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The effect of grinding type on roughness and coagulation of magnesite particles

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Abstract: Coagulation is one of the most important processes for aggregating the fine particles in a dispersed system via different mechanisms. Many factors such as particle size, morphology, pH, coagulant type, and concentration may affect particle-particle interactions, which will then be effective on their coagulation characteristics. In this study, the effect of grinding type on the roughness of magnesite particles under 38 microns was evaluated for its role in the coagulation of particles under different salt types and concentrations. The results of grinding tests showed that rougher particles could be obtained with a rod mill compared to the particles ground in a ball mill. After grinding of particles, an increasing trend was obtained in terms of settling rate for different salts in an order of $NaCl<CaCl_2<AlCl_3$ during coagulation tests under all concentrations (10^{-3}, 10^{-2}, 10^{-1} and 1 M) at a constant pH value as 8.01±0.2. In sum, the results of this study clearly showed that adjusting the grinding conditions is not only effective on the size and morphology of the particles but also plays a significant role in particle-particle interactions and accordingly their coagulation characteristics.

Keywords: grinding, roughness, coagulation, salt

1. Introduction

Coagulation and flocculation are used to remove the insoluble and colloidal heavy metal precipitates formed during the precipitation step. By way of explanation, coagulation is the process of making the particle less stable by neutralizing its surface charge, thus encouraging the initial aggregation of colloidal and finely divided suspended matter. It leads to bring particles into closer contact in the form of bunches and aggregates (US Army Corps of Engineers, 2001). In other words, coagulation is the process of stabilizing a given suspension or solution. Moreover, in recent years, there is an increasing trend on the number of researches about that process, mainly due to its low cost, ease of operation, and energy savings (Bratby, 1980; 2016; Xue et al., 2021; Vasiljevic et al., 2023).

These processes enhance operational efficiency by facilitating the aggregation of very fine-sized particles, particularly before sedimentation and filtration (Khazaie et al., 2022). However, for the success of this process, not only chemical parameters but also the physical properties of particles should be considered (Vaziri Hassas et al., 2021, Uysal et al., 2021, Celik et al., 2024). While numerous studies in the literature focus on chemical parameters affecting coagulation efficiency, there is limited information available regarding the influence of particle morphology on these processes (Vaziri Hassas et al., 2021).

However, in literature, the effect of particle morphology and the variation on particle size distributions is generally investigated based on grinding type and afterwards their contribution on flotation characteristics of different samples. For instance, Gu et al., (2025) reported that upon controlling of the grinding parameters for particle shape, the flotation kinetics could be adjust to higher values for lower ash coal particles. Bu et al., (2019) investigated the effect of both dry and wet grinding

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conditions of coking coal on their responded flotation recovery, and induction time values. They found that while more irregular and smooth surfaces were obtained by wet-ground particles, conversely rougher particles were obtained by dry grinding process. Accordingly, it was presented that shorter induction times and higher flotation recoveries were obtained by wet-ground particles due to the better particle-particle and particle-bubble interactions, which were attributed to different breakage mechanisms. In another study by Ulusoy and Bayar (2022), the effect of different grinding techniques such as vibrating disc and ball milling on the morphology of quartz particles in two size fractions (coarse and fine) was investigated with dynamic image analysis. It was found that more rounded particles were obtained for vibratory disc compared to ball milling regardless of particle size range. This in turn clearly presented that the selection of grinding medium is an important parameter not only for particle size in the same grinding time but also the morphology of the particles. In addition to the particle morphology, Bu et al., (2020) investigated the effect of wet and dry grinding conditions of coal in a laboratory scale ball mill in terms of their particle size distribution. They characterize the distribution by adapting commonly used distribution functions as Gates-Gaudin Schumann (G-G-S), Rosin-Rammler-Bennett (R-R), Gaudin-Melody (G-M) and Swrebec functions to find the fitting performance for cumulative particle size curves for the products obtained from different conditions. As a result, they found that while R-R function could be well-applied in a short grinding time, the G-G-S and G-M function became more appropriate for longer grinding times. This in turn showed that upon the variation of dominant breakage mechanism from nipping to abrasion, chipping or attrition during grinding processes, the response of particle size distribution will differ and can be explained with the help of different functions. In another study by Ni et al., (2022) the grinding characteristics of spent carbon anode from aluminum electrolysis in both ball and rod mill were investigated in terms of variations on shape factor and surface roughness of ground samples. They found that while particles with higher elongation ratio and roughness were obtained by rod milling of samples, ball milling provided smooth surfaces due to the dominant abrasion mechanism, which in turn resulted in finer sized particles. Apart from these studies, a very recent review paper by Sun et al., (2023), reported that the contribution of preparation methods, assay a significant importance on particle roughness values, which come into the front while evaluating the particle-bubble interactions, which in turn, flotation recoveries and kinetics. In addition to their reports on experimental results, they also showed that a considerable decrease could be obtained for energy barrier heights upon higher roughness values, which in turn resulted in higher flotation recoveries and lower induction times.

