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MAGNESIUM HYDROXIDE RECOVERY FROM MAGNESIA WASTE BY CALCINATIONS AND HYDRATION PROCESSES

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Abstract: In this study, hydration behavior of the magnesia waste, supplied from the electrostatic bag house of the sintering unit of MAS Company, Eskisehir-Turkey was studied to find out re-usability of the material as a source of magnesium hydroxide ($Mg(OH)_2$) production. According to chemical and crystalline phase analyses, MgO content of the sample was determined as 49.9 wt% and consists of mainly raw magnesite ($MgCO_3$), sintered magnesia-periclase (MgO) and small amount of serpentine (lizardite) phases. In hydration process, the reactivity of magnesium oxide determines the rate and extent of hydration of the sample. Therefore, the waste samples both calcined and uncalcined forms were hydrated in water and the efficiencies were compared according to sample source. The hydration experiments were carried out at temperatures ranging from 30 °C and 80 °C to evaluate the influence of temperature on particle morphology and surface areas of the products. The efficiency of the hydration was evaluated by using thermogravimetric (TG) and scanning electron microscopy (SEM) analyses. The test results suggest that the magnesia waste could be used as a source material for production of magnesium hydroxide.

***Key words**: magnesium hydroxide, hydration, magnesia waste, characterization*

Introduction

Magnesium hydroxide is an important compound that finds application in various industries such as additives in pulp-paper production, pharmaceutical products and flame retardant in polymeric materials (Rothon, 2000; Dong et al., 2010). The use of catalyst, catalysts support, sorbent for chemical and destructive adsorption of various pollutants, adsorbent or pH regulator for wastewater treatment (Gulkova et al., 2004; Climent et al., 2007; Gao et al., 2008) and a source material for the production of magnesium oxide, metal or chemicals are also common application areas (Shand et al., 2006; Aphane et al., 2007). In recent years, the advances in imaging, engineering and

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manipulating system at the nanometer scale have led to numerous research studies dealing with synthesis of $Mg(OH)_2$ nanoparticles or nanosurfaces. For example, the utilization of superfine $Mg(OH)_2$ as a smoking and toxic-free flame retardant filler in plastic, rubber, and other halogen-free polymeric materials has attracted much attention due to the improved properties such as a good thermal stability, good fire retardant and low toxicity (Li et al., 2014). Recent studies performed by Dong et al. (2010 and 2011) showed that nano-sized $Mg(OH)_2$ can be an antibacterial agent. Pan et al. (2013) also reported that the nano-sized $Mg(OH)_2$ is adsorbed on bacterial surface by charge attraction first, and then destroys the integrity of cell walls resulting in death of bacteria.

In literature, many methods were proposed to produce nano-sized $Mg(OH)_2$. However, the wet precipitation was proved to be the most simple and a low-cost way to synthesize $Mg(OH)_2$ crystals (Fernandez et al., 2014). $Mg(OH)_2$ with different morphologies such as rods, tubes, needles, and lamina have been synthesized by hydrothermal reaction using different magnesium sources such as magnesium powder, $MgSO_4$, and $Mg(NO_3)_2 \cdot 6H_2O$. In the study performed by Beall et al. (2013), magnesium chloride, magnesium acetate and metallic magnesium have been used as the magnesium source. The synthesis results showed that production of pure hydromagnesite, utilizing hydrothermal methods, is much simpler and faster. The effect of type and concentration of modifiers on the physicochemical and functional properties of magnesium hydroxide obtained by precipitation, and its calcinate magnesium oxide, was studied by Pilarska et al. (2013). In this study, magnesium hydroxide obtained from magnesium sulfate and sodium hydroxide was subjected to thermal decomposition at 450 ^oC. The modifiers were non-ionic compounds representing poly(ethylene glycols) including PEG 200, 8000, and 20000. It was found that the modifier introduced into the reaction system had a direct influence on the morphological and dispersive properties of the products as well as their surface character and high surface area. Qiang et al. (2014) also reported that hexagonal $Mg(OH)_2$ nanoflakes were successfully synthesized in the presence of polyethylene glycol 20,000 (PEG-20,000) surfactant. Recently Fernandez et al. (2014) released a paper in which $Mg(OH)_2$ nano-flakes, with different morphologies, were synthesized via the hydrothermal method. It was found that synthesis temperature and reaction time have a strong influence on the product morphology, particle size, agglomeration level and crystallographic structures. It was also suggested that the prolongation of reaction time improves the crystalline degree of magnesium hydroxide particles. Besides than the particle morphology, the agglomeration behavior of the nano-sized particles within the polymer matrix was also defined as an important parameter that determines the usability of the synthesized product. An exemplary modification applied to reduce water absorption of magnesium hydroxide precipitate with addition of octadecyl dihydrogen phosphate as a surface modifier was proposed by Dongmin et al. (2009). Song et al. (2010) reported that hydrophobic magnesium hydroxide nanoparticles can be synthesized by the simultaneously addition of reactant solutions into an organic phase instead of an aqueous phase. Later, Pilarska et al. (2011) proposed that the PEGs can be used to modify the surface properties of $Mg(OH)_2$.

