

## Electrochemical Process Investigation of Synthesizing Magnesium Oxide from Bittern Solution for Solid Oxide Fuel Cells Sealant

Sulistyo<sup>1\*</sup>, Slamet Saefudin<sup>2</sup>, Sulardjaka<sup>1</sup>, Reza Abdu Rahman<sup>1,3</sup>

<sup>1</sup> Department of Mechanical Engineering, Universitas Diponegoro, Semarang 50275, Indonesia

<sup>2</sup> Department of Mechanical Engineering, Universitas Muhammadiyah Semarang, Semarang 50275, Indonesia

<sup>3</sup> Department of Mechanical Engineering, Faculty of Engineering, Universitas Pancasila, DKI Jakarta 12640, Indonesia

\* Corresponding author's e-mail: [sulistyo@lecturer.undip.ac.id](mailto:sulistyo@lecturer.undip.ac.id)

### ABSTRACT

Magnesium oxide (MgO) is an essential material for producing solid oxide fuel cells (SOFC) sealant. It can be derived from bittern waste. The common approach uses membrane electrolysis, which requires complex equipment and high energy costs. Alternatively, direct electrolysis can be taken using proper parameters to maximize the production rate. This work analyzes the process according to the input voltage, which varies between 10 and 16 V. The designed working voltage is suitable for direct conversion from renewable sources such as photovoltaic. The evaluation shows that the working voltage notably affects the reaction rate of the bittern solution. The working voltage of 16 V has the lowest power factor (2.58), while the working voltage of 10 V indicates the highest power factor of 3.56. It makes the reaction rate for the working voltage of 10 V extremely low, causing the lowest production rate of MgO with only 4.27 g. Oppositely, the suitable working voltage improves the production of MgO up to 75%. Microscope evaluation indicates that the produced MgO from the process has a lower agglomeration concentration after heat treatment at 700 °C, which is desirable to ensure effective fuel transfer in fuel cell apparatus.

**Keywords:** brine, bittern, electrolysis, magnesium hydroxide, photoelectric

### INTRODUCTION

Seawater is processed through desalination to produce fresh water. The process is vital for regions with limited water supply. Desalination consumes energy, which can be supplied from conventional power plants and other alternative sources. The recent trend indicates that the desalination process can be performed using solar power to reduce the pollution of the conventional model. Solar energy is harvested using photovoltaic [1] or concentrated thermal [2], which can be used as the energy source for desalination. Moreover, the system is possible to operate continuously with the help of an energy storage system [3–5]. However, one crucial problem still occurs in the desalination process, which is related to the waste produced (bittern). The typical process for handling bittern

is directly discharged into the open sea. It causes severe damage to the environment due to the high salinity of the solution.

The bittern solution is high in sodium and magnesium content. The two materials are essential for the modern era, such as sodium-based energy storage [6], magnesium hydride for hydrogen storage [7] and magnesium oxide for solid oxide fuel cell (SOFC) sealant [8]. In case of SOFC sealant, magnesium is considered an ideal candidate considering its vast availability and technical suitability, such as density, conductivity and redox stability [9–11]. Also, global society focuses on alternative mining techniques with minimum environmental impact, particularly for magnesium [12]. Thus, reprocessing bittern waste is ideal for reducing the potential impact of the waste [13].

The high magnesium content in bittern solution is generally processed through a membrane electrolysis process [14]. The process involves several steps, using electricity as the main power source for separation [15]. Despite its effectiveness, membrane electrolysis requires complex equipment and high energy costs. Thus, it mostly applies to producing high-economy metals such as lithium [16]. Another process uses a chemical reaction to recover magnesium from the bittern solution [17]. The process is relatively fast and reliable but incurs extra costs regarding the additional reactant. Alternatively, direct electrolysis can be used to extract magnesium from bittern solution [18]. The process is simpler than membrane electrolysis and still in developing phase to improve its performance as a separation technique in the metal production process.

