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Interfacial phenomena of magnesium hydroxide micro phases

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ABSTRACT

The magnesium hydroxide aqueous suspensions were prepared over a wide range of different types of dispersants with different dosages percentage. The effect of dispersant types and dispersant dosages on the rheological behavior and stability of magnesium hydroxide aqueous suspensions have been investigated by means of viscosity, sedimentation measurements, flowability, Total organic carbon (TOC) and zeta potential. The results showed that considering the four types of dispersants, Calcium lignosulfonate (Ca-lig) enhance the viscosity and flowability of the slurry the most. Also it decreases the pH of slurry. The study gives insights on important application process optimization.

1. Introduction

The main focus of this paper is on magnesium hydroxide slurry and its rheological behavior. Magnesium hydroxide can be prepared either by physical or chemical methods. Examples of most common chemical methods of preparation are by direct precipitation, hydrothermal and hydration methods [1–4]. Magnesium hydroxide is known for its poor water solubility. It releases its alkalinity slowly and when heated, it can decompose. Therefore, it is used in wastewater treatment instead of caustic soda and lime. It is also used in flue gas desulfurization, as flame resistant material, and in many other fields [5].

Purification of wastewater and liquid waste is one of the most intensively developed applications of magnesium hydroxide. Moreover, one of the common uses of magnesium hydroxide is in the conservation of paper. Paper and related products are subject to adverse changes in their physical, chemical and mechanical properties as a result of their storage and use [6].

The magnesium hydroxide suspension has to have a high solid loading and be reasonably stable with adequate fluidity to prevent aggregation and sedimentation of particles over a broad range of solid loading. To produce a pumpable slurry, the addition of a dispersant is typically required to lower the slurry viscosity. Unless the suspension is stable, the solid will have the tendency to settle to the bottom of the storage vessel, and the settled solids can be easily re-suspended and do not form a hard cake at the container bottom. Fundamental aspects as the interaction of magnesium hydroxide slurry in novel dispersants are studied to understand the mechanical behavior of these slurries.

In this study, four types of dispersants were mainly used based on the previous studies [7]. The influence of four types on the rheological behavior of the slurry was studied in detail. Sodium citrate (Na-Cit) with molecular weight M ~ 258 Da, sodium phosphonate (Na-Phos) with molecular weight M ~ 600 Da – 800 Da, sodium lignosulfonate (Na-Lig) with molecular weight M ~ 35 kDa – 45 kDa and calcium lignosulfonate (Ca-Lig) with molecular weight M ~ 30 kDa – 40 kDa. are the main four dispersants used in this study and their molecular structures are shown in Fig. 1(a), (b) and (c). Na-Cit, also identified by the chemical name trisodium citrate dehydrate, is a tribasic salt of citric acid. Sodium citrate is widely used in foods preservatives, beverages and various technical applications mainly as buffering or emulsifying agent. It is used medically as anticoagulant in blood storage, and for urine alkalization in the prevention of kidney stones [8].

Phosphonates are a class of chelating agents and scale inhibitors. Typical variants are aminotris(methylene phosphonic acid) (ATMP) or diethylenetriamine penta(methylene phosphonic acid

