Understanding the agglomeration behavior of nickel laterite and gold ores using statistical design of experiments

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Abstract
The drum agglomeration of nickel laterite and gold ores has been optimized through the design of experiments (DOE) using a Taguchi L16 (4^5) orthogonal array to determine the optimum conditions for maximizing average agglomerate size and minimizing the amount of fines. The effects of controllable operating factors including moisture content (nickel laterite ore: 34-37%; gold ore: 7-10%), retention time (2-3.5 min), drum speed (15-45% critical speed), drum load (nickel laterite ore: 8-32 %; gold ore: 6-22%) and acid concentration (150-600 g/L) on the performance of the agglomeration process were studied.

For nickel laterite ore, maximum average agglomerate size and minimum percent fines (-1 mm) occurred under the following conditions: drum load (23.7%), moisture (36.5%), time (3 min), drum speed (30% critical speed) and acid concentration (150 g/L). Under the studied nickel laterite ore conditions, the most effective parameters for maximizing average agglomerate size and minimizing the amount of fines were found to be drum load and acid concentration, respectively. Drum speed had a statistically significant effect on minimizing the amount of fines. Maximum average agglomerate size and minimum percent fines (-1 mm) for gold ore occurred under the following conditions: drum load (19.3%), moisture (8.5%), time (2 min 15 s) and drum speed (40% critical). The most significant factors for maximizing average agglomerate size and minimizing the amount of fines for gold ore were found to be drum load, time and moisture.

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Introduction
Several heap leach operations have experienced problems in terms of poor recovery due to percolation issues caused by low-grade complex ores, tailings and clayey deposits. Poor percolation can lead to low metal extraction due to solution channeling or the development of impermeable/dead zones within the heap (Dhawan et al., 2012; Kappes, 2005). Improper heap building practices such as the use of loose agglomerates and inadequate attention toward the presence of clay minerals have been among the main reasons for percolation issues. During the transport of ore material, severe segregation of the material can occur. To overcome percolation problems, a major improvement was made through the introduction of agglomeration prior to ore placement. If the ore particles and agglomerates are of similar size, segregation can be avoided to a great extent (Dhawan et al., 2013; Kinard and Schweizer, 1987; McClelland and van Zyl, 1988). Agglomeration improves the uniform percolation of solution through ore heaps and is applicable to many ores, wastes and milled tailings (Bouffard, 2005; Dhawan et al., 2013). Manning and Kappes (2005) reported agglomeration/stacking accounts for ~14% of the total heap leaching operating cost. The cost of binder is a primary contributor toward the total cost. Also, the lack of consistent quality control tests for agglomerate often leads to operational problems. The details are mentioned elsewhere (Dhawan et al., 2013). Considering the significant contribution of the agglomeration/stacking step toward the total heap leach operating cost, it is appropriate to study the agglomeration behavior and its optimization for two different ores. The reason to study the same parameters for different ores is the fact that significant variability in the agglomeration behavior of different ores has been reported to exist in industrial operations.

It is well understood that a wide feed particle size distribution (PSD) is not ideal for consistent high-quality agglomerates, when it is known that agglomerates must undergo mechanical

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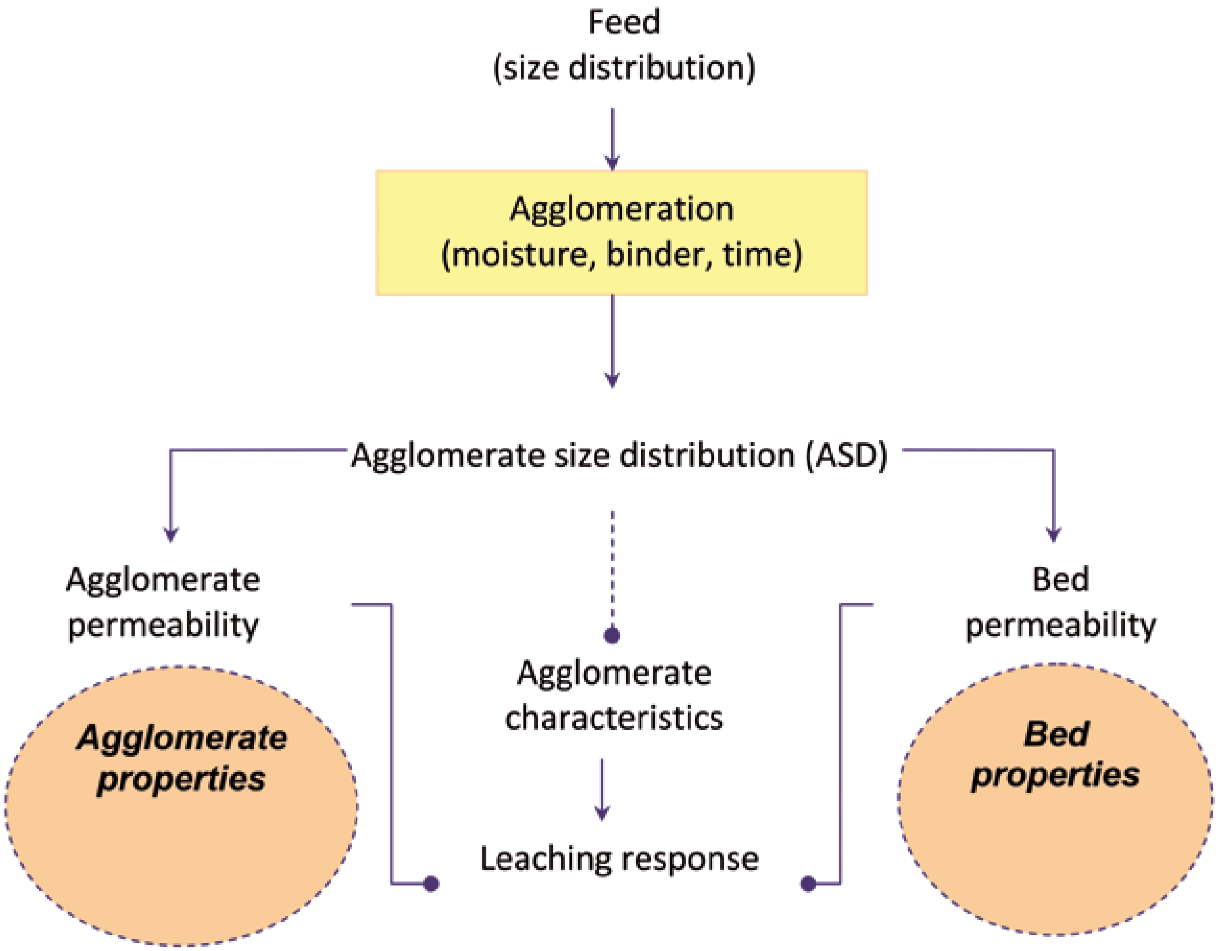

Figure 1 — Role of particle size in crushed ore agglomeration-heap leaching systems (ASD: agglomerate size distribution).

Table 1 — Consequences of improper agglomeration in heap leach operations.

Heap characteristic	Loose fines	Segregation during stacking	Compaction	Deteriorated permeability
Voidage	Decreased	Variable	Decreased	Uneven lixiviant flow
Hydraulic conductivity	Decreased	Variable (low/high)	Decreased	Severe ponding
Density	-	Variable (pockets)	Increased	Dead zones
Saturation	Increased	Issue/poor percolation	-	-

handling such as stacking and drop-off from conveyors before actually being placed on heaps (Herkenhoff and Dean, 1987). To achieve the desired size distribution or appropriate bed characteristics, crushed ore agglomeration has been used as a pretreatment step for the heap leaching of gold, copper, nickel and uranium ores containing significant amounts of fines and clay minerals (Bouffard, 2005; Dhawan et al., 2013).

Crushed ore agglomeration takes place through either the adherence of fine particles to coarse particles (rim agglomerates) or the adherence of fine particles to each other (nucleated/conglomerates) or a combination of both. The rim agglomerates were reported to be more stable and preferred for leaching (Tibbals, 1987). Due to the small amount of fines (up to 30%) in crushed ore agglomeration, rim agglomerates seem to be more prevalent. During drum agglomeration, residence time and binding agents dominate the growth and mechanism of agglomeration. The lack of quality agglomerates is one of the major reasons for several heap leach failures (Kappes et al., 2000). The effects of improper agglomeration on heap properties are summarized in Table 1 (Dhawan et al., 2013; Guzman et al., 2008; Kodali et al., 2011; Velarde, 2005).