Direct electrolysis operates based on an electrochemical separation technique. The key benefits of the process are design simplification, high production rates, and low environmental impact [19]. Moreover, the method is relatively flexible since the initial results of the process produce magnesium hydroxide ( $\text{Mg}(\text{OH})_2$ ), which is useful for various applications. For example, Amrulloh et al. [20] utilized  $\text{Mg}(\text{OH})_2$  from bittern solution for Pb and Cd separation processes, showing a good kinetic rate of the process using nanostructured  $\text{Mg}(\text{OH})_2$ . Further modification is performed by doping the produced magnesium with zinc oxide to produce hydrogen from wastewater recovery [21]. Thus, reprocessing bittern solution is considerably effective in supporting magnesium's usability in the modern era.

Optimization is performed to ensure the effective direct electrolysis for bittern solution. Guo et al. [22] employed liquid metal to accelerate the reaction rate, resulting in a lower carbon emission of the process with only 20.6 kg of  $\text{CO}_2/\text{kg}$  Mg. Choi et al. [23] took a different process indicator, which investigated an energy-based model of combined electrolysis and carbonation, indicating that 91.3% of  $\text{CO}_2$  could be recovered from the process using the final products  $\text{CaCO}_3$  and  $\text{MgCO}_3$ . The key aspect of the direct electrolysis is the reaction mechanism of the solution. Cai et al. [24] examined the mechanism of magnesium ion release at the magnesium anode, demonstrating the direct relation between the applied electricity and the kinetic rate of reaction. Technical aspects such as sediment formation, power factor, and solution composition are crucial to ensure

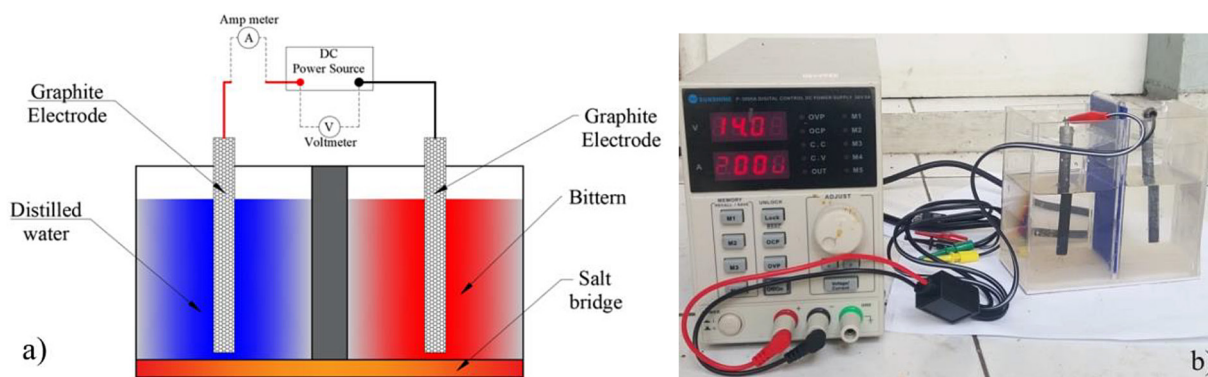
effective direct electrolysis [25]. Thus, the operational consideration and composition of the bittern solution are critical aspects that affect the quality of the reaction process.

The basic concept of electrolysis is related to ion exchange, causing variations in pH level, temperature, and chemical composition of the solution throughout the process. For example, the study performed by Li et al. [26] showed the relation between the working voltage and boiling rate of the electrolysis process. Another study focused on the variation of working voltage (2 and 3 V) for the process, showing a direct impact on the kinetic rate and changes in the solution during the process [27]. Different work focuses on numerical modelling for optimizing the technical parameters of the electrolysis process. The work here [28] analyzed numerically the critical aspect of direct electrolysis, confirming that the working voltage and current density are the major factors which significantly affect the quality of the process. A successful improvement in optimizing electrolysis for hydrogen production leads to a significant achievement in this system [29]. The same consideration is important for optimizing the electrolysis process for magnesium production, particularly from waste sources, to support the development of recycling and green mining processes.