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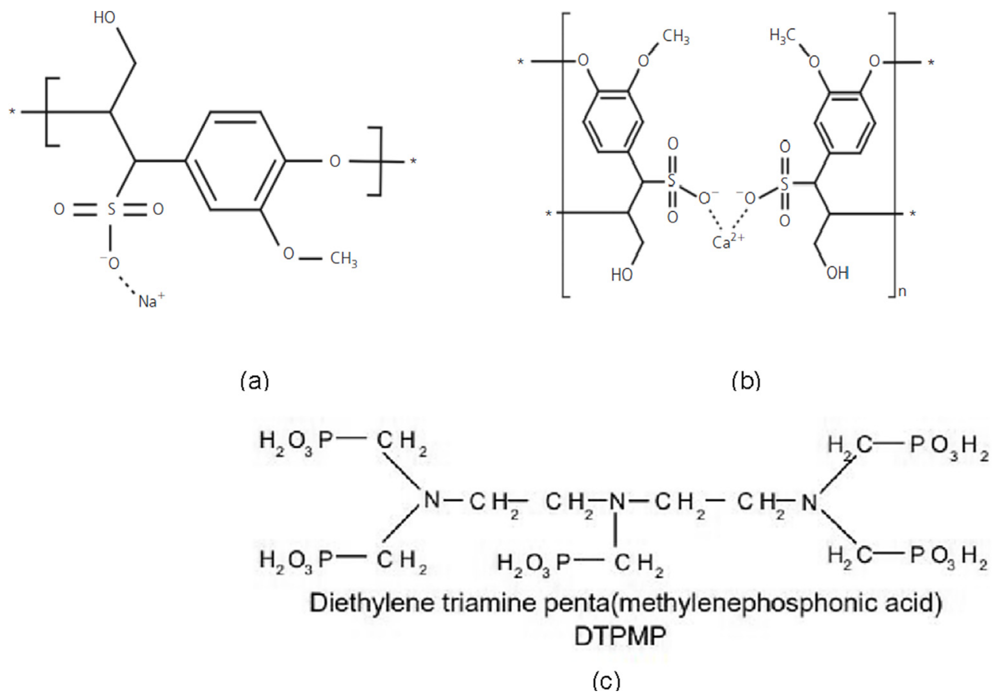

Fig. 1. Molecular structure for the used dispersants (a) sodium Lignosulfonates, (b) calcium Lignosulfonates (c) methylene phosphonic acid.

(DTPMP). They are used in household cleaning products, and as water treatment additives in many applications [9].

Lignosulfonates are by-products of the timber industry, often used in cost sensitive applications and dispersing scenarios. Sodium and calcium types are common in e.g. construction chemical formulations and products [8]. The used product exhibits a weight average molecular weight in the range of 40,000 to 65,000 with greater than 90% ranging from 1000 to 250,000 [9].

The intent of this paper is to examine the effect of small (Sodium citrate and sodium phosphonate) and large molecules (sodium lignosulfonate and calcium lignosulfonate) as dispersants for aqueous magnesium hydroxide suspension. The rheological behavior and stability of magnesium hydroxide suspensions were studied by measuring the viscosity, sedimentation behavior, pour out test, particle size, zeta potential, total organic carbon and pH.

2. Materials and experimental procedures:

For the experimental work two types of magnesium dihydroxide (MDH) $Mg(OH)_2$ were used, commercially caustic calcined magnesia (CCM)(MgO) based magnesium dihydroxide (MDHs) powder and brucite magnesium dihydroxide (MDH) $Mg(OH)_2$ powder. The mean volume diameter of particle is 6.9 μm of the brucite, and 8.5 μm of the production sample measured by Malvern Mastersizer 2000(Malvern Instruments Ltd., UK). The BET surface area is 26.01 m^2/g (ASAP2020 M, Micrometrics Instrument Corp., US). The morphology of brucite and caustic calcined magnesia (CCM) based magnesium dihydroxide (MDHs) was investigated by means of transmission electron microscopy in order to identify specific differences between the two untreated hydroxide sources. The micrographs were taken in the boundaries of the particles in order to specifically locate and identify agglomeration tendency, layer stacking thickness and circumference of the primary crystallites.

2.1. Particle size

Particle size distribution of the solid particles within the suspensions was determined using a Malvern "Mastersizer 2000" equipped with a "Hydro 2000G" dispersion unit (Malvern Panalytical). Result calculation is based on "Mie theory" for laser scattering and a general purpose model with enhanced sensitivity for irregular shaped particles was applied. The measurements were conducted in water, where several drops of suspension were introduced into the dispersion device until the signal to noise ratio was within the acceptable limits. Results are reported as volume based median values (D_{50}).

2.2. Viscosity measurements

Viscosity measurements were performed using a Brookfield LV series viscometer instrument from Brookfield Engineering Labs. All samples were investigated with spindle LV4 or V73, depending on their dynamic viscosity magnitude range, ramp procedure, room temperature, time.

2.3. Sample preparation

200 g of 40% (w/w) magnesium hydroxide slurry were homogenized by an IKA Ultra Turrax (T18, digital) at 10.000 rpm for 60 s. When an addition of dispersant was undertaken, the dispersant was also introduced with the help of 10.000 rpm Ultra Turrax dispersion. Prior to measurement the samples were furthermore left to relax for another 5 min before starting the measurement cycle (up/down shear rate ramp). Dispersant dosages were lying in the range of 0.01% to 0.7% (dosage by weight of solid hydroxide).