The natural form of magnesium hydroxide (brucite) deposits are scarce and often contaminated by fibrous minerals. Therefore, it is mainly produced by the hydration of magnesia (MgO) obtained either from a heat treatment of mineral magnesite, precipitation from seawater or magnesium containing brines. The use of seawater as starting material is more costly because of the huge amount of water required and has some difficulties in the crystallization stages. Therefore, it preferentially produce by calcinations of $MgCO_3$ for decomposition to MgO, and than, it is subsequently hydrated in water to convert MgO to $Mg(OH)_2$ (Thorp and Gilpin, 1949). Due to a wide spectrum of application areas as discussed above, the hydration mechanism of $Mg(OH)_2$ obtained from magnesia has been received an interest by several researchers in literature. In general, it was found that calcination temperature and treatment time prior the hydration, surface area, particle size of starting material, slurry density, hydration temperature and the hydration time were the main parameters that can influence the hydration performance and product properties. The term of hydration can be referred as a formation of a hydrated compound from the reaction of water with a solid compound. Therefore, most of studies were carried out with water. The earlier study performed by Maryska and Blaha (1997) showed that the hydration rate of MgO, obtained from the calcination of magnesium hydroxide carbonate, is strongly affected by both calcination and hydration temperatures. The effect of calcination temperature on the surface area, activity of MgO and response of these parameters to MgO hydration was studied by Birchal et al. (2000). It was reported that calcination temperature is the main variable affecting the surface area and reactivity of MgO, where both parameters increase with increasing temperature up to 900 ^{o}C. Later, Rocha et al. (2004) reported that the hydration performance of high purity magnesia increases with increasing of hydration temperature. Strydom et al. (2005), also studied the effect of calcining temperature on the hydration of industrially obtained MgO. It was found that degree of hydration is influenced significantly by temperature and contact time. Recently, Pilarska et al. (2014) showed that the higher reactivity and better hydration performance of the calcined sample could be caused by microstructural changes.

Although much attention has paid to produce low cost material for large scale applications, current synthetic version of magnesium hyroxide still tent to be costly. Furthermore, the waste material produced from metallurgical and mining activities has been taken into consideration not only by the governments related with the stringent environmental legislations but also by the producers to maintain the sustainable and cost efficient production. The recent studies performed by Fernandez et al. (2009) and Formosa et al. (2011) showed that low-grade magnesium hydroxide, an industrial by-product obtained in the calcination of magnesia, can be used as cheap filler in polymeric materials. Turkey has about 160 teragrams of the world reserve of 3 600 tergrams. Raw, calcined, dead burned and fused form of magnesite are the main products of Turkey. In 2012, Turkey ranked second in magnesite exports in the word with a share rate of 32%. MAS magnesite company (located in Eskisehir) one of the biggest sintered magnesia producers of Turkey. The company processes yearly 150 gigagrams of sintered product. With a help of recently installed rotary furnace, the company is planning to increase the processing capacity near 250 gigagrams in 2014. The sintered magnesia produced by crushing, screening, drying, classification, magnetic separation and sintering at horizontal rotary furnace. By this route, every year, apprx. 20 Gg of waste material is collected in the furnace electrofilters and the amount of waste stock has being progressively increased year by year due the lower recycling rate of it. Therefore, the objective of the study is to determine the recyclability of magnesia waste as a cost effective relatively pure magnesium hydroxide for filler applications.

