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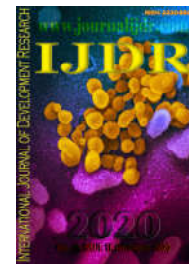
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RESEARCH ARTICLE

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ANALYSIS OF MAGNESIA HYDROXYLATION HEAT AS SOURCE OF ENERGY TO CONCENTRATE AND/OR DRY MAGNESIUM HYDROXIDE PULP

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ABSTRACT

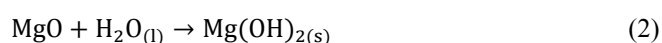
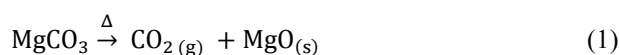
Magnesium hydroxide is an important inorganic compound that is largely used in many industrial processes. It can be obtained through the hydroxylation of magnesite, followed by comminution and drying step. In this context, the present work aims to evaluate if the excess of hydroxylation reaction heat can be used in the subsequent step of product drying, developing and simulating the mass and energy balances in the system. Hydroxylation tests were carried out with magnesite from mineral source, in a non-adiabatic reactor with 25 % initial solids concentration. The behavior of the pulp in one adiabatic system was simulated in the Excel software, varying the initial pulp content between 25 and 35% solids, considering the hydroxylation conversion varying from 0 to 100%. The reaction heat of caustic magnesium hydroxylation can promote a relevant change in the pulp concentration if an adiabatic reactor is used instead of a non-adiabatic one. The energy released from the reaction was enough to heat the system until 100 °C, vaporize partially the water and concentrate the pulp until 57%. The hydroxylation heat can promote significant energy savings to obtain the traditional magnesium hydroxide in pulp (52%) and powder (26%). In the non-adiabatic reactor, this heat would be lost.

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INTRODUCTION

Magnesium hydroxide (MH) is largely applied as flame retardant and effluent neutralizer. It can be obtained from magnesite, through the following steps: magnesite calcination (Equation 1), magnesite hydroxylation (Equation 2), comminution, and drying. During hydroxylation, the magnesite is transformed in magnesium hydroxide, the solid structure expands, and the pulp solid concentration increases (Shand, 2006; Kitamura, Onizuka & Tanaka, 1995).



The hydroxylation process generally takes place in two steps, using magnesite that has elevated magnesium oxide grade: I. Dissolution of the magnesite in the grain boundary, the region with the greatest free energy; II. Supersaturation of solution with subsequent precipitation of magnesium hydroxide, which promotes volumetric expansion in the grain boundary, generating tensions that cause breaking bonds inside

the crystals (Birchal *et al.*, 2001; Salomão, Bittencourt & Pandolfelli, 2006). When magnesite is added to water, the pH of pulp raises up to 10.0 - 10.3, which is due to the dissolution of MgO, hydroxylation of magnesite, and liberation of hydroxyl ions into the solution (Shand, 2006; Chen & Tao, 2004). Magnesium hydroxide precipitation causes particle expansion and intermolecular tensions on the oxide surface derived from larger molar density of Mg(OH)₂ (24.30 cm³.mol⁻¹) when compared to magnesite (11.25 cm³.mol⁻¹) (Khangaonkar, Othman & Ranjitham, 1990). When the hydroxylation is carried out in pulp, with solid concentration up to 50%, the mechanism involves the dissociation of magnesite, that supersaturates the aqueous solution due to the release of magnesite (Mg²⁺) and hydroxyl ions (OH⁻), so the magnesium hydroxide precipitates, occurring after heterogeneous and homogeneous nucleation (Birchal, Mansur & Ciminelli, 2001; Rocha, Mansur & Ciminelli, 2004). Thus, the reaction progress depends on process variables, such as temperature, viscosity, and mineral impurities (Santos, Andrade & Rocha, 2017; Matabola *et al.*, 2010; Amaral *et al.*, 2007; Liu *et al.*, 2012; Valle-Zermeño *et al.*, 2012).

