Understanding Lateritic Ore Agglomeration Behaviour as a Precursor to Enhanced Heap Leaching*

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Abstract

Although nickel (Ni) laterite ores constitute the majority of Ni mineralization resource world-wide, in contrast to Ni sulphides, their processing via conventional beneficiation (e.g. multi-gravity and flotation) and hydrometallurgical routes is intractable as they are predominantly low grade and complex, both mineralogically and chemically. Due to their physico-chemical characteristics, low grade lateritic ores require more aggressive but costly chemical and hydrometallurgical techniques (e.g., leaching in high pressurized tanks) for value metal (Ni and Co) extraction. Processing such ores through cost-competitive heap (4-10 m high) leaching as an alternative, requires successful agglomeration of the feed into robust and porous granules. To date, producing of granules with desirable attributes poses a major geotechnical challenge to industry. In the present work, we investigate agglomeration behaviour of siliceous goethite Ni laterite ore and selected oxides and clay minerals (hematite, quartz and kaolinite) which constitute the predominant host gangue phases of typical low grade Ni laterite ores. Fundamental knowledge and understanding of the agglomeration mechanisms and kinetics which are essential for producing robust real ore granules, and pivotal to the subsequent heap leaching process, are gleaned. Isothermal, batch agglomeration tests involving 30 and 44 % w/w sulphuric acid solution as a binder indicated that 5 – 40 mm granules of differing roughness and morphologies were produced in 8-14 min. The results showed feed characteristics (e.g., mineralogy and particle size distribution) and binder content (15-25 wt.%) dependent agglomeration behaviour. Slow agglomerate nucleation and growth were displayed by the kaolinite clay mineral whilst the oxides exhibited faster agglomeration kinetics. Siliceous goethite feed ore fine/coarse ratio, H_2SO_4 binder dosage and acid content, product drying temperature and aging conditions, all showed significant impact on agglomeration mechanisms (e.g., particle wetting, nucleation and growth processes) and granule attributes (e.g., size and strength). Agglomerates strength increased with increasing fine/coarse particle ratio.

1 Introduction

Sulphidic and lateritic ores are the two major sources of global nickel (Ni) mineralization. The sulphidic ores constitute about 40 % of the world's Ni reserves but account for about 60 % of total Ni production. In contrast, the lateritic ores constitute about 60 % of the world's Ni reserves but account for only 40 % of total Ni production. The historic preference of sulphidic ores over lateritic ores is due to the fact that, the latter is mostly chemically and mineralogically variable and complex as well as lower in grade, posing major economic and technical challenges during processing (Kim *et al*., 2010; Elias, 2002; Mudd, 2010; Quaicoe et al., 2011). Despite the challenges, the current demand for commodity metals (e.g., Ni, Co) and depletion of high grades ores (e.g., Ni sulphide ores) have necessitated the need to process Ni laterites as alternative ores (Kuck, 2009; Kim *et al*., 2010; Elias, 2002; Mudd, 2010; Lee *et al*., 2005; Golightly, 1981; Horton, 2008). Conventional beneficiation unit operations (e.g. flotation, electrostatic and magnetic) are mostly employed initially to concentrate or upgrade these ores where possible. For complete value metal recovery, aggressive chemical / hydrometallurgical techniques such as Heap Leaching (HL), Atmospheric Leaching (AL) and High Pressure Acid Leaching (HPAL) are usually employed. Due to relatively low operating and capital cost involved in HL as well as operational flexibility, it is mostly considered as suitable processing route for complex, low grade ores (Lewandowski and Kawatra 2008, 2009). In spite of the advantages offered by HL, several persisting scientific and technological challenges make it ineffective and economically non-viable processing technology. Some of these are linked to poor permeability issues usually associated with finely ground feed ore bed and the presence of acid consuming clay minerals (Chamberlin, 1986; Dixon, 2003; Eisele and Pool 1987; Lewandowski and Kawatra, 2008, 2009; Kappes, 1979). Poor permeability caused by fine particles usually occurs through segregation during heaping and fine particles migration with leachate through the heap (Chamberlin, 1986; Dixon, 2003; Eisele and Pool 1987; Lewandowski and Kawatra, 2008; Kappes, 1979). The migration of fine particles clogs the natural flow channels, and form impermeable layers within the heap that restrict lixiviant percolation. Consequently, the leachate flows through paths of least resistance, leading to poor solution distribution and hence, low metal recovery. Fine mineral particles agglomeration is mostly used to

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minimised or eliminate poor permeability issues caused by the fine particles which tend to improve heap leaching performance (Chamberlin, 1986; Dixon, 2003; Eisele and Pool 1984; Lewandowski and Kawatra, 2008, 2009; Kappes, 1979).

Despite the economic relevance of agglomeration pre-treatment, its successful application to complex, low grade Ni laterite ores heap leaching is limited (Readett and Fox, 2009 a and b). To date, there are only two commercial plants in minerals industry operating full-scale heap leaching of agglomerated Ni laterite ores, the Murrin Murrin (Western Australia) and Caldag (Turkey) operations. Fundamental studies are required to understand the agglomeration behaviour which determines the geotechnical and hydrometallurgical characteristics of the heap granules of typical low grade Ni laterite (e.g., siliceous goethite) ores.

