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## Characterization of MgO Calcined from Mg(OH)<sub>2</sub> Produced from Reject Brine

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### ABSTRACT

Reactive magnesia (MgO) has received attention as a cement binder due to the lower calcination temperatures used during its production and its ability to absorb and store CO<sub>2</sub> permanently. While a majority of the MgO is produced from the calcination of magnesium carbonate, an alternative route involves its extraction from reject brine, a concentrated by-product obtained from treating brackish water or seawater in desalination plants. Brine is preferred for the recovery of MgO due to its local global abundance and its high Mg<sup>2+</sup> content. The proposed process involves the synthesis of Mg(OH)<sub>2</sub> from waste brine collected from a desalination plant via the addition of different amounts of aqueous ammonia (NH<sub>3</sub>H<sub>2</sub>O). The results include the determination of an optimum amount of NH<sub>3</sub>H<sub>2</sub>O for a high reaction rate, leading to the synthesis of a high amount of pure Mg(OH)<sub>2</sub>. The obtained Mg(OH)<sub>2</sub> was then calcined to produce MgO at different temperatures (500-700°C) and durations (2-48 hours). The morphologies of the synthesized Mg(OH)<sub>2</sub> plates and MgO particles produced under a range of conditions were investigated in detail, indicating the feasibility of MgO production from waste brine and highlighting the factors influencing the properties of the final product.

### INTRODUCTION

The concentration of carbon dioxide (CO<sub>2</sub>) in the atmosphere has increased from 325 parts per million (ppm) at the beginning of the industrial era in 1970 to 398 ppm in September 2015 measured in Mauna Loa Observatory (Conway and Tans 2015). The average global CO<sub>2</sub> level is expected to reach 400 ppm in 2016 (Conway and Tans 2015). As the environmental impact of construction materials is becoming a serious concern, the development of novel alternatives with lower energy requirements and associated CO<sub>2</sub> emissions is more crucial than ever. Due to the wide availability of its raw materials and easy manufacture, concrete made from Portland cement (PC) is the most used material in the world, with a consumption rate of nearly 3 billion tonnes per year (USGS 2013). Cement production contributes to roughly 9.5% of anthropogenic CO<sub>2</sub> emissions worldwide (Jos G.J. Olivier and Marilena Muntean 2014). The industry manufacturing accounts for 4.8% plus the fuel combustion related to cement production contributes to approximately same level of CO<sub>2</sub> emissions, mainly due to the high calcination temperatures (~1450 °C) and energy required for the decomposition of limestone (Jos G.J. Olivier and Marilena Muntean 2014).

Several sustainable improvements ranging from sustainable energy sources usage to partial replacement of cement with low-carbon materials have been suggested within the cement industry. The utilization of sustainable energy sources such as wind and solar power has firstly been mentioned in the context of switching from traditional fossil fuels to renewable energy sources as the importance of solar energy got recognized in 1911 (Jones and Bouamane 2012). One approach includes the replacement of PC with industry by-products such as PFA and GGBS. Another approach of the sustainable initiatives in the cement industry involves the development of green cement binders such as geopolymers and reactive MgO (MgO) cements. Recently developed reactive MgO (MgO) cements require significantly lower calcination temperatures (~700 °C) and have the ability to absorb CO<sub>2</sub> in the form of stable carbonates while gaining strength (Unluer and Al-Tabbaa 2013, Unluer and Al-Tabbaa 2014).

Different grades of MgO exist and are categorized according to the calcination temperature used during the production process. Light-burned (reactive or caustic-calcined) MgO is calcined between 700 and 1000 °C, resulting in the highest reactivity and largest surface area amongst all grades. Hard-burned MgO is calcined between 1000 and 1400 °C, with a comparatively lower reactivity and surface area. Dead-burned MgO is calcined above 1400 °C, leading to the lowest surface area and hence unreactivity possible, and is the grade found in PC usually consumed in refractory industries.

