Laboratory Settling Tests Applied to Define Bauxite Consumption Strategy in Alumina Refinery

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Abstract

Alumina refineries are usually designed to consume a specific source of bauxite, however the usage of bauxites with different grades of available alumina, reactive silica and iron content is becoming more prevalent as a requirement to balance financial and technical goals. The mineralogical composition of the bauxite is also one of the main parameters used to evaluate the capability of each alumina refinery with respect to the bauxite source. Performance within the clarification area can be affected by the mineralogy of the solids that must be separated. This paper outlines work that has combined data from XRD analysis with laboratory settling test results to establish flocculant dosage and slurry pumping requirements to help the refinery to define a consumption strategy for different bauxite sources. This strategy can be oriented/viewed in terms of bauxite blending in the refinery feed and the required flocculant dosages or co-dosages (combination of different flocculant products). Settling rates, compaction and clarity in the overflow (supernatant) have been evaluated for each combination of bauxite feed, adjusting flocculants dosages. The conclusion of this work is that it is possible to apply lab settling tests with success to get an estimate of how different bauxite grades can be blended and added into the refinery feed, minimizing impacts on refinery production and raw materials performance.

Keywords: Bauxite; Bayer; settling rate, compaction, clarification

1. Introduction

Every alumina refinery is originally designed to process a predetermined kind of bauxite in a tight range of variability with regards to its mineralogical and chemical characteristics. With reference to the refinery observed in this article, traditionally only bauxite from Northern region of Brazil was consumed until a few years ago. More recently, a significant effort has been undertaken to understand how to take advantage of other bauxite sources which, from time-to-time, may have more favorable economics with regards to availability and transportation.

The bauxite from other deposits or having originated from a different deposition process, mainly sourced from Africa, obviously have different chemical and mineralogical composition such as variations in the boehmite and goethite content. Prior to consuming a new bauxite, a lot of information needs to be studied to define the strategy of consumption, such as blending with the local bauxite, and also flocculant usage/dosage requirements.

Therefore, some laboratory test work is required to be undertaken to:

1. Identify the mineralogical and chemical composition of the said ore and,

2. Gain an insight into the settling characteristics of the residue produced from the digestion of the new bauxite. This is done by performing bench settling tests simulating the conditions of the clarification process within the refinery.

It is important that the above work is carried out well in advance of processing the ore or blend of ores so that when a given different bauxite starts to feed the refinery the strategy of blending and flocculent usage/consumption will be already defined.

2. Settling and Clarification Process

Clarification (solid-liquid separation) is a key step in the Bayer process. This process consists of separating the bauxite residue from the green liquor containing soluble sodium aluminate ensuring that upon arrival to the Precipitation circuit, the liquor is completely devoid of any solid particles. Usually alumina refineries combine two subsequent steps to perform the solid-liquid separation: thickening plus filtering. The thickening of residue from digestion step is conducted inside tanks named thickeners (other alumina operators may refer to these as decanters or settlers but the function remains the same) with subsequent "washing" of the dense underflow occurring in washers that works with a water counter-current flow against the residue with the objective to recovery soda and also to further thicken the slurry before sending it to the residue lakes [1].

To achieve effective solid-liquid separation, flocculants (macromolecular polymers) are applied to induce aggregation of the residue particles. The polymer acts by rapidly adsorbing onto the surface of multiple particles causing the fine suspended solids to form into larger structures (aggregates) which settle faster. The separation of solids and green liquor must be conducted quickly or alumina trihydrate will precipitate during clarification. This is known as auto-precipitation and results in loss of production. It is vital to alumina production that clarification be effective. Poor flocculation can lead to excessive fines in the overflow which can blind the filter–cloths in the security filtration building leading to lower production rates. Additionally, any solids which pass through the filter cloth can contaminate the product. Ineffective flocculation may also lead to higher loss of caustic by entrainment within the residue [2].

The flocculant dosages employed are related to the composition of Bayer residue with reference to the mineralogy. Most common minerals are hematite, goethite and quartz with anatase, calcite and non-digested aluminates also being found.

