The precipitate was oven dried at 115°C for 6 h, and then weighed. The results are presented in Figure 2.

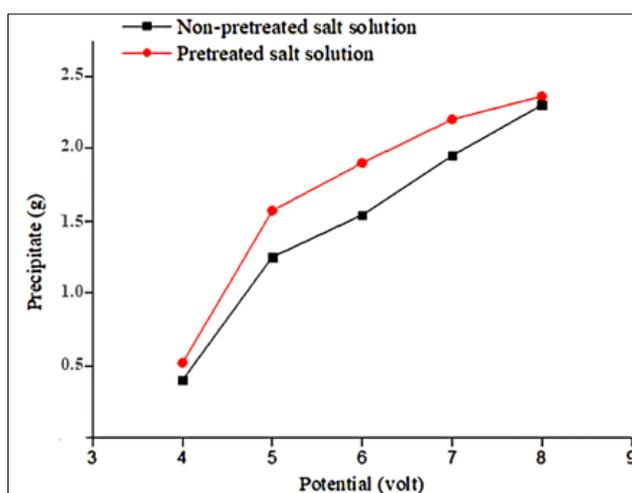


Figure 2. The experimental results showing the effect of potentials on the mass of precipitate

As indicated in Figure 2, the two treated samples exhibit the same trend, in which progressive increase in the mass of precipitate was observed following an increase in potential applied, which is in agreement with general features of an electrochemical process [34, 35]. It is also observed that the mass of the precipitate obtained from the BaCl₂-pretreated sample is higher than that of the precipitate obtained from the sample without pretreatment, suggesting that removal of SO₄²⁻ is beneficial to a certain extent. The mass of precipitate increased from 0.40 to 2.30 g and from 0.52 g to 2.36 g for non-pretreated and pretreated samples, respectively.

The second set of experiments was performed to study the effect of electrolysis time. The experiments were conducted at 15, 30, and 45 min. The experiments were run using the solutions with the same concentration as previously used and at fixed potential of 8 V. The experimental results of 60-min electrolysis are presented in Figure 3.

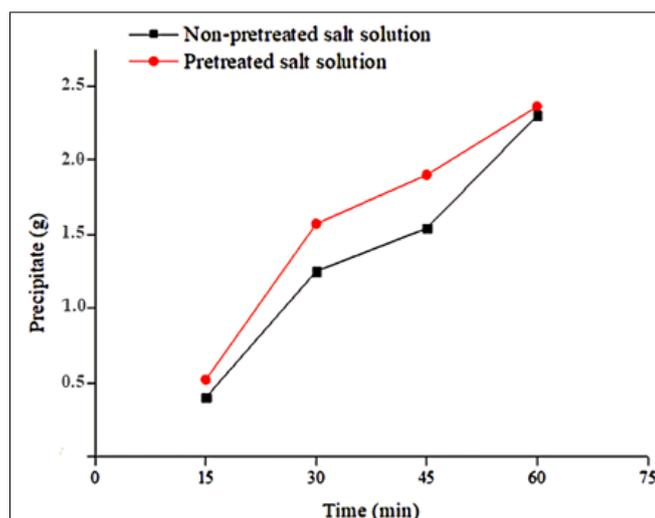


Figure 3. Experimental results showing the effect of electrolysis time on the mass of precipitate

As can be observed, the trends of experimental results presented in Figure 3 are very similar to those observed for the results of experiments at different potentials shown in Figure 1. A continuous increase in the mass of precipitate was observed up to 60 min, which is the longest electrolysis time applied. For the experiments using non-pretreated solution, the mass of precipitate increased from 0.40 to 2.31 g, whereas for those using the pretreated sample, an increase from 0.52 to 2.37 g was observed.

Figure 4 presents the results of experiments that investigated the effect of salt concentrations. The experiments were conducted using various concentrations of salt solution at 200, 300, and 400 mg/L, potential of 8 V, and electrolysis time of 60 min. The results of the previous experiment with salt concentration of 500 mg/L, potential of 8 V, and electrolysis time of 60 min are also included in the figure.

As can be seen in Figure 4, the mass of precipitate produced increases up to the salt concentration of 400 g/L (2.30 g for non-pretreated and 2.56 g for pretreated samples), followed by decrease with the use of solution with concentration of 500 mg/L. The decrease in the mass of precipitate with the use of salt solution with concentration of 500 g/L is most likely due to increased viscosity of the solution, resulting in a decrease in diffusion of ions to be deposited in the cathode. Such phenomena have been reported by others [36, 37] during electrowinning of copper powder which reported its effect on the morphology of deposits of Zn.