In this study, the agglomeration behaviour of selected clay (kaolinite) and oxide (hematite and quartz) minerals which typically constitute the predominant host gangue phases in certain, low grade Ni laterite ores were investigated in tandem with a real Ni laterite (siliceous goethite) ore. Specifically, the effect of binder content and its composition (30 vs. 44% w/w H_2SO_4) and post-agglomeration treatment conditions (e.g., drying) on agglomeration behaviour and granule properties (size, integrity, strength) were studied. Particularly, the influence of feed characteristics (chemical / mineralogical composition and primary particle size distribution) on granule growth behaviour and binder on agglomerate properties (e.g., size, morphology and compressive strength) were examined.

2 Materials and Methods

2.1 Model Minerals

Three model polydispersed minerals: kaolinite (clay), quartz and hematite (oxide) were used. Tables 1 and 2 show the properties of these powders. The specific surface area was determined by a 5 point N_2 BET (Brunauer *et al.*, 1938) analysis (Coulter Omnisorp 100, Hialeah FI. USA). The particle size distributions (PSD) of the minerals (Fig. 1) were determined by laser diffraction method using Malvern Mastersizer 2000A. X-ray Fluorescence (XRF) method was also used to determine their individual minerals' chemical compositions. The measurements were conducted using a Panalytical MiniPal 4 EDXRF Spectrometer using default condition sets for 60 s for each condition. The XRF equipment was calibrated using pure oxides.

2.2 Nickel Laterite Material

Polydispersed, -2 mm and -150 µm siliceous goethite (SG) laterite ore (~1.0 wt.% Ni) from Western Australia were used in this study as received. Quantitative X-ray powder diffraction QEMSCAN analyses showed complex mineral associations where the quartz, goethite, nontronite and serpentine comprise

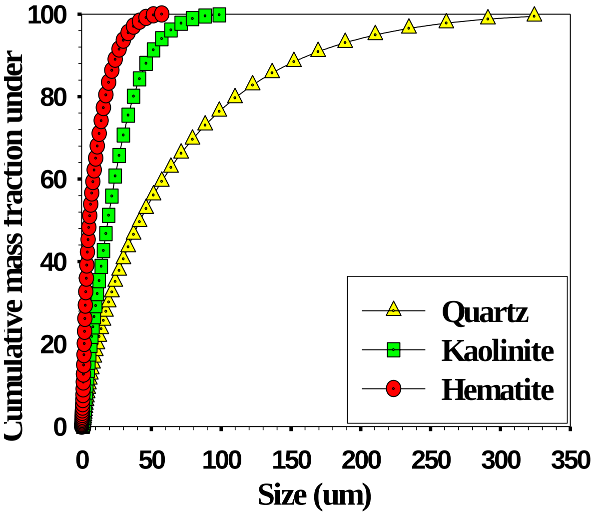


Fig. 1 Primary Particle size Distribution of Hematite, Kaolinite and Quartz Minerals.

Table 1: Initial Feed Properties of Quartz, Hematite and Kaolinite Minerals and Amount of Acid used for Agglomeration.

Properties	Quartz	Hematite	Kaolinite
D_{10} (µm)	5.2	1.4	3.8
D_{50} (µm)	6.3	6.3	18.9
$D_{4,3}$ (µm)	65.6	11.1	23.3
$D_{3,2}$ (µm)	13.5	3.1	9.7
BET surface area (m^2/g)	1.0	14.5	24.8
Dry mass (g)	500	500	500
True density (g/cm^3)	2.7	5.3	2.6
Bulk density (g/cm^3)	1.7	2.0	0.8
Mass of acid used (g)	109.8	88.2	214.3
Volume of acid used (cm^3)	91.5	73.5	174.2
Initial bed pore volume (cm^3)	108.8	155.0	432.7

*Loss of ignition

Table 2: Chemical Composition of Quartz, Hematite, and Kaolinite Minerals.

Major oxide	Quartz	Hematite	Kaolinite
SiO_2	99.1	2.4	45.8
TiO_2	-	-	1.9
Al_2O_3	-	3.1	35.6
Fe_2O_3	-	93.0	1.2
MgO	-	-	0.2
CaO	-	-	0.1
K_2O	-	-	0.2
Na_2O	-	-	0.2
LOI*	0.9	1.5	14.8

the dominant and hematite, asbolane and kaolinite comprise the minor mineral phases (Table 3). It also established the dominant, sub-dominant and minor gangue mineral phases with some size dependency in the sample, where bimodal particle size distributions of fine and coarse size fractions were displayed.

The distribution of nickel, cobalt and manganese in the SG ore based on chemical analysis is given in Fig. 2. Details of the Quantitative X-Ray Diffraction data for the main components of the ore are given in Figure 3. As shown in Fig. 2, the dissemination of the three elements is greater for the -1.18 to 0.038 mm and C6 and C7 size fractions than for the C1-C5 size fractions. The dominant phases are goethite across all the sizes with quartz being the predominant phase in the coarse sizes (Fig. 3). Smectite and serpentine clay minerals are indicated, with both largely showing up in the fine fractions as expected.

Table 3 Mineralogical Composition of -2 mm Siliceous Goethite Ore.

Mineral phase	Mass %
Quartz	36.06
Kaolinite	0.21
Mg-bearing silicates (e.g., serpentine)	8.71
Nontronite (smectite group)	18.77
Goethite	27.43
Hematite	2.81
Asbolane	0.40
Total Nickel	1.1
LOI*	5.64

2.3 Binder

For the model minerals (hematite, quartz and kaolinite), 30 % w/w H_2SO_4 solution was used as the binder for all the tests. In the case siliceous goethite (SG), 30 and 44 % w/w H_2SO_4 solutions were used as the binders at mass contents in the range 15 – 25 wt.%. These corresponded to a binder dosage of 250 – 480 kg H_2SO_4/t solid (wet basis). Due to the difference in minimum binder saturation requirement for effective agglomeration for the model minerals, as a result of their initial porosities and true densities, the binder contents used for successful agglomeration varied: 15, 18 and 30 wt. %, respectively for hematite, quartz and kaolinite.