Considering those findings and the lack of knowledge in literature, the effect of particle roughness on coagulation processes would provide a different point of view on adjusting the grinding conditions not only for their flotation characteristics but also their coagulation and flocculation characteristics upon the addition of relevant chemical to the system. In this context, the most common traditional laboratory procedure for assessing solid-liquid separation is the batch cylinder test. Batch settling tests (BSTs) are used to obtain detailed information on the sedimentation behaviour (e.g. settling velocity) of suspensions. Generally, a suspension is flocculated within a measuring cylinder and the settling rate is determined from the rate of fall of the mudline. With a given initial settling rate and the value of critical point, BSTs have been a major step in sizing thickeners and clarifiers. These tests are important steps in estimating compressive yield stress and hindered settling function. It is also reported that the coagulation of particles before filtration increases the filtration efficiency (Vaziri Hassas et al., 2021).

Thus, in this paper, the effective role of particle roughness parameter for magnesite mineral obtained from different grinding conditions will be examined through coagulant type and concentration, which in turn would determine the effect of roughness in particular for particle-particle interactions constituting the heart of coagulation processes.

2. Materials and methods

Magnesite samples used in this study were obtained from KÜMAŞ Magnesite Co., Kütahya, Turkey. X-ray diffraction Analysis (XRD) using the Rigaku-Miniflex XRD unit determined the crystallinity of the sample. The radiation applied was CuKα from a long fine focus Cu tube, operating at 40 kV and 15 mA (i.e., 600 W x-ray tube) while the 2θ range (5-90°) and the scanning speed were adjusted to 2°/min. The

XRD results of the sample revealed that the samples were mainly composed of magnesite with impurities in trace quantities (Fig. 1).

Before grinding tests, the size distribution of the feed sample was determined via sieves with apertures of 1, 0.500, 0.212, 0.150, 0.106, 0.075, and 0.038 mm. As shown in Fig. 2, the d_{80} of the sample was very close to 0.300 mm while the d_{50} of the sample was found as 0.150 mm. Considering these values, the particle size distribution of crushed material indicated that the presence of fines (<0.038 mm) in the feed material was negligible. Following the determination of feed size, to adjust the size range suitable for coagulation tests, the sample was ground under 0.038 mm by adapting different grinding medium types such as rod and ball mill. In these tests, in addition to size reduction, the contribution of the grinding type on the roughness of the particles was investigated.