Currently, the main production of MgO, performed at an amount of 12.5 million tonnes in 2003 (COMMISSION 2010) via the calcination of natural carbonates such as dolomite (CaMg(CO<sub>3</sub>)<sub>2</sub>) or magnesite (MgCO<sub>3</sub>). MgO can also be obtained synthetically from seawater or brine (Bhatti, Dollimore et al. 1984). This contributes to ~14% of the global MgO production (Kramer). This process involves recovery of abundant magnesium ion from seawater to precipitate Mg(OH)<sub>2</sub> or MgCO<sub>3</sub>, which then undergoes calcination decomposed to MgO at the end. Another production route of MgO is from magnesium silicates, which has received attention due to of the associated CO<sub>2</sub> sequestration potential during production (Simons and Vlasopoulos 2012, Baglioni, Ferraro et al. 2014). Novacem patented a novel integrated low-carbon MgO production process based on magnesium silicates raw materials in 2012 (Simons and Vlasopoulos 2012). This novel process involves: (i) producing magnesium silicate slurry, (ii) contacting carbon dioxide and precipitating MgCO<sub>3</sub> (iii) heating MgCO<sub>3</sub> to MgO and recycle carbon dioxide to step ii, representing an environmentally friendly cement binders production process. Although these methods present a great potential for the production of MgO on a larger scale, there is still some concern regarding the high energy requirements and high amounts of raw materials needed, regardless of the low calcination temperatures. A potentially more sustainable alternative production route for MgO can incorporate the use of industry waste and by-products, especially those with high magnesium contents such as waste brine or produced water (A. Al-Zahrani, Almutaz and Wagialia 1990, Ahmed, Arakel et al. 2003, Tran, Van Luong et al. 2013).

In coastal regions with limited amounts of fresh water resources such as Singapore, desalination is considered as a feasible alternative for the production of fresh water to meet residential and industrial demands. Desalination involves the removal of salts from saline water to produce fresh water. Reject brine is a concentrated by-product obtained from treating brackish water or seawater in desalination plants. At the end of this process, the most common way of disposing the waste brine involves its discharge back into the sea. However, the high concentration of salt within the brine, which is much denser than the feedstock supply, accumulates at the bottom of the sea and creates a severe impact on the environment when it is discharged back into the sea (Ahmed, Arakel et al. 2003). Several strategies such as deep well injection and mechanical or thermal evaporation have been proposed to deal with the disposal of brine instead of its direct discharge into the sea (El-Naas 2011). However, a permanent solution to eliminate the environmental impacts of this waste brine through a sustainable method has not yet been proposed.

The chemical concentration of brine varies according to the surrounding environment and usually consists of a much higher salt concentration than seawater (Tran, Van Luong et al. 2013). The chemical reaction

between reject brine and certain additives (e.g. lime solution) for the recovery of salts provides a feasible and environmental friendly use for this waste material. At the end of this reaction, the reject brine is converted into valuable and useful solids, while the remaining brackish water can be used for irrigation (El-Naas 2011). The abundant concentration of  $Mg^{2+}$  in brine serves as an excellent source for the recovery of MgO. One method to repurpose waste brine involves the production of  $Mg(OH)_2$  from the existing composition. This is realized by adding an alkali source such as, lime milk (CaO solution) (E, A et al. 1946), sodium hydroxide (NaOH) (Turek and Gnot 1995), or ammonia ( $NH_3H_2O$ ) (Henrist, Mathieu et al. 2003) to seawater/brine, thereby precipitating  $Mg^{2+}$  in the form of  $Mg(OH)_2$ . The precipitated  $Mg(OH)_2$  can be used as a flame retardant (Fellner, Hiveš et al. 2011), heavy matter leaching precipitant with several industry applications (Mironyuk, Gun'ko et al. 2006, Guo, Pei et al. 2015) or can be further calcined to produce MgO (Kotsupalo, Ryabtsev et al. 2010).