2.4 Batch Agglomeration Equipment

The agglomeration tests were carried out batch-wise in a horizontal, stainless-steel drum granulator of 0.3 m internal diameter and length of 0.2 m (Fig. 4A) operated at a constant rotational speed of 60 rpm. The drum wall is fitted with six 5 mm high baffles spaced evenly around the interior to aid in tumbling of the feed charge. To enable visual observations during the agglomeration process, Perspex material was used as cover plates for both drum openings. As a safety precaution, the granulator was operated in an enclosed guarded cage as shown in Fig. 4B.

2.5 Agglomeration Procedure

Approximately, mineral feed charge consisting of 500-800 g of dry powder with a known amount of H_2SO_4 binder was used for each agglomeration test. This loading corresponds to about 4-7 % of the total effective drum volume. The powder was pre-mixed over 2 min with a pre-determined amount of the binder in an acid resistance glass ware before transferred into the drum granulator. For most of the agglomeration tests, a maximum batch time of 14 min was used. It is worth mentioning that due to the tendency of the wet material sticking to the drum wall, a regular scraping of the material (~ 30 s intervals) was found to be sometimes necessary for efficient tumbling and agglomeration.

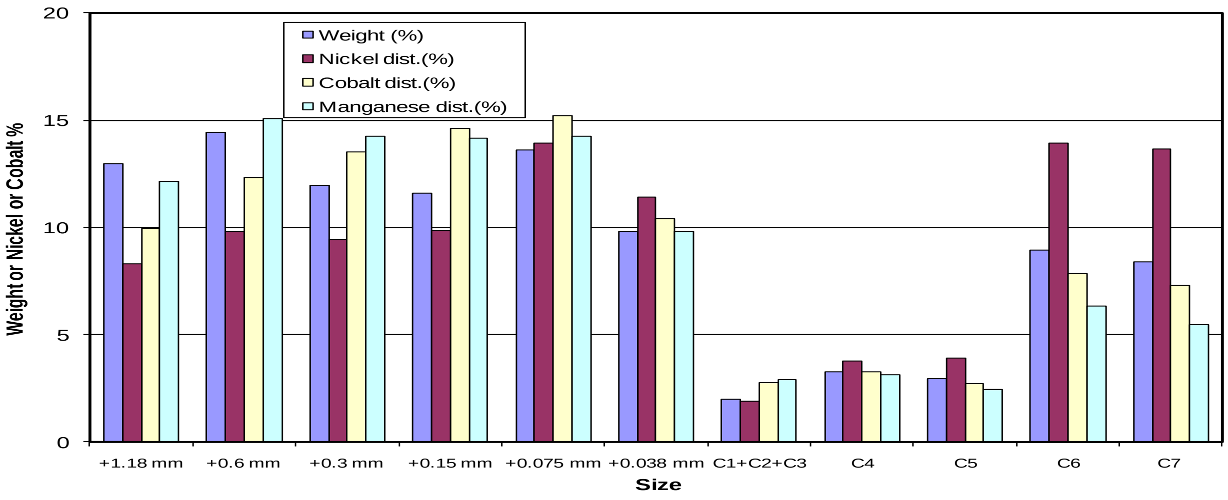


Fig. 2: Sizing and Metal Distributions in the Siliceous Goethite Sample

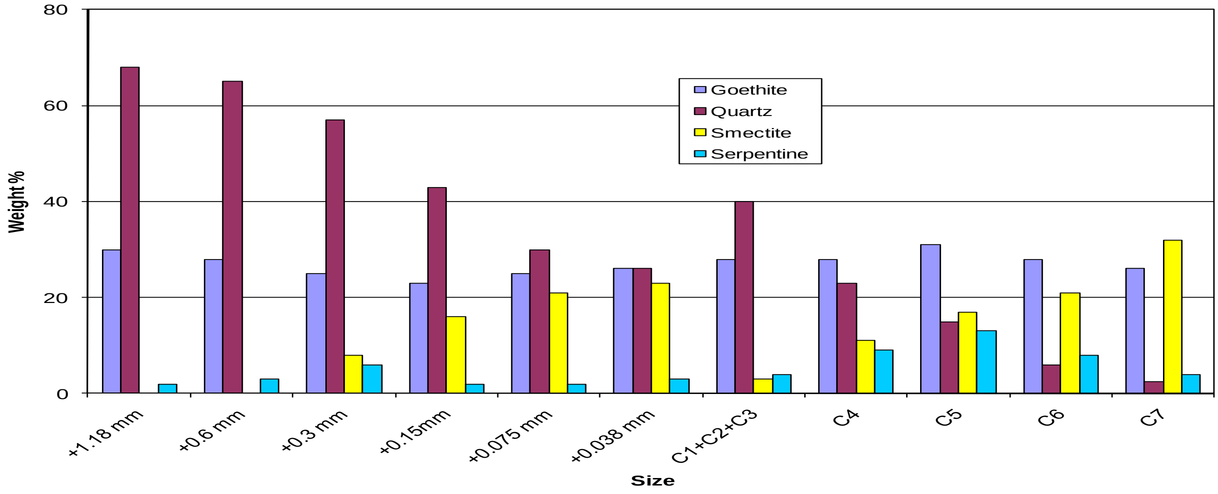
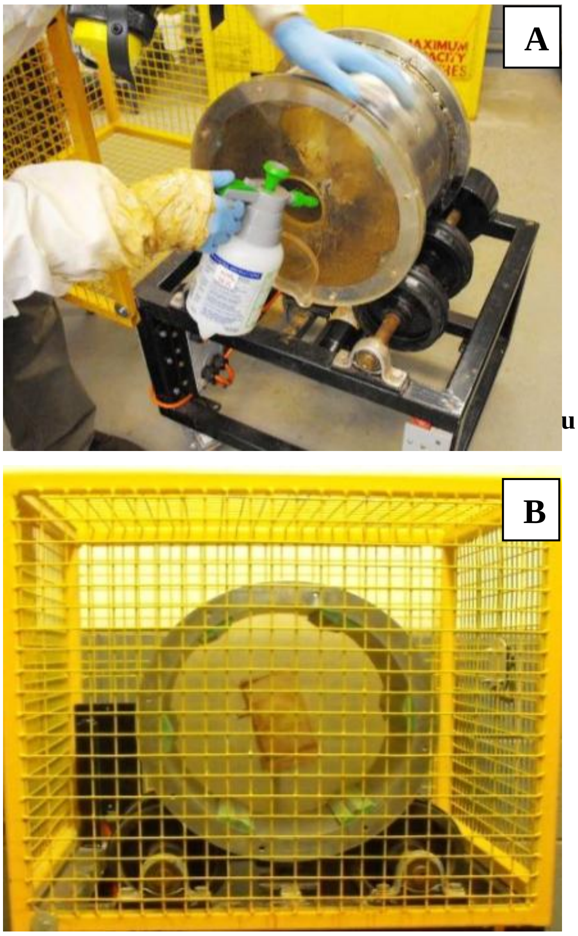