2.4. Pour out test

60 mL polypropylene screw cap bottles were filled with approx. 50 g of sample and the corresponding weight and filling height of

the sample was recorded. The samples were left untouched for 14d until the flow out behavior was tested. First, the height of the sediment was noted vs. the total height of suspension. Secondly, the bottle was opened and the suspension was left flowing out for approximately 30 s. The corresponding residual weight of the bottle and remaining suspension was noted. Then the fluid suspension was put back into the bottle and the whole sample was shaken up. Subsequently the second pour out was undergone for another 30 s and again the remaining weight of the bottle and sample was determined. The calculation of the pourable portion of the sample is solely based on the sample weights after deduction of the bottle and the screw cap.

2.5. Total organic carbon

TOC measurements were conducted on a "liquiTOC II" machine from Elementar Analysensysteme GmbH to determine the distribution of the dispersants within the solid liquid matrix. In order to calculate the adsorbed amounts of dispersants, the carbon contents for the magnesium hydroxide suspensions and the dispersants were determined first and afterwards the mixture of magnesium hydroxide with 7% (w/w) dispersants were investigated. The supernatants of all suspension samples were gained by centrifugation at 8500 rpm during 10 min using a Biofuge primo R centrifuge from Heraeus.

The dispersants were pre-diluted to approx. 2% (w/w) solutions. Then all liquids (suspension supernatants and dispersant solutions) were further diluted in a way that 15 mL of 0.1 M hydrochloric acid was added to 2.5 mL of liquid sample before introduction to the TOC machine.

2.6. Zeta potential

Zeta potential determination was conducted on a DT1200 electroacoustic spectrometer from Dispersion technology. 380 g of a 40% (w/w) magnesium hydroxide suspension was introduced into the machine and 30 mL of dispersant solution was titrated to the sample during the running measurement. For the brucite based suspension the dispersant dosage range was 0.07% (w/w) and for the caustic magnesia based suspension the dosage range was 0.7%. During the measurement the sample was constantly stirred at 250 rpm to prevent settling and to ensure distribution of the dispersant homogeneously.

For the calculation of the zeta potential a density of the solid of 2.39 g/mL was assumed and the measured particle size distribution was also incorporated. During the titration the evolution of pH was monitored and recorded simultaneously.

3. Results and discussion

3.1. The morphology of brucite and CCM MDH

Electron micrographs were taken on a JEOL JEM-1400 120 kV TEM using a hydrophobically modified copper grid sample holder. As shown in Fig. 2 very distinct and finely structured features of platelets and hexagonal sheets for the hydrated CCM sample, whereas the brucite sample showed mainly irregular shapes and bold structures which indicated lower crystallinity and lower ordering of the crystallites.

The morphology of brucite MDH showed no significant preference in regard of agglomeration. Moreover, no noteworthy hexagonal features were observed in the sample. During the preparation of the respective TEM specimen the thin hydroxide sheets preferably aligned horizontally towards the surface and the overall appearance could be described as amorphous with diameters of visible particles greater than 100 nm. The crystallites of CCM MDH were of the order of approx. 100 nm in diameter and the stacking thickness of layers was clearly visible and estimated at approx. 30–50 nm. Agglomeration was pronounced at the edges and corners of CCM MDH and yielded a collapsed "house of cards" like structure, which was not observable at the brucite based hydroxide.

3.2. Effect of dosage on the particle size of the slurry

In contrast to the findings of the morphological investigation, the particle size determination via laser scattering technique indicated a coarser particle size for the untreated CCM MDH vs the brucite sample (Table 1). The median volume based D_{50} was found to be 6.9 μm for the naturally occuring hydroxide and 8.7 μm for caustic hydrated material. Therefore the difference of both material sources was not very pronounced.

Additional admixture dosage was creating a range of median D_{50} results from 5.4 μm to 9.6 μm (min/max) and the average result for the whole dosage range and admixture matrix was 6.4 μm in the case of brucite MDH. The respective values for the hydrated sample were 4.6 μm and 10.4 μm (min/max), as well as 7.7 μm as average result for an addition of a certain dispersant at a certain dosage amount. The most influence of dispersant type was observed for Na-Cit in the case of brucite MDH and for Na-Lig in the case of CCM MDH.