Methodology

Material

The waste sample (EFD) used in the experiments was supplied from the electrostatic bag house of MAS Company, Eskisehir-Turkey. The chemical composition of representative sample (determined by XRF analyses) indicated that SiO_2 and CaO are the main impurities together with little amount of Fe_2O_3 and Al_2O_3. The magnesium oxide content of the sample was determined as 49.70 wt%. However, MgO to $MgCO_3$ ratio (0.185), determined by the carbon content of the sample (LECO furnace analysis), showed that unreacted $MgCO_3$ still is the main mineral component.

Table 1. Chemical analysis results of magnesite dust sample

Oxide %	EFD	Oxide %	EFD
SiO_2	4.45	CaO	2.79
Fe_2O_3	1.04	MgO	49.70
Al_2O_3	0.06	LOI*	41.94

*LOI, loss of ignition

The detected by XRD analyses mineral phases are given in Fig. 1. These result confirmed that the chemical analyes date and it was found that the sample mainly consisted of raw ($MgCO_3$) and sintered magnesite (periclase) and small amounts of serpentine (lizardite) phases. Particle sizes distribution of the sample was determined by wet screening. It was found that the D_{80} value's of sample was about 0.130 μm.

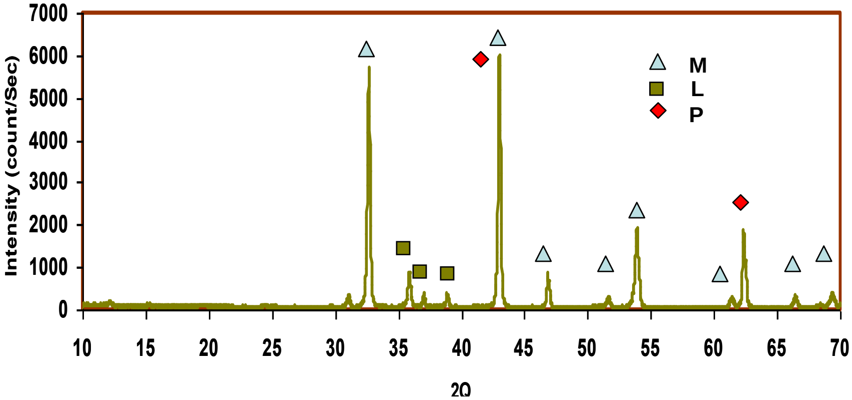

Fig. 1. XRD diffraction pattern of EFD; M: magnesite, L: lizardite, P: periclase

Methods

The hydration experiments were performed in a closed 250 cm^3 glass beaker. In order to determine the effect temperature variation on hydration reaction, the beaker was immersed in water containing another glass beaker placed on a temperature-controlled magnetic stirrer. In each test 10 g of sample as calcined or received form, was added in to the 250 cm^3glass beaker containing 100 cm^3 of aqueous solution.

The temperature of the aqueous solution was previously adjusted to predetermined value (40 ^oC, 60 ^oC and 80 ^oC) and kept constant during the hydration. The hydration temperatures selected in this study were determined based on the previous studies (Van der Merwe, 2006; Fillippou, 1999; Aphane, 2007; Pilarska, 2012). In hydration tests, the slurry was continuously stirred at a constant rate of 250 rpm for two hours and pH variations of the solution was recorded according to time intervals. The products were then filtered under vacuum, dried in an oven at 100 °C for two hours and weighted. The concentration of dissolved Ca and Mg ions in solution after hydration were analyzed by the complexometric method. The TG analyses of the samples were carried out with a NETZSCH STA 409 PC/PC instrument. A heating rate of 10 K min^{-1} in a nitrogen atmosphere from 30 to 1000 ^oC was applied. The percentage of $Mg(OH)_2$ in the products was obtained from the curves of mass loss as described earlier by (Aphane, 2007). A NOVA 1000e surface analyzer with nitrogen gas as an adsorbent was used to determine the BET surface areas of the products. The samples were degassing at 240 ^oC during 16 hours. Scanning electron microscope (SEM) analysis of samples was investigated by using a high vacuum electron microscope ZEISS SUPRA VP 50. In order to prevent the charging, samples were firstly coated with sputter coater using Au-Pd target. The e-beam used for analysis was 20 keV and working distance was 12.7 mm. At these working conditions 5000- and 30000-fold magnified images were taken at the secondary electron image mode (SEI).