The importance of quality agglomerates is evident. Because of the lack of fundamental knowledge or controlled studies, crushed ore agglomeration is still considered more of an art rather than a science. This has been well stated by Lu et al. (2007), "The agglomeration practice seems to fall largely in the realm of experience and practice." The role of particle size from an agglomeration-heap leaching point of view is schematically shown in Fig. 1 (Dhawan et al., 2012). It can be seen from Fig. 1 that the macroporosity (bed permeability) of the agglomerated ore for the heap is responsible for percolation in the leaching process, whereas the microporosity (agglomerate permeability) of the individual agglomerates may control transport processes such as diffusion in the leaching response.

One of the main challenges in the analysis, control and optimization of heap leaching operations is the precise determination of agglomerate size. Special care is needed while handling wet agglomerates. The sampling and sieving may involve interference such as coalescence and breakage of agglomerates, which alter the size distribution. One of the possible ways to overcome the handling issue involves the rapid freezing of the agglomerates with liquid nitrogen (Bouffard, 2005; Dhawan et al., 2013). Other studies reported drying the agglomerate sample and then sieving for the determination of agglomerate size distribution (ASD) (Bouffard, 2005; Kinard and Schweizer, 1987). Kodali et al. (2011) reported air drying of newly formed agglomerates below 30° C for 24 h to obtain dried agglomerated samples, which were further screened on a ro-tap shaker for 3 min at a very low shaking speed to avoid breakage. The use of a ro-tap shaker may be detrimental for nonbinder agglomerates, considering the weak nature and instability of these agglomerates.

Bouffard (2005, 2008) reported wet screening to identify the particles that make up the original agglomerates. Although longer mixing times involve growth and breakage of agglomerates, the overall effect of extended agglomeration times has been reported to be detrimental. Also, it was stated that higher moisture content results in a narrower size distribution of agglomerates and vice versa. However, the procedure for the determination of the ASD was not reported.

The applicability of population balance models for describing the size enlargement processes such as pelletization or crushed ore agglomeration has been highlighted (Bouffard, 2005). Published literature regarding the control and modeling studies of crushed ore agglomeration is very limited. The reasons may be the lack of understanding of the process, experimental difficulties and, also, the ambiguous role of size. In addition, it seems that the determination of the ASD of the nonbinder agglomerates is a tedious job, considering the moist state of the agglomerates, representative sample acquisition challenges, wide size spectrum (microns to inches) and stability issues (Dhawan et al., 2012).

Most agglomerators are selected based on solids residence time rather than degree of agglomeration (Miller, 2010). This statement indicates the lack of information available on agglomeration procedures and fundamental understanding in this field.

Numerous studies have been conducted on pelletization of fines. Agglomeration can be seen as "welding" of fine particles onto bigger particles and joining of fine particles to grow as a lump particle or a combination of various sizes joining to make a conglomerate. Pelletization is a form of agglomeration where pellets are formed by congregating mineral particles around a center.

Pelletization studies involve a narrow feed size distribution (~ micron size range). Due to a similar environment during agglomeration, the agglomerate growth is more uniform and controlled (due to less stochastic events, i.e., minimum collision between large agglomerates and small agglomerates in the drum). On the other hand, crushed ore agglomeration involves wide feed size distributions, which lead to significant differences in agglomerate growth mechanisms (layering and growth breakage). Moreover, the tumbling action in agglomeration drums, the meta-stable state of agglomerates (moist, friable on drying) and the measurement of agglomerate size significantly contribute to the stochastic nature of drum agglomeration. Recently, Nosrati et al. (2012) reported the agglomeration behavior of fine-size (feed -1 mm) nickel laterite ore. It closely resembles pelletization conditions rather than crushed ore agglomeration. Also, the process optimization was not evaluated.

Review of the literature indicates that no systematic studies regarding the optimization of drum agglomeration have been published. Furthermore, few authors have commented on the significance of agglomerate size because of the difficulty in measuring agglomerate size and the stochastic nature of the process (it is worth noting that wet greenball/agglomerate sizing has not been a problem in other industries). Moreover, the real challenge is to determine the quantitative effect of each parameter on the response and also the possible interactive effect of parameters in optimizing agglomerate size. This is often impossible through the conventional step-by-step optimization procedure, where all other parameters have to be fixed while studying the effect of a certain parameter. Considering these aspects, it becomes clear that the determination of the optimal conditions for the agglomeration of a specific ore with traditional experimental procedures is a cumbersome task, and more robust statistical designs should be considered.

The agglomeration of crushed ore with water, acid or polymeric flocculent ensures uniform wetting of surfaces and also promotes the swelling of clay particles prior to placement in the ore bed (Watling et al., 2011). Readett and Fox (2011) reported the agglomeration step as the most critical variable in the successful establishment of the nickel laterite Murrin Murrin heap leach plant in Australia. There are significant differences in characteristics/properties of copper, nickel and gold ores that impact heap leaching (Watling et al., 2011). These differences (such as moisture content, clay content, acid consumption and mineral dissolution) have been reported in detail elsewhere (Dhawan et al., 2013).

Therefore, the present study aims to understand the agglomerate size response for two different ores agglomerated in a laboratory batch drum. For this purpose, a design of experiments (DOE) procedure (Taguchi's L16 orthogonal array design composed of five factors at four levels) was used to determine conditions for maximizing average agglomerate size (d_{50}) and minimizing the amount of fines. The factors considered in this study include:

- Moisture content (nickel laterite ore: 34-37%; gold ore: 7-10%).
- Retention time (2-3.5 min).
- Drum speed (15-45% critical speed).
- Drum load (nickel laterite ore: 8-32%; gold ore: 6-22%).
- Acid concentration (150-600 g/L).

The analysis of variance (ANOVA), which is commonly used to establish the relationship between the experimental conditions and responses, was performed, and the significant experimental factors were identified.

Materials and methods

Materials. Nickel laterite and gold ores provided by two mining companies were used in this study. The ores were screened into various size fractions by the providers. The particle size distributions of ores used in the experiments are shown in Fig. 2. It can be seen from Fig. 2 that gold ore had a finer particle size than the nickel laterite ore. The weight percentages of -200 mesh for nickel laterite ore and gold ore were 15% and 11%, respectively. This indicates that nickel laterite ore had more very fine particles.

In the present work, fines are considered to be -1 mm particles instead of the -200 mesh (-75 micron) portion. This decision was made because of the limitation posed by dry screening of the agglomerated material. Moreover, on average, the fines remaining after agglomeration were less than 2%. Therefore, it was decided to limit the size measurement to a minimum

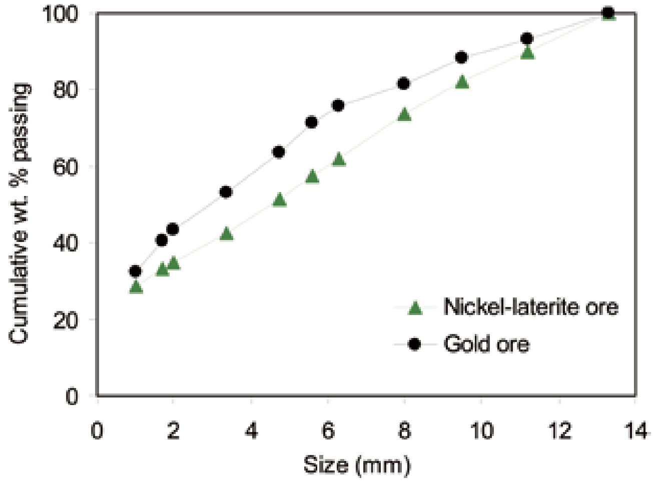

Figure 2 — Feed size distributions of both ores used in the experiments.

particle size of 1 mm.

The top size fed to the laboratory agglomerator was limited to 12.5 mm. This was done for two reasons: 1) to ensure that agglomerates were not too large for subsequent column leaching and 2) due to the limited drum size and capacity used in this study. It is worthwhile to mention that, in small drums, the presence of large particles hindered the growth of other agglomerates.

The bulk densities of the nickel laterite and gold ores were ~1.17 g/cm^3 and ~1.67 g/cm^3, respectively. Also, the natural moisture content of the nickel laterite and gold ore was measured to be 13.4% and 1.4%, respectively. About 0.5 kg of the ore sample filled approximately 30% of the drum, which is a typical maximum value according to the literature (Perry and Green, 2006). In nickel laterite ore, nickel is associated with different mineral phases, with chlorite/smectite as the major gangue minerals. For gold ore, quartzite/silica is the major gangue mineral. The nickel laterite ore contained a 24-wt.% clay minerals fraction. Analytical grade sulfuric acid (EMD Chemicals, 95-98%) and sodium cyanide (NaCN) solution (1,000 ppm) were used for nickel ore and gold ore agglomeration, respectively. DI water was used throughout the agglomeration experiments.