Friedrich et al. (E, A et al. 1946) patented a simple process to precipitate MgO in the form of  $Mg(OH)_2$  from seawater at a pH of 10.5 using a lime solution in 1943. Martinac et al. (Martinac, Labor et al. 1996) utilized dolomite composed of 57.6% CaO and 42.3% MgO to precipitate  $Mg(OH)_2$  from seawater, which was then calcined at  $950^\circ C$  to produce MgO. Dave and Ghosh (Dave and Ghosh 2005) used hydrated lime ( $Ca(OH)_2 \cdot 2H_2O$ ) to precipitate  $Mg(OH)_2$  from seawater at a pH ranging between 7.0 and 7.5. All these studies introduced  $Ca^{2+}$  into the seawater/brine solution, which resulted in the formation of gypsum ( $CaSO_4 \cdot 2H_2O$ ) along with other precipitates due to the presence of sulphate in the solution. The contamination of the final product due to the formation of gypsum necessitates desulfation via the addition of  $CaCl_2$  into the seawater/brine. Marian Turek (Turek and Gnot 1995) utilized sodium hydroxide (NaOH) to precipitate  $Mg(OH)_2$  from brine and reported an improved precipitation at lower temperatures and higher NaOH concentrations. This improved sedimentation was attributed to the high viscosity of NaOH solution and brine, which impeded the contact between the precipitating agent and the brine. Henrist et al. (Henrist, Mathieu et al. 2003) studied several parameters such as the chemical nature of the utilized bases, type of counter-ions and temperature on the morphological characteristics of  $Mg(OH)_2$ . Addition of NaOH to  $MgCl_2$  solution led to a globular cauliflower-like morphology, while the use of aqueous ammonia ( $NH_3H_2O$ ) resulted in plate-like shapes due to the structural difference of cations present in the solution. Single and circular plate-like particles were observed at lower temperatures while particles had a tendency to intergrow at  $60^\circ C$  (Henrist, Mathieu et al. 2003). Most of these methods that focused on precipitating  $Mg(OH)_2$  from saline water suffered from low reaction kinetics and poor dewatering and filtration properties, which limited their use on an industrial scale.

Although there are numerous studies on the reaction of  $MgCl_2$  or brine with different additives, limited research has been reported on the recovery of valuable metals from actual reject brine. This research focuses on the treatment of reject brine collected from a local desalination plant.  $NH_3 \cdot H_2O$ , which facilitates the formation of a buffer solution once conjugated with its acid ion, was utilized to maintain a stable pH for the precipitation of  $Mg^{2+}$ . Other advantages of  $NH_3 \cdot H_2O$  included the lack of impurity ions like  $Ca^{2+}$ , which resulted in the formation of a high purity MgO; and its ability to form a closed-system in which it can be reutilized as suggested in the modified Solvay process (El-Naas 2011). At the end of this process,  $NH_3$  can be almost fully recycled, thereby fully eliminating the generation of waste (El-Naas 2011).

The overall goal of this study is to synthesize MgO from locally sourced waste brine and identify the parameters influencing this process. To achieve this, a comprehensive analysis on the synthesised  $Mg(OH)_2$  and MgO was performed. X-ray diffraction (XRD) was used to study the compositions and crystal structures of the precipitates obtained under different conditions. Field emission scanning electron microscopy (FESEM) was used to analyse the morphologies of the precipitates, whereas thermogravimetric analysis (TGA) was used to study the transformation of precipitates.

This paper focuses on the characterization of MgO calcined from  $Mg(OH)_2$  under a range of calcination conditions. MgO is directly calcined from  $Mg(OH)_2$  which is precipitated from reject brine. A comprehensive investigation and comparison of physical and chemical properties of MgO with commercial

MgO is illustrated in this research. In this research, a fully characterization of MgO in terms of X-ray diffraction pattern, chemical compositions, textural properties, morphologies and reactivity is detailed.

## EXPERIMENT

**Materials.** The main materials used in this research were reject brine (supplied by Tuaspring desalination plant in Singapore) and ammonia hydroxide (NH<sub>3</sub>·H<sub>2</sub>O) solution with ACS reagent grade (25.0% NH<sub>3</sub> basis, supplied by Sigma-Aldrich-Singapore). The chemical and physical properties of these materials were obtained from the suppliers and analysed where necessary.