Table 2. The test design employed for studying the effect of individual parameters on the hydration processes

Sample code	Temperature (°C)	
N1-40R	40	Received form
N2-60R	60	Received form
N3-80R	80	Received form
N4-40C	40	Calcined at 900 °C for 2 h
N5-60C	60	Calcined at 900 °C for 2 h
N6-80C	80	Calcined at 900 °C for 2 h

Results and discussion

pH variation of hydration solution and product weight

The variations of solution pHs during hydration processes are given in Fig. 2. Although relatively higher values were measured for calcined samples, in general, a similar trend was observed for all samples subjected to hydration processes. The pH's of the solutions slightly decreases with increasing treatment time up to two hours, and then, remains almost constant for further progressing contact times. It was also observed that the increase of the solution temperature leads to a decrease of the solution pH's for both calcined and un-calcined samples.

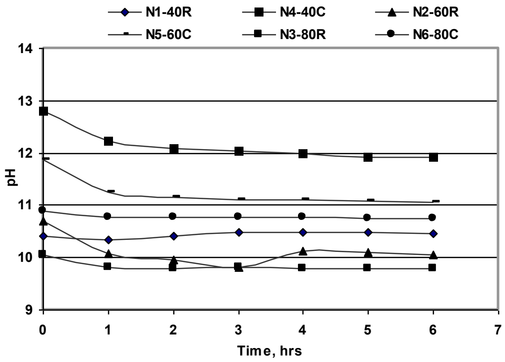

Fig. 2. pH variation of solution during the hydration process

The hydration reaction of MgO to $Mg(OH)_2$ can be given as following chemical equation:

$$MgO_{(s)}+H_2O_{(l)} \rightarrow Mg(OH)_{2(s)} . \tag{1}$$

According to the stoichiometric calculation, it can be expected that weight of the product should be higher than that of the starting material proportionally to the hydration efficiency. The higher weights measured for the calcined samples and given in Fig. 3 clearly indicated the importance of calcinations step prior the hydration process. The material used in this study is a by-product of the sintering processing of $MgCO_3$ and may expected to react with water for hydration. However, the results showed that effective conversion of to $Mg(OH)_2$ requires calcination steps prior to hydration in water. As discussed earlier, the received sample consists of mainly raw magnesite, little amounts of sintered magnesia and impurities. The lower weights measured for the un-calcined samples indicated limited reactivity of the starting material for hydration in water. For hydration of the calcined sample, it was observed, that the weight of the products increases with increasing solution temperatures. However, these are limited and can be concluded that increase in solution temperature has a slight affect on hydration processes within the studied temperature range.

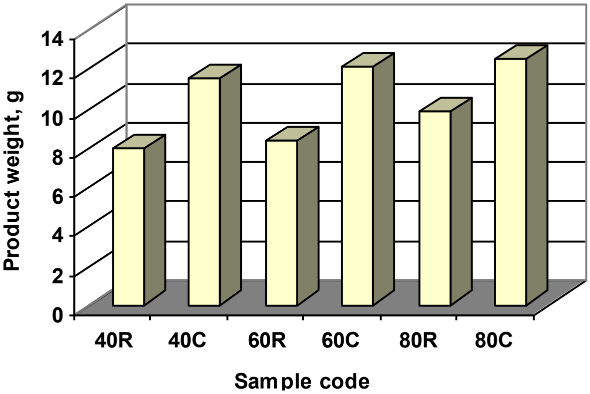

Fig. 3. Effect of sample source and hydration temperature on product weight

Thermogravimetric analyses

The TG curves of the hydrated samples both received and calcined forms according to employed hydration temperatures are given in Fig. 4a and 4b, respectively. For uncalcined samples (Figure 4a), a number of weight loss steps up to a temperature of 600 °C was observed. These steps can be ascribed to moisture loss (between 50 and 200 °C), $Mg(OH)_2$ decomposition (after 200 °C to 450 °C) and $MgCO_3$ decomposition (450 °C to 600 °C). The TG curves of calcined samples completely match the characteristic MgO - $Mg(OH)_2$ transformation mass loss-temperature curve between 200 °C and 450 °C. Although the mass losses are lower than the theoretical value obtained for pure MgO (30.9%), they are confirmed by the formation of $Mg(OH)_2$ after hydration processes as previously reported by several researchers (Aphane, 2007).