In general, higher doses of admixture yielded slightly lower median D_{50}, probably due to better stabilization or dispersion and lower agglomeration during the measurement.

In conclusion it has to be stated that the laser scattering results were complementary to the morphological findings, as both methods show different aspects of size and shape of both materials (bulk vs. boundary).

3.3. Effect of dispersant types and dosages on rheological behavior:

The viscosity of the magnesium hydroxide suspension was measured at 250 rpm for two types of magnesium hydroxide, namely, brucite MDH and CCM MDH by dispersing it with different types as Na-Cit, Na-phos, Na-Lig and Ca-Lig. For the determination of general rheological properties and the overall dosage range of the two different hydroxide sources, a stirring speed applicable to typical stirring conditions in processing steps was chosen (e.g. stirred vessel at 250 rpm).

Furthermore, the spindle selection was accomplished by some minor testing to be suitable over the entire measurement range and all admixture types. Spindle V73 was chosen in the case of brucite, whereas LV4 was used in the CCM MDH experiments, according to higher viscosity in some dosage and stirring speed situations.

All samples underwent a certain shear scenario according to an upwards and downwards ramping procedure to yield a reproducible shear history for the samples during the measurement [10].

Therefore the highest shear rate and stirring speed of 250 rpm lay within the center of the programmed ramp. The pure hydroxide samples yielded dynamic viscosity values of approx. 200 cP and 400 cP as shown in Fig. 3.

Generally a dosage of organic compounds was expected to lower the viscosity of the magnesium hydroxide suspension. This assumption was confirmed for the brucite based system at dosages up to 0.07% already. In contrast, the anticipated behavior was not observed for dosages of 0.01–0.07% of all organic admixture systems in the caustic hydrated hydroxide. Therefore, the dosage range was increased by a factor 10. Subsequently all discussion related to brucite vs CCM are based on the different dosage ranges

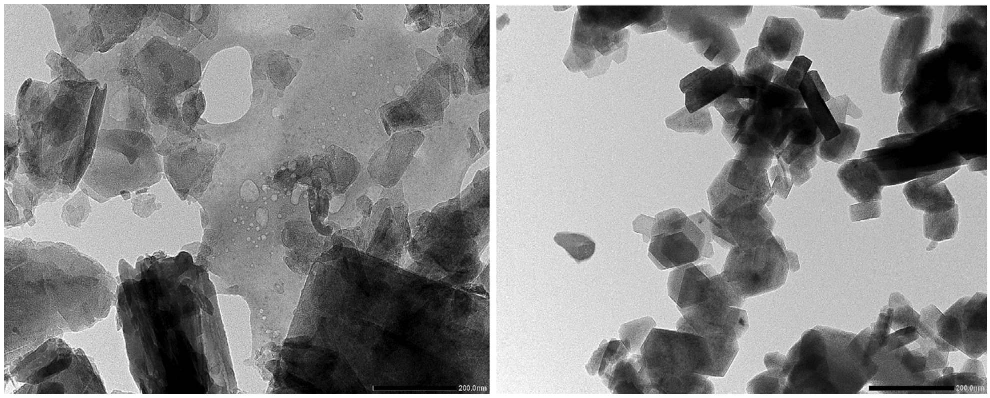

Fig. 2. TEM image of brucite MDH (left) and CCM MDH (right) (magnification 50 k, scale 200 nm).

Table 1
Influence of admixture type and dosage on median particle size D_{50} of brucite and CCM MDH suspensions.

Brucite					CCM MDH			
Dosage [%]	**D_{50} / μm**							
	Na-Cit	Na-Phos	Na-Lig	Ca-Lig	Na-Cit	Na-Phos	Na-Lig	Ca-Lig
0.0	6.9	6.9	6.9	6.9	8.7	8.7	8.7	8.7
0.1	9.6	6.7	6.5	5.9	7.3	8.2	6.0	9.5
0.3	5.5	7.1	5.8	5.4	7.5	8.5	5.1	10.1
0.5	7.6	5.9	6.2	5.8	7.4	7.9	5.7	8.6
0.7	6.0	5.9	6.1	5.6	7.7	10.4	4.6	8.1