3. Mineralogical Characterization – Qualitative Analysis by XRD

XRD diffraction patterns are characteristic of a substance and can therefore be used for identification when compared to a standard diffractogram. The ICDD – International Centre for Diffraction Data - has a database containing 848 000 diffractometric standards with interplanar distances and intensities diffracted to the planes *hkl* of the crystalline structure listed [3].

The procedures to handle the identification of the crystalline phases in a sample were based on the Hanawalt method, detailed by Cullity [3]. Nowadays automatic systems are applied in crystalline phase identification where the most intense peaks of each phase contained in the datasheet are compared to the diffractogram obtained to the sample. Results are expressed according to the compatibility comparative probabilities [4]

4. Evaluated Samples

Table 1 illustrates the chemical composition of a number of different bauxite sources successfully consumed in Alumar Refinery. These results have been used to define strategies for processing these bauxites or blends of these bauxites in the future.

Table 1. Approximately chemical composition of different bauxite sources.

Samples/originated from	Al_2O_3 (%)	SiO_2 (%)	Fe_2O_3 (%)	TiO_2 (%)	Available Al_2O_3 (%)	Reactive SiO_2 (%)
01 – South America / Brazilian North	52.5	4.7	13.5	1.3	48.0	3.8
02 – South America / Brazilian Southeast	57.1	6.0	4.8	2.6	50.1	4.9
03 – South America / Amazon Region	48.6	5.5	14.6	2.4	45.1	2.1
04 – Africa / Awaso	49.7	2.0	19.4	1.7	45.3	1.6
05 – Africa / Guinea	47.4	2.0	23.8	2.6	43.2	1.1

5. Experimental Procedure

5.1. XRD

5.1.1. Sample Preparation

Samples were previously submitted to crushing and pulverizing before micronizing. A micronizer MCCrone 1/30 HP – 1400 RPM was used containing agate grinding bodies for 12 minutes. Ethanol was added as refrigerating liquid in order to avoid changes in mineralogical phases. After that samples were dried in air circulation oven at the temperature of 40° for 8 hours. A hydraulic Herzog press was used to press the pellets applying 200 kN in a tungsten ring as support.

5.1.2. Analysis on XRD

A scan of each pressed pellet was performed by using a Cubix3 – Panalytical XRD through a monochromator installed before XCelerator detector. Instrumental conditions are summarized in the Table 2, below.

Table 2. General instrumental conditions to perform XRD scans.

Condition	Parameter
Radiation	Cu Kα (λ = 1,54186 Å)
Energy into X-ray tube	45 kV x 40 mA
Angular window (°2θ)	2.5 to 70°
Angular step (°2θ)	0.02°
Time per step	5 s
Total time	1 h 5 mim40 s
Rotation (*spinner)*	0.5 rev/s

Interpretation of the scans obtained was based on already known mineralogical composition. Thus, the main minerals observed were: gibbsite, boehmite, hematite, goethite and quartz. The structures used as crystallographic standard reference were obtained from software High Score Plus Version 2.0a – 2004, by comparison with PDF2 from ICDD – International Centre of Diffraction Data (2003). The codes are listed below:

- Gibbsite – cód. PDF2 000-120-460;
- Boehmite – cód. PDF2 00-005-0190;
- Hematite – cód. PDF2 00-006-0502;
- Goethite – cód. PDF2 00-017-0536;
- Quartz – cód. PDF2 00-050-490.

The main peaks are presented on the schematic diffractogram showed below in Figure 1.