Fig. 3 Quantitative X-Ray Diffraction Data for Major Host Gangue Mineral Species in the Siliceous Goethite ore Sample.



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lator (A) without and (B) with Guarded Cage (for Safe Operation).

2.6 Agglomerate Characterisation

After each agglomeration, the granules' size distribution (GSD) on mass basis was determined by a conventional sieving technique. The cumulative mass fractions undersize were then plotted against granule size (defined as mesh size of retaining sieve). A bench-top tensile-compressive strength machine (Hounsfield, UK) shown in Fig. 5 was used to load the agglomerates in diametric compression. This was achieved by applying a load (force) to a granule held between two parallel flat surfaces, one of which is held stationary and the other attached to a constant velocity drive (Fig. 5). A load cell attached to the upper drive surface enables the measurement of the resultant force at a maximum load setting of 1000 N and velocity of 10 mm/min. Based on the agglomerate diameter and the force at which breakage occurred (as measured by the machine), the compressive strength was calculated from equation (1).

$$\sigma_s = 4P/\pi d_g^2 \qquad (1)$$

where σ_s is compressive/failure strength, d_g is granule diameter (m) and P is the applied load or force (N)

Three sets of agglomerates were used for strength measurements, first set was air-dried at ambient temperature (~ 22 – 25 °C), second set oven dried at 40 °C, and the last set kept wet in air-tight plastic all over 24 h.

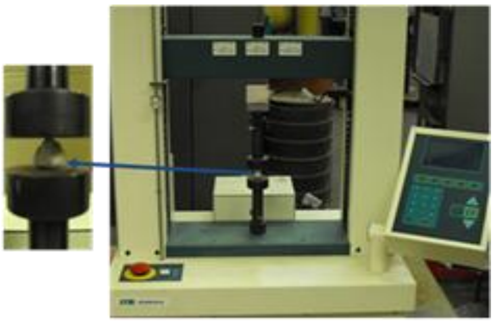


Fig. 5 Apparatus for Loading Agglomerates in Diametric Compression.

3 Results and Discussion

3.1 Model Minerals Agglomeration Behaviour

The model oxide and clay minerals agglomeration behaviour was investigated by analysis of the evolution of their granules' size distribution (GSD) after 2, 4, 8 and 14 min (Fig. 6 and 7). The results show smooth, spherical granules of hematite (Figure 6A) and quartz (Fig. 6B) in contrast with kaolinite granules (Fig. 6C) which have irregular shape and rough surfaces.

Fig. 7 displays the GSD, reflecting marked differences. The results show that hematite granule nucleation was more intensive than those of quartz and kaolinite. Noticeable amounts of fine (feed) particle fractions were observed in the PSD of quartz (Fig. 7B) and kaolinite (Fig. 7C) up until 8 min of agglomeration. On the other hand, there was a significant shift to larger granule size over the same time for hematite (Fig. 7A). This indicates that hematite displays shorter nucleation induction time than quartz and kaolinite, a behaviour which depends upon the time for the liquid binder to be distributed from the granules core to the surface (Iveson, 1997). The results further show that after nucleation (time > 2 min), quartz and kaolinite particles followed similar agglomeration sub-processes of pseudo-layering growth between 2-4 min, followed by non-random coalescence (4-8 min) and pseudo-layering growth between 8-14 min. The hematite particles, on the other hand, displayed non-random coalescence behaviour between 2-4 min and 8-14 min.

These growth behaviour displayed by the ores may be attributed to binder-ore interaction characteristics (Benali, 2009). Whilst constant binder surface tension and composition and drum speed were used, different binder volumes were involved. Apart from the differences due to ore mineralogy, there is a noticeable difference in the PSDs of the three samples. The mean particle sizes (D3,2 and D4,3) decrease as follows: quartz > kaolinite > hematite. Feed PSD has influence on granule growth, with granule dynamic strength and growth rate increasing with decreasing mean particle size at saturation (Benali, 2009). Broader PSD of feed also causes the powder bed to be more densely packed as the smaller particles easily fill the inter-granular gaps among the larger particles. Consequently, this leads to difficulty in the liquid binder distribution and wetting of particles resulting in poor or slow agglomeration growth behaviour. Therefore the difference in the growth behaviour exhibited by the ores can be attributed to the differences in feed ore characteristics such as porosity, particle density, primary particle size distribution, mineralogy and volume of binder used.

