UMSAEP UM-UWC Linkage Report

April – June, 2018

Visit at MISSOURI S&T, Rolla

Toward a collaboration to investigate the flotation of Rare Earths metals (REMs) from phosphogypsum and fly ash

Submitted by:

Jean-Luc Mukaba, Leslie Petrik

Department of Chemistry, (UWC)

Hosted by Dr. Lana Alagha

Department of Mining and Nuclear Engineering, S&T

1. Introduction

The present report gives an overview about different activities achieved during the visit of Jean-Luc Mukaba to the University of Missouri Science and Technology (M S&T) campus of Rolla. This visit is as the results of academic collaboration between the University of Missouri and the University of the Western Cape on the approved project “Toward a collaboration to investigate the flotation of Rare Earths metals (REMs) from phosphogypsum and fly ash”. It is within this collaboration that he had the opportunity to spend about two months in the Department of Mining and Nuclear Engineering. In this Department he was exposed to some of the techniques such as froth flotation which is related to his current PhD study consisting of recovering rare earths metals from phosphogypsum. Froth flotation is one of the most widely used techniques in mineral processing to concentrate valuable metals prior to hydrometallurgical treatment. It consists of selectively separate valuable metals from low value material associated with the desired metal. This technique also strongly relies upon the surface properties of the gangue minerals. Thus, a thorough characterization of the phosphogypsum in this regard is required in order to efficiently perform the froth flotation process. In order to perform the flotation test, some characterisations such as zeta potential analysis is crucial. This characterisation allows determining both, the charge status of the surface based on the pH values and also the type of the collectors (anionic or cationic). The following section gives the main objective of this visit and also other activities undertaken at Missouri S &T.

2. Objective

As stated in the initial UMSAEP proposal, this visit aimed to understand the surface properties of phosphogypsum waste and also to investigate the floatability of the rare earths metals contained in this feed at both micro scale and bench scale. This task is part of the current PhD study which consists of recovering the rare earths metals from phosphogypsum with particular attention to Cerium, Lanthanum and Neodymium. This visit also aimed to enforce the current research collaboration between Prof. Leslie Petrik (UWC) and Dr. Lana Alaga (S&T) who are respectively his project supervisor and advisor on the topic “Toward a collaboration to investigate the flotation of Rare Earths metals (REMs) from phosphogypsum and fly ash”. Other activities during the month of April include participation in the seminars (presentations) held on a weekly basis in the Department of Mining and Nuclear Engineering and in which Jean-Luc also had the opportunity to present his research work to the faculty members and post-graduates students on the 16th of April, 2018.

2.1 Tasks achieved

During the first month of the visit (2nd- 27th of April, 2018), the main focus was on understanding the theoretical aspect of froth flotation of phosphogypsum with regard to rare earth recovery. This allowed determination of the experimental conditions shown in Table 1 and the experimental setup shown in Figure 4 for a micro flotation test. These conditions were set for froth flotation (micro and bench scale) and for the zeta potential experiments in which only some of the conditions had to be applied. In addition to this task, a few preliminary analyses were also performed, namely the particle size distribution (for suspension), gravimetric distribution of dry phosphogypsum (sieving) and the XRD analysis of different fractions of phosphogypsum. This last analysis was carried out on different fractions (pan < 45 µm < 63 µm < 90 µm < 106 µm) to investigate the variability of phases as a function of the particle size with regard to rare earths element content. The XRD results, the gravimetric distribution of the particles and particle size distribution in suspension are discussed in section 2.2 below.

Table 1: Experimental setup

Feed	Phosphogypsum
Mass of the feed (% w)	0.5- 200 g
Collectors*	1) Sodium Oleate (0.5 -1.5 g/L) 2) Benzohydroxamic acid (0.5 -1.5 g/L)
pH modifier	NaOH and HCl (0.1-1 M)
depressant	Na_2CO_3, $H_2C_2O_4$
Particle size required	-106+38 µm
Frother	Methyl Isobutyl Carbinol (MIBC), Xylenol, 1-pentanol
Stirring speed (Impeller)	500-2000 rpm
Conditioning time	15-30 min
Air flow rate	30-45 mL/ min
Flotation cell capacity	100-1000 mL