Limited work focuses on the experimental evaluation of the operational parameter and the quality of the produced magnesium from the bittern solution. Therefore, the present study aims to experimentally investigate the effect of the parameter process on the direct electrolysis of bittern solution for producing magnesium. The study focuses on the operational aspect that can be supported by renewable sources, such as photovoltaic, to drive the electrolysis process [30]. The detailed analysis of the process and characterization of the produced magnesium are expected to bring positive contributions to the development of the green electrolysis process, reducing the risk of pollution caused by bittern solution and maximizing the potential of renewable sources to support the green mining process for extracting magnesium from alternative source.

## MATERIALS AND METHOD

This work used a direct electrolysis reactor consisting of electrodes, bittern solution, distilled water, and a power source (Figure 1a). The



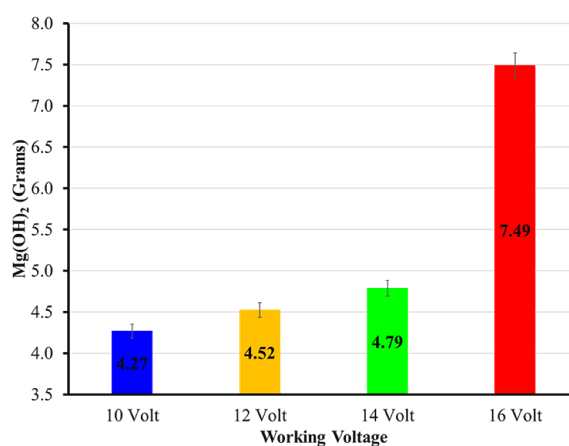
**Figure 1.** Schematic design for the electrolysis process using graphite electrodes

proposed model has one major benefit regarding the simplicity of the apparatus and process control as discussed in this work [31]. Graphite was used as an electrode since it has good electrical conductivity and corrosion resistance to the high concentration of salt water [32]. Figure 1b shows the photograph of the experimental process. The compartment was made of acrylic (thickness 2 mm) with net volume of 200 ml for each compartment. The bittern solution was located in the negative compartment (cathode), while the distilled water was in the positive compartment (anode). A salt bridge was prepared at the bottom of the compartment to ensure the continuity of the electrolysis process.

The bittern solution was obtained from local salt producer. The solution was filtered several times to remove the impurities. The bittern was characterized using XRF (X-ray fluorescence) to provide a detailed basis. The major contents from the solution were Na (6.4284%), Mg (2.2759%), Cl (11.646%), K (0.3168%), and Ca (0.4457%). Then, the electrolysis was performed. The applied current from the power source was limited to 2 A throughout the process, while the working voltage was set between 10 to 16 V (interval 2 V). The electrolysis changes the current and voltage from power source to the reactor [33]. Thus, both values were measured simultaneously to determine the power factor and voltage drop. The temperature, pH, and total dissolved solids (TDS) were measured every fifteen minutes. The total duration of the process was two hours.

## RESULTS AND DISCUSSION

The magnesium hydroxide obtained from the process is used as the initial evaluation. As seen in Figure 2, there is a positive trend in



**Figure 2.** Voltage impact on the synthesis capability of magnesium hydroxide

the increment of working voltage and the produced magnesium hydroxide. The value varies between 5.97% and 12.16% under the designed working voltage of 10–16 V. Interestingly, the produced magnesium hydroxide increases less than 10% between 12 and 14 V. However, it improves significantly using 16 V, where the magnesium hydroxide produced increases by around 65.9%. Since the process was taken using the same current, it confirms that working voltage is the most crucial factor that drives the reaction rate throughout the process [34].

The detailed profile of the process is plotted in Figure 3, which presents the power factor and voltage drop. The power factor increases at the initial process (Figure 3a). However, the profile decreases after a certain time, representing the kinetic reaction changes. The working voltage of 10 V is the only model that shows a continuous increment, indicating a slow reaction rate causing the lowest production rate (Figure 2). Figure 3b plots the voltage drop for each working voltage,

corresponding to the decrement in power factor. The highest voltage drop occurs with the working voltage of 16 V, confirming the effective process of ion exchanges during the electrolysis. It also indicates that the higher voltage leads to a substantial reaction time, increasing the production rate of magnesium hydroxide.

