A Novel Experimental Apparatus for Red Side Studies

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Abstract

This paper presents work within Hydro's Bauxite and Alumina (B&A) R&D group to develop a novel experimental apparatus for red side laboratory scale investigations, through collaboration with the filtration company, Gaudfrin. Essentially, the equipment involves an autoclave system, capable of desilication-digestion studies, connected to a pressure filtration rig designed and produced by Gaudfrin, capable of filtration and washing studies with the autoclave product. Through operating with larger volumes than conventional lab equipment, the current set up allows for essential studies to be conducted, including new bauxite evaluation and filtration characterization. The apparatus is detailed, and some commissioning and calibration results are shown to highlight its capabilities.

Keywords: laboratory apparatus, red side, autoclave, pressure filter.

1. Introduction

Most Bayer laboratories are equipped with some sort of pressurized digestion equipment to routinely measure the relevant available alumina and reactive silica in their feed bauxite. For such routine investigations which follow standard methodologies regarding temperature, caustic concentration, residence time as well as analytical methodology for the final solution, small scale bombs with block digesters analogous to Figure 1 can be used.

This kind of apparatus does however have some limitations for non-standard test work, largely associated with the volume of slurry generated. Performing detailed kinetic studies can prove difficult, where ideally both liquor and solids analysis is performed to assist in mass balancing. For a typical small-bomb digest with say, 3 g of bauxite added, the solid residue retrievable can drop to around 1 g. To generate sufficient residue to conduct mineralogical, elemental and wet-chemical analyses, the number of bombs required increases considerably. While this can be done, running multiple bombs for the same condition in this sort of equipment can present other difficulties. For example, for a 5-minute digest, while it can be simple to extract one bomb from such an apparatus, safely removing (for example) 12 bombs, without too broad a residence time distribution, can prove quite impractical. Beyond kinetic investigations or test work requiring significant amounts of solid residue, perhaps the main drawback for Bayer Process and Bayer R&D labs is the inability to generate enough digested slurry for settling tests.

The typical settling test procedure utilizes 1000 mL of slurry, to which diluted flocculant is added and shear applied so that the settling rate and settled slurry volume can be measured (see for example [1]). Typically, at one time, five settling tests can be performed to compare the effects of variables such as flocculant dosage, solids concentration and flocculant type. While this sort of test work has its flaws and the literature points out associated misinterpretations [2], the test procedure remains a staple for both flocculant developers, researchers (see for example [3]), as well as refiners that can process different types of bauxite. As is perhaps obvious,

generating 5000 mL of slurry simultaneously with the small-scale bomb digester is a practical impossibility. These considerations and the difficulties associated with the small-bomb apparatus, prompted our group to develop a larger scale digestion system.

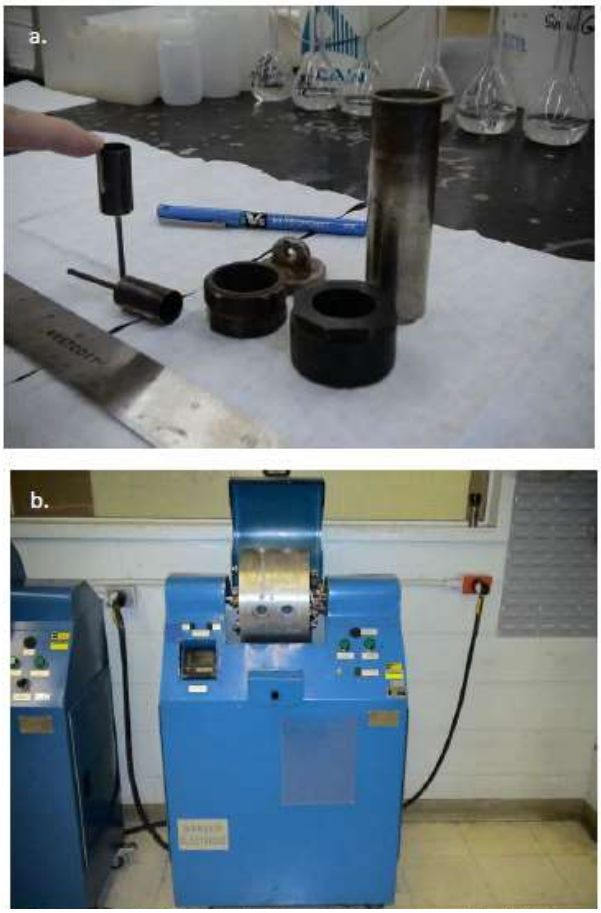

Figure 1. a) 45 mL Parr® bomb and b) block digester for routine bauxite analysis.

When producing larger volumes of slurry, and then subsequently separating and recovering both the solid and liquid fraction, the laboratory solid-liquid separation step can prove a technical challenge. Bayer digestion residues are notorious for slow filtration using conventional vacuum equipment and conditions. This, combined with an unstable slurry associated with digestions that exceed gibbsite solubility at atmospheric temperatures, leads to an experimental difficulty. It is possible that the aluminate in liquor can precipitate as gibbsite or bayerite, preventing a functional mass balance to be achieved. While it is possible to stabilize the digested slurry using low dosages of organic reagents such as mannitol (see for example [4]), unpublished work within Hydro's laboratory has suggested that for very unstable liquor, this is still not sufficient to prevent gibbsite reversion during the filtration step. Two improvements present themselves in this situation:

1. Separate the solids using flocculants, analogous to the settling test procedure described, and filter the smaller volume of settled material.
2. Filter the slurry under higher pressure to accelerate the process.

Step 1 is routinely practiced in our laboratories, but (i) a significant amount of material is rejected in the "clear" supernatant and (ii) accurate analyses of the solid material for particle size distribution is no longer possible. These weaknesses, combined with the relatively recent developments in the industry toward pressure filtering bauxite residues (see for example [5]), prompted the pursuit of pressure filtration as part of an integrated lab apparatus. The objective was to assist with the laboratory solid-liquid separation, while allowing for measurement of

4. Conclusions and Next Steps

The safety and viability of both the autoclave and pressure filter, operating in isolation and unison has been demonstrated. Test work is now underway in various facets of both R&D projects and refinery technical support, utilizing either or both apparatus described here. After some usage, areas for improvement have been identified and will be introduced in the future. The current oil bath system for heating the pressure filter is an open system with a rather primitive manual temperature controller. To prevent the unpleasant odors and improve the safety even further, a closed system with a PI temperature controller has been designed and is under construction. A data logger for the temperature profile of the autoclave is also under consideration which would make the data recording for the experimental analysts much less onerous. Fundamental to the sustainability of the system as a whole is the preventative maintenance program to calibrate necessary equipment and replace/check different components. This program is still underway and to be finalized in the near future.

5. Acknowledgments

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