(*): The molar concentrations differ based on the molecular weight of each collector

2.2 Characterization attained at S&T

Figure 1 shows the XRD results of five fractions of particles (pan < 45 µm < 63 µm < 90 µm < 106 µm). The existence of a singular mineral phase can be seen from the diffractograms, proven through the matching of the major diffraction peaks located at 14.7, 25.6, 29.7 and 31.8 2θ. These peaks are characteristic of the phosphogypsum mineral phase. Since no other detectable mineral phase was identified by qualitative XRD, an additional characterization technique such as the quantitative XRD analysis is required. This is because the phosphogypsum feed investigated in this study has initially been found to exist in three different forms with regard to the amount of molecular water (gypsum: $CaSO_4$ $2H_2O$; bassanite: $CaSO_4$ $0.5H_2O$ and anhydrite: $CaSO_4$). It is therefore important to perform a quantitative XRD on these fractions to further understand the distribution of each phase ($CaSO_4$ $2H_2O$; $CaSO_4$ $0.5H_2O$ and $CaSO_4$) within the variation of the particle size.

Besides the XRD analysis, the size distribution analysis is crucial in flotation in order to get an indication upon the weight (%) of the solid and also the particle size to use in the slurry. Therefore, the particle size distribution using dried phosphogypsum was also carried out prior to flotation. The results shown

in Figure 2 revealed that 50 weight % of the particles were smaller than 96 μm (P_{50}= 96). The particle size distribution in a suspension of phosphogypsum and ethanol was also determined prior to zeta potential analysis. These results are displayed in Figure 3, and showed that the distribution of particles after grinding the phosphogypsum at 5 min and 15 min were respectively D_{80}=31.3 and D_{80}=22.6. The D_{80}=31.3 distribution obtained at 5 min was selected to be used for the zeta analysis due to short time of grinding.

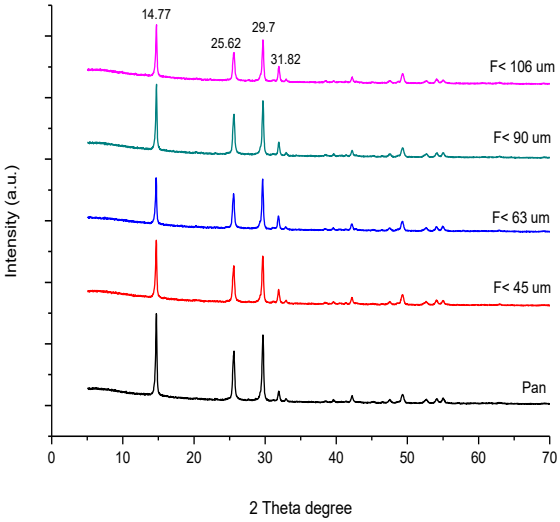

Figure 1: Qualitative XRD analysis of fractions

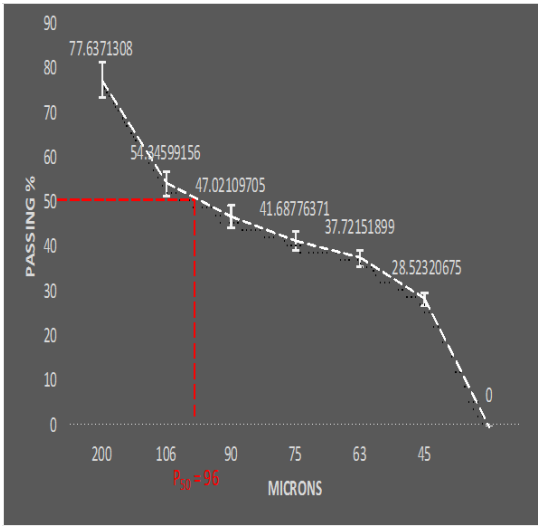

Figure 2: Particle size distribution of dry phosphogypsum (P_{50}=96 μm)