Electrolysis is an endothermic process [35] we demonstrate the full abatement of  $Mg^{2+}$ , with concomitant recovery of magnesium hydroxide. We have studied the influence of three operational variables, current density, temperature and forced mass transport in the coulombic efficiency, cell voltage between the electrodes, solid purity and lithium ions loss. Response surface methodology was used to model the system. Our results show that both in terms of energy efficiency, product purity and minimal  $Li^+$  loss it is best to work at very low current density, high flow rate, and high temperature ( $32 A \cdot m^{-2}$ ,  $60 \text{ }^\circ C$ , and  $18 L \cdot h^{-1}$ , resulting in temperature increment within the compartment. The initial temperature for each compartment was  $30 \text{ }^\circ C (\pm 0.5 \text{ }^\circ C)$ . The temperature

of the anode compartment (Figure 4a) increases steadily for the working voltage between 10 and 14 V, especially after reaching  $50 \text{ }^\circ C$ . The high kinetic reaction with 16 V causes the maximum temperature at  $56.6 \text{ }^\circ C$ . It supports the justification of a higher kinetic rate as presented in the power factor and voltage drop (Figure 3) for the same voltage. Also, the reaction occurs in the cathode at a higher rate, making the temperature at this compartment higher (Figure 4b). One crucial factor relates to the composition of the bittern, which probably leads to a faster temperature distribution within the compartment. As a result, the final temperature is relatively similar, particularly between 14 and 16 V. Compared to the produced magnesium hydroxide (Figure 2), it is clear that the temperature increment is not proportional to the reaction process of the bittern solution. Thus, choosing a suitable working voltage becomes crucial to maintaining the effective reaction rate during the process.

The effect of the reaction process changes the pH value for each compartment. As shown in Figure

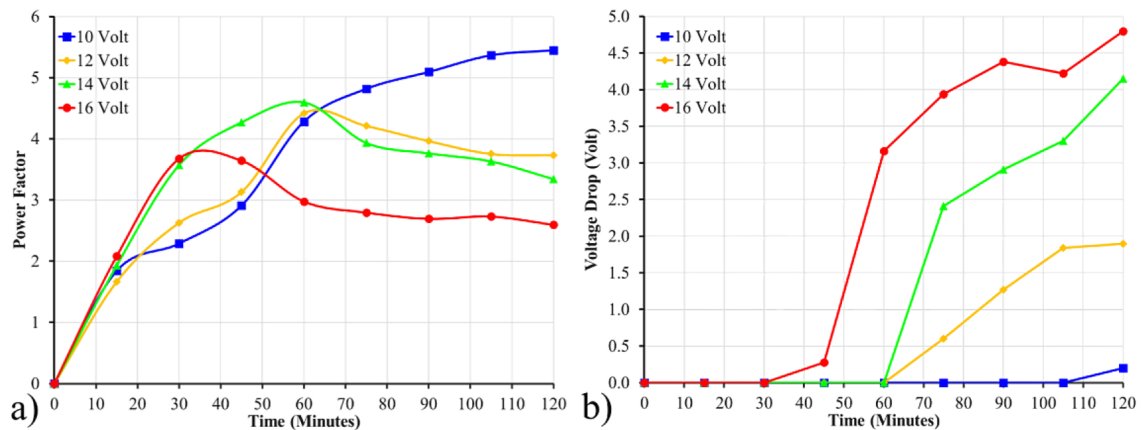


Figure 3. The power factor (a) and voltage drop (b) profile from the electrolysis process