In order to ensure reliability of the observed trends, reproducibility of the agglomeration behaviour was checked. Three replicate experiments were performed at three different times for each material used. Evidently, the results (Fig. 7D) showed that the agglomeration behaviour was reproducible because substantial similar size distributions were obtained after 14 min in all the three replicate agglomeration tests. The model minerals agglomeration results are useful for benchmarking our understanding of the real nickel laterite ores. The striking agreement between the behaviour of the quartz and hematite and that of siliceous goethite laterite ores shown below (Fig. 10), is worth noting.

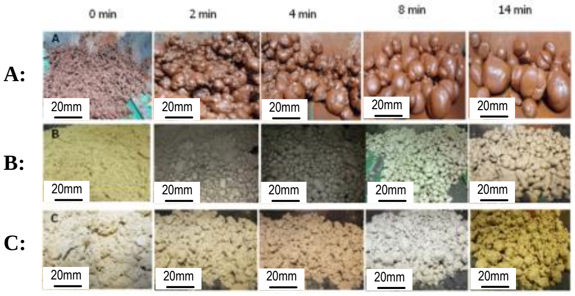
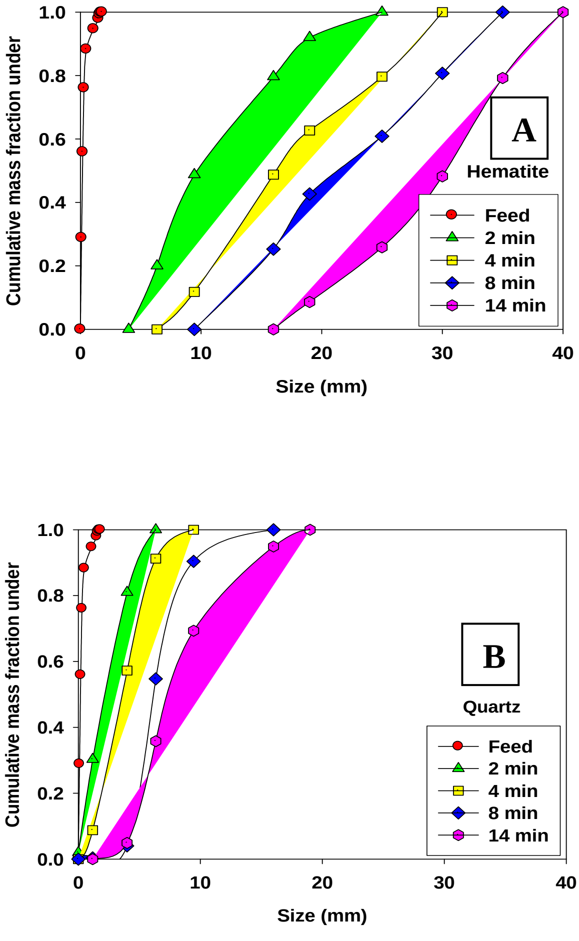


Fig. 6 Digital Images of (A) Hematite (B) Quartz and (C) Kaolinite Granules inside Drum Agglomerator.



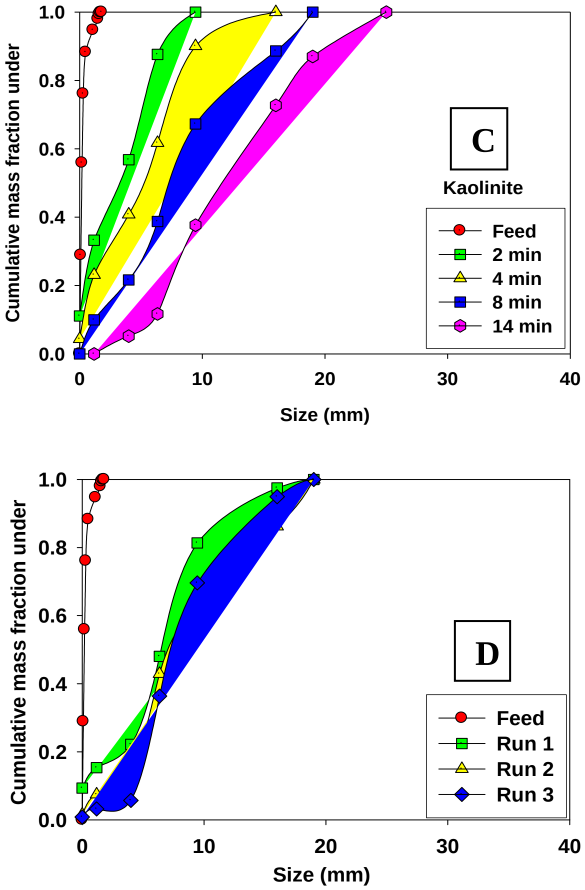


Fig. 7 Granules size Distribution of (A) Hematite, (B) Quartz and (C) Kaolinite as a Function of Time and (D) Three Replicate Quartz Agglomeration Tests at 14 min.