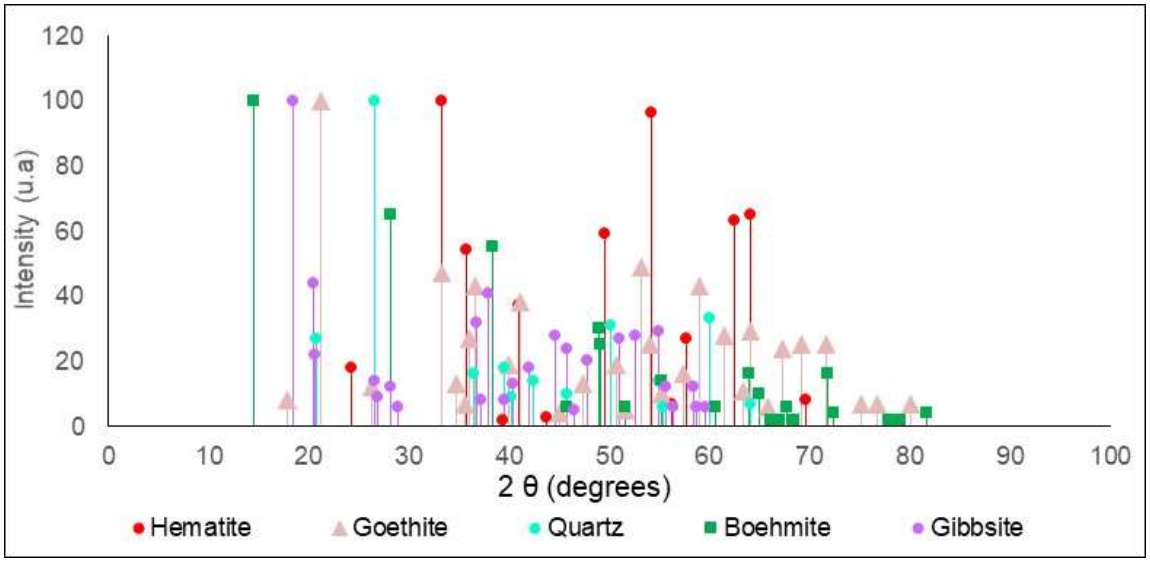

Figure 1. Crystallographic references (peaks).

Diffractograms are plotted in the graph showed in the Figure 2, below, with the reference peaks to the main crystallographic phases searched.

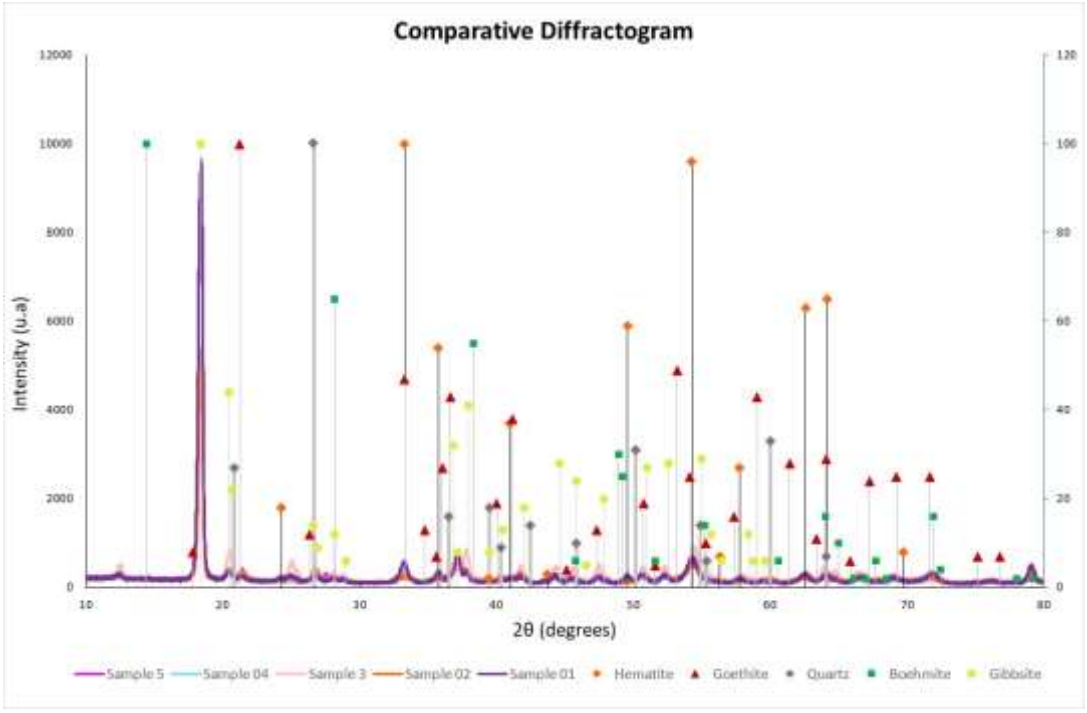

Figure 2. Diffractograms from XRD scans and the main minerals.