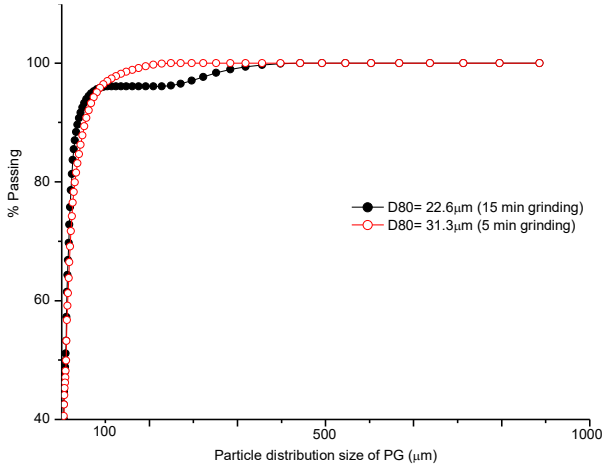

Figure 3: Particle Size distribution in a suspension (5 min and 15 min grinding)

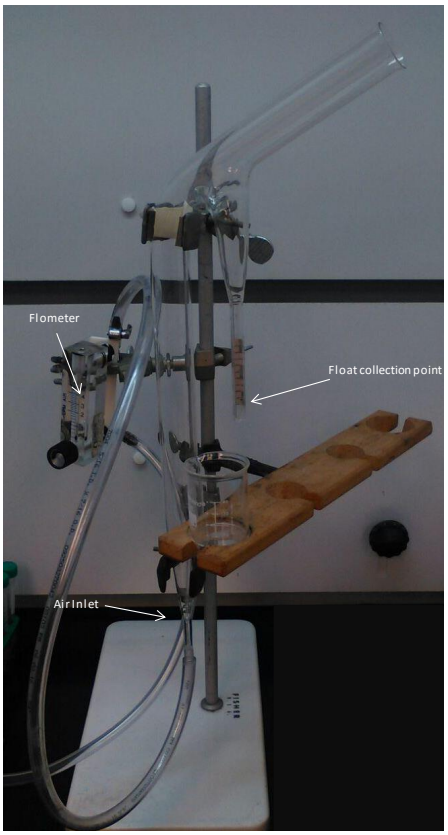

Figure 4: Set up of Hallimond tube for micro flotation

2.3 Challenges

As stated in section 1.2, one of the objectives of this visit was to perform a few flotation tests at micro and bench scales using respectively Hallimond tube and Denver flotation cells to recover the rare earths elements from phosphogypsum. Unfortunately this task was not achieved due to a few instrumental constraints related to the technical aspects and also the running cost in terms of the characterization of the flotation products (floats and tails). The main characterizations required at this stage were the ICP-OES and XRF for mineral and chemical composition; and the QEM-SCAN was also required for mineral liberation analysis of the phosphogypsum. ICP-OES or XRF analyses were repeatedly required at each flotation test for the characterization of the products while QEM-SCAN was only needed for a once-off characterization purpose. This last analysis is important in mineral processing prior to flotation but the cost of analyses, which was not budgeted for, and also the limited time frame of the visit could not allow these analyses to be carried out.

3. Benefit and Recommendation

Despite the fact that some of the main goals of this visit were not achieved; the overall exposure during the visit has brought to us an additional knowledge in particular within the flotation technique and the setup can be reproduced in our laboratory at UWC. This is in an attempt to complete some unattended tasks. In addition to the research aspect, this visit has also allowed us to build a network with some post-graduate students at S&T in general and in particular in the research group that is ran by Dr. Lana Alaga and also with some faculty members at S&T for prospective post-doctoral position and collaboration in terms of analysis and other assistance. This UMSAEP linkage program has been helpful and utmost valuable for Jean-Luc's development as a UWC student at academic and cultural level. However, we do suggest that the program be restructured to strengthen research collaboration by including a running cost component in the budgeting to allow UWC students in particular those in science, to also perform experiments and analyses while in Missouri. This was actually one of the major challenges Jean-Luc faced while visiting Missouri S&T due to the lack of the running cost of analysis. In our experimental plan, the ICP-OES or XRF was required for regular elemental composition analysis and the cost is between $25-30 per sample. The other important analysis prior to flotation was the QEM-SCAN which could not also be carried out due to the cost ($400 per sample).

Acknowledgements

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Souvenirs…

Dr. Lana's research group, S&T

Mrs Cindy Boles (International office, S&T)

UMSAEP's team (Missouri& UWC)

Department seminar presentation, S&T