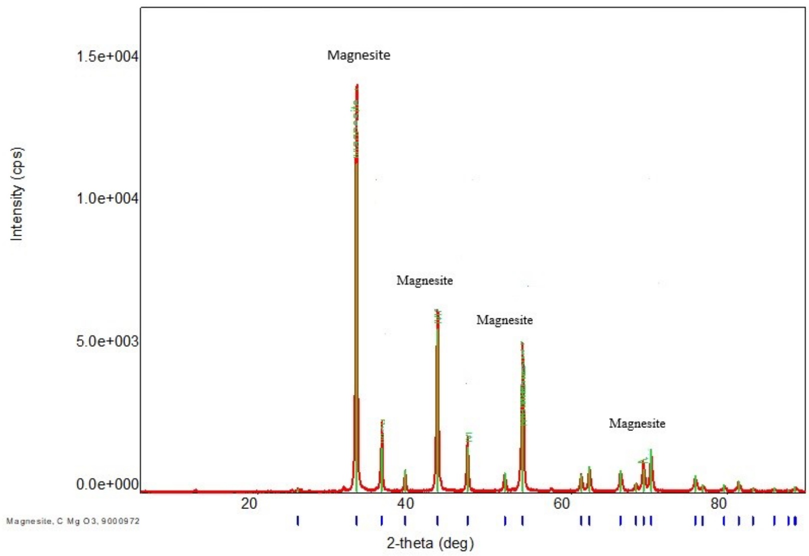


Fig. 1. XRD pattern of the magnesite sample

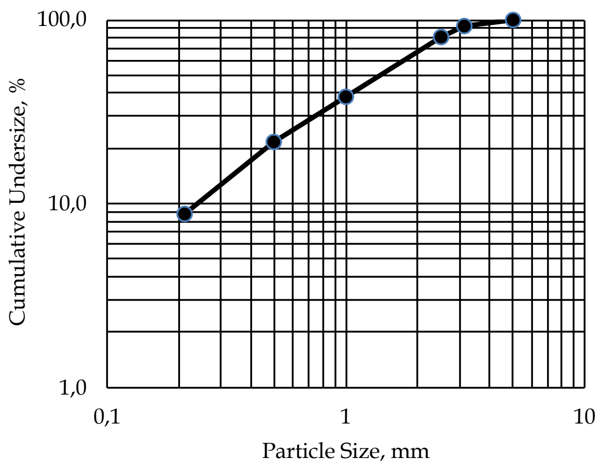


Fig. 2. The size distribution of feed sample to grinding processes

During these tests, a laboratory-type ball mill with a volume of 5224 cm^3 (D=19.5 cm, H=17.5 cm) was used with a mixture of steel balls (which were 30, 25, and 20 mm in diameter and 816 g in weight), and a laboratory-type rod mill with a volume of 7850 cm^3 (D=20 cm, H=25 cm) with steel rods (which were 31.7 mm, 24.6 mm, 15.9 mm, and 9.8 mm in diameter). In addition, as reported in a recent paper (Gu et al., 2025), the critical speed of the mill is another important parameter to be considered for the grinding processes and affects not only particle size but also the shape of the particles. The diameter of the laboratory-type ball and rod mills was 19 cm, and using that value the critical speed of the mill was calculated using Eq. (1).

$$Nc = 420/\sqrt{(D)} \tag{1}$$

Nc is the critical speed of the mill (rpm), and D is the diameter of the mill (cm). All the grinding tests were performed at 75% of the critical speed of the mill, at around 72.3 rpm.

The mill feed sub-samples were ground for different durations under both grinding conditions, after which material below 0.038 mm was separated from each grinding step material using wet screening. Consequently, size analysis was performed using the Malvern Particle Sizer 3000 with a wet analysis unit (Fig. 3.).

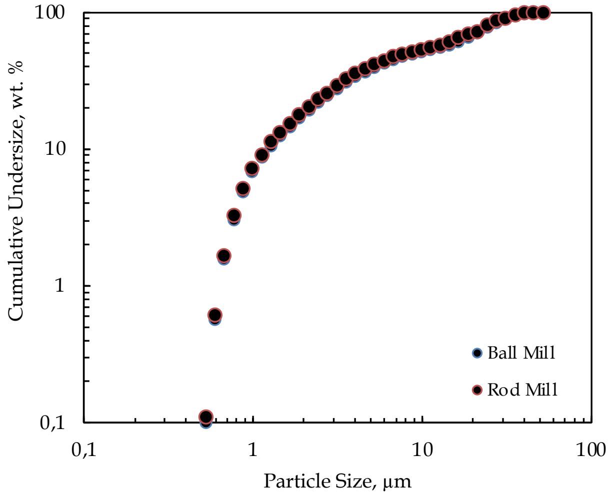


Fig. 3. The size distribution of the ground sample using the Malvern Particle Sizer 3000 with a wet analysis unit.

As can be seen from the particle size distribution (PSD) graphics of ground particles in the ball and rod mill, while the d_{80} sizes were around 25 microns for each one, the d_{50} sizes of the particles were measured as 6 microns on average. From this point of view, it can be concluded that the roughness can be studied while the effect of the size distribution for different grinding mediums can be dismissed during the evaluation of results.

2.1. Profilometer

The surface roughness values of pellets formed with the ground particles with different grinding mediums were determined with the contact profilometer device (Mitutoyo SV-2100 model, Japan) (Fig. 4.). The details of the experiments were reported in one of our previous papers (Uysal et al., 2021).

Fig. 4. The photo of profilometer from sideview (left) rod and ball mill ground magnesite particles in pellets formed (right)

2.2. Settling tests

For each sedimentation test, 100 ml of deionized water was used at a solid concentration ratio of 1%. Sedimentation tests were applied to samples taken from two different mill outputs to observe the effect of grinding and morphology on coagulation. All tests had the same conditions for both ball and rod mill output samples. Parameters considered included pH and salts with different concentrations. The selection criteria for these parameters can be well ascribed to the two main mechanisms during coagulation as neutralization of surface charge and double-layer compression. Thus, the variation on the valence of salts are the most effective parameter for double-layer thickness (Partheniades, 2009). To induce coagulation and observe the effect of pH on the prepared salt solutions was initially measured before and after the addition of magnesite sample. As mentioned above, in the first series of tests, the effect of pH value was examined to determine the effective pH value for the coagulation of magnesite. Different salt types and concentrations were considered to observe the effect of the second mechanism on coagulation. In these tests, the pH was adjusted using NaOH to create basic conditions, with a pH value set at approximately 8. The second part of these tests involved different types of salts (NaCl, $CaCl_2$, and $AlCl_3$) at various concentrations. Sedimentation tests were conducted for all salt concentrations.