According to these diffractograms it is possible to conclude that sample 01 (bauxite from South America – Brazilian North) has no boehmite phase, which was also observed by chemical analysis. However, sample 04 and sample 05 (bauxite from Africa) showed peaks according to the boehmite standards.

In general, the samples from South America are fairly similar except for the goethite content in the sample 03 (bauxite from South America – Amazon Region) where the peaks showed higher intensities [5].

5.2. Laboratory Settling Test

Bauxite samples were digested in the laboratory using a 5 L digestion bomb PARR reproducing the conditions of Bayer process, in terms of caustic, temperature and time. After digestion the exit slurry product was combined with 1st washer overflow in order to achieve the theoretical solids concentration of the feedwell (thickener feed) in the refinery. The characteristics of the slurry obtained were evaluated through the analysis of soluble alumina, total caustic and alkaline plus solids concentration before and after the dilution.

After mixing with 1st washer overflow to target the desired solids concentration the mixture was kept inside a 1 L glass cylinder flask inside a hotbox at the temperature of 95 °C and allowed to equilibrate prior to flocculant being added. Applied flocculant dosages were calculated from available alumina in digestion feed and the amount of expected residue solids.

A perforated disc plunger was used to suspend/homogenize the solids within each 1 L flask before the flocculant dosages were applied. Once the flocculant had been added the slurry was again gently plunged to mix the flocculant with the slurry and induce aggregation. When the plunger was taken from the cylinder the falling/moving interface was monitored. A chronometer/timer was started when the interface passed the 900 mL graduation. The chronometer was kept running until the 700 mL mark when it was stopped. Thus, it was possible to calculate the settling rate (expressing in m/h) knowing the length of the interval

between the referenced graduations. It is important to note that to each bauxite/batch of settling tests two different flocculant regimes were used, the first being a singular dosage of one flocculant product (expressed as D1 in the chart legends) and the second being a combination (co-dosage) of two different flocculant products (expressed as D2 in the chart legends). The axis in this chart, which unit is g/tonne, refers to the total dosage (one or two flocculants).

Settling rates are plotted in the graph contained in the Figure 3, below. The lines referenced to the sample 01 (bauxite from South America – Brazilian North) can be considered a reference to this refinery. The evaluation of new bauxites, the participation of a new bauxite through a blend with a known bauxite and the flocculant dosages and/or co-dosage required are benchmarked to these reference curves/lines.