Agglomeration procedure. All agglomeration experiments were conducted using a small batch agglomerator, as shown in Fig. 3. The agglomeration drum was made using a 1-L fluorinated polyethylene (FLPE) Nalgene bottle 9 cm in diameter by 17 cm in length. Three polytetrafluoroethylene (PTFE) lifters placed 120° apart were attached in the drum. Each lifter was 2 cm wide, 14.3 cm long and 6 mm thick. The small batch agglomerator was driven with a 24-V dc motor and operated at variable speeds up to 76 rpm. The drum was mounted in a horizontal position and the rotation was set at the desired speed.

Each batch experiment consisted of two distinct steps, mixing and agglomeration. The feed was first mixed or homogenized at the experimental drum speed prior to agglomeration. Following mixing, the required solution (sulfuric acid for nickel laterite ore and cyanide for gold ore) was then added using a peristaltic pump while the drum was in motion. The solution was introduced using a distributor held along the central axis of the drum. The distributor had openings of 1 mm placed

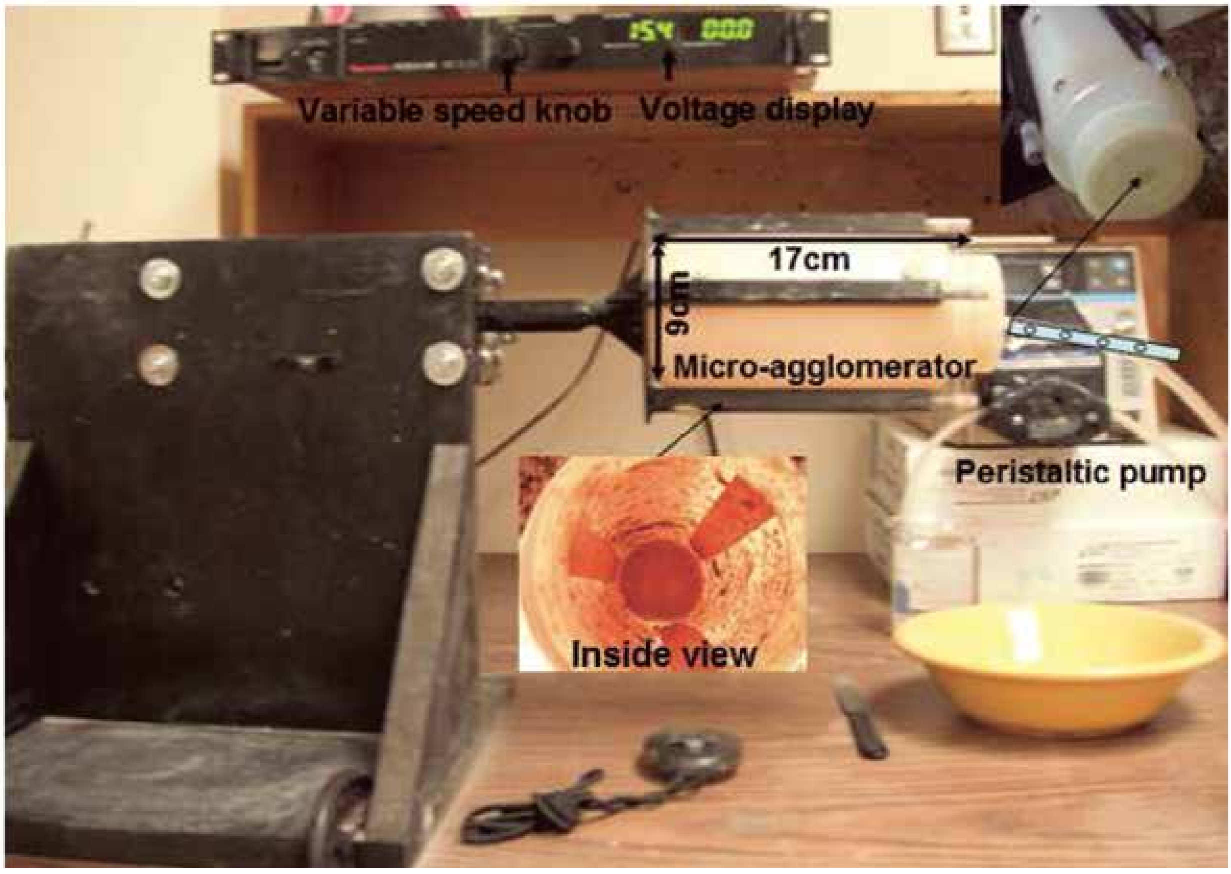

Figure 3 — Image of the small batch agglomerator and ancillary equipment used in this study.

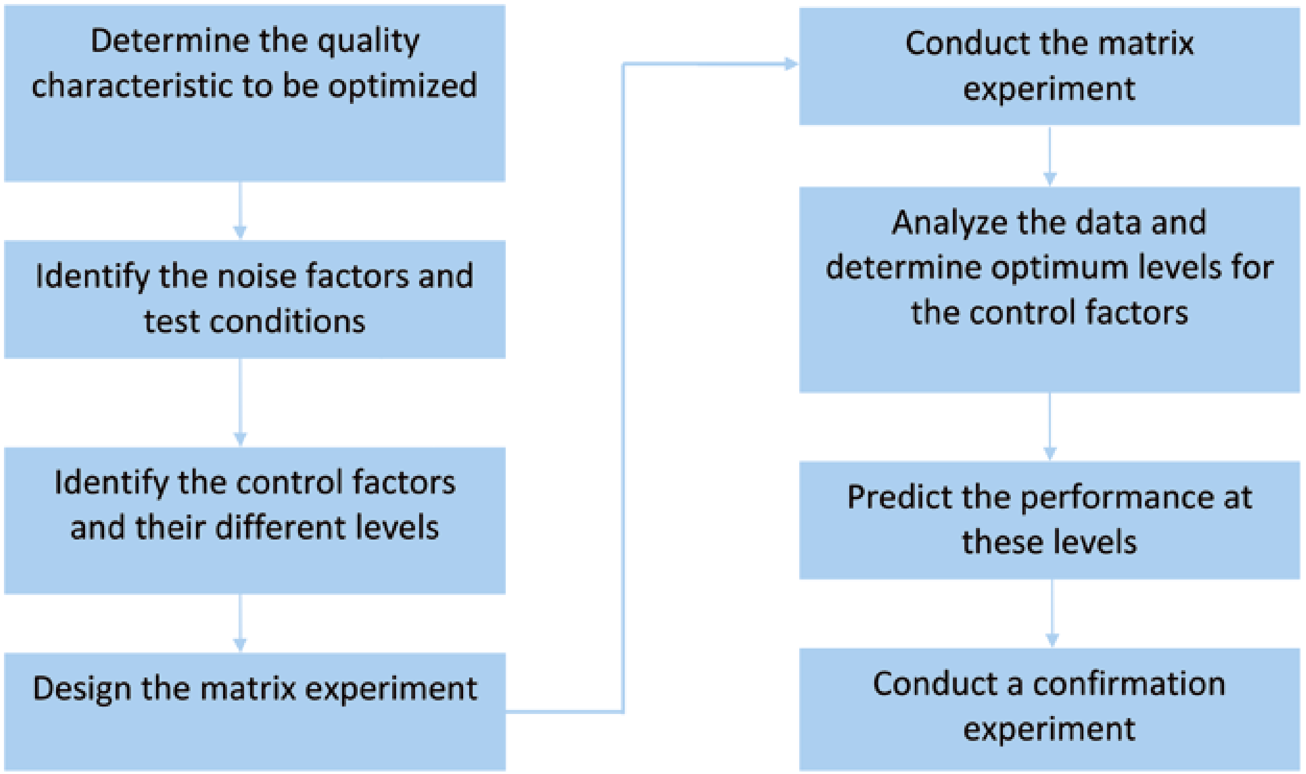

Figure 4 — Steps involved in the Taguchi design of experiments (adapted from Nik et al., 2012).

with 25 mm spacing on centers facing toward the charge. The liquid was distributed evenly across the ore within the drum for the first third of the retention time.

Following the agglomeration time, the agglomerates were unloaded from the drum and placed on a tarp at room temperature (~23-25° C) for one day. The entire sample was then air dried and manually sieved softly to determine the ASD. A ro-tap machine was only used while screening the run-of-mine ore. Regular screens (US Tyler mesh) from 19 mm to 1 mm were used for sieve analysis. After each experiment, a photograph of the product was taken.

Experimental design. The Taguchi method possesses distinct advantages over conventional experimental design methods, as it minimizes the variability around the target value. It utilizes orthogonal arrays from experimental design theory to study a large number of variables with a small number of experiments. Orthogonal arrays are subsets of the full factorial experiments, which are balanced in such a way that each variable parameter occurs the same number of times and no two experimental runs are the same, except for the repetition tests. The design significantly reduces the number of experimental configurations to be studied (Roy, 1995; Safarzadeh et al., 2007, 2008). The steps involved in Taguchi's approach are shown as a box diagram in Fig. 4.