The similarity of the decomposition curves and slight increase of mass losses with increasing solution temperatures showed that hydration reactions were affected by the solution temperature. It was found that the mass losses increase from 19.05 to 19.78%

with increasing solution temperature from 40 ^{o}C to 80 ^{o}C. However, the relatively lower mass losses analyses of the calcined sample and the effect of temperature contribution to the hydration reactions compared with the previous reports, can be attributed the lower particle size of the waste material, presence of some impurities in the starting material and lower solubility tendency of water as hydrating agent. Van der Merwe and Strydom, (2006) studied the effect of hydration temperature on the percentage of $Mg(OH)_2$ by using different hydrating agents and reported that the increase on hydration temperature enhances the performance of the process for all hydrating agent. However, the hydration performed in water was the least temperature dependent. Hydration performed in magnesium acetate was found more intensive than any of the other hydrating agents. Although the product obtained from the hydration of MgO in magnesium acetate solutions contains also some unreacted magnesium acetate, Aphane **(**2007) also released a similar report later, and proposed that the magnesium acetate was a better hydrating agent than water. These reports concluded that the solubility of the various compounds plays an important role in the mechanism of the process, and that the rate of hydration is controlled by the rate of the chemical reactions taking place at the MgO particle surface.

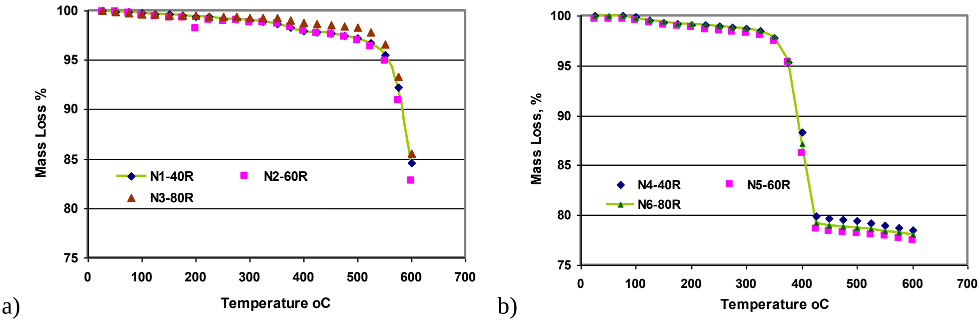

Fig. 4. TG results of samples: uncalcined (a) and calcined (b)

Due to relatively complexity of the hydration of MgO in water, the rate of the hydration process is receiving more interest and concern as a key parameter for the hydration reaction occurring on the solid surface and also it’s important effect on the final product morphology. In an early study, Kitamura et al. (1969) proposed that a modified version of the chemical shrinking core model could be used for describing the reaction kinetic. This model has been found adequate to fit kinetic data at high temperatures (between 135 ^{o}C and 200 ^{o}C), but could not fit kinetic data for hydration in water at lower temperatures. Birchal et al. (2001) developed an alternative, semi-empirical model, that also assumes chemical control of the reaction with no diffusive contribution. This model assumes an increasing resistance to the hydration process caused by the filling of the porosity inside and between the MgO particles with the $Mg(OH)_2$ product. Later Rocha et al. (2004) reported that two distinct hydration processes may occur depending on the temperature. At high temperatures, the hydration of magnesia seems to be initially governed by the oxide dissolution (chemical control). As the reaction progresses, both surface and pores of magnesia particles are covered progressively by the hydroxide produced, changing the porosity of the solid. As a result, the diffusion of water is hindered inside particles, thus reducing the overall reaction rate (diffusive control). At low temperatures, the hydration is purely chemical-controlled. These studies clearly indicated beneficial effect of temperature contribution to the chemical reaction. In a recent study, Thomas et al. (2014) suggested an interesting approach and reported that variation with temperature more than one mechanism is controlling the hydration rate in the temperature range. They applied an alternative model known as the boundary nucleation and growth (BNG) model that has previously been applied to the hydration of Portland cement and tricalcium silicate. Although, the analyses results showed that the hydration kinetics of both light and hard-burned MgO can be fit using a BNG model, the experiment performed with using light-burned MgO was found to be an almost ideal BNG system within the temperature range from 20 ^{o}C to 50 ^{o}C. The calorimetric analyses results for the light-burned MgO between 20 ^{o}C to 50 ^{o}C showed that the hydration reaction is affected by temperature variation. The values of G (the linear rate of growth of a product region in any direction) at 30 ^{o}C and 50 ^{o}C are not too different for these two powders. This indicates that it is primarily the higher surface area of the light-burned powder that leads to the much faster hydration at a given temperature. Greater surface area results in more nucleation, and thus more regions of hydration product growing at the same time. Beside the calcinations affect, the particle size of the solid material that is subjected to hydration also determines the reaction rate. The smaller particle size supports the dissolution of material and subsequent nucleation of $Mg(OH)_2$ on MgO. Higher surface resulting from lower particles size would lead to a faster reaction.