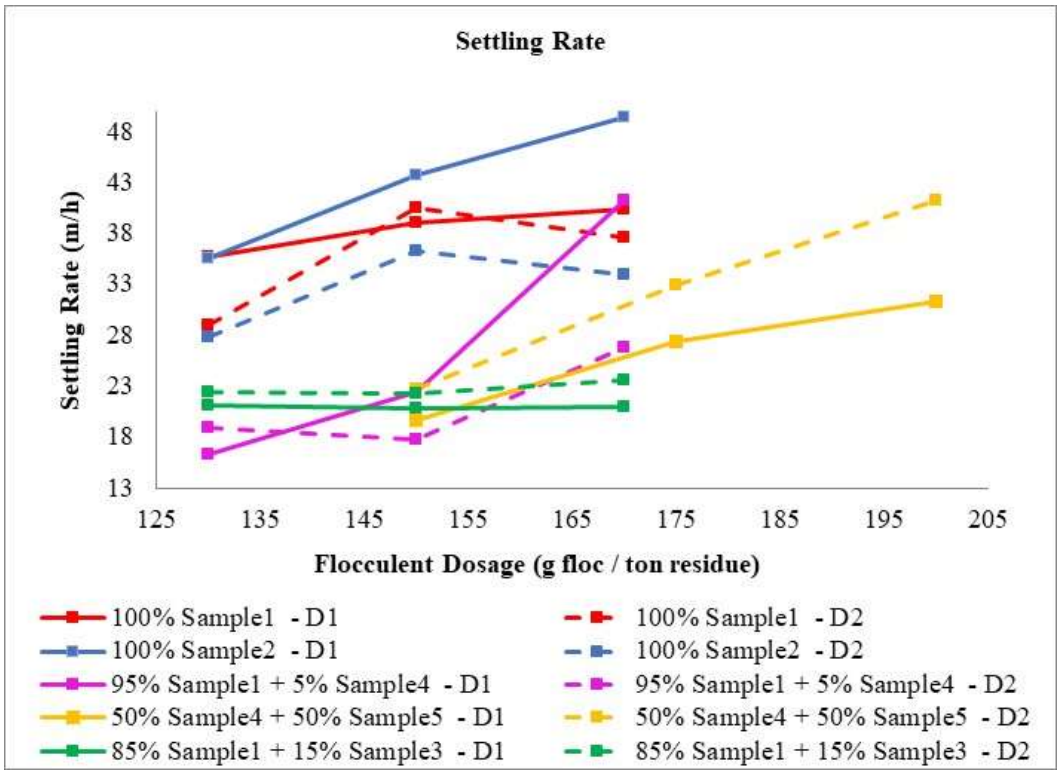

Figure 3. Settling rates curves from lab settling test.

The compaction or settled volume of slurry at the bottom of the cylinder was measured after 10 minutes of respite and it was expressed as mL. After this measurement, an aliquot of the supernatant (overflow) liquor at the top of the cylinder was taken in order to evaluate the clarity via a turbidity measurement. An internal correlation between turbidity and solids concentration were applied to convert NTU to g/kL.

From these curves, it is possible to conclude that some bauxites require higher flocculant dosages/co-dosages, for example, sample 04 and sample 05 (bauxite from Africa) even when blended at as low as 5 % in refinery bauxite feed.