The magnesium hydroxide concentration influences the pH and the viscosity of the pulp, which can be controlled by the use of dispersants at temperatures ranging from 30 to 80°C. Inorganic and organic dispersants, such as acetic acid, cause appreciable effects in magnesia hydroxylation, controlling the pulp viscosity and increasing the reaction rate (Matabola *et al.*, 2010). The temperature is an essential parameter to speed the hydroxylation, which is associated with nucleation and growth phenomena that control hydroxylation carried out up to 80 °C. The increase in temperature can take place because of the exothermic reaction heat, so it can modify the evolution of hydroxylation (Birchal *et al.*, 2001; Rocha, Mansur & Ciminelli, 2004; Matabola *et al.*, 2010; Amaral *et al.*, 2007; Liu *et al.*, 2012). Magnesia hydroxylation is an exothermic and spontaneous reaction under normal condition (25°C and 1.0atm): the reaction heat corresponds to -37.13kJ.mol⁻¹ and the Gibbs energy is -27.65kJ.mol⁻¹ (Atkins & De Paula, 2003). The increase in solids concentration in the reaction pulp can affect the hydroxylation heat transfer in the reactor and the temperature tends to rise.

In the regular process, the reaction step is followed by the solid comminution that is an essential to get a product under 45 µm. After that, the magnesium hydroxide pulp goes to the concentration step or drying, depending on the final product to be obtained. It is an essential step of the process when part of the water vaporizes to produce a concentrated pulp (65% solids) or a powdered product (5% humidity) (Shand, 2006; Matabola *et al.*, 2010; Gibson & Maniocha, 2007; Ozkan, Yekeler & Calkaya, 2009; Tromans, 2008; Wills & Nappier-Munn, 2006; Nedmag, 2017; Premier, 2017; Khangaonkar, Othman & Ranjitham, 1990). While the drying step requires energy to evaporate the water, the reaction step generates heat that could be used as energy source. So, the main objective of this work is to analyze the extent in which the hydroxylation heat can be used to promote the concentration and/or drying of the magnesium hydroxide pulp, by energy and mass balance in hydroxylation of a high purity magnesia.

Research Elaboration

Hydroxylation tests and characterization: Magnesium oxide hydroxylation experiments were carried out in a 2.0 L ceramic reactor at a speed of 64 rpm. The pulps were prepared using a high-concentration caustic calcined magnesia (93.47% w/w MgO) and the temperature of the system was registered by a thermocouple for 8.0 hours. The tests were performed according to the parameters shown in Table 1.

Table 1. Test parameters of magnesia hydroxylation in the mill

Parameters	Values
Magnesia mass (kg)	0.175
MgO content (%)	93.490
MgO mass (kg)	0.163
H ₂ O mass (kg)	0.525
Pulp mass (kg)	0.699
MgO (mol)	4.060
Reaction total enthalpy at 298.15 K (kJ)	-150.770

The quantitative chemical analysis was performed by X-Ray fluorescence spectrometry (XRF) on PHILIPS PW-2510 spectrometer. A tablet of the solid was prepared by mixing 1.0 g of the sample with boric acid and the analysis was performed using an X-ray beam from a rhodium anode. The thermogravimetric analysis (TGA) was performed using a

platinum crucible under a nitrogen atmosphere. The heating rate applied in the analysis was 20 °C.min⁻¹ from the initial temperature of 19 °C to the final temperature of 900 °C, using a Shimadzu Thermogravimetric Analyzer (TGA 50) (Földvári, 2011). X-ray diffraction was done to identify the crystalline mineral phases (Philips - PW 1710).

Heat analysis of the real process (non-adiabatic): The energy balance of the caustic magnesium hydroxylation process and the behavior of the pulp was developed considering the magnesia chemical analysis and for the initial solid concentration of 25%. The sensible heat (ΔH) of the reaction was expressed as a function of the heat capacity coefficients A, B, C, D, E of the pulp components (H₂O, MgO and Mg(OH)₂) along the hydroxylation, the initial temperature (T₁) and the final temperature of each test (T₂), according to equations 2 and 3. The heat capacity coefficients of the pulp components are shown in Table 2 (Smith, Van Ness & Abbott, 2007; NIST, 2020).