Surface area

Figure 5 shows the variation of the surface area of $Mg(OH)_2$ formed by hydration of the samples, both in received and calcined forms, at elevated solution temperatures. It was found that variation of BET values follows a similar trend with thermogravimetric analysis results, as discussed above. According to the applied hydration temperature, the surface areas of the hydrated products increased from 5.04 m^2/g to 6.34 m^2/g for uncalcined samples. On the other hand, for calcined samples, considerably higher values were determined. The surface areas of the products increase from 18.32 m^2/g to 19.20 m^2/g by increasing temperature from 40 ^{o}C to 80 ^{o}C, respectively. The higher surfaces areas measured for calcined samples can be attributed to higher conversion rates compared to un-calcined samples due to lower surface area. The surface areas of the samples were determined as 3.36 m^2/g and 39.15 m^2/g for un-calcined and calcined samples, respectively. As discuses previously (Birchal et al., 2000; Rocha et al., 2004; Strydom et al., 2005; Aphane, 2007), calcination temperature is the main variable that affects the surface area and reactivity of MgO during hydration process. The results show that by hydrating MgO calcined material between 650 and 1000 °C, about the same percentage of $Mg(OH)_2$ was obtained, whereas at higher calcination temperatures (1200 °C or more) the percentage of magnesium hydroxide decreases considerably. In the first stage of the hydration the oxide dissolution occurs within particles, changing porosity within this stage leads to creation of supersaturation, nucleation and growth of $Mg(OH)_2$ at the surface or internal boundaries of MgO particles.

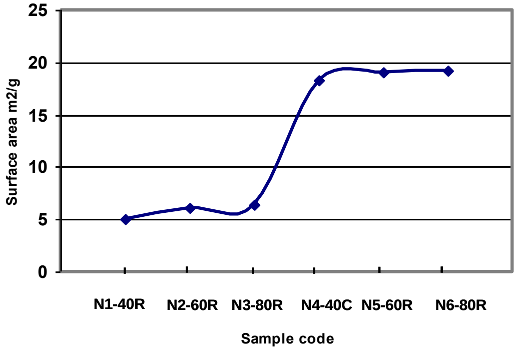

Fig. 5. Surface area variation of samples according to sample source (calcined and un-calcined) and hydration temperature

Product morphology

In general, the crystalline $Mg(OH)_2$ belongs to the hexagonal system and is characterized by platelet–like crystals arrangement. However, it may also adopt several morphologies and sizes which offer to them advantageous for industrial applications. The morphologies of selected samples, analyzed by SEM, are given in Fig. 6.

The observed cryptocrystalline form of magnesite crystals for the sample N1-40R, suggested that the sample still conserves the previous form. This indicates that the inefficiency of the hydration processes on the waste sample as received form (Fig. 6a and b). On the other hand, the calcined samples have a spherical morphology and consist of aggregated irregular and tiny crystals, which reflect the characteristic crystallographic structure of brucite. The morphology of these aggregates results from rapid rates of precipitation rate under high supersaturating conditions. As previously reported by Rocha et al. (2004), during hydration of MgO in water, the supersaturation is reached rapidly in a few minutes. If the hydration is performed at low temperatures, the higher initial supersaturation level leads to formation of a significant amount of fine magnesium hydroxide particles with the high nucleation rate. Water diffuses rapidly into MgO particles and $Mg(OH)_2$ precipitates inside the whole magnesia particle, so higher conversion levels are obtained at short periods of time. In contrast, with increasing hydration temperatures the morphology of the product is changed and result in a slightly more compact particles as observed in Fig. 6e.