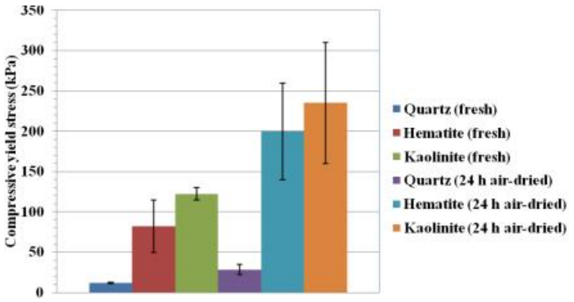


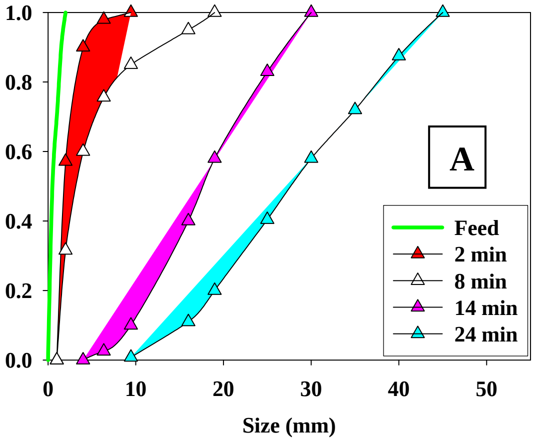
Fig. 8 Compressive Strength of Wet and Air-dried Quartz, Hematite and Kaolinite Agglomerates.

3.3 Nickel Laterite Agglomeration Behaviour: Effect of Binder Solution Composition/ content

To investigate the effect of binder solution composition (i.e., acidity) and content (wt.% of binder solution) on agglomeration behaviour of SG ore, two H_2SO_4 solutions (30 and 44% w/w) were used. For agglomeration tests conducted with 44% w/w H_2SO_4 binder (water/acid ratio: 1.27), 15 wt.% binder content led to insufficient wetting and hence, no agglomeration was observed in the course of 24 min. At 20 wt.% binder content, the inherent moisture was adequate for successful agglomeration (Figure 9A). The data clearly indicate that despite rapid nucleation observed during first 2 min, granule size growth was slow where only ~20% of granules had size > 5 mm after 8 min. This was partly due to strong adherent tendency of the nuclei towards the drum walls, reducing the number of nuclei – granule collisions. These collisions are believed to facilitate granule size growth via mechanisms such as pseudo-layering and/or coalescence.

The results in Fig. 9A also show that granules of 5 – 25 mm size range were produced within 8 – 14 min, whilst further agglomeration up to 24 min resulted in the formation of oversize agglomerates. The increase of the 44% w/w H_2SO_4 binder solution content from 20 to 22.5 wt.% enhanced the binder saturation of the feed ore and led to noticeably faster nucleation and growth process (Fig. 9B). The data indicate that 2 – 15 mm size granules were produced after 2 min, with ~80% of 5 – 25 mm size granules formed within 8 min. A further increase of binder content to 25 wt.%, dramatically intensified the agglomeration process (Fig. 9C). This led to massive nucleation which started during of dry feed and binder solution mixing and hence, formation of 5 – 15 mm size granules after 1 min and thereafter, coarse granules (5 – 40 mm). It is worth mentioning that the main mechanism for granule size growth changed from pseudo layering to coalescence upon 2.5 – 5 wt. % binder content increase.

In contrast, tests conducted with 30% w/w H_2SO_4 solution showed that agglomeration was insignificant at 15 wt.% binder content due to poor wetting of the powder. 20 wt.% or higher binder content however, was effective. Fig. 10A clearly shows that agglomerates in the size range 5 – 25 mm were produced within 8 min whilst longer time of 14 min led to the formation of markedly coarser agglomerates (> 40 mm). At higher binder contents of 22.5 wt.% and 25 wt.%, larger agglomerates were produced within 8 and 2 min, respectively (Fig. 10B).



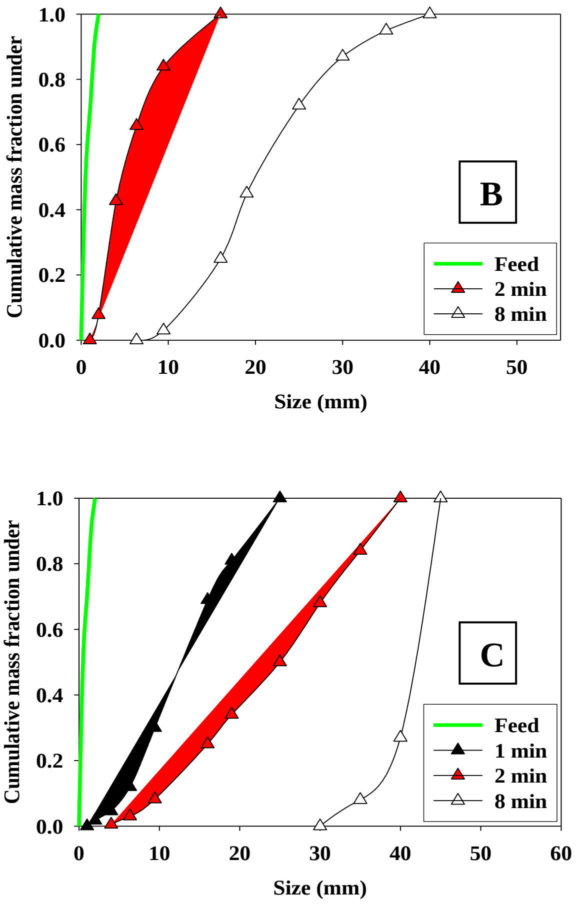


Fig. 9 **The Granule size Distribution for the SG Ore as a Function of Agglomeration Time with 44% w/w H_2SO_4 Solution at (A) 20 wt.%, (B) 22.5 wt.% and (C) 25 wt.% Binder Content.**