2.2.1. Settling rate tests

These experiments were conducted in a cylindrical glass cell of 100 mL (Fig. 5.). In all experiments, the total solution (solid+liquid) was prepared in the form of 100 mL mixtures at a certain % solids by wt. as 1% and salt concentrations at a natural pH of respective mineral suspensions using distilled water (8.01±0.03). The same procedure was followed with the one that was reported in a previous publication (Celik et al., 2024; Li et al., 2024). The average values obtained were recorded for constructing distance-time curves. The average settling rate in that particular collector concentration and percentage solids by wt. was obtained from the slope of the curve, and concentration-dependent logarithmic "Sedimentation Rate" curves were drawn.

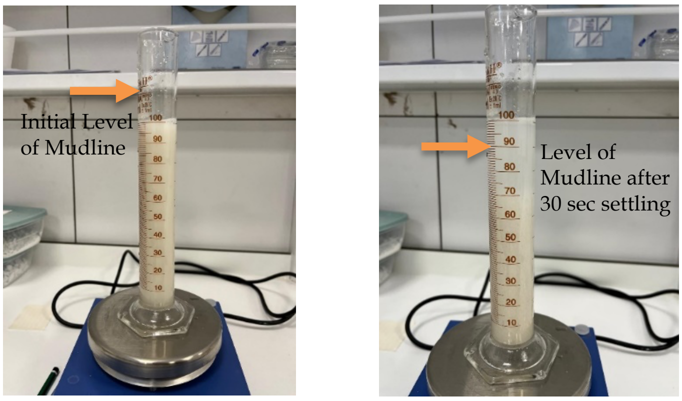


Fig. 5. Settling rate experiments of magnesite particles

2.2.2. Jar tests

The amount settled against settling time was performed in a 200 mL glass graduated cylinder of 40×40×320 mm in size, which has a tap to discharge liquid at a level corresponding to approximately 100 mL. The same procedure was followed with the one that was reported in a previous publication (Celik et al., 2024). The prepared suspension was transferred into the graduated cylinder and the time at which suspension became stable was recorded, e.g. 30 sec. for -38 µm sample. The valve was opened and the 100 mL portion of it discharged, filtered and the remaining solid on the filter paper was dried in an oven set at 105oC upon which the agglomeration efficiency (Ec) was calculated using Eq. 2.

$$E_C = \frac{W_i - W_f}{W_i} x100 \quad (2)$$

where Wi represents the initial % solids corresponding to the suspension volume remaining in the 100 mL filtrate and W_f symbolizes % solids in the suspension remaining in the filtrate after the settling process.

3. Results and discussion

3.1. Profilometer

Roughness characterization includes measurements such as average roughness (Ra) and other roughness parameters like skewness (Rsk) and kurtosis (Rku). While the average roughness value is generally used to express the roughness characteristics of materials, other parameters like skewness and kurtosis are used for more specific surface characterization. The roughness values of magnesite samples are provided in the table below.

Table. 1. Roughness Values (Ra) of Magnesite samples

Material type and grinding media	Average roughness (Ra) Values (μm)
Magnesite (Rod Mill)	2.2391
Magnesite (Ball Mill)	1.8835

3.2. Coagulation tests

3.2.2. Settling rate tests

Before tests for the effect of salt type, and salt concentration on the coagulation of fine-ground magnesite particles with varying roughness values, different series of tests were adapted as a function of pH and solid wt. % in the suspension with distilled water to determine the optimum conditions for targeted tests. In these primary tests, the settling rate method was used. The results of these tests are shown in Fig. 6. respectively.