**Equipment.** The crystal structures and chemical compositions of MgO calcined from Mg(OH)<sub>2</sub> were firstly tested using X-ray diffraction (XRD) which provides a qualitative and quantitative analysis of the crystal phases. MgO samples were grinded and vacuum dried before analysis, performed via a Bruker D8 Advance with a Cu K $\alpha$  source under the operation conditions of 40 Kv and 40 mA, emitting radiation with a wavelength of 1.5405 Å, scan rate of 0.02° /step, and 2 $\theta$  range of 5~70°. The quantitative analysis of chemical compositions of MgO was carried out using thermogravimetry/differential thermal analysis (TG/DTA). TG/DTA was performed via a PerkinElmer TGA 4000 to analyse the thermal decompositions of the prepared samples at a heating rate of 10 °C/min under air. The morphologies of MgO were studied by imaging fracture surfaces under a JSM-7600F thermal Field Emission Scanning Electron Microscopy (FESEM) on gold-coated specimens. The textural properties in terms of specific surface area (SSA), pore volume and mean pore radius were determined from nitrogen adsorption – desorption isotherms on a QuadrasorbEvo automated surface area & pore size analyzer. The SSA was determined using Brunauer-Emmett-Teller (BET) method while pore distributions were obtained using Barrett-Joyner-Halenda (BJH) method.

**Experiments procedures.** Mg(OH)<sub>2</sub> was synthesized from reject brine in the same procedure as section 5.2.3 illustrated at an optimized ratio of 6:1. This ratio was chosen because of the acceptable Mg<sup>2+</sup> sequestration rate and comparatively low Ca<sup>2+</sup> precipitation. Mg(OH)<sub>2</sub> precipitated through this process was then calcined to produce MgO, as shown in



The calcination conditions are illustrated in Table 1.

**Table 1. Calcination conditions**

Calcine temperature	500 °C	600 °C	700 °C
Duration time (h)	2	2	2
Duration time (h)	24	24	24
Duration time (h)	48	48	48

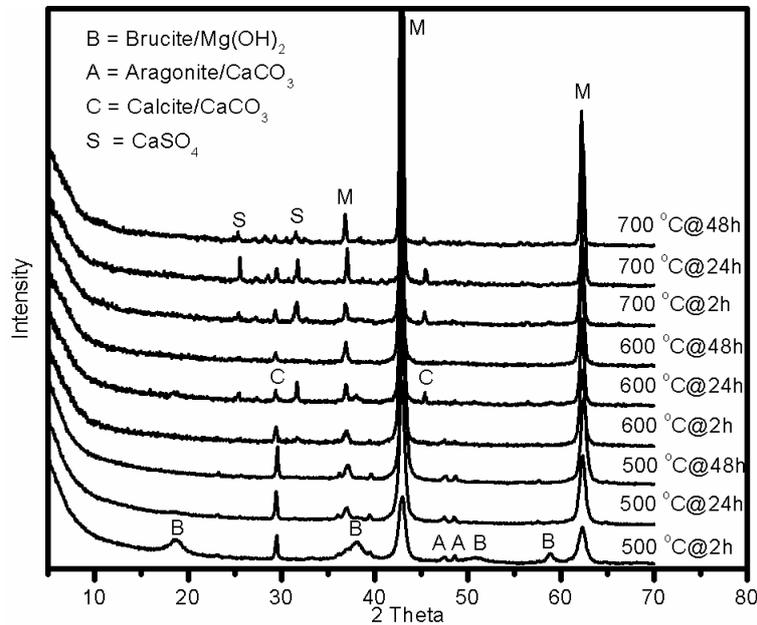
*Heating rate: 10 °C/min and slow cooling*