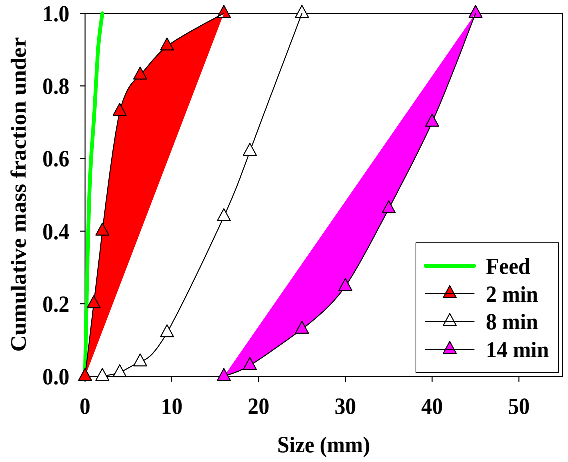


Fig. 10a The Granule size Distribution for the SG Ore as a Function of Time with 30% w/w H_2SO_4 Solution at 20 wt.% Binder Con tent.

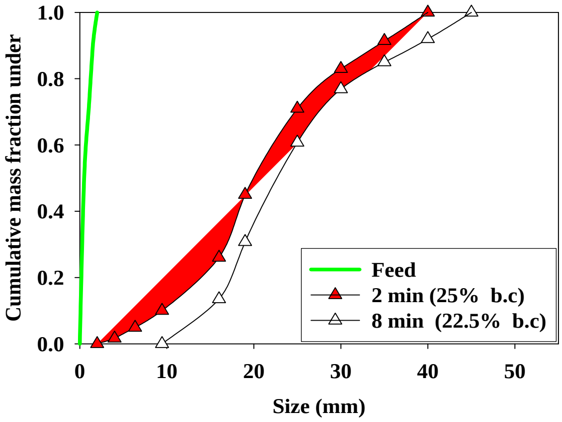


Fig. 10b The Granule size Distribution for the SG Ore as a Function of Time with 30% w/w H_2SO_4 Solution at 22.5 and 25 wt.% Binder Content.

For the latter, the agglomerates coalesced rapidly forming two or three large lumps after 3 min. The results in Fig. 9, 10a and 10b suggest that the higher the H_2SO_4 binder content, the faster the agglomeration rate at a given batch time. These observations underscore the key role of binder content and acid strength in wetting the ore particles and controlling their bonding mechanisms during the agglomeration process. Strong cohesive forces (capillary forces arising from negative Laplace pressure) become increasingly dominant as the wetted particles move from pendular through funicular to capillary states, fostering strong agglomeration.

3.4 Nickel Laterite Agglomeration Behaviour: Effect of Fines to Coarse Particles' Ratio

Increasing the fine (F) (-150 µm)/ROM coarse (-2 mm) particles' mass ratio of the SG ore feed (at a fixed binder content):

(i) slows down the overall agglomeration rate and granule size growth (Fig. 11A vs 11B) where smaller granules of narrower size distribution are form at a given time .

(ii) enhances the nucleation stage, where more nuclei form per unit mass of feed ore during the initial stage, leading to larger number of smaller size of granules (Fig. 12A vs 12B).

(iv) changes the granule growth mechanism from coalescence to pseudo layering due to decreased wetting (Fig. 11A vs 11B).

(v) requires higher binder content to maintain faster agglomeration rate at a fixed time (Figure 11A vs 11B

3.5 Failure Strength of Siliceous Goethite Agglomerates

The effect of binder composition (acidity) and content on compressive/failure strength (diametric loading) and integrity of agglomerate are shown in Tables 4. The data in Table 4 indicate that the compressive strength of fresh agglomerates slightly de-

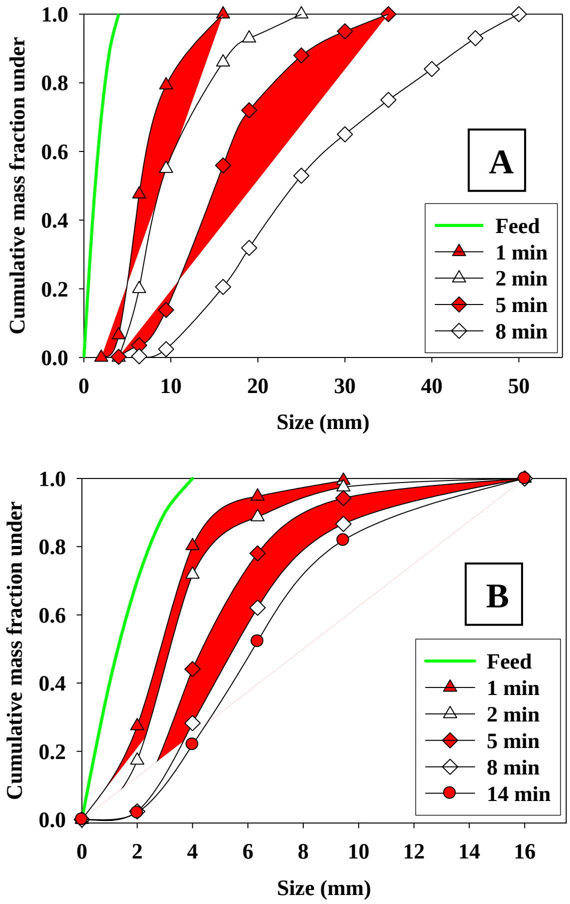


Fig. 11 The Granule size Distribution for the SG Ore with (A) 20%F/80%ROM and (B) 60%F/40%ROM Produced with 25 wt.% Binder Content (30% w/w H_2SO_4) as a Function of Time.