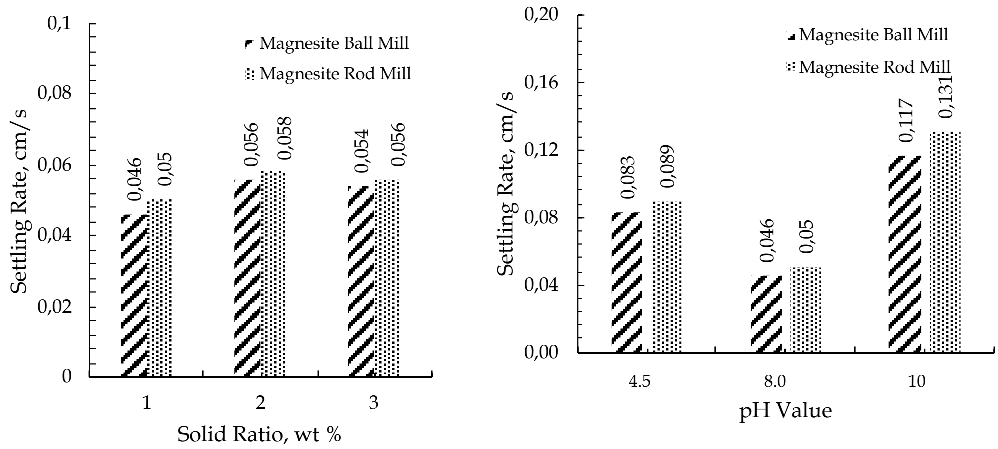


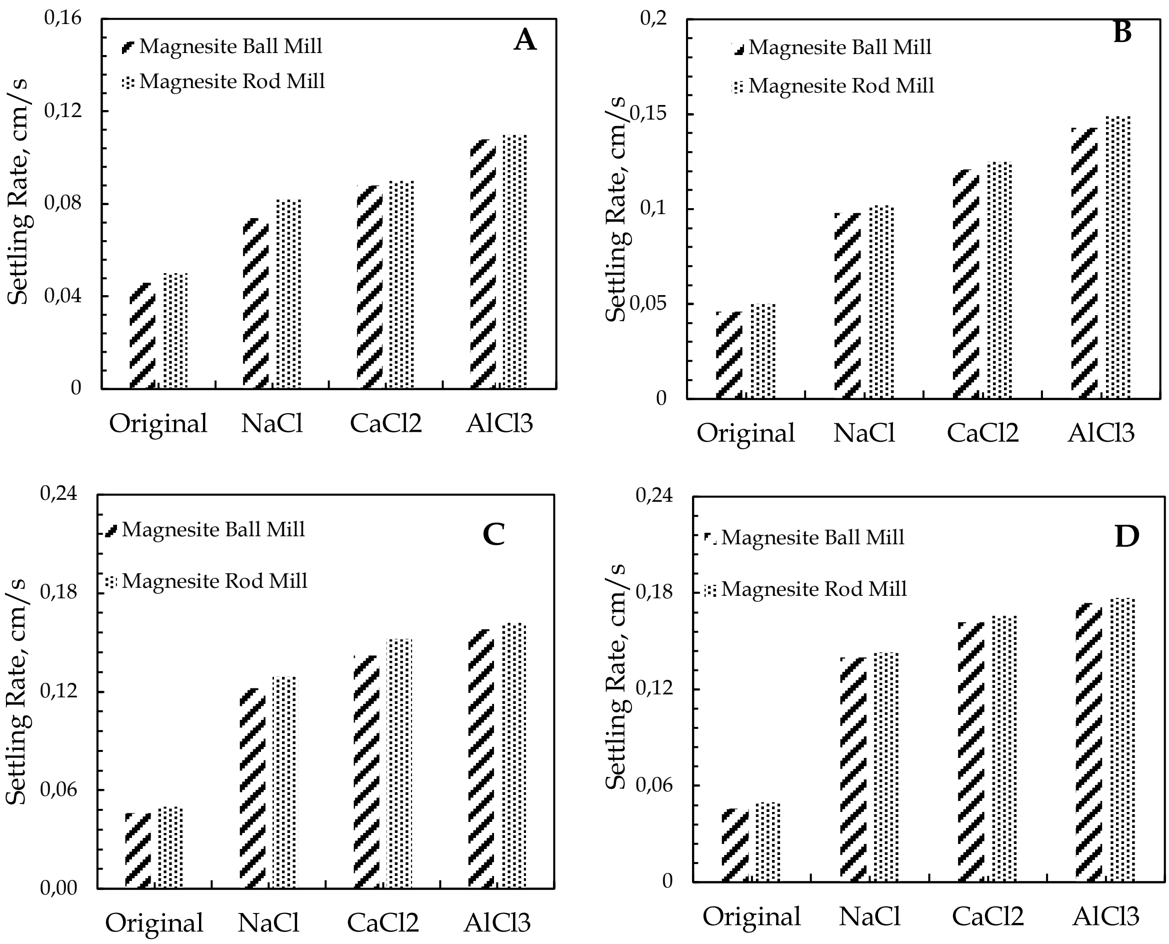
Fig. 6. The effects of solid ratio and pH value on coagulation characteristics of magnesite samples ground in different grinding medium types

As can be seen from the results shown for ball and rod-milled samples, the general trend is that the rod-milled products settle faster than ball-milled products, which in turn is a function of their roughness values. On the other hand, considering the values obtained for different conditions as solid wt. percentage (1 wt. %, 2 wt. % and 3 wt. %) and pH (4.5, 8.0, and 10.0), generally, the minimum values obtained in solid wt. percentage parameters as 1 wt. % were taken as a reference for future tests to show the contribution of salt concentration and accordingly the contribution of double-layer compression for coagulation efficiency and therefore particle-particle interactions. Although lower settling rate values were obtained within acidic pH range values, remaining tests were performed at the original pH values of the samples to prevent the possible compression effect of salts during the coagulation of particles. Hence, in this way, a better comparison could be obtained for different salt types and roughness values of particles.