The Taguchi method makes use of a loss function to measure the performance characteristics deviating from the target value. Then, the value of the loss function is transformed into a signal-to-noise (*SN*) ratio (Roy, 1995; Safarzadeh et al., 2007, 2008). Further, the *SN* ratio is used as a standard to indicate the ratio of sensitivity to variability and is also used to optimize the process (Nik et al., 2012). Generally, the *SN* ratio analysis can be performed in three forms of performance characteristic:

1. Smaller is better;
2. Larger is better, and
3. Nominal is the best.

The *SN* ratio for each level of the process parameters is calculated based on the *SN* analysis, as shown in Eqs. (1) and (2).

$$SN_S = -10\log\left(\frac{1}{n}\sum_{i=1}^{n} Y_i^2\right) \quad (1)$$

$$SN_L = -10\log\left(\frac{1}{n}\sum_{i=1}^{n} \frac{1}{Y_i^2}\right) \quad (2)$$

where SN_S and SN_L are the performance characteristics, n is the number of repetitions performed for an experimental combination and Y_i is the performance value of *i*th experiment. SN_L is used if the system is optimized when the response is as large as possible, whereas SN_S is used when the response is as small as possible.

Regardless of the category of the performance characteristic, the larger *SN* ratio corresponds to the better performance characteristic. In its simplest form, the *SN* ratio is the ratio of the mean response (signal) to the standard deviation (noise). It should be noted that *SN* ratios are merit functions that take into account response average and response variability. As the *SN* ratio increases, the variability of the response decreases. Therefore, the optimal level of the process parameters is the level with the highest *SN* ratio (Safarzadeh et al., 2007, 2008).

All experiments were carried out in random order to avoid noise sources that could take place during an experiment and bias the results. The interactive effect of parameters was not taken into account in the theoretical analysis, because some preliminary tests indicated that they could be neglected. The

Table 2 — L16 (4^5) orthogonal Taguchi array. The numbers 1-4 indicate four different levels of the parameters (A) moisture content, (B) time, (C) drum speed, (D) drum load and (E) acid concentration.

Experiment No.	Experimental factors A	B	C	D	E
1	1	1	1	1	1
2	1	2	2	2	2
3	1	3	3	3	3
4	1	4	4	4	4
5	2	1	2	3	4
6	2	2	1	4	3
7	2	3	4	1	2
8	2	4	3	2	1
9	3	1	3	4	2
10	3	2	4	3	1
11	3	3	1	2	4
12	3	4	2	1	3
13	4	1	4	2	3
14	4	2	3	1	4
15	4	3	2	4	1
16	4	4	1	3	2

validity of this assumption was checked by confirmation experiments conducted at the optimum conditions.

An orthogonal array, L16 (4^5) — five parameters with four levels each — was selected, as it is the most suitable for the conditions being investigated. Each experiment was repeated twice under the same conditions at different times to monitor the effects of noise sources in the agglomeration process. The L16 orthogonal array as shown in Table 2 is a table of integers whose column elements (1-4) represent the four levels of the column factors. Each row of orthogonal array represents a run, which is a specific set of factor levels to be tested.

Experimental parameters and their levels were determined by scoping experiments. The preliminary tests were carried out under standard room temperature conditions. Furthermore, humidity and temperature were not controlled during the experiments. For these reasons, humidity and temperature were not taken into consideration as parameters and any effect will appear as a noise factor. The temperature within the lab was approximately 23-27° C, while the humidity was around 10-40% relative humidity (RH). Noise factors are those parameters that are either uncontrollable or are too expensive to control such as variation in environmental operating conditions. Noise factors may or may not have a negative impact on system performance.

Based on the published information for crushed ore agglomeration and the preliminary experiments performed, (A) moisture content, (B) time, (C) drum speed, (D) drum load and (E) acid concentration were chosen as the five factors to be investigated at four levels, as shown in Table 3. It should be noted that the range of variables were selected from preliminary experiments to produce only agglomerates of "good" appearance (i.e., not too dry or too wet).

Based on scoping experiments, the amount of cement addition was fixed for gold ore experiments at 8 kg/t of ore. A 1,000-mg/L sodium cyanide solution was used for gold ore. The pH of the solution was adjusted to 11.5 using sodium hydroxide solution. No binder was used in the nickel ore.

It should be noted that in the Taguchi method, the experiment corresponding to optimum working conditions might not have been done during the experimental design stage. In such cases, the performance value corresponding to optimum working conditions can be predicted by using Eq. (3). The confidence interval at a chosen error level can be calculated from Eq. (4).

$$Y_{opt} = \frac{T}{n} + \left(\bar{A}_i - \frac{T}{n}\right) + \left(\bar{B}_j - \frac{T}{n}\right) + \ldots \quad (3)$$

$$C.I. = \pm\sqrt{\frac{F(1,n_2)V_e}{N_e}} \quad (4)$$

where n is total number of trials, T is the sum of all responses and $\bar{A}_i$, $\bar{B}_j$,… the average of responses at levels i, j, etc. F is the statistical F value at the desired confidence level with degrees of freedom of 1 and degrees of freedom of error, n_2; V_e the variance of error and N_e the effective number of replications.

Table 3 — Factors and their levels for the experimental design.

Factors	Levels: Nickel laterite 1	2	3	4	Gold ore 1	2	3	4
Moisture (%)[a]	34	35	36	37	7	8	9	10
Time (mins)	2	2.5	3	3.5	2	2.5	3	3.5
Drum speed (%Nc)[b]	15	25	35	45	15	25	35	45
Drum load (%)	7.9	15.8	23.7	31.6	5.5	11	16.6	22
Acid concentration (g/L)	150	300	450	600	-			

[a] $$\text{Moisture }(\%) = \frac{M_{wet} - M_{dry}}{M_{wet}} \times 100$$

$M_{wet} - M_{dry}$ is the weight of the solution used in the agglomeration and M_{wet} is the weight of the ore samples after solution has been added.

[b] $$N_c = \frac{42.3}{\sqrt{D}}$$

N_c is the critical speed or speed at which the material will be carried around the drum by centrifugal force and D is the inside diameter of the drum (m).

Results and discussion

The d_{50} and <1 mm (%) responses including replicates 1 and 2 from the four-level L16 (4^5) orthogonal design for nickel laterite and gold ores are listed in Table 4. The collected data were analyzed using a Microsoft Excel spreadsheet to evaluate the effect of each parameter on the optimization criteria.

Taguchi recommends analyzing the variation using an appropriately chosen signal-to-noise ratio (*SN*). In these experiments, the system is optimized when the response is as large as possible for average agglomerate size; therefore, SN_L is considered and factor levels that maximize the SN_L ratio are optimal. In contrast, the system is optimized when the response is as small as possible for fines, SN_S is considered and factor levels that maximize the SN_S ratio are optimal.

While the ANOVA calculations were performed using an Excel spreadsheet, the performance statistics curves were obtained using Minitab 15.0 software (Minitab Inc., USA). In statistics, random errors are assumed to produce residuals that are normally distributed. Also, the residuals should fall in a symmetrical pattern and have a constant spread throughout the range. After analysis, noise terms were determined to be normally distributed based on the acceptable trend line obtained in normal probability plots.

In Taguchi optimization, researchers often make use of analysis of variance (ANOVA) to determine the factors that influence the average response and also the signal-to-noise ratio (Nik et al., 2012; Roy, 1995; Safarzadeh et al., 2007, 2008). ANOVA is also used to estimate the variance of error and to quantify the effect of each factor on the performance statistics. The ANOVA procedure results in the calculation of the sum of squares (S), degree of freedom (DOF), mean square (variance) and associated *F*-test for significance (*F*). It is generally performed to see whether the process parameters are statistically significant. The *F*-value for each process parameter indicates which parameter has a significant effect on the agglomeration process and is simply a ratio of the squared deviations to the mean of the squared error. Usually, the larger the *F*-value, the greater the effect on the agglomeration process (average size and percent fines) due to a change in the process parameter. Optimal combination of the process parameters can be predicted using ANOVA analysis and performance characteristics. *S* (sum of squares), *V* (mean square variance), *F* (variance ratio), *S'* (pure sum of squares) and *P* (percentage contribution on response) were calculated based on SN_L and SN_S data.

It will be noticed that some of the parameters are pooled from ANOVA analysis. If a factor's percentage contribution is small, the sum of squares (*S*) for that factor should be merged with the error. This procedure is known as "pooling," and is accomplished by removing the smallest sum of squares from ANOVA analysis. The pooling process continues with the factors having larger effects, until the pure sum of squares (*S'*) for the rest of the factors becomes a positive value. Pooling should be performed until the DOF of error is approximately half the total DOF of the experiment (Roy, 1995).