$$\Delta H = \sum(x_i \cdot A_i) \left(\frac{T_2 - T_1}{10^3} \right) + \sum(x_i \cdot B_i / 2) \left(\frac{T_2^2 - T_1^2}{10^3} \right) + \sum(x_i \cdot C_i / 3) \left(\frac{T_2^3 - T_1^3}{10^3} \right) + \sum(x_i \cdot D_i / 4) \left(\frac{T_2^4 - T_1^4}{10^3} \right) - \sum(x_i \cdot E_i) \left(10^3 / (T_2 - T_1) \right) \quad (2)$$

$$\Delta H = \sum(A_{pulp}) \left(\frac{T_2 - T_1}{10^3} \right) + \sum(B_{pulp} / 2) \left(\frac{T_2^2 - T_1^2}{10^3} \right) + \sum(C_{pulp} / 3) \left(\frac{T_2^3 - T_1^3}{10^3} \right) + \sum(D_{pulp} / 4) \left(\frac{T_2^4 - T_1^4}{10^3} \right) - \sum(E_{pulp}) \left(10^3 / (T_2 - T_1) \right) \quad (3)$$

Table 2. Heat capacity coefficients of the main components in the caustic magnesia hydroxylation pulp

Component	Heat capacity coefficient (J.mol ⁻¹ .K ⁻¹)				
	A	B	C	D	E
H ₂ O	-203.606	1523.290	-3196.413	2474.455	3.855
MgO	47.260	5.682	-0.873	0.104	-1.054
Mg(OH) ₂	84.903	74.440	-68.928	26.632	-2.175

(NIST, 2020)

The solid phase composition changes during the progress of magnesium oxide hydroxylation. The molar compositions were calculated for each test (0 to 8h) as well as their respective: capacity coefficients, temperature variation and the real sensible enthalpy of the pulp (ΔH_{real}) - proportional to the measured temperature variation. Besides, the theoretical reaction heat was determined ($\Delta H_{\text{theoretical}}$), considering the heat produced if magnesium oxide hydroxylation reaction was 100% of conversion. Consequently, it was possible to determine the heat loss.

Heat analysis of the adiabatic process

The pulp temperature variation is a function of the reaction heat. Actually, during the reaction process, the pulp solids content increases, due to the magnesium hydroxide formation. But, if the reaction heat is used to heat the pulp, this energy can warm the pulp, vaporize the water, so the pulp solids content rises. The concentration process considers three followed steps, according to the reaction heat (Equation 5): I - the pulp heating; II - water vaporization; III - the solid product and steam heating. Therefore, the system temperature can be calculated according to Equation 6.

The maximum pulp temperature that could be reached is equal to the boiling temperature of pure water (100 °C at 1.0 atm). Dta for pure water was used considering the very low solubility of magnesium oxide and hydroxide, that would not be enough to change significantly the solution composition thus causing the effect of boiling point elevation. When all the water in the system could be vaporized, the heat should promote a temperature increase of the steam and the solid product, represented by Equations 5 and 6. The energy balance considered the parameters: ΔH_{pulp} - sensible heat variation of the system, ΔH_{steam}^{water} - water vaporization heat, ΔH_{solid} - sensible heat of the solid; ΔH_{steam} - sensible heat of the water vapor.

$$\Delta H_{reaction} = \Delta H_{pulp} + \Delta H_{water}^{steam} + \Delta H_{steam} + \Delta H_{solid} \quad (5)$$

$$\Delta H_{reaction} = \int_{298}^{37} C_{p,pulp} dt + \Delta H_{water}^{steam} + \int_{37}^{T_f} C_{p,steam} dt + \int_{37}^{T_f} C_{p,solid} dt \quad (6)$$

The total hydroxylation conversion (100%) could generate the maximum reaction heat (ΔH_{ideal}) equal to 150.77 kJ, considering the initial caustic magnesia quantity (174.75 g). The process simulation was developed considering the hydroxylation rate varying from 0 to 100% and the increment was 1%. Three different pulps were analyzed, using the initial solid concentrations: 25, 30 and 35%. The initial solid concentration was studied under 40% because the magnesia hydroxylation pulp tends to have a high viscosity. The process generally requires the use of dispersants, which occurs in low concentration (under 1.0 mol. L⁻¹), so the dispersants were not considered for the present analysis. The heat capacity coefficient was calculated for each pulp - considering the pulp is composed of magnesium oxide, magnesium hydroxide, and water. Besides, the energy balance was calculating through simulation in the Excel software:

- I -The reaction enthalpy for distinct hydroxylation conversions(0 - 100%);
- II -The sensible pulp heat (which would be available to heat the system to a maximum temperature of 100 °C);
- III -The required enthalpy for water vaporization;
- IV -The percentage amount of water capable to be evaporated relative to the amount of water in the final pulp;
- V -The variance of solids concentration (considering the water vaporization and the reaction).