## RESULTS AND DISCUSSIONS

### XRD Analysis

Figure 1 shows the XRD diffractograms of MgO calcined under different conditions. While MgO is the main component obtained in the final precipitate, CaCO<sub>3</sub> and CaSO<sub>4</sub> is also spotted in smaller quantities due to the presence of bicarbonates in the solution converting to carbonates which attacked Ca<sup>2+</sup> to form CaCO<sub>3</sub>. The peak positions of the final product matches the reference peaks of MgO (JCPDS card No. 89-7746) well. Mg(OH)<sub>2</sub> was not fully decomposed at 500 °C for 2 hours while either increasing the calcination temperature or duration time can kill all the Mg(OH)<sub>2</sub> decomposed to MgO. CaCO<sub>3</sub> is spotted since the decomposition temperature is ~ 700 °C and increasing the duration time gradually kills the CaCO<sub>3</sub> decomposed to CaO at 700 °C. CaCO<sub>3</sub> is in the form of aragonite and calcite at 500°C while aragonite gradually transits into calcite with increasing calcination temperature. That's because aragonite will spontaneously convert to calcite when heated to 400 °C if pressure does not change (Dasgupta 1964).

The presence of calcium sulfate (CaSO<sub>4</sub>) at higher calcination temperature is due to SO<sub>4</sub><sup>2+</sup> compounded with Ca<sup>2+</sup> from the brine solution. CaSO<sub>4</sub> has a poor solubility in water (0.21g/100ml at 20 °C for anhydrous and 0.24g/100ml for dihydrate form) (Gangolli and Chemistry 1999) and remains stable at high temperature.



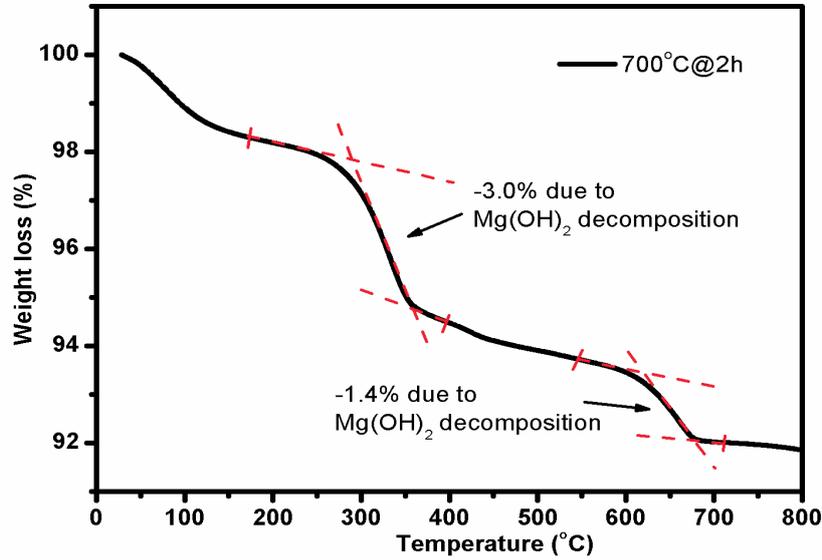
**Figure 1. XRD diffractograms of MgO calcined under different conditions.**

### TGA Analysis

**Table 2. TGA and DTA results of MgO calcined under different conditions**

Calcining conditions	Peak temperature (°C)	Weight loss between 340-440°C (%)	Peak temperature(°C)	Weight loss between 650-750°C (%)
500 2h	403.8	0.43	725.4	2.3
500 24h	406.8	1.1	702.9	2.3
500 48h	409.8	1.4	738.1	2.2
600 2h	407.3	5	706.9	1.1
600 24h	393.4	0.9	690.9	0.3
600 48h	393.4	5	690.9	1.8

700 2h	407.3	3.0	706.9	0.1
700 24h	393.4	1.7	690.9	0.3
700 48h	393.4	0.3	690.9	0



**Figure 2. TGA profile of MgO obtained after calcining Mg(OH)<sub>2</sub> at 700 °C for 2 hours.**

The quantitative analysis of the chemical compositions of MgO obtained from calcination from Mg(OH)<sub>2</sub> was further analysed under TGA to assess its degree of formation and record the content of undecomposed Mg(OH)<sub>2</sub>. The TGA results of MgO calcined under different conditions are shown in

Table 2. The decomposition of the MgO calcined at 700 °C for 2 hours is further analysed, as presented in

**Figure 2.** The dehydration of Mg(OH)<sub>2</sub> and decarbonation of CaCO<sub>3</sub> are observed at ~400 and ~700°C, respectively. The TGA results show the presence of small amount of undecomposed Mg(OH)<sub>2</sub> and CaCO<sub>3</sub> after calcination.