Table 4 — Experimental values for average agglomerate size (d_{50}) and percent fines.

	Responses (d_{50} (mm))				Responses (<1 mm (%))			
	Nickel laterite		Gold ore		Nickel laterite		Gold ore	
Run	1	2	1	2	1	2	1	2
1	4.82	4.69	4.31	4.27	11.62	12.64	10.03	10.93
2	5.32	4.74	5.44	5.13	16.47	10.53	3.72	3.83
3	6.81	7.81	5.50	6.26	6.90	3.98	2.04	6.26
4	7.22	7.34	7.09	7.32	3.83	3.80	1.32	7.33
5	5.92	7.17	8.13	8.28	7.50	4.80	1.38	1.02
6	5.85	7.22	9.32	8.63	14.14	9.46	1.41	1.69
7	6.05	5.50	5.38	4.95	6.37	5.55	5.84	4.95
8	7.94	7.57	6.73	6.88	1.52	1.73	2.18	6.89
9	7.54	10.01	10.96	10.41	6.39	5.50	1.38	1.83
10	8.45	8.92	9.11	7.84	1.49	0.86	1.29	1.44
11	6.22	6.95	8.94	8.23	5.90	2.67	1.53	8.24
12	5.15	5.81	5.83	5.59	7.33	6.24	4.56	5.59
13	7.62	7.41	7.25	7.84	2.97	3.16	3.73	2.18
14	7.44	5.04	6.06	5.66	6.34	5.95	4.43	4.18
15	11.99	11.85	12.01	9.01	2.13	2.28	2.90	9.01
16	5.97	7.21	9.73	8.97	11.23	7.17	0.63	0.51

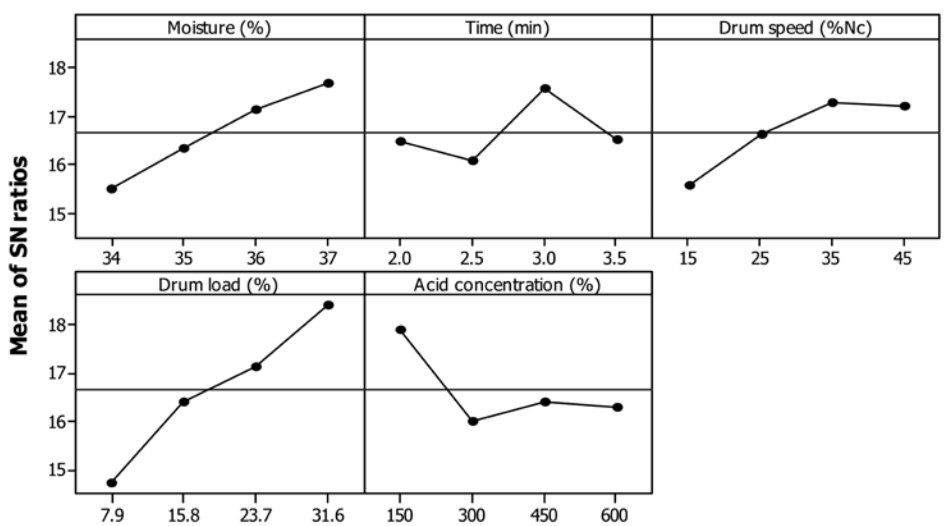

Figure 5 — Effect of controllable factors on performance statistics (SN_L for d_{50}) for nickel laterite ore.

Nickel laterite ore. The Taguchi method uses graphs of the marginal means of each factor, as shown in Fig. 5. The usual approach is to examine the graphs and pick the best performer (winner). In Fig. 5, the effects of controllable factors on SN_L for the average agglomerate size (d_{50}) are shown. According

Table 5 — Analysis of variance (ANOVA) based on SN_L data for nickel laterite and gold ores (d_{50}).

Factors	Nickel laterite (F = 4.49)						Gold ore (F = 4.38)					
	S	*f*	*V* (*S/f*)	*F*	*S′*	*P*	*S*	*f*	*V* (*S/f*)	*F*	*S′*	*P*
Moisture (%)	10.76	3	3.59	2.21	5.88	9.89	28.38	3	9.46	49.44	27.81	34.04
Time (min)			Pooled						Pooled			
Drum speed (% N_c)	7.23	3	2.41	1.48	2.35	3.95	0.77	3	0.26	1.34	0.19	0.24
Drum load (%)	27.99	3	9.33	5.74	23.11	38.84	51.41	3	17.14	89.54	50.83	62.22
Acid concentration (g/L)	8.66	3	2.89	1.78	3.78	6.36	-					
Error/others	4.87	3	1.62	1.00	-	40.96	1.15	6	0.19	1.00		3.51
Total	59.51	15	-	-	-	100	81.70	15	-	-	-	100

Table 6 — Analysis of variance (ANOVA) based on SN_S data for nickel laterite and gold ores (percent fines).

Factors	Nickel laterite (F = 4.49)						Gold ore (F = 4.38)					
	S	*f*	*V* (*S/f*)	*F*	*S′*	*P*	*S*	*f*	*V* (*S/f*)	*F*	*S′*	*P*
Moisture (%)	81.10	3	27.03	2.61	50.01	8.86	126.89	3	42.30	5.72	104.70	22.04
Time (min)			Pooled				177.65	3	59.22	8.01	155.46	32.72
Drum speed (%N_c)	192.09	3	64.03	6.18	160.99	28.51	33.82	3	11.27	1.52	11.63	2.45
Drum load (%)	58.40	3	19.47	1.88	27.30	4.83	114.59	3	38.20	5.16	92.40	19.45
Acid concentration (g/L)	201.96	3	67.32	6.49	170.86	30.26	-					
Error/others	31.10	3	10.37	1.00	-	27.54	22.19	3	7.40	1.00		56.07
Total	564.65	15			-	100	475.14	15				100

to Fig. 5, it is evident that with increasing drum load and moisture, SN_L responses increase significantly. The effects of acid concentration and drum speed are smaller on SN_L. Time had minimal effect on SN_L over the range examined.

Also, according to ANOVA results (Table 5), based on *P* values (i.e., percent contribution), drum load followed by moisture has the greatest effect on the SN_L response, as confirmed through performance statistics observations. For maximizing the agglomerate size, only the *F*-value of the drum load factor in nickel laterite ore is greater than the extracted *F*-value (F = 4.49) from the table for a 95% confidence level. This means that the variance of only the drum load factor is significant compared with the variance of error and only moisture has a meaningful effect on the response. It is believed that these effects caused changes in agglomerate charge dynamics. Higher drum filling and higher moisture content lead to a higher probability of coalescence of particles or more growth due to overcrowding, hence to larger agglomerates (e.g., larger SN_L).

Increasing drum speed causes an increase in SN_L, followed by a plateau in values. This may be due to increased energy, which may inhibit the further growth of agglomerates through breakage. This observation of the effect of drum speed is the opposite of what was recently reported by Nosrati et al. (2012). These authors reported that higher drum speeds result in higher kinetic energy, as well as a higher number of interparticle collisions. However, it is important to mention that their work resembles pelletization conditions (feed < 1 mm) rather than crushed ore agglomeration. Also, the ore type (nickel laterite; high clay content) and drum characteristics were different. On the other hand, in the present study, feed characteristics are quite different, as mentioned in the materials section.

Increasing acid concentration led to a decrease in SN_L, followed by a plateau in values. This behavior is due to less solution being available at the higher acid concentration. In terms of maximizing the SN_L for nickel laterite ore, time (3 min), drum load (31.6%), drum speed (35% N_c) and acid concentration (150 g/L) were selected from this analysis. Moisture does not exhibit a distinct optimum value over the range tested.

For nickel laterite ore, the plots of SN_S for percent fines (-1 mm) versus the controllable factors are shown in Fig. 6. Based on this figure and the ANOVA results (Table 6), it is clear that, with decreasing acid concentration, the SN_S response decreases sharply and then increases. This could be due to a decrease in solution volume at higher acid concentration. Also, with increasing drum speed, the SN_S response significantly increases (e.g., more -1 mm particles are present after agglomeration). It is believed that at higher drum speeds, fines probably adhere in a weak manner or are loosely bound.