RESULTS

Chemical characterization: The caustic magnesia contains a high amount of magnesium oxide (98.15%), Table 3. The magnesium oxide was the main component of the sample which was confirmed by the intense peaks of periclase in several characteristic bands (100, 96, 12, 10 and 5), Figure 1. The magnesium mass balance was performed to obtain the chemical composition and determine the amount of available magnesium oxide for hydroxylation. The caustic magnesia sample was composed mainly of periclase (93.49%) and brucite (6.44%), representing 99.93% of the sample. Then, for the heat analysis development, the sample was considered as a solid composed of only MgO and Mg(OH)₂.

Table 3. Chemical composition of caustic magnesium (dry basis), determined by FRX analysis

Component	(%w/w)
MgO	98.1500
Mg	59.1680
Si	0.0044
Ca	0.0140
Fe	0.0022
Mn	0.0003
Al	0.0003

Table 4. Composition of caustic magnesium and determination of MgO available for hydroxylation

Component	MgO (%w/w)	Mg (%w/w)
Mg(OH) ₂		2.70
Al ₂ Mg ₅ Si ₃ O ₁₀ (OH) ₈		0.07
MgCO ₃		0.09
Unavailable	-	2.86
Total	98.15	59.17
Available	93.34	56.31

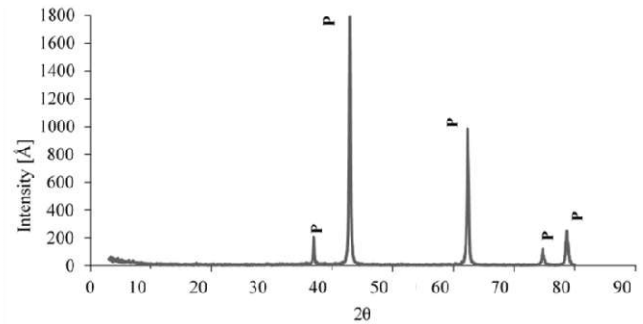


Figure 1. XRD pattern of Brazilian caustic magnesium: P = Periclase

The initial liquid phase presented metal ions, according to plasma source mass spectroscopy (ICP-MS), Table 5. It revealed the slight solubilization of some metals, due to the low solubility in water (at 20 °C) of the minerals identified in caustic magnesia (Antonelli et al., 2006; Wesolowski, 1984; Meyers & Speyer, 2003; Morey, 1962; Zhao et al., 2010; Whitney & Eberl, 1982; Fao, 2016; Labsynth, 2020; Itaplana, 2020; Bénézech et al., 2011). Magnesium was the main element present in the aqueous phase, $[Mg]_{total} = 58.7 \text{ mg.L}^{-1}$. Dissolved calcium and silicon were under 10 mg.L⁻¹. Aluminum, iron and manganese concentrations were below 0.2 mg.L⁻¹, the detection limit of the ICP-MS technique. Thus, the heat analysis considered the pulp liquid phase as pure water during the hydroxylation process.

Table 5. Concentration of metal ions in pulp of caustic magnesia hydroxylation samples

Ion concentration (mg.L ⁻¹)					
Mg	Ca	Si	Al	Fe	Mn
58.7	7.5	2.4	<0.2	<0.2	<0.2

Energy balance: The enthalpy variations were calculated for each pulp obtained through magnesia hydroxylation as a function of time. The initial enthalpy (H₁) was the same for all tests (-3.395 kJ), at 298 K, and the initial solid concentration of the pulp (magnesia and water) was 25% solids. The final enthalpy (H₂) was determined by the temperature reached in

Table 6. Variation of real enthalpy and reaction heat during the hydroxylation of caustic magnesia - 25% of solids