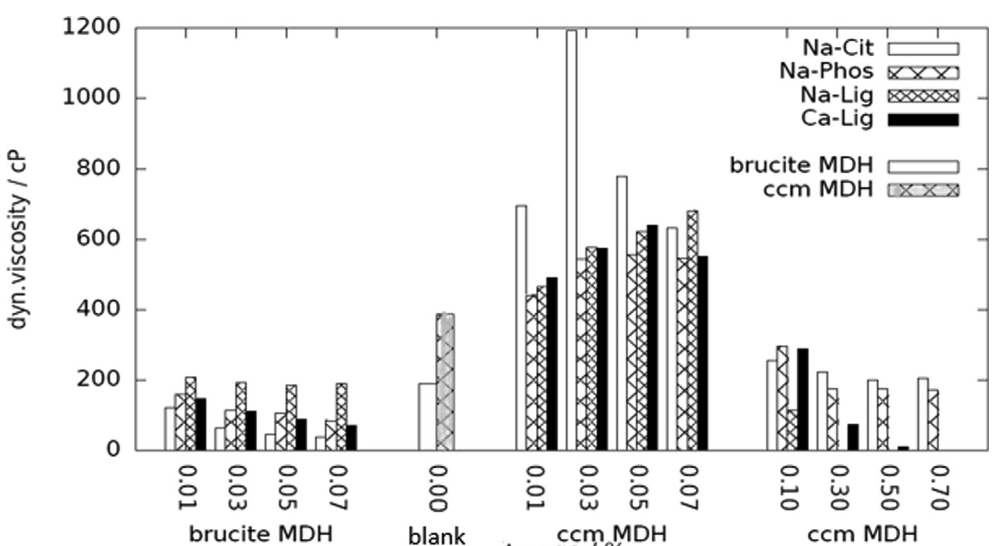

Fig. 3. Influence of admixture type and dosage on median dynamic viscosity of brucite and CCM MDH suspensions at high shear (250 rpm) [1,12].

of 0.01% – 0.07 vs 0.1% – 0.7% in the case of magnesia based magnesium hydroxide.

The variation of stirring rate of the slurry affected the rheologyical behavior of the slurry as at 25 rpm stirring rate the maximum value of viscosity approx. 1700 cP, while at 250 rpm stirring rate the maximum value was 200 cP. It indicates that at high shear the flowability of the slurry increases and the viscosity decreases.

The rheologyical behavior of small molecules group was remarkable than in large molecules group at same stirring rates. However, the rheologyical behavior of CCM MDH at stirring rate 25 rpm the maximum value of viscosity was 2400 cP, while at 250 rpm stirring rate the max value was 380 cP.

It is revealed that large molecules decrease the viscosity of CCM MDH suspension more than small molecules. In general, the low stirring rate causes rise in viscosity and poor liquidity. As reported, the increase of the dosages % of dispersants resulted in decreasing the viscosity of suspension.

3.4. Sedimentation behavior

The sedimentation behavior of brucite MDH and CCM MDH with different dispersant dosages and types is shown in Fig. 4. The sedimentation height shows reverse trends with the increase of dispersants dosage, the sedimentation height slightly decreases as the dispersants dosage is increased, leading to a more