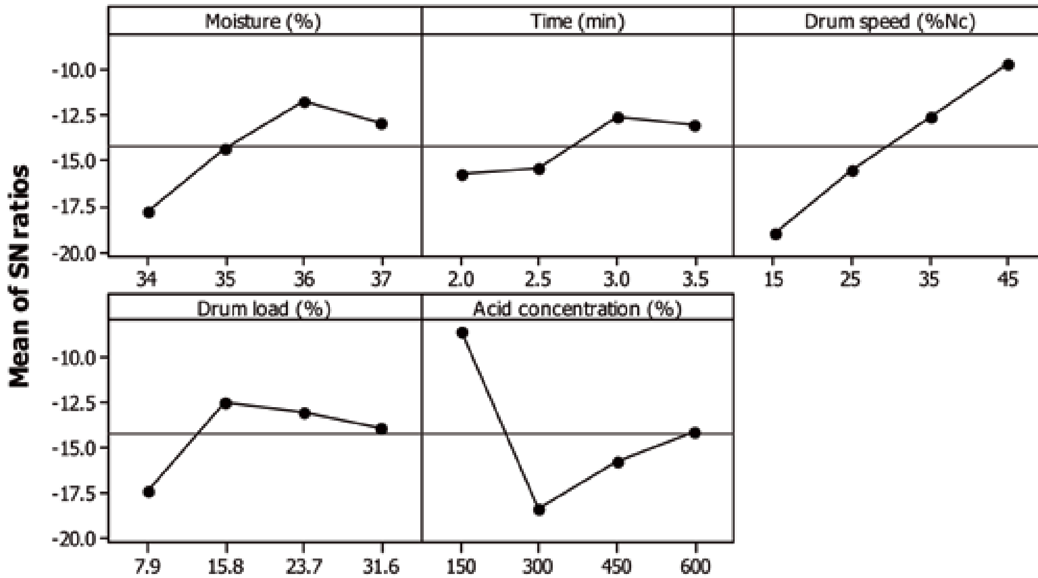

Figure 6 — Effect of controllable factors on performance statistics (SN_S for -1 mm particles) for nickel laterite ore.

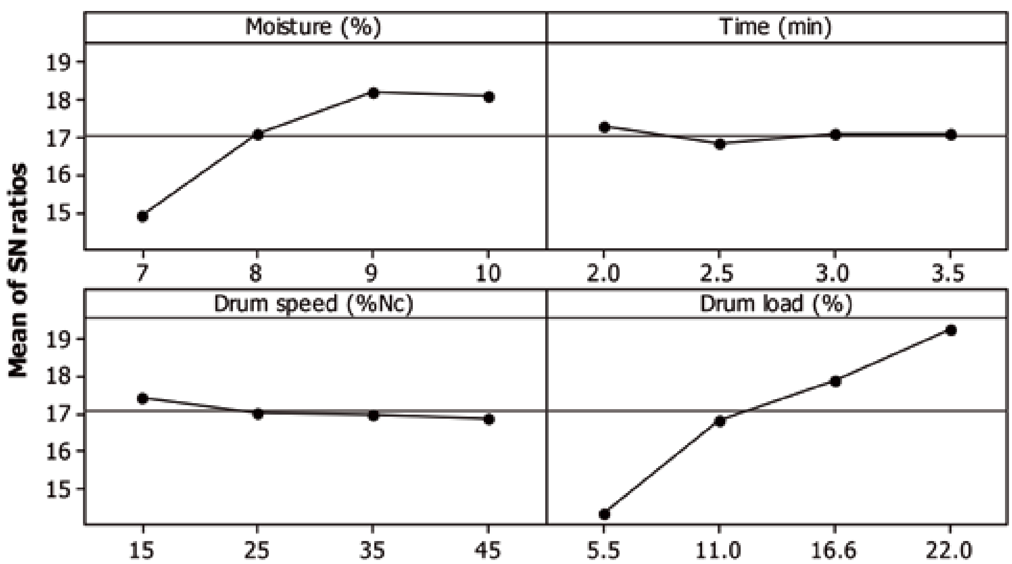

Figure 7 — Effect of controllable factors on performance statistics (SN_L for d_{50}) for gold ore.

Increasing moisture also led to an increase in the SN_S response, as indicated by the ANOVA results (Table 6). This response was expected, as more liquid usually helps with fine particle attachment due to the presence of more solution for liquid bridging. But at the last step, the SN_S response decreases, which may be because the added liquid saturated the particle surfaces. This decreased the capillary forces holding the smaller particles to the large particles in the agglomerates. Overall, the strength-moisture relationship loosely seems to follow results for iron ore pelletization strength as discussed by Nosrati et al. (2012). The other parameters have marginal effects on the SN_S for percent fines (-1 mm). In terms of maximizing the SN_S for nickel laterite ore, time (3 min), drum load (15.8%), drum speed (45% N_c), acid concentration (150 g/L) and moisture (36%) were selected as the best conditions.

Gold ore. The effects of controllable factors on SN_L for average agglomerate size (d_{50}) for gold ore are shown in Fig. 7. According to this figure and the ANOVA results (Table 5), it is evident that, with increasing drum load, the SN_L response increases significantly. This response is similar to that observed for nickel laterite ore and, again, may be caused by the higher probability of coalescence of particles and more growth with longer residence times. Moisture also had a meaningful effect on SN_L over the range examined. One possible reason is the liquid phase viscosity as a factor in increasing the "stickiness" of the gold ore. Alternatively, the higher surface area of the laterite ore may prevent the "free" moisture for sequestering stray fines.

Unlike nickel laterite ore, gold ore did not exhibit a trend between SN_L and time and drum speed. This difference may be due to the mineralogy of the ores or stable product formation due to cement as binder in the gold ore. Due to the presence of cement binder, the gold ore produced better agglomerates that were more "sticky." This property of the ore may have negated the effect of drum speed and time. In terms of maximizing the SN_L for gold ore d_{50}, time (2 min), drum load (22%), drum speed (15% N_c) and moisture (9%) were selected.

Figure 8 displays the effects of controllable factors on SN_S for percent fines (-1 mm) from gold ore agglomeration. Based

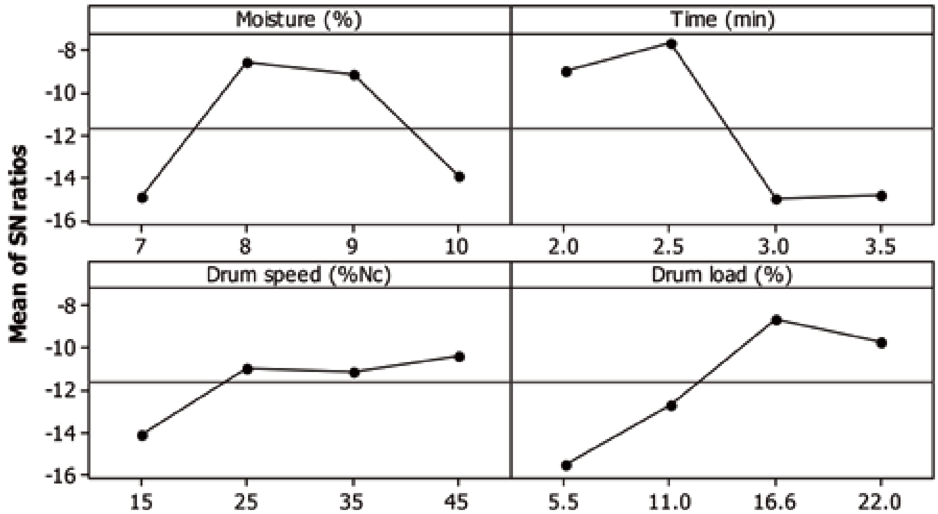

Figure 8 — Effect of controllable factors on performance statistics (SN_S for -1 mm particles) for gold ore.

on ANOVA results (Table 6), the *F*-test results for minimizing the fines with gold ore indicate that time, moisture and drum load had meaningful effects on the response. This could be due to better mixing achieved at an optimum combination of time, moisture and drum load. Based on the data, only drum speed had a negligible effect. This is probably caused by the general lack of fine material (all values are less than 1%) after agglomeration. The lack of fine material after agglomeration is encouraging from a practical standpoint considering the clay contents in ores. The very fine particles in nickel laterite ore do not agglomerate well compared to the agglomerates produced from gold ore, primarily due to the presence of cement as a binder in gold ore. From a visual inspection standpoint, gold ore possessed a more sticky nature than nickel laterite ore. In terms of maximizing the SN_S for gold ore, time (2 min), drum load (22%), drum speed (15 % N_c) and moisture (9%) were selected.

It is interesting to observe that, for the two given ores, different parameters affect the agglomeration response differently based on percent contribution (*P* value). One of the primary reasons for these differences is likely the significant difference in clay content in nickel laterite ore versus gold ore. Besides mineralogy, another difference is the natural moisture content (nickel laterite ore (13.4%) > gold ore (1.4%)) and, of course, the presence of cement as a binder in gold ore. From visual observation and experience, gold ore seems to be more sticky. Moreover, based on scoping experiments at longer retention times (>4 min), nickel laterite ore was also observed sticking on drum walls.