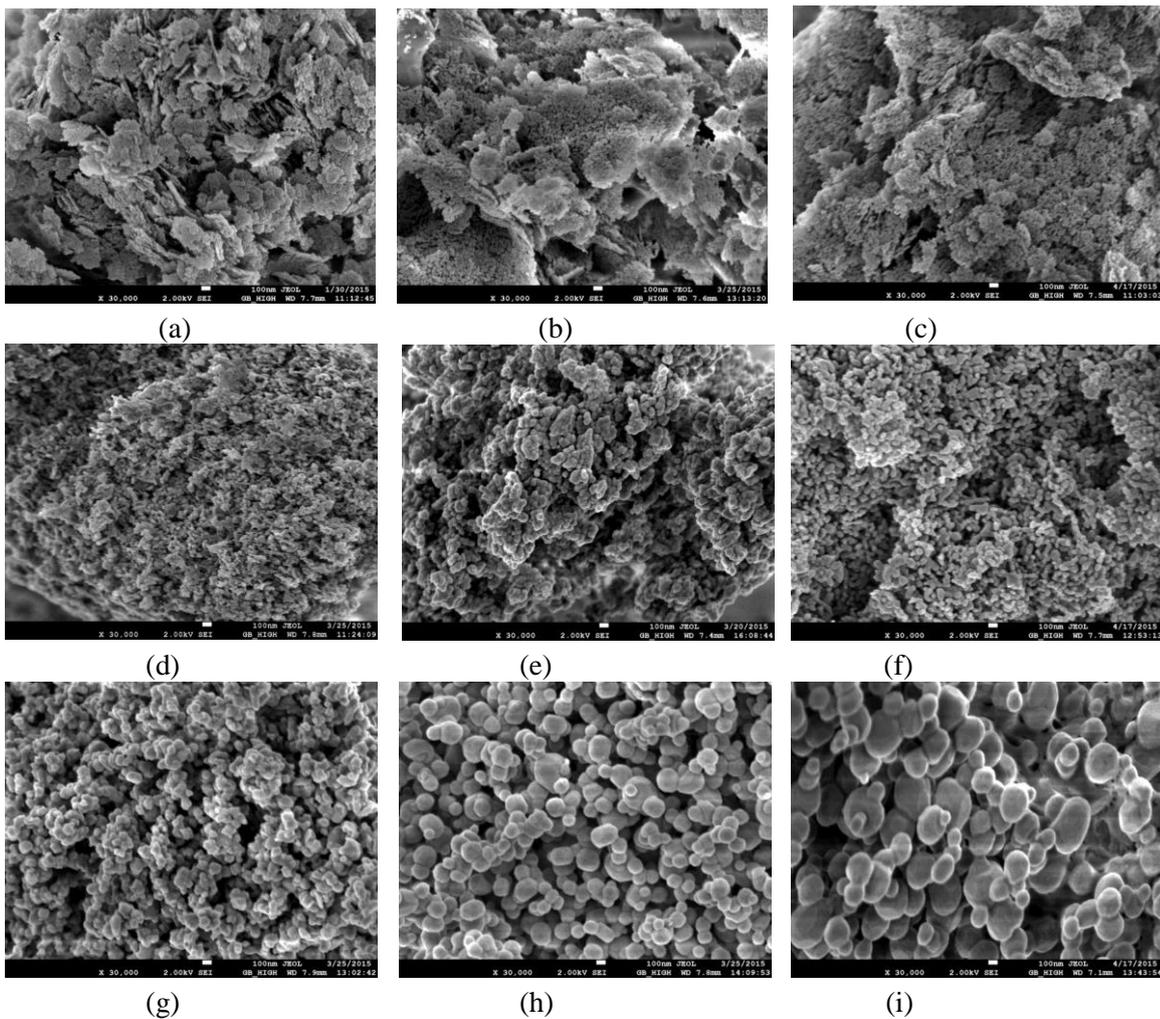
The first weight loss associated with the dehydration of Mg(OH)<sub>2</sub> started at 343.5°C and resulted in the release of H<sub>2</sub>O. The second weight loss, attributed to the decarbonation of CaCO<sub>3</sub> resulting in the release of CO<sub>2</sub> at 649.5-750 °C. The results shown in