As mentioned above, finely ground magnesite samples in different mills presented different roughness values. To calculate the settling rate based on roughness, solutions with different salt types and molar values were prepared. These prepared solutions are respectively NaCl, $CaCl_2$, and $AlCl_3$ salt concentrations.

3.3. The effect of salt type and concentration on settlement rate of magnesite

The effects of NaCl, $CaCl_2$, and $AlCl_3$ salts on the precipitation rates of magnesite samples are compared. In this comparison, the precipitation rates of products from the ball mill and rod mill were analyzed using solutions with different molarities. The results of these tests are shown in Fig. 7. When distilled water is used, the lowest precipitation rates are observed. The results of these tests showed that while the settling rate of the samples ground via ball mill was 0.046 cm/s, and it increased to 0.050 cm/s for samples ground with rod mills. These results indicate that natural water does not sufficiently reduce the electrostatic repulsion forces between particles, hence resulting in lower precipitation rates.



Salt conc. (M)	Ball mill (Settl. rate, cm/s)			Salt conc. (M)	Rod mill (Settl. rate, cm/s)		
0 (Original)	0.046			0 (Original)	0.05		
	NaCl	$CaCl_2$	$AlCl_3$		NaCl	$CaCl_2$	$AlCl_3$
10^{-3}	0.074	0.088	0.108	10^{-3}	0.082	0.09	0.11
10^{-2}	0.098	0.121	0.143	10^{-2}	0.102	0.125	0.149
10^{-1}	0.122	0.142	0.158	10^{-1}	0.129	0.152	0.162
1	0.14	0.162	0.174	1	0.143	0.166	0.177

Fig. 7. The effects of NaCl, $CaCl_2$, and $AlCl_3$ salts on the precipitation rates of magnesite samples (salt concentrations, A: 10^{-3} M; B:10^{-2} M; C:10^{-1} M; D: 1 M; Values shown separately in Table for legibility)

When NaCl solution is used, the precipitation rates increase compared to distilled water. As can be seen from the Figure, while the product from the ball mill has a rate of 0.074 cm/s, it increased to 0.082 cm/s for the products of rod mill. This increasing trend clearly shows that NaCl salt reduces the electrostatic repulsion forces between particles, as described specifically in the literature (Zhang et al. 2017), thereby increasing the precipitation rate. $CaCl_2$ solution provides slightly higher precipitation rates compared to NaCl solution. As can be seen from Fig. 7., while the product from the ball mill has a rate of 0.088 cm/s, and the product from the rod mill, has a rate of 0.090 cm/s. This indicates that $CaCl_2$ more effectively reduces double-layer compression than NaCl, resulting in faster precipitation.

Among the various types of salts tested, the $AlCl_3$ solution yields the highest precipitation rates. For magnesite, the underflow from the ball mill records a rate of 0.108 cm/s, while from the rod mill, it reaches a rate of 0.110 cm/s. $AlCl_3$ effectively minimizes the electrostatic repulsion forces between particles, thereby maximizing the precipitation rate.

As a basic chemical knowledge, the hydrated size for alkali metal/metal counter-ions has an order: Na+>Ca++>Al+++, just the opposite of the variation of counter-ion binding degree. Therefore, the same concentration of $AlCl_3$ more effectively decreases the electrostatic repulsion of surfactants than NaCl, benefit for the formation of aggregates, which is mentioned before in the literature (Zhang et al., 2017; Srinivasan and Blankschtein, 2003), in detail.

In summary, the graph demonstrates that both the type of salt and the milling method significantly influence precipitation rates. $AlCl_3$ results in the highest precipitation rates, followed by $CaCl_2$ and NaCl, while distilled water provides the lowest rates. Additionally, products from rod mills generally exhibit higher precipitation rates than those from ball mills, suggesting that rod mills produce rougher particles that facilitate faster precipitation. These findings underline the crucial role of both salts and grinding methods in optimizing particle precipitation rates.

3.4. Jar tests

In addition to settling tests, a series of tests were adapted with the «Jar Test» methodology in order to prove the optimum conditions obtained in «settling rate» tests and to evaluate the effect of roughness from a different point of view.