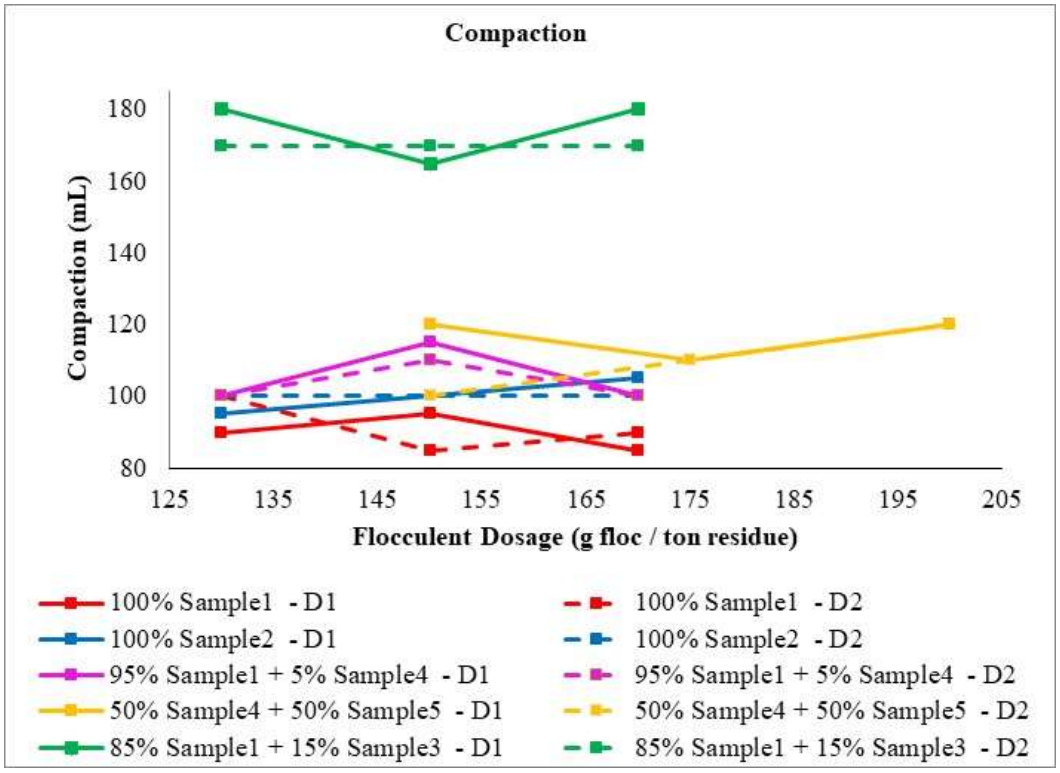

Figure 4. Compaction curves from lab settling test.

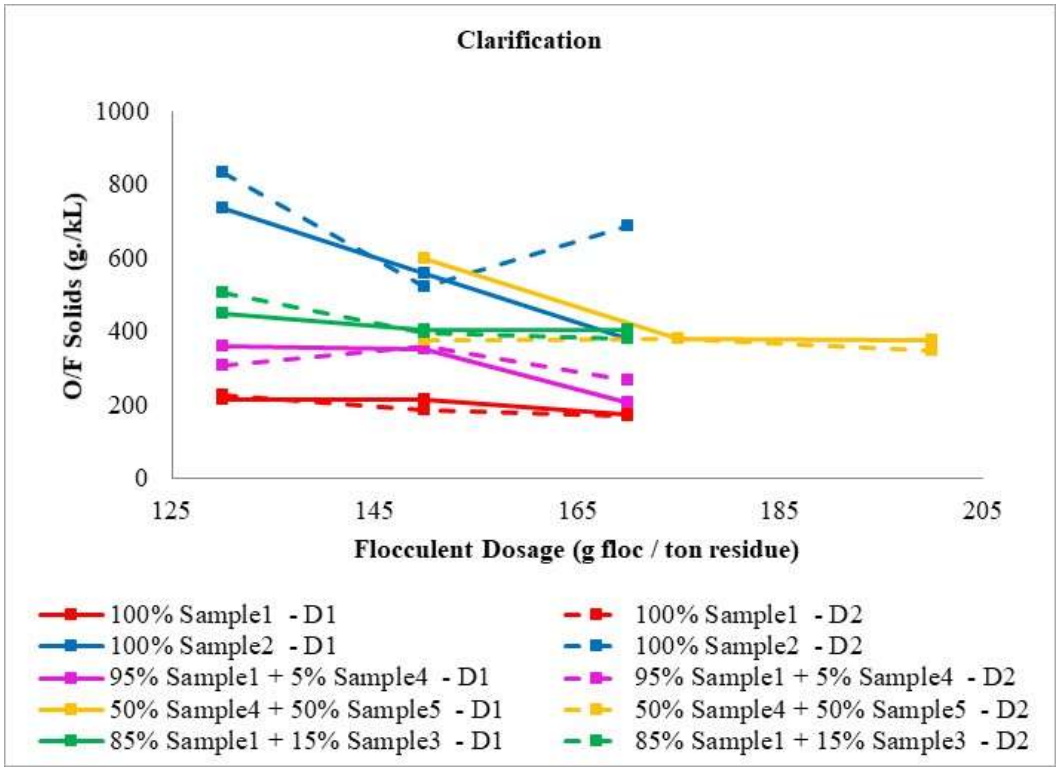

Figure 5. Clarity in overflow (O/F solids g/kL) curves from lab settling test.

Variations in mineralogy and chemical composition can explain the behavior of the South America bauxites. Sample 02 (bauxite from South America – Amazon region) presented results meaningfully different when comparing to the other South America bauxite samples. Sample 02

showed high goethite intensities combined with lower total iron content. From Figure 3 above, it is possible to conclude that when using a feed blend that constitutes 15% (or more) of this bauxite combined with "normal" bauxite there is an expectation that there will be difficulties with regards to the settling behavior of the resulting residue.