To examine the ore mineralogy differences, X-ray diffraction (XRD) analysis was performed for feed materials and agglomerates. The analyses did not detect the formation of any new phases. However, the formation of new phases cannot be ruled out, because these phases may form at very low amounts below the detection limit of XRD. Lu et al. (2007) observed the formation of new phases ($FeSO_4 \cdot 5H_2O$ or $MgSO_4 \cdot 4H_2O$) after agglomeration and acid curing (two weeks' rest period for ore after agglomeration) for copper ores (Zaldivar ore, Placer Dome, South America). However, the authors of the current paper used only one day of air drying for the different ore types, which may be the reason for the lack of such observation.

Optimizing agglomeration and validating the design results. Finally, using these findings and modeling significant effects with the Taguchi method, results for all combinations of levels can be predicted. Then, these predictions should be confirmed by further experiments. The confidence intervals (at 95% confidence level or 5% risk) were calculated using Eq. (4). The calculated confidence level values for average agglomerate size and fines content for both ores are shown in Table 7. Table 7 also shows the experimental and predicted values for both ores for average agglomerate size and percent fines at optimum conditions predicted through the Taguchi design. It is noted that the amount of fines for nickel laterite and gold ore predicted from Eq. (3) is negative, which does not have any physical meaning. However, when they are added to the calculated confidence interval, they give reasonable numbers. Therefore, it is possible to achieve acceptable results with implementation of the Taguchi design of experiments.

Extremely large agglomerate size can be detrimental based on percolation behavior (larger agglomerate size increases void space) but, at the same time, it is well understood that fines should be minimized, but not completely eliminated. This is because, if the heap has a large void space, the solution will pass through without the complete reaction inside the ore particles/agglomerates and, hence, there will be issues with leaching recovery and solution management. On the other hand, fines are necessary to "spread" the applied lixiviant and provide wetting of the ore away from drippers. Therefore, there exists a trade-off between these criteria, and the optimum experiments were selected based on the average of the parameter levels for maximum SN_L and SN_S values in each ore.

It is quite clear that the experimental response values in Table 7 are within the range of predicted values (calculated using Eq. (3)) from the Taguchi method. The results showed that, for nickel laterite ore, maximum average agglomerate size and minimum percent fines occurred near the conditions of drum load (D_3: 23.7%), moisture ($A_{3.5}$: 36.5%), time (B_3: 3 min), drum speed ($C_{3.5}$: 40% N_c) and acid concentration (E_1: 150 g/L), whereas for gold ore, maximum average agglomerate size and minimum percent fines occurred under the following conditions; drum load ($D_{3.5}$: 19.3%), moisture ($A_{2.5}$: 8.5%), time ($B_{1.5}$: 2 min 15 s) and drum speed ($D_{3.5}$: 40% N_c). These results were acceptable within the confidence limits that were calculated in each case (Table 7). The subscripts following the capital letters mean the level of factor under consideration. In

Table 7 — Comparison of experimental and predicted results.

Ore	Experiment	Average agglomerate size (d_{50}) Run 1	Run 2	Average	Predicted	Fines (%) Run 1	Run 2	Average	Predicted
Nickel laterite	$A_{3.5}B_3C_{3.5}D_3E_1$	7.50	8.75	8.12	11.07 ± 2.97	1.16	1.10	1.13	-0.77 ± 2.09
Gold	$A_{2.5}B_{1.5}C_{1.5}D_{3.5}$	10.00	10.11	10.05	10.96 ± 1.27	1.36	1.99	1.68	-0.04 ± 1.70

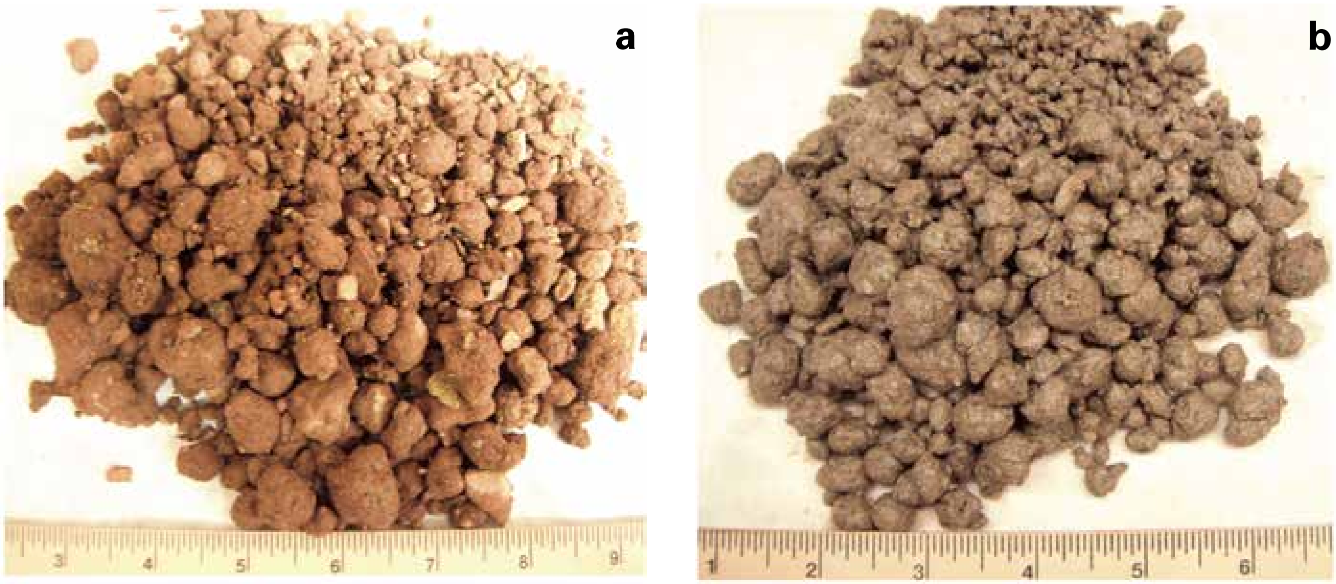

Figure 9 — Agglomerates obtained under the optimum process conditions for (a) nickel laterite ore and (b) gold ore.

the case of rational subscripts, they mean the level between the indicated integer and the next integer. For example, $D_{3.5}$ means that the level considered for drum speed is in between levels 3 and 4.

Figure 9 shows the images of the agglomerates produced under the conditions explained in Table 7 for nickel laterite and gold ores. It can be seen from Fig. 9 that both ores possess a smooth surface, but gold ore has a distinct surface sheen. From an operator point of view, an ideal agglomerate is usually one that appears to be visually good, i.e., neither too wet nor too dry. One of the ways to determine optimum agglomeration moisture is through visual inspection and the appearance of the agglomerate surfaces (Guzman et al., 2008; Kodali et al., 2011; Velarde, 2005). Due to the presence of high clays in nickel laterite ore, surface sheen was never observed (Fig. 9a). Also, gold ore produced more regular shaped agglomerates than nickel laterite ore, probably due to the presence of cement as a binder. According to Guzman et al. (2008) adequate moisture content leads to a change in the ASD, a reduction in the surface roughness and, thereby, an increase in the surface reflectance of the agglomerate.

Based on the fact that the results obtained from the confirmation experiments are nearly within the calculated confidence intervals for both ores (see Table 7), it can be concluded that experimental results are within the predicted range. This indicates that interactive effects of parameters are most likely negligible (Roy, 1995).

Conclusions

Crushed ore agglomeration has been used as a pretreatment step for the heap leaching of ores containing significant amounts of fines and clay minerals. The primary objective of crushed ore agglomeration is the creation of a size distribution (agglomerates of uniform size) that leads to minimum segregation, improved percolation and uniform solution distribution.

The effect of controllable operating factors including moisture content (nickel laterite ore: 34-37%; gold ore: 7-10%), retention time (2-3.5 min), drum speed (15-45% N_c), drum load (nickel laterite ore: 8-32%; gold ore: 6-22%) and acid concentration (nickel laterite ore 150-600 g/L) on nickel laterite and gold ore agglomeration were evaluated using the Taguchi method described in this paper with the application of an L16 (4^5) orthogonal array. As a result, higher moisture, higher retention time, higher drum load, intermediate drum speed and lower acid concentration within the ranges studied are recommended for nickel laterite ore small-scale batch agglomeration. Optimum operating conditions for maximizing agglomerate size for nickel laterite ore are moisture (37%), retention time (3 min), drum speed (35% N_c), drum load (31.6%) and acid concentration (150 g/L). On the other hand, optimum operating conditions for minimizing the amount of fines are moisture (36%), retention time (3 min), drum speed (45% N_c), drum load (15.8 %) and acid concentration (150 g/L).