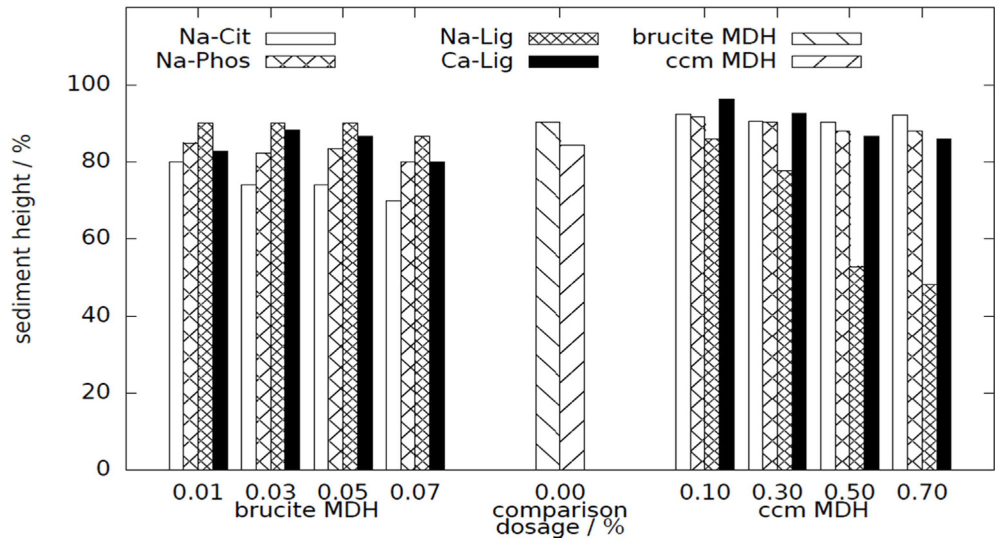

Fig. 4. Influence of admixture type and dosage on sedimentation height after 14 d storage of brucite and CCM MDH suspensions.

and more dense particle packing gradually. This means that the suspensions become more and more stable. For example, brucite MDH suspension with Na-Cit at 0.07 wt% the sedimentation height % is 70, while for CCM MDH suspension with Na-Lig at 0.7 wt% the sedimentation height % is 48.

3.5. Pour out test

First pour out test was measured for brucite MDH samples without shaking it and the amount of slurry poured out of the bottle was calculated. As shown in Fig. 5 that at 0.07 wt% dosage of the dispersant Na-Cit was 95.6. Then the bottle was shaken to stir the remaining slurry for one revolution. The remaining slurry was then poured out over approx. 30 s. The amount of all slurry poured out for both pours can be determined as the second pour out. At 0.07 wt% dosage of the dispersant of Na-Cit was 99.4.

On another note, for the first pour out test for CCM MDH samples prior shaking it, the amount of slurry poured out of the bottle was calculated. As shown in the Fig. 6 that at 0.7 wt% dosage of the dispersant of Na-Lig was 50. As the second pour out, 0.7 wt% dosage of the dispersant of Na-phos is 99.

For brucite samples the sodium citrate is best dispersing for pour out test and the sedimentation behavior of the all brucite samples are soft. While, for CCM MDH samples the Na-Phos is the best for pour out test, the pour out behavior of Na-Lig and Ca-Lig are higher than Na-phos but, the sedimentation behavior are so sticky and Na-Lig formed hard a cake at the bottom of the vessel.

3.6. TOC (total organic carbon)

The TOC analyses are conducted to measure the organic carbon concentrations of the studied dispersants as Ca-Lig [11–12] from large molecules and Na-Phos from the small molecules group were measured. It can be seen from Table 2, that the total organic carbon (TOC) of dosage 0.7 wt% of dispersant in CCM MDH system is mea-

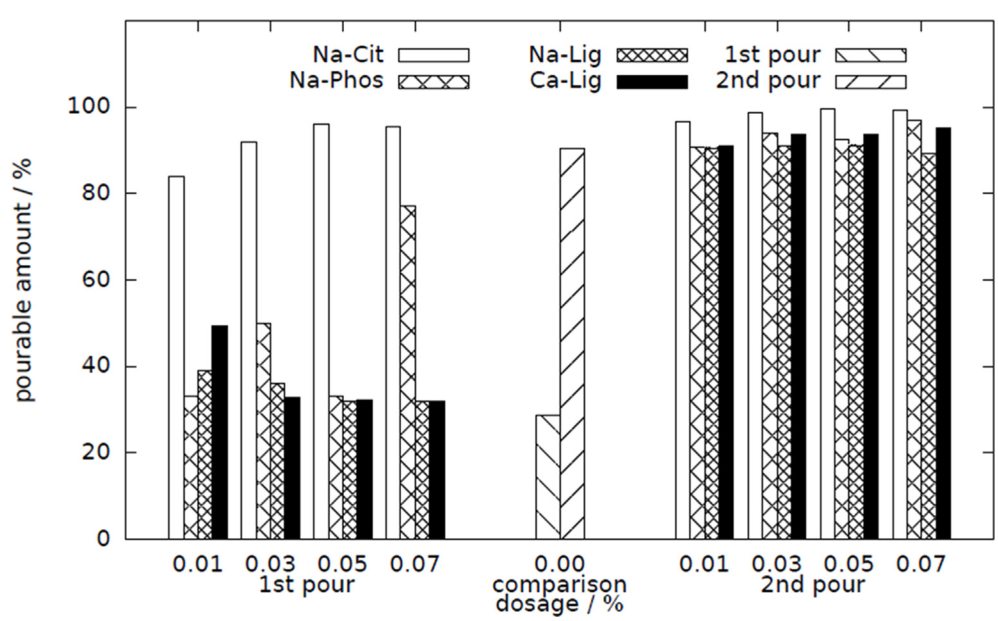

Fig. 5. Influence of admixture type and dosage on flowability after 14 d storage of brucite MDH suspension.