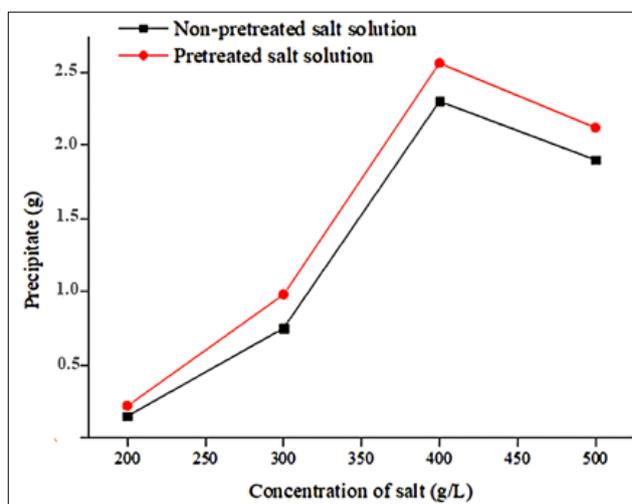


Figure 4. The experimental results showing the effect of salt concentration on mass of the precipitate produced

From the three sets of experiments conducted, the largest amount of precipitate was produced from the experiments using salt solution with the concentration of 400 g/L, potential of 8 V, and electrolysis time of 60 min. The precipitates obtained from these particular experimental conditions were then analyzed using XRF techniques, and the results are presented in Table 2.

Table 2. Elemental composition of precipitates obtained from non-pretreated and pretreated salt solution

Element	Content (%)	
	Non-pretreated salt solution	Pretreated salt solution
Mg	38.420	45.851
S	1.509	1.108
Cl	34.930	35.411
K	0.614	0.032
Ca	12.870	4.702
Br	0.063	6.619
Fe	0.537	0.171
Ba	0.224	1.137
Si	0.492	0.407
P	0.932	0.969
Ni	0.444	0.051
Mn	0.731	0.284
Al	0.788	3.013
Ag	1.025	0.039
Pd	5.135	0.000
Cu	0.795	0.176
Others	0.491	0.03

As can be seen in Table 2, the elemental composition of both samples is marked by the emergence of Mg as the main constituent, which implies that electrochemical precipitation of Mg^{2+} in the form of $Mg(OH)_2$ was achieved as expected. It is also obvious in the results that pretreatment of salt solution using $BaCl_2$ was useful to increase the Mg content significantly, and accompanied by decrease in S, K, and Ca content. With respect to this compositional change, the results obtained demonstrated the possibility that $BaSO_4$ precipitate produced by pretreatment of the salt solution with $BaCl_2$ also functions to entrap K and Ca effectively. For Cl, no significant change in the content was observed, most likely because Cl^- ions are known as a highly electroactive species and, therefore, these ions were anodically oxidized into gaseous Cl_2 during the electrolysis.

Both precipitates were then subjected to calcination at $700^\circ C$ for 5 h. This thermal treatment was carried out primarily to convert $Mg(OH)_2$ into MgO [19, 38], and at the same time to activate MgO as a catalyst [15]. The calcined precipitates were then characterized using the XRF technique and the results are presented in Table 3.

Table 3. Elemental composition of calcined precipitates obtained from non-pretreated and pretreated salt solution

Element	Content precipitate from salt solution (%)	
	Non-pretreated	Pretreated
Mg	74.228	88.870
S	1.211	0.474
Cl	4.992	5.014
K	0.149	0.036
Ca	10.625	2.890
Br	2.556	0.001
Fe	0.041	0.107
Ba	1.285	1.065
Si	1.216	0.369
P	0.399	0.324
Ni	0.303	0.034
Mn	0.051	0.180
Al	2.752	0.360
Others	0.0192	0.276

By comparing the results obtained for uncalcined samples (Table 2) with those obtained for calcined samples (Table 3), the calcination resulted in a highly significant change in composition of

the samples. Calcination treatment sharply increased the Mg content, suggesting the conversion of $\text{Mg}(\text{OH})_2$ into MgO by releasing the water molecules. A sharp decrease in Cl content was also observed, which implies that calcination treatment also removed this element from the samples, most probably in the form of gaseous Cl_2 . The existence of Mg as the most abundant component is also a strong indication of the existence of MgO as the main constituent of the samples and, therefore, for simplification, the two precipitates were termed MgO .

3.3. Transesterification

A simple approach to evaluate the activity of the catalyst is to calculate the conversion of the vegetable oil into biodiesel by using the equation (1) [39]:

$$\%conversion = \frac{V_i - V_u}{V_i} \times 100 \quad (1)$$

where V_i = initial volume of oil (mL)

V_u = volume of unreacted (residual) oil (mL)

Using the above equation, we found that 90% conversion was achieved using the MgO produced from the salt solution without pretreatment and 98% using the MgO produced from pretreated salt solution. These achievements indicate that both catalysts can be considered as having high catalytic activity and comparable with more advanced catalysts.