	Pulp (h)	0	1	2	3	4	5	6	7	8	Ideal
Process	Hydroxylation (%)	0.00	10.23	21.91	20.63	48.34	88.28	88.23	88.92	88.82	100.00
	$\sum n$ (mol)	33.37	29.73	26.56	23.34	21.25	20.77	20.29	19.84	19.39	19.39
MgO	mass (g)	163.49	146.76	127.67	129.76	84.46	19.16	19.24	18.11	18.28	0.00
	n (mol)	4.06	3.64	3.17	3.22	2.10	0.48	0.48	0.45	0.45	0.00
	n_{consumed} (mol)	0.00	0.41	0.89	0.84	1.96	3.58	3.58	3.61	3.60	4.06
	x (mol/mol)	0.12	0.12	0.12	0.14	0.10	0.02	0.02	0.02	0.02	0.00
H ₂ O	mass (g)	524.61	459.01	401.95	343.95	306.20	297.63	289.03	280.93	272.76	272.76
	n (mol)	29.12	25.48	22.31	19.09	17.00	16.52	16.04	15.59	15.14	15.14
	n_{consumed} (mol)	0.00	0.41	0.89	0.84	1.96	3.58	3.58	3.61	3.60	4.06
	x (mol/mol)	0.87	0.86	0.84	0.82	0.80	0.80	0.79	0.79	0.78	0.78
Mg(OH) ₂	mass (g)	11.26	46.72	74.35	71.33	136.88	231.36	231.24	232.87	232.64	259.08
	n (mol)	0.19	0.61	1.08	1.03	2.15	3.77	3.77	3.80	3.80	4.25
	$n_{\text{generatade}}$ (mol)	0.00	0.41	0.89	0.84	1.96	3.58	3.58	3.61	3.60	4.06
	x (mol/mol)	0.01	0.02	0.04	0.04	0.10	0.18	0.19	0.19	0.20	0.22
Heat capacity coefficient (J.mol ⁻¹ .K ⁻¹)	$\sum x_i.A$	-171.44	-166.98	-161.94	-156.27	-149.61	-145.44	-144.08	-142.68	-141.26	-140.38
	$\sum x_i.B$	1330.43	1307.77	1283.29	1250.04	1226.72	1225.30	1218.28	1211.47	1204.16	1205.77
	$\sum x_i.C$	-2789.88	-2741.03	-2687.94	-2617.65	-2564.17	-2555.01	-2539.91	-2525.14	-2509.42	-2511.02
	$\sum x_i.D$	2159.52	2121.31	2079.67	2025.16	1982.25	1973.05	1961.25	1949.67	1937.39	1938.01
	$\sum x_i.E$	3.22	3.13	3.02	2.91	2.76	2.65	2.62	2.59	2.56	2.53
Variance of heat capacity coefficient (J.mol ⁻¹ .K ⁻¹)	$\sum x_i.A$	0.00	-2.60	-5.54	-8.85	-12.73	-15.17	-15.96	-16.78	-17.61	-18.12
	$\sum x_i.B$	0.00	-1.70	-3.54	-6.04	-7.80	-7.90	-8.43	-8.94	-9.49	-9.37
	$\sum x_i.C$	0.00	-1.75	-3.65	-6.17	-8.09	-8.42	-8.96	-9.49	-10.05	-10.00
	$\sum x_i.D$	0.00	-1.77	-3.70	-6.22	-8.21	-8.63	-9.18	-9.72	-10.29	-10.26
	$\sum x_i.E$	0.00	-2.89	-6.19	-9.67	-14.39	-17.88	-18.76	-19.68	-20.59	-21.40
Temperature (K)	$T_1.10^{-3}$	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	-
	$T_2.10^{-3}$	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	0.30	-
Energy (kJ)	ΔH_{real}	0.00	0.20	0.24	0.27	0.38	0.36	0.29	0.33	0.25	-
	$\Delta H_{\text{reaction}}$	0.00	15.42	33.03	31.10	72.88	133.10	133.03	134.07	133.92	150.77
	Q_{loss}	0.00	15.22	32.80	30.83	72.50	132.74	132.74	133.74	133.66	-
	Q_{lost} (%)	0.00	98.69	99.28	99.13	99.47	99.73	99.78	99.76	99.81	-

the final of each test, enabling the calculation of the real energy variation in the system (ΔH_{real}). The reaction heat ($\Delta H_{\text{reaction}}$) was calculated according to the hydroxylation conversion achieved in each test. In an adiabatic system, the reaction heat would be accumulated in the system and warm the pulp. So, the heat lost during the hydroxylation process was determined by the difference between $\Delta H_{\text{reaction}}$ and ΔH_{real} . The temperature variance was moderate, between 1.0 C and 6.0 °C, Table 6. The heat loss was significant, and the medium value achieved 99.76% during the process, preventing the increasing in temperature. The heat capacity coefficients showed a small variance, which was explained by the large concentration of water in the pulp, which was 75% initially.