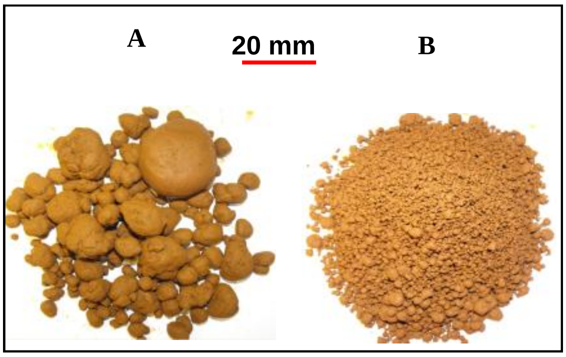


Fig. 12 Digital Images of (A) 20%F/80%ROM and (B) 60%F/40%ROM SG Ore after 8 min of Agglomeration with 25 wt.% Binder Content (30% w/w H_2SO_4).

creased with decreasing binder acid content. This may be partly attributed to the greater density and more viscous binder liquid within the granules at higher binder acid strength (44% w/w H_2SO_4). Minor increase in binder content (e.g., 20 to 22.5 wt.%) had no significant effect on the agglomerate strength. In contrast, the strength of air-dried agglomerates was significantly higher than that of fresh agglomerates and increased slightly with increasing binder content. The higher strength observed for air-dried agglomerates is due to the stronger solid bridges which form between particles when the acid-mediated leached species solidify (crystallize) within the agglomerate with decrease in porosity upon drying.

Table 4 The Compressive Strength of Fresh and Air-dried SG Agglomerates Produced with different Binder Liquid Type and Content.

Binder Liquid	Binder Content (wt.%)	Agglomerate Failure Strength (kPa)	
		Wet (sealed container)	24 h Air-dried (ambient)
44% w/w H_2SO_4	20%	35 ± 5	110 ± 10
	22.5%	30 ± 5	120 ± 10
	25%	35 ± 5	150 ± 10
30% w/w H_2SO_4	20%	25 ± 5	160 ± 10
	22.5%	20 ± 5	180 ± 10

3.6 Effect of Fines/coarse Particles' Mass Ratio on Dry Agglomerate Strength:

Fig. 13 shows that increasing fine (-150 µm)/coarse particles' mass ratio in -2 mm feed ore leads to increased agglomerate/pellet density (decreased porosity) as smaller particles easily fill the pore spaces between the large particles. This also enhances agglomerate/pellet dry compressive strength.

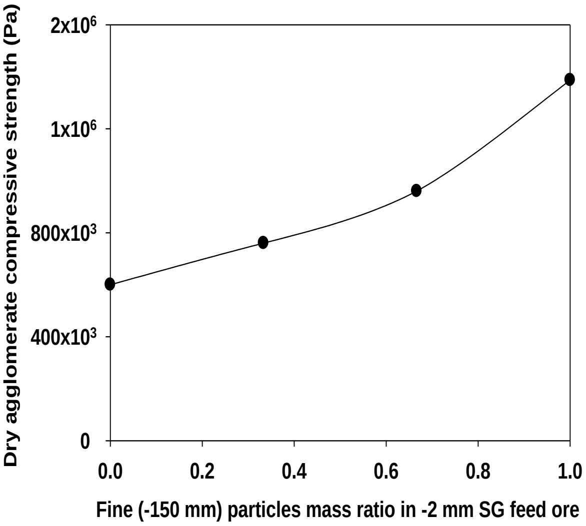


Fig. 13 Effect of Fine (-150 µm) to Coarse (-2 mm) Feed Particles' Mass Ratio on Dry SG Agglomerate Compressive Strength.

3.7 Column Leaching Behaviour of SG Agglomerates:

Both cured (48 h air-dried) and uncured SG agglomerates displayed good re-wetting stability during 100 days of laboratory scale column leaching tests (with 0.5 m bed height) where ~80% Ni recovery was achieved (Addai-Mensah et al., 2011). Despite some cracks appearing on agglomerates in the column during early stages of leaching with ≈ 15 % heap slump, the SG agglomerate beds retained their 8.5 $dm^3/m^2/.h$ over 100 days. The agglomerates beds also appeared to be reasonably stable when extra loads applied on top of the 0.5 m columns to mimic heap height of 4 m which is normally used in real plant applications. The complementary column leaching tests indicate that SG agglomerates produced in this study are robust and strong enough to enable construction of 4 m heaps for Ni laterite plant.

Conclusions

In this paper, the influence of feed and binder characteristics on agglomeration behaviour and granule properties was studied fundamentally. The results revealed that:

- Feed ore mineralogy, binder content and primary particle size distribution have a decisive impact on agglomeration behaviour and product properties (size, shape, surface morphology and failure strength).
- Model mineral hematite exhibited relatively faster nucleation and stronger agglomerate growth behaviour in comparison with quartz and kaolinite
- Robust, 5 – 40 mm siliceous goethite laterite ore agglomerates may be readily produced within 8-14 min of agglomeration, with 30 – 44 % w/w H_2SO_4 at binder content of 250 – 480 kg / t solid (wet basis).
- Post-agglomeration drying or curing enhances the strength of the agglomerates.
- The agglomeration growth behaviour exhibited by the model and real Ni laterite mineral ores was found to be highly reproducible.

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