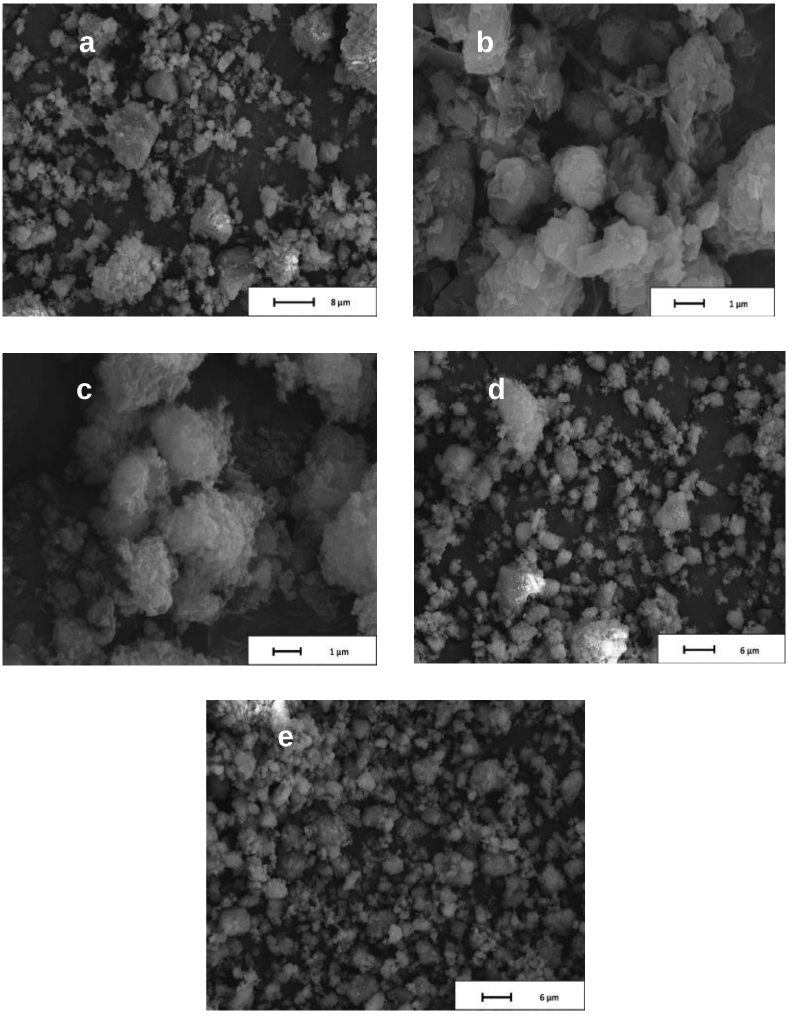

Fig. 6. SEM images of hydrated N1-40R, N5-60C and N6-80C

Conclusion

In this study, the recyclability of magnesia waste as a relatively pure magnesium hydroxide was investigated. According to hdyration tests result it can be concluded that effective conversion of them to $Mg(OH)_2$ requires calcination steps prior the hydration in water. The increase of hydration temperature has a positive effect on the hydration efficiency and grain texture. However, it was found that this effect was not significant. The mass losses increase from 19.05 to 19.78% with increasing solution temperatures from 40 ^{O}C to 80 ^{o}C, respectively. This can be attributed a higher activity of the calcined samples. Higher surface area of the powder leads to much faster hydration at lower temperatures. On the other hand, as observed from SEM images, the increase of hydration temperature caused a slightly more dense coverage of the surface and internal boundaries with the nuclei of hydrated products. The results suggested that the hydrated product could be considered as filler material for plastic industry. However, the polarity and stability of the additives in plastic mixtures are important parameters, therefore, further studies should also be performed on the electrokinetic properties of the hydrated product.

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