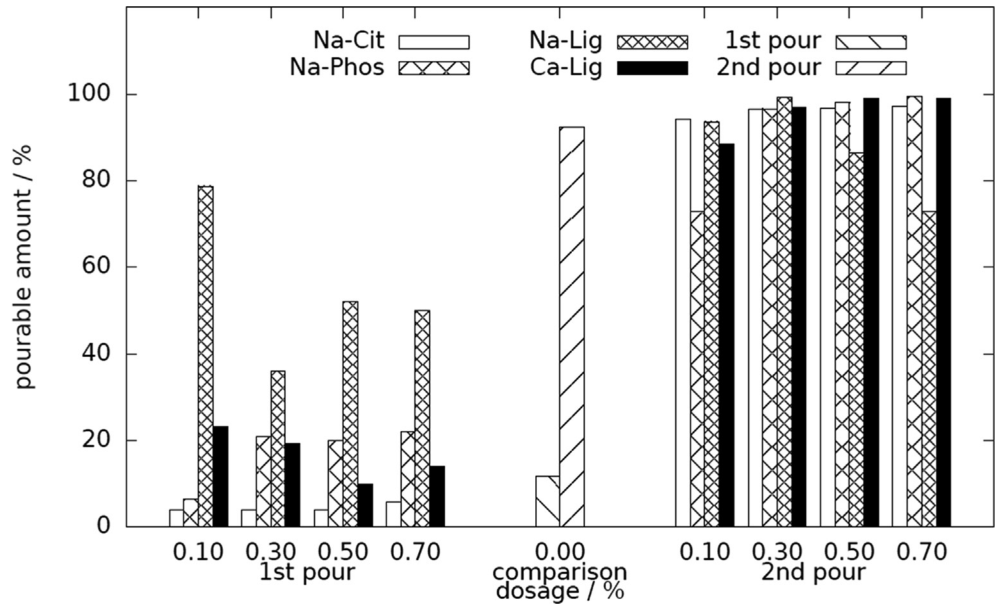

Fig. 6. Influence of admixture type and dosage on flowability after 14d storage of CCM MDH suspension.

Table 2
Adsorptive behaviour of Na-Phos and Ca-Lig determined by TOC in the system water vs CCM MDH suspension at 7% admixture dosage.

	CCM MDH					
	TOC / mg/L		**dilution factor**		**C content / g/L**	
System	Water	CCM	water	CCM	water	CCM
Na-Phos	96.7	42.1	292.0	249.6	28.2	10.5
Ca-Lig	87.4	287.6	3353.2	250.0	293.1	71.9