Table 2 confirm the complete decomposition of Mg(OH)<sub>2</sub> and CaCO<sub>3</sub> under 700°C for 24 hours.

**FESEM Analysis.** The morphology of MgO under difference calcination conditions is investigated by FESEM analysis, as shown in Figure 3. The FESEM examination shows the presence of two different morphologies of MgO (plate-like and spherical shape) obtained under different calcination conditions. A flake-like structure dominates at lower calcination temperature while a spherical structure dominates at higher calcination temperature. Duration time significantly affects the size of the MgO particles. Calcined at 500 °C, MgO maintains the similar plate-like morphology as mother Mg(OH)<sub>2</sub>, while the plate-like shape gradually transits into fine spherical particles as calcination temperature goes higher to 700 °C. As the duration time increases from 2 hours to 48 hours at 700 °C, the spherical particles grow larger from 100nm to 300nm. The size of each spherical particle is around 300nm with homogeneous distribution under 700°C for 48 hours calcination.

**Particle size and specific surface area analysis.**

Table 3 shows the effects of calcination temperature and duration time on the specific surface area (SSA) of MgO. It can be clearly seen the SSA decreases and particle size increases when the calcination temperature and duration time increases in which SSA and particle size is closely related. During the decomposition of Mg(OH)<sub>2</sub>, porous structures are formed as water vapour escapes and then the MgO particles grow due the continuous calcination which causes the reduction of pore volume and enlargement of pore size, therefore shrinking the SSA(Mo, Deng et al. 2010).



**Figure 3. FESEM images of MgO calcined under different conditions: (a) 500 °C at 2h, (b) 500 °C at 24h, (c) 500 °C at 48h, (d) 600 °C at 2h, (e) 600 °C at 24h, (f) 600 °C at 48h, (g) 700 °C at 2h, (h) 700 °C at 24h,(i) 700 °C at 48h.**

**Table 3. Particle size and specific surface area of MgO calcined under different conditions.**

Conditions	d(0.5) um	SSP (BET)m <sup>2</sup> /g
500 2h	28.5	78.78
500 24h	40.4	43.54
500 48h	45.6	43.99
600 2h	25.8	26.84
600 24h	35.2	21.7
600 48h	60.2	20.75
700 2h	59	10.25
700 24h	67.9	6.25
700 48h	71.3	5.5

## CONCLUSION

The results of this study demonstrate the feasibility of obtaining high purity nano-sized MgO from waste brine. The results shed a light on the significant potential of brine in the recovery of Mg<sup>2+</sup> and the synthesis of pure MgO with a wide range of industrial applications. The resulting MgO powder composed of homogeneously distributed spherical particles with an average size of 300nm. The calcination temperature and duration time has a significant effect on the microstructure and reactivity of MgO. The size of MgO particles grow bigger at higher temperature. This MgO obtained from waste brine can be a candidate for utilization as catalysts, bactericides, as well as toxic chemical adsorbents (Aramendia, Borau et al. 1996, Yang and Lieber 1996, Bhargava, Alarco et al. 1998, Kolusheva, Boyer et al. 2000, Xu, Wei et al. 2001, Makhluf, Dror et al. 2005) due to its nano-size and high purity.

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