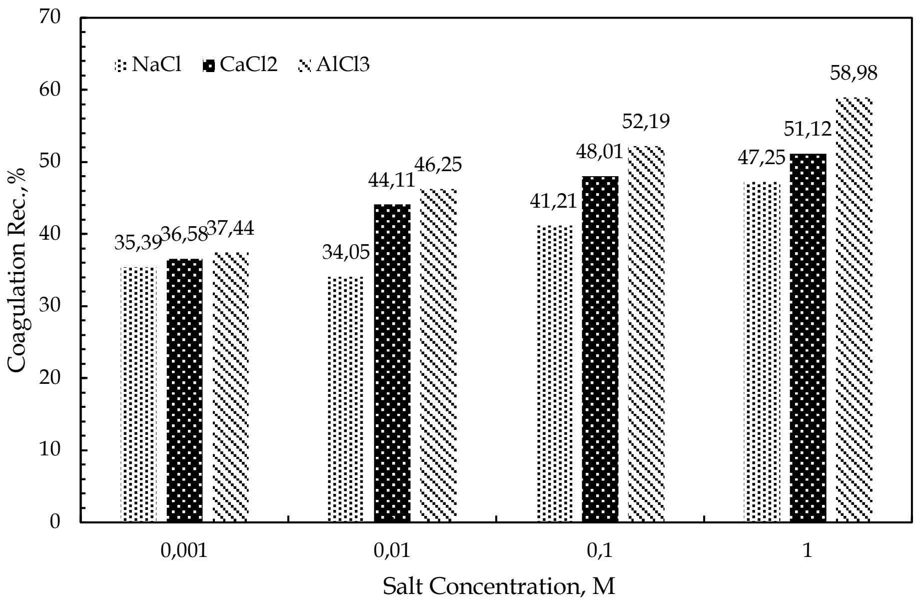


Fig. 8. Coagulation tests for magnesite-rod milled

The Fig 8. compares the coagulation efficiencies (Coagulation Rec., %) of three different salts (NaCl, $CaCl_2$, and $AlCl_3$) at various concentrations (0.001 M, 0.01 M, 0.1 M, and 1 M) for a magnesite- rod mill. When NaCl is used, the coagulation efficiency at a concentration of 0.001 M is approximately 35.39%. As the concentration increases, the coagulation efficiency also increases, reaching about 47.25% at a concentration of 1 M. When $CaCl_2$ is used, it shows higher coagulation efficiency than NaCl even at low concentrations. At a concentration of 0.001 M $CaCl_2$, the coagulation efficiency is around 36.58%, and as the concentration increases (0.01 M, 0.1 M, and 1 M), the coagulation efficiency steadily increases, reaching about 51.12% at 1 M. $AlCl_3$ shows the highest coagulation efficiency at all concentrations. With 0.001 M $AlCl_3$, the coagulation efficiency is around 37.44%, increasing to approximately 49.22% at 0.01 M and 0.1 M, and exceeding 55.78% at a concentration of 1 M. Overall, NaCl has the lowest coagulation efficiency, $CaCl_2$ is more effective than NaCl and provides moderate coagulation efficiency, while $AlCl_3$

shows the highest coagulation efficiency. As the salt concentration increases, the coagulation efficiency increases for all salts.