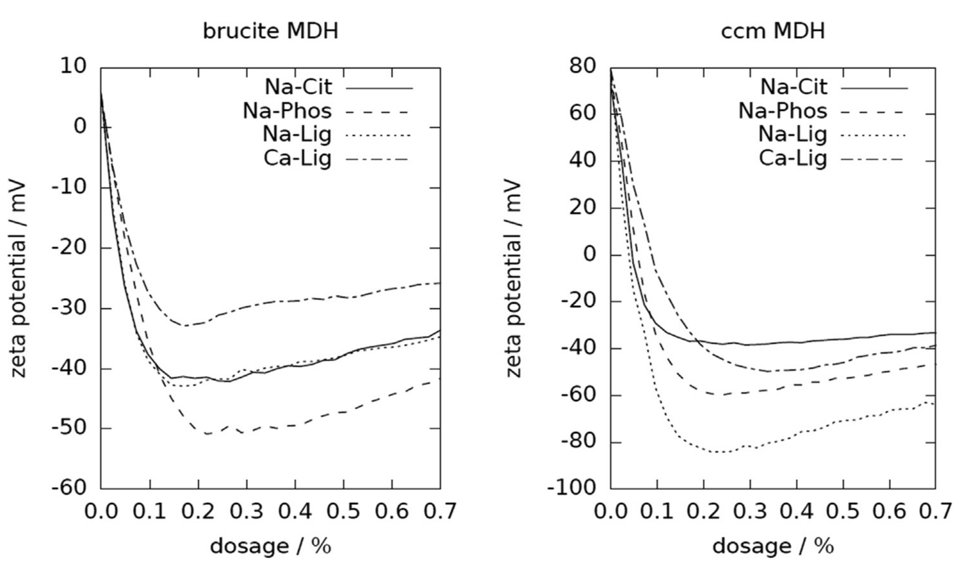

Fig. 7. Influence of admixture type and dosage on zeta potential of brucite and CCM MDH suspensions [13].

sured as Ca-Lig is 71.8 g/l, while Na-phos is 10.4 g/L. However, the total organic carbon of dispersants in water system is 293.2 g/L for Ca-Lig and 28.2 g/L for Na-phos. This result indicates the amount of non adsorbed carbon% of Ca-Lig is 24.5% and Na-phos is 37%. It is revealed that Ca-Lig has more absorbtion for carbon content than Na-Phos.

3.7. Zeta potential

Zeta potential measurements were performed to determine the surface charge of the magnesium hydroxide particles in the presence of varying dosages of the dispersants [13]. The brucite MDH suspension of 40 wt% was treated with Na-Cit, NA-phos, Na-Lig

Table 3
Influence of admixture type and dosage on pH of brucite and CCM MDH suspensions.

	brucite				CCM MDH			
Dosage [%]	**pH**							
	Na-Cit	Na-Phos	Na-Lig	Ca-Lig	Na-Cit	Na-Phos	Na-Lig	Ca-Lig
0.0	9.8	9.8	9.8	9.8	11.6	11.6	11.6	11.6
0.1	10	9.9	10	9.8	12.5	12.2	12.1	11.5
0.3	10.3	9.9	10	9.8	12.5	12.1	12.0	11.3
0.5	10.4	10	10	9.8	12.4	12.4	11.9	11.2
0.7	10.5	10.1	10.1	9.8	12.4	12.5	11.8	11.1

and Ca-Lig. Addition of increasing amounts of dispersant resulted in a decrease of the zeta potential as shown in Fig. 7. The decrease is strongest for Na-phos and less strong for Na-Lig. For example, addition of 20 mL of Na-phos decreases the zeta potential only to approximately −47.3 mV whereas the same amount of Na-Lig produces zeta potential of about −41.7 mV.

The CCM MDH of 40 wt% was titrated with Na-Cit, NA-phos, Na-Lig and Ca-Lig. Addition of increasing amounts of dispersant resulted in a decrease of the zeta potential as shown in Fig. 7. The decrease is strongest for Na-Lig and less strong for Na-phos. For example, addition of 20 mL of Na-Lig decreases the zeta potential only to approximately −71.3 mV whereas the same amount of Na-phos produces zeta potential of about −52.7 mV.

3.8. pH measurements

Variation of pH values and dosages % of disperesnets added to brucite MDH and CCM MDH.As the pH values of the brucite MDH samples are ranged from (9.5–11.3) as shown in Table 3.But pH values of the CCM MDH samples are different from each other as the range (10.4–12.4). The pH values for Ca-Lig and Na-ligdecreases, while Na-phos and Na-Cit increases in the production samples. The large molecules group decreases.

4. Conclusion

In this study, the influence of dispersants on the rheological behavior of magnesium hydroxide slurry was investigated. From the results of this study, Four types of dispersants were used to obtain most stable magnesium hydroxide slurry with low viscosity. Ca-Lig was the most suitable dispersant regarding viscosity, flowability and pH. The viscosity of magnesium hydroxide slurry decreases as the dosage of dispersants increases. Large molecules of dispersants decreases pH but the same molecules group of dispersants increases pH.

Declaration of Competing Interest

The author declare that there is no conflict of interest.

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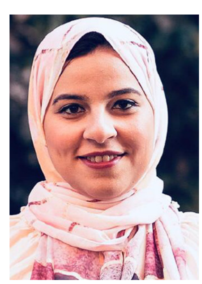

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