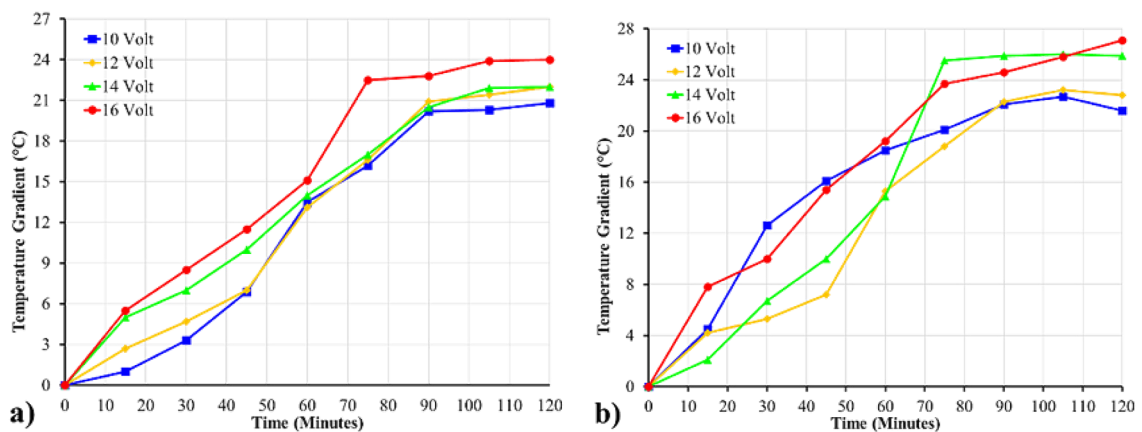


Figure 4. Temperature changes during the electrolysis process on the anode (a) and cathode (b) sides



5, each compartment shows the opposite characteristic of the pH value. The pH value for the anode compartment increases due to formation of magnesium hydroxide (Figure 5a). The slow reaction rate for a working voltage of 10 V makes the pH value increase by  $3.8 \times 10^{-2}$ /minute. It confirms the lowest formation of magnesium hydroxide based on this parameter. Interestingly, the decrement in pH level on the cathode side tends to be faster at the beginning of the reaction (Figure 5b). It indicates that the solution becomes acidic, particularly for the first period of the reaction process (15 minutes). The value ranges between 2.5 and 2.66, which can be observed for all working voltages.

The opposite pH value profile is shown for each compartment according to the total dissolved solids (TDS, Figure 6). The low content of TDS for the anode side (Figure 6a) is affected by the reaction process, which occurs mainly on the cathode side. It confirms the temperature profile for the anode side, which is steadily increased compared to the cathode side (Figure 4a). Since the rate of change of TDS and the reaction rate are closely

correlated, it is evident that the voltage of 10 V has the slowest rate of reduction (16.1 ppm/minute). However, significant variation is observed for the higher voltage. For example, the working voltage of 14 V experiences fluctuation, while the working voltage of 12 and 16 V show a steady decrement. The cathode side experienced an increase in TDS due to the formation of magnesium hydroxide on the electrode side (Figure 6b), confirming the decrement in pH value (Figure 5b). The stable value over  $4 \times 10^3$  ppm is achieved after 45 minutes with a working voltage of 16 V. The low reaction rate of the working voltage of 10 V causes a slower TDS increment. In addition, the final TDS value ranges between  $4.633$  to  $4.734 \times 10^3$  ppm, showing that the electrolysis process for the bittern solution can be performed with the given working voltage.

The electrolyte from the electrolysis process was processed for heat treatment. The aim was to observe the microstructure and crystal profile to examine the quality of the produced magnesium oxide. The first heat treatment was performed at a temperature of 110 °C to eliminate impurities.