Adiabatic process: The variations of the heat capacity coefficients throughout the simulation were small and it can be explained by the large quantity of water in the pulp, Figure 2. So the thermal characteristic of the pulp could be similar during hydroxylation.

- The pulp parameters were determined for three different initial solid concentration (25, 30, 35%), Figures 3, 4 and 5. The available energy for water vaporization was expressed by the difference between the reaction heat and the sensible heat, thus it showed the same values for all systems. The maximum value was 145.60kJ
- The vaporized water was different for each pulp and the systems with higher initial solid concentration would have a more significant impact on water vaporization and, consequently, it could promote higher energy savings. The maximum levels of vaporized water for the different systems (25, 30, 35% of initial solids) were: 13.18, 18.02, 24.36%
- The solid concentration was directly influenced by the water vaporization and by the reaction, it increased during the hydroxylation. For each pulp was observed the gain in solid concentration considering the adiabatic condition and if the reaction was complete (100%), the solid concentration could reach the following values for each pulp (25, 30, 35%) respectively: 38.72, 47.42, 56.53%. Then, the solid concentration could be near the required value for the as traditional magnesium hydroxide in pulp (65.00%).

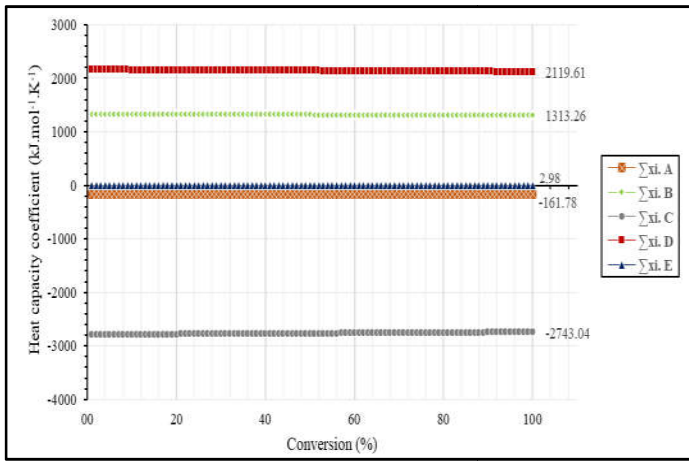


Figure 2. Variation of heat capacity coefficients during caustic magnesia hydroxylation in adiabatic system, for pulp with initial 25% solids content.

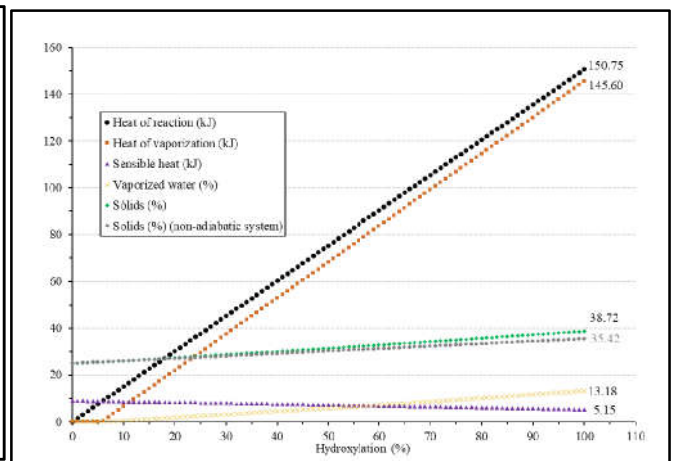


Figure 3. Heat parameters during caustic magnesia hydroxylation simulation for pulp with initial 25% solids content in adiabatic system

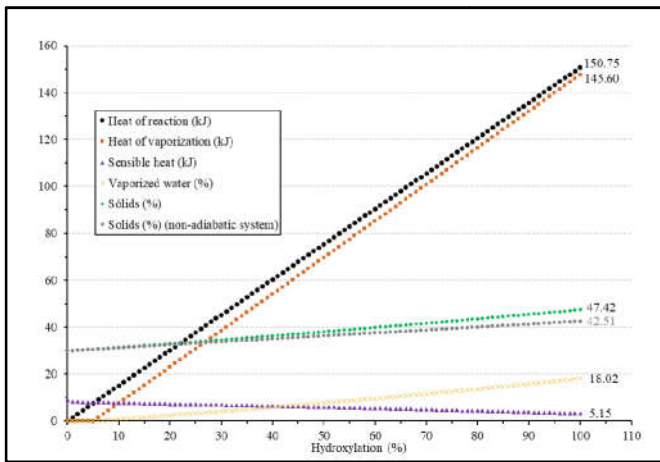


Figure 4. Heat parameters during MgO hydroxylation simulation, for pulp with initial 30% solids content in adiabatic system