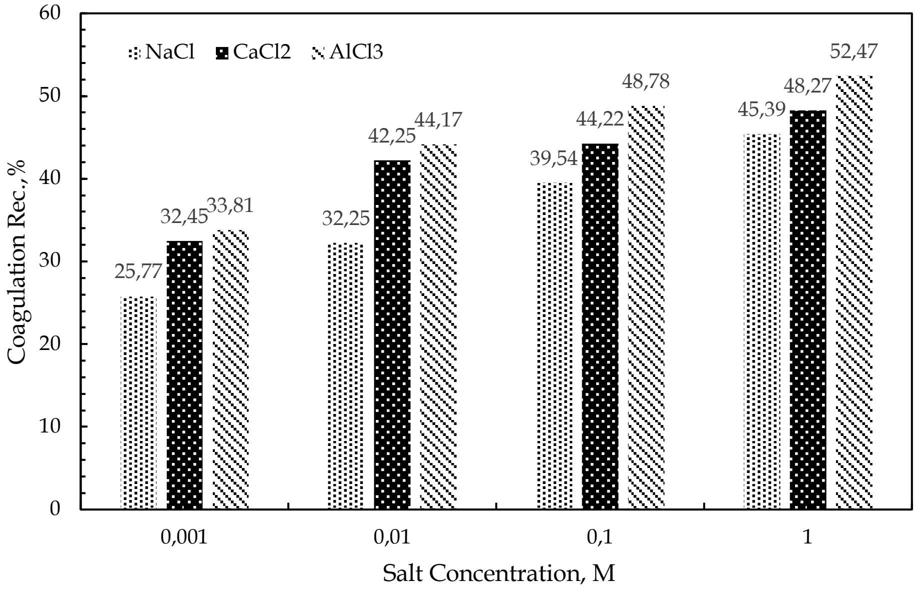


Fig. 9. Coagulation tests for magnesite-ball milled

The Fig 9. compares the coagulation efficiencies (Coagulation Rec., %) of three different salts (NaCl, $CaCl_2$, and $AlCl_3$) at various concentrations (0.001 M, 0.01 M, 0.1 M, and 1 M) for a magnesite-ball mill. When NaCl is used, the coagulation efficiency at a concentration of 0.001 M is approximately 25.77%. As the concentration increases, the coagulation efficiency also increases, reaching about 45.39% at a concentration of 1 M. When $CaCl_2$ is used, it shows higher coagulation efficiency than NaCl even at low concentrations. At a concentration of 0.001 M $CaCl_2$, the coagulation efficiency is around 32.45%, and as the concentration increases (0.01 M, 0.1 M, and 1 M), the coagulation efficiency steadily increases, reaching about 48.27% at 1 M. $AlCl_3$ shows the highest coagulation efficiency at all concentrations. With 0.001 M $AlCl_3$, the coagulation efficiency is around 33.81%, increasing to approximately 46.48% at 0.01 M and 0.1 M, and exceeding 52.47% at a concentration of 1 M. Overall, NaCl has the lowest coagulation efficiency, $CaCl_2$ is more effective than NaCl and provides moderate coagulation efficiency, while $AlCl_3$ shows the highest coagulation efficiency. In summary, as the salt concentration increases, the coagulation efficiency increases for all salts.

4. Conclusions

- The roughness values of magnesite particles increased with the usage of a rod mill compared to grinding with a ball mill.
- The settling rate of particles exhibited a minimum at low solid wt. % while an average value was obtained under the original pH values of particles.
- Adding mono and multi-valent salts enhanced the coagulation of rougher and smoother particles in the order of $NaCl<CaCl_2<AlCl_3$. However, rougher particles produced higher settling rates in the presence of the same salt concentration.
- In a scientifically simple and clear way, which is previously mentioned in the literature, the order of hydrated sizes for alkali metal and metal counter-ions is $Na^+ > Ca^{2+} > Al^{3+}$, which is inversely related to their binding strength. As a result, $AlCl_3$ is more effective than NaCl and $CaCl_2$ at reducing electrostatic repulsion among surfactants, aiding in aggregate formation ($NaCl<CaCl_2<AlCl_3$).
- In addition to settling tests, the same trend was also obtained as the results of jar test experiments.
- As shown in the Figure below (Fig. 10), the overall results suggested that the adjustment of milling methods to obtain rougher particles is one of the key parameters to control the particle-particle interaction and accordingly their coagulation characteristics regardless of coagulant type.

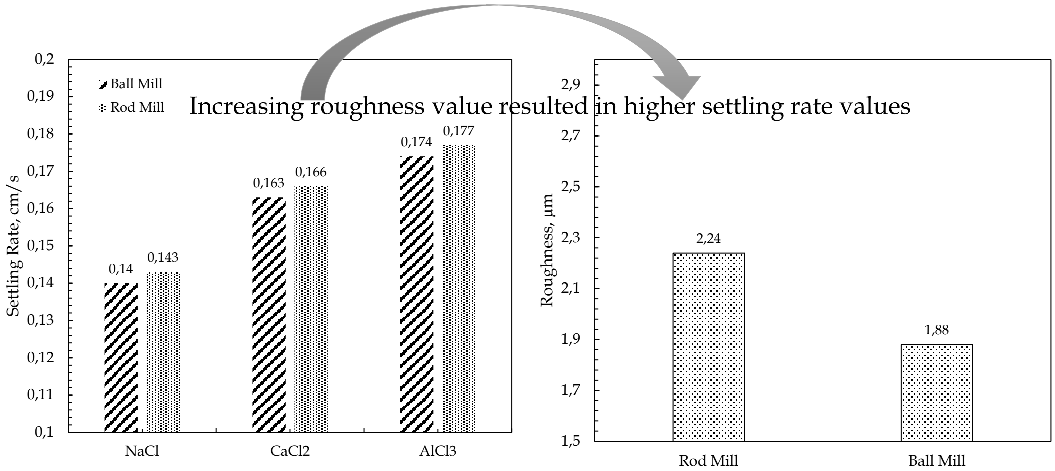


Fig. 10. The relation between settling rate and roughness values of magnesite particles as an overall result (salt concentration: 1 M)

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