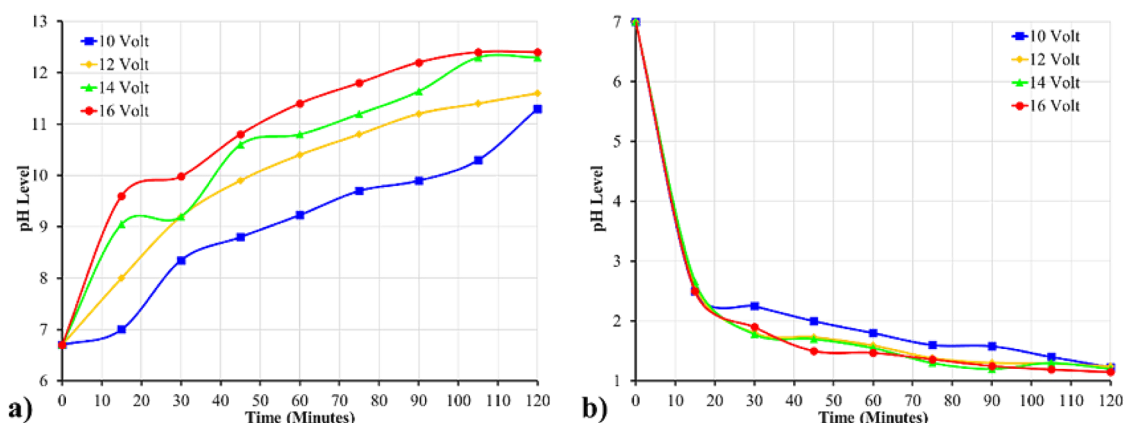


Figure 5. pH variations on the anode (a) and cathode (b) sides during the electrolysis process

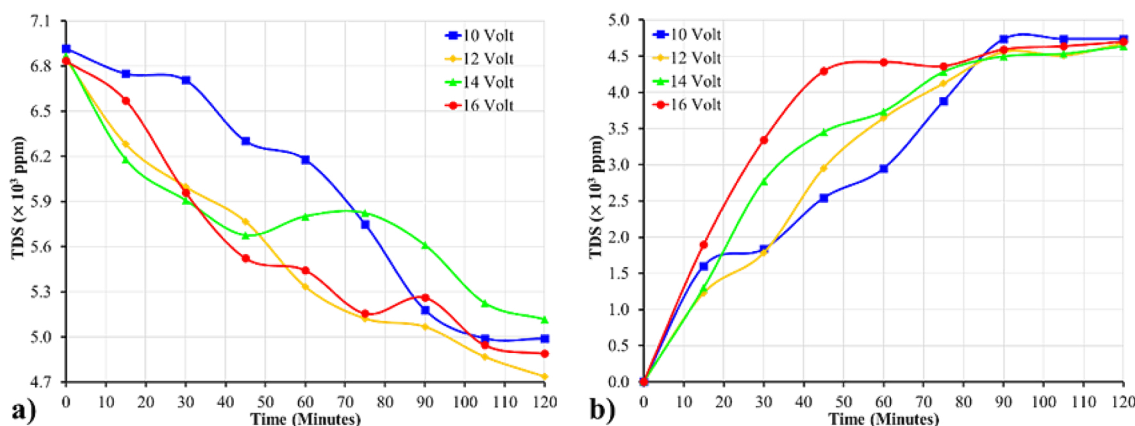
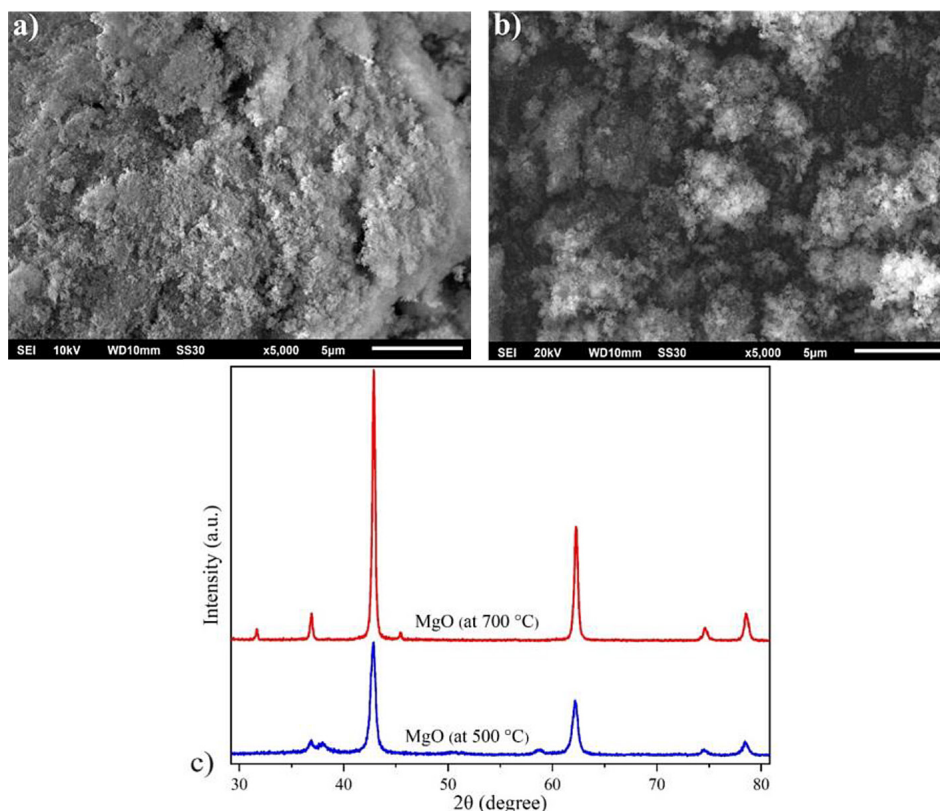