However, for gold ore, different conditions are recommended as optimal operating conditions for small-scale batch agglomeration. Optimum operating conditions for maximizing the agglomerate size were found to be moisture (9%), retention time (2 min), drum speed (15% N_c) and drum load (22%). On the other hand, optimum operating conditions for minimizing the amount of fines are moisture (8%), retention time (2.5 min),

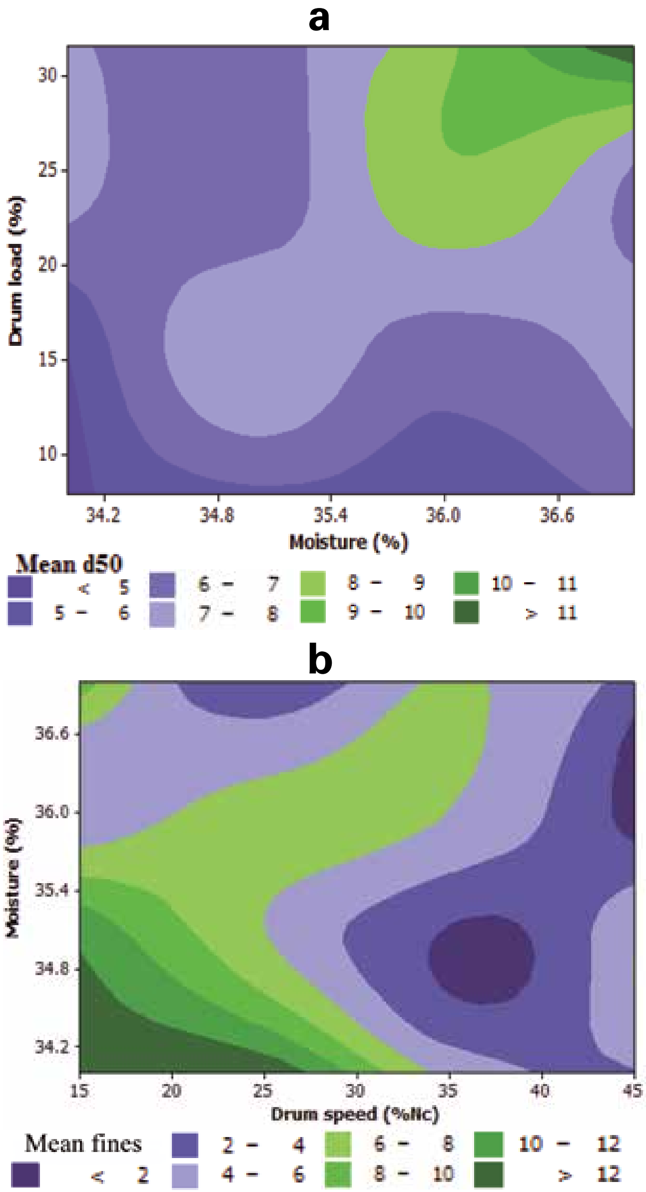

Figure 10 — Contour plots showing response surfaces for (a) d_{50} (mm) against moisture and drum load and (b) percent fines (-1 mm) against moisture and drum speed for nickel laterite ore.

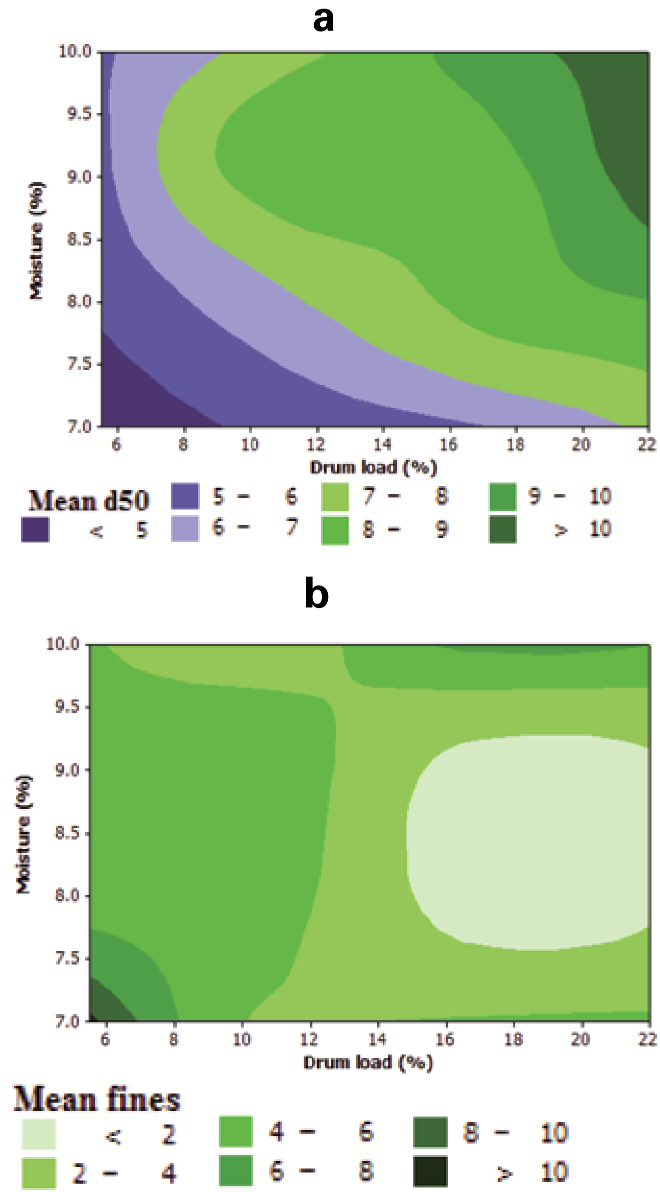

Figure 11 — Contour plots showing response surfaces for (a) d_{50} (mm) against moisture and drum load and (b) percent fines (-1 mm) against moisture and drum load for gold ore.

drum speed (45% N_c) and drum load (16.6%).

Analysis of variance (ANOVA) indicated that some parameters had a more significant effect on the response than other variables. For the nickel laterite ore, drum load affected the maximum average agglomerate size. For minimizing fines, acid concentration and drum speed were statistically significant. For the gold ore, drum load and moisture were the only factors that statistically affected the maximum average agglomerate size. Time, moisture and drum load were statistically significant toward minimizing the fines. This difference in behavior is believed to be caused by the presence of clays in nickel laterite ore and the presence of cement as binder in gold ore. Other possible reasons can be differences in bulk density (nickel laterite ore: 1.17 g/cm^3, gold ore: 1.67 g/cm^3) and natural moisture (nickel laterite ore: 13.4%, gold ore: 1.4%).

With the help of contour plots, as shown in Fig. 10a, the areas where maximum average agglomerate size (d_{50}) can be obtained for nickel laterite ore include higher moisture content and drum load. In terms of agglomeration process dynamics, it may be explained according to the fact that higher moisture content and higher drum load may ease the formation of larger agglomerates due to the higher probability of adherence of fine material. In Fig. 10b, the area where minimum fines percent in nickel laterite ore can be obtained occur with higher drum speed (35-40% N_c) and lower moisture (up to 35%). This can be explained based on the fact that at higher drum speeds and lower moisture content the agglomerate charge will undergo more tumbling, which may promote more adherence of loose fines to existing charge/agglomerates.

Areas where maximum average agglomerate size (d_{50}) can be obtained for gold ore include higher moisture content and drum load (Fig. 11a). However, the area where minimum fines percent in gold ore can be obtained occurs at a higher drum load (16-22%) and lower moisture content (up to 9%) (Fig. 11b).

It is well known that plant operators have been making use of subjective tests such as visual inspection to identify the quality of agglomerates (Dhawan et al, 2013; Guzman et al., 2008). Based on their inspection, changes are made to agglomerating parameters (using control knobs such as agglomerating reagents, drum load, drum speed and time) to achieve better results. Considering these facts, certainly, these contour plots can be of importance for plant operators to achieve the desired agglomerate characteristics.

From this test work, it is observed that nickel laterite and gold ores reacted differently to agglomeration parameter changes. This indicates that optimization of each ore must be considered individually.

Additionally, the change of acid concentration is often made with regard to leaching performance and acid consumption found in column leaching. This data indicates that changes in acid concentration can have an effect on agglomerate sizes and thus could affect heap permeability. Obviously, the Taguchi design used lab-scale results to understand the agglomeration behavior but more testing needs to be done using large scale drums to verify the findings reported in this study. Based on similar works such as iron ore pelletization, batch lab-scale drums have contributed significantly toward the understanding of growth kinetics studies. Nevertheless, based on the present research results, it seems plausible that high clay containing ores should emphasize acid concentration as a control factor and, in general, the effect of drum load should not be ignored. Needless to say, there are more quality control tools, such as mechanical stability of agglomerates, compression strength and porosity, to be studied in future work.

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