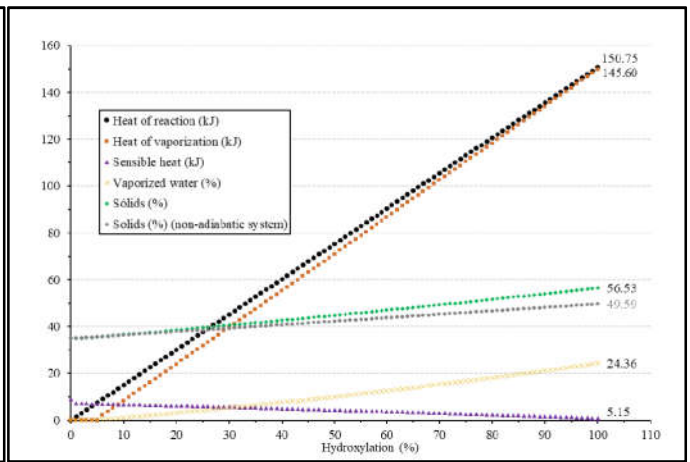


Figure 5. Heat parameters during MgO hydroxylation simulation, for pulp with initial 35% solids content in adiabatic system

Table 7. Mass analysis of processes for concentration of magnesium hydroxide pulp for each initial solid concentration, considering the total hydroxylation (100%)

System	25		30		35		Pulp	Powder
	non-adiabatic	adiabatic	non-adiabatic	adiabatic	non-adiabatic	adiabatic		
Final pulp - solids (%)	35.42	38.73	42.51	47.42	49.59	56.53	65.00	95.00
m _{total} (g)	699.51	639.84	582.91	522.55	499.69	438.34	381.22	260.84
m _{H₂O} (g)	451.61	392.04	335.12	274.76	251.89	190.55	133.43	13.04
m _{Mg(OH)₂} (g)	247.80	247.80	247.80	247.80	247.80	247.80	247.80	247.80
Water to remove - pulp (g)	318.18	258.62	201.69	141.33	118.46	57.12	-	-
Water to remove - powder (g)	438.56	379.00	322.07	261.72	238.85	177.51	-	-
Removed water - pulp (%) /	-	18.72	-	29.93	-	51.78	-	-
Energy economy (%)	-	-	-	-	-	-	-	-
Removed water - powder (%) /	-	13.58	-	18.74	-	25.68	-	-
Energy economy (%)	-	-	-	-	-	-	-	-

The energy produced by the hydroxylation could be enough to heat the system up to 100°C and vaporize partially the water. When the solid concentration of the pulp increases, the effect of the reaction heat is greater. The vaporized water increased for each pulp (with 25, 30 and 35% of solids) and it could achieve the following values: 13.18%, 18.02% and 24.36%. Thus, the adiabatic process can remove part of the required water to obtain the commercial pulp (65% solids) or to produce the powder neutralizer (95% solids/ 5% humidity). If the initial system had a larger solid concentration, more significant would be the water removal and directed associated with energy savings to vaporize the water, table 7.

The energy savings for the drying step could be relevant, reaching the values: pulp - 51.78%; powder - 25.58%.

CONCLUSION

The real process in the ceramic reactor showed a high heat loss (99.98%) and the heat capacity of pulp demonstrated a low variance during the hydroxylation process. The reaction heat of caustic magnesium hydroxylation can be used to promote a relevant impact on the magnesium hydroxide pulp concentration, it could promote a significant energy saving to obtain the traditional magnesium hydroxide pulp (52%) and

powder (26%). This energy is enough to heat the system up to 100°C, vaporize the water partially and concentrate the pulp up to 57%, when the initial solids is 35%.

Acknowledgments

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Nomenclature

CSTR - Continuous stirred tank reactor.

FRX - X-ray fluorescence.

HM - magnesite hydroxide.

TG - thermogravimetry.

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