Figure 6. TDS value variations on the anode (a) and cathode (b) sides during the electrolysis process



**Figure 7.** Scanning electron microscope (SEM) for MgO after heat treatment at 500 °C (a) and 700 °C (b), and (c) its X-ray diffractometer (XRD) pattern

Then, the remaining powder was heated at temperatures 500 °C and 700 °C. The temperature represents the working temperature of low and medium temperature SOFC [36].

The SEM profile for the heated magnesium oxide at 500 °C shows large clumps (Figure 7a). It is commonly affected by the agglomeration process of magnesium oxide. In contrast, the treated magnesium oxide at 700 °C indicates a lower concentration of agglomerated profile (Figure 7b). During the heat treatment, the mass of the powder was decreased around 0.484 g. Thus, a higher temperature is desirable to minimize the formation of agglomeration within the produced magnesium. A lower agglomeration concentration is advantageous for SOFC since it requires suitable possibility to ensure effective fuel transfer during the operation [37]. The XRD pattern shows the intensity at  $2\theta$  of 36.8°, 42.8°, 62.2°, 74.5°, and 78.4° (Figure 7c). Both XRD patterns indicate a lower intense peak compared to its base ( $\text{Mg}(\text{OH})_2$ ) [38], showing the suitable quality of the produced magnesium oxide from this work. Thus, the given parameter in this work is feasible to drive the reaction process to harvest magnesium oxide from bittern solution.

## CONCLUSIONS

The present study demonstrates that the bittern solution is feasible to be processed through direct electrolysis for magnesium oxide production. The key parameter process is highly related to the working voltage. It shows a significant improvement in the produced magnesium oxide up to 75.45% using a working voltage of 16 V. The lowest power factor of the given voltage indicates the effective reaction rate. This is followed by a high temperature increment in the reactor compartment, including changes in pH and TDS values. The average temperature indicates the variation in each compartment only 2.8 °C and 3.8 °C, showing the given working voltage resulting in different reaction rates of the process. The same phenomenon is also observed for the pH and TDS values.

The morphology of the produced magnesium oxide indicates agglomeration, which can be reduced through heat treatment. The XRD pattern shows a lower number of intensity peaks, indicating the suitable quality of the produced magnesium oxide. The low agglomeration profile is essential to improve the fuel transfer rate for the fuel cell. Thus, the finding from this work is

applicable to producing magnesium oxide from bittern solution. Further evaluation is advisable by examining a large-scale model, including a detailed energy cost and improvement on the heat distribution within the compartment to improve the direct electrolysis process.

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