Similar conclusions can be drawn when observing the graphs of compaction and clarity, shown above in the Figures 4 and 5, respectively. Another relevant point is when observing the response of the different flocculation regimes (singular or co-dosage) for the same bauxite samples. Depending on the bauxite sample, a co-dosage can result in a better settling rate nevertheless the response from clarity is better without co-dosage.

6. Bauxite Consumption Strategy Definition

The main outputs from this kind of work, i.e. interpreting the results from settling tests with help from mineralogy, are to provide enough information to help in the consumption strategy of new bauxites. In this work, four different bauxite samples were studied prior to their usage in the refinery to allow recommendations to be made as to how to operate with them in the refinery.

The recommendations were based on:

- The contribution of the new bauxite in the refinery feed i.e. blend ratio,
- The required dosages to achieve satisfactory settling and overflow solids, and,
- Whether or not a single flocculant product or a combination (co-dosage) of two flocculant products was needed to achieve the required settling and overflow solids criteria.

From the lab test data and resulting recommendation, there was subsequently no operating problems when the processed in the refinery. Thus, a standardized flow was defined for new bauxite evaluation as illustrated in Figure 6 below.

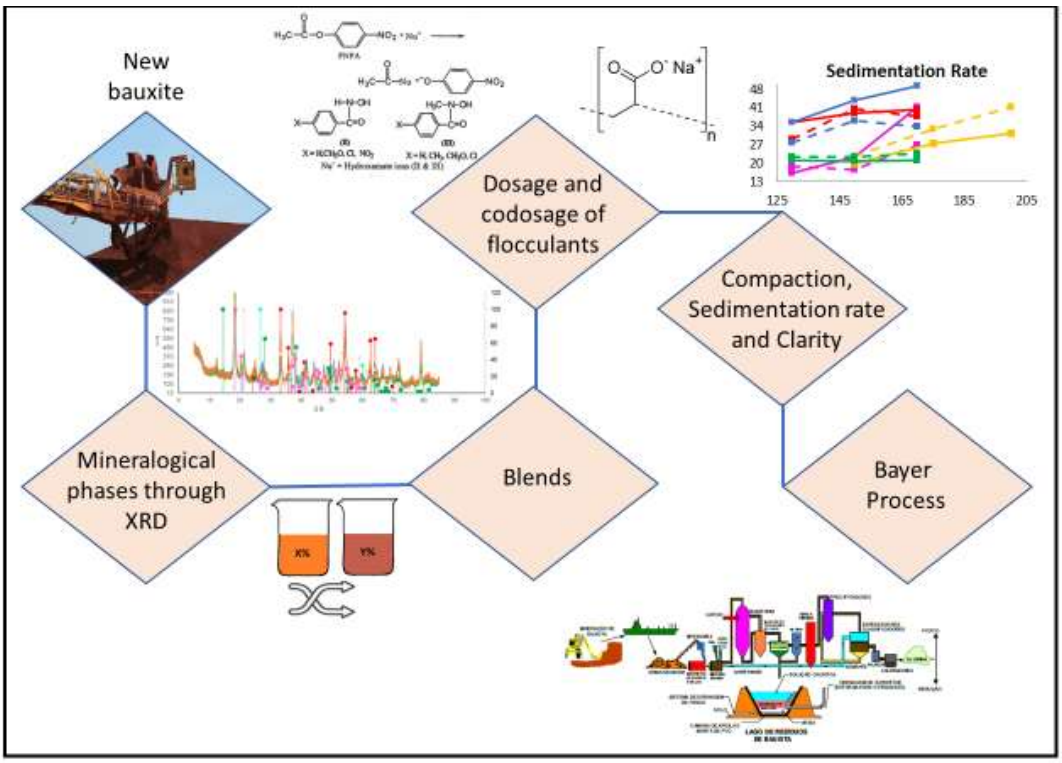

Figure 6. Schematic for new bauxite evaluation through lab settling tests + analysis.

7. References

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