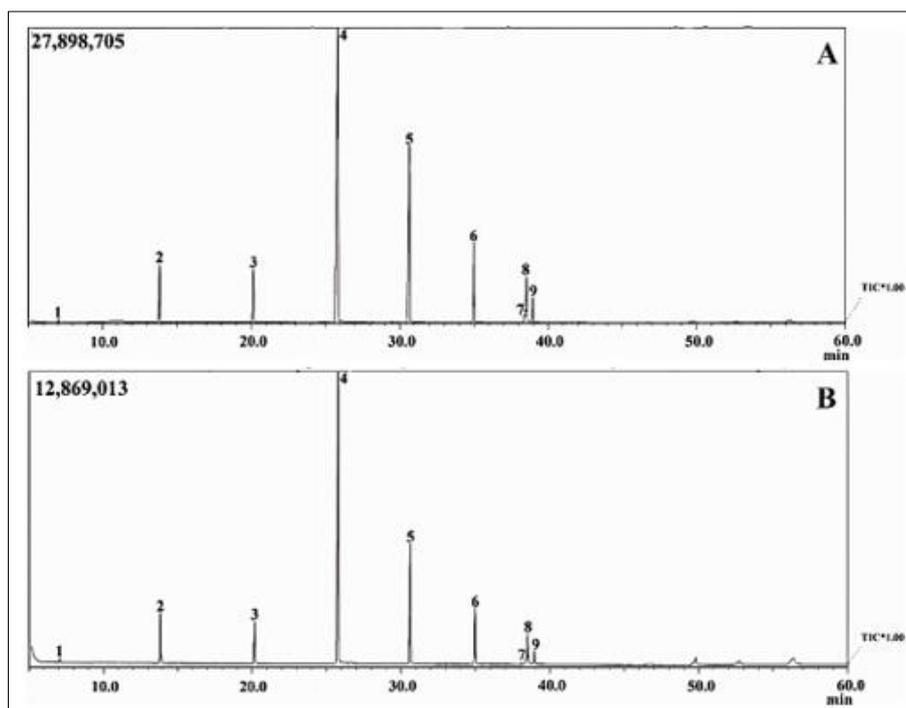


Figure 5. GC chromatogram of coconut oil transesterification product using MgO derived from non-pretreated salt solution (A), and MgO derived from pretreated salt solution (B)

Transesterification products were then analyzed by GC-MS, producing GC chromatograms as presented in Figure 5. As demonstrated by the results in Figure 5, the two chromatograms have the same patterns and each contains nine peaks, indicating that the two samples are composed of the same compounds. The components of the samples were then identified using software MS Library System NIST62, Wiley229 database, by comparing the MS spectra of the samples to those of standard compounds in the library system. The relative amount (relative percentage) of each component was



estimated by dividing the peak area of the component with the total peak area of all components identified. The results are compiled in Table 4.

Table 4. The components of coconut oil transesterification products using MgO produced as a catalyst

Peak number	Compound name	Molecular formula	Relative percentage (%)	
			Sample A	Sample B
1	Methyl caproate	C ₇ H ₁₄ O ₂	0.23	0.53
2	Methyl caprylate	C ₉ H ₁₈ O ₂	4.81	6.68
3	Methyl caprate	C ₁₁ H ₂₂ O ₂	4.54	5.54
4	Methyl laurate	C ₁₃ H ₂₆ O ₂	54.06	53.07
5	Methyl myristate	C ₁₅ H ₃₀ O ₂	20.33	19.00
6	Methyl palmitate	C ₁₇ H ₃₄ O ₂	7.90	8.10
7	Methyl linoleate	C ₁₉ H ₃₄ O ₂	0.81	0.87
8	Methyl oleate	C ₁₉ H ₃₆ O ₂	5.24	4.34
9	Methyl stearate	C ₁₉ H ₃₈ O ₂	2.08	1.85

In Table 4, sample A refers to transesterification product using MgO produced from salt solution without pretreatment and sample B refers to the product of the experiment using MgO derived from pretreated salt solution as a catalyst. As can be seen in Table 4, all components of the two products are FAMES, confirming the potential of the MgO produced in this study as a competitive catalyst in biodiesel production. Considering the very low price of raw salt and appreciating the simplicity of electrochemical method, it is important to acknowledge that raw salt is a highly prospective alternative raw material for MgO production.

4. Conclusions

The present investigation revealed the prospective potential of raw salt as an alternative source of MgO, and the opportunity of application of electrochemical precipitation process as production method. The results indicate that optimum mass of precipitate was produced from salt solution with concentration of 400 mg/L electrolyzed using a potential of 8 V for 60 min. The experimental results demonstrated that MgO with purities of 74.23% and 88.87% was produced from salt solution without and with BaCl₂ pretreatment, respectively. Another important finding of the present study that should be mentioned is high catalytic activity of the MgO produced in transesterification reaction, with yield of 90% using the MgO produced from salt solution without pretreatment and 98% using MgO produced from salt solution with pretreatment. These yields reflect the potential of the MgO produced as an alternative to the more advanced and expensive catalysts.

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