



# MINERAL PROCESSING ABSTRACTS

*A quarterly abstract service*

Issue 1, May 2014

Volume 1

A contribution from students of  
DEPARTMENT OF FUEL AND MINERAL ENGINEERING  
INDIAN SCHOOL OF MINES, DHANBAD

## JOURNALS

[International Journal of Mineral Processing](#) (Volume 126)

[Minerals Engineering](#) (Volume 55)

[Fuel](#) (Volume 127)



## CONTRIBUTIONS

**Prof [Dr] Nikkam Suresh**

Vice-President IIME-Student Chapters

Arvind Garg (II Year)

Shreevardhan Daga (II Year)

Priyank Jain (III Year)

Piyush Bapana (III Year)

Shubham Dwivedi (III Year)

Shubham Ashok Gandhi (III Year)

### **Disclaimer**

*All the studies presented below are a work of the authors (cited in the abstract) and a copyright of ELSEVIER. The work was collected and compiled by the persons mentioned above with a sole aim of academic use and non-commercial distribution.*



## PREAMBLE

Dear all,

It's my pleasure to bring it to the knowledge of all the members of IIME that the student members of IIME-Student Chapter, ISM-Dhanbad, have made their sincere efforts and profoundly contributed in bringing out this '**Abstract Service**'. This pleasant effort from Students, helps Mineral Engineering fraternity, working in different corners of our country, particularly those who are in remote areas.

**The other Students' Chapters, are also getting their Programmes into "some Frame Work" and would join this main stream.**

We thank the President IIME, **Prof B K Mishra**, for his encouragement and freedom given to Students' Chapters so that the next Generation Mineral Engineers can take active role in IIME and look back with pride.

We look forward to your suggestions and new ideas to make Students' Chapters more vibrant.

Best wishes to all Members.

**[Prof Nikkam Suresh]**  
Vice-President, Student Chapters-IIME

## Dear All

'**Requirements**' and '**Adjustments**' are the two key factors in leading the life peacefully. Somehow, we are always habituated to lead the life for sake of peace by making adjustments with minimum requirements. In this context, I appreciate the innovative Ideas of Mineral Engineering students from ISM, that within their limitations they have found a way to serve the Mineral Engineering fraternity with whatever best they could do to make the basic requirements of Mineral Engineers. I strongly believe in saying '**Imagination is more important than knowledge**'. So, this volume is a typical example to it. More importantly, they have released the requirement and importance of the **knowledge and its dissipation** and augers well for the future of MLE.

I wish the entire student body for their best future and endeavor.

**[Prof (Dr) B K Mishra]**  
Director, IIMT, Bhubaneswar and  
President – IIME

# INTERNATIONAL JOURNAL OF MINERAL PROCESSING

Volume 126

January 2014

Copyright © 2014 Elsevier Ltd. All rights reserved



Provided by:  
DEPARTMENT OF FUEL AND MINERAL ENGINEERING  
INDIAN SCHOOL OF MINES, DHANBAD

---

## TABLE OF CONTENTS

1. RECOVERY OF HIGH GRADE IRON COMPOUNDS FROM LD SLAG BY ENHANCED MAGNETIC SEPARATION TECHNIQUES .....	3
2. MODELING VALIDATION OF CALICHE ORE LEACHING USING SEAWATER .....	4
3. AN IMPROVED ESTIMATION OF SHEAR RATE USING ROTATING COAXIAL-CYLINDER FANN VISCOMETER: A RHEOLOGICAL STUDY OF BENTONITE AND FLY ASH SUSPENSIONS .....	5
4. INFLUENCE OF BACTERIA–COAL ELECTROSTATIC INTERACTION ON COAL CLEANING .....	6
5. A TECHNOLOGY OF PREPARING HONEYCOMB-LIKE STRUCTURE MgO FROM LOW GRADE MAGNESITE .....	7
6. PARTICLE SIZE DISTRIBUTION AND STRUCTURAL CHANGES IN LIMESTONE GROUND IN PLANETARY BALL MILL .....	8
7. RAPID ORE BREAKAGE PARAMETER ESTIMATION FROM A LABORATORY CRUSHING TEST.....	9
8. RECOVERY OF MANGANESE AND NICKEL FROM POLYMETALLIC MANGANESE NODULE USING COMMERCIAL EXTRACTANTS.....	10
9. RECOVERY OF RARE EARTH METALS FROM VOICE COIL MOTORS USING BIS(TRIFLUOROMETHYLSULFONYL)AMIDE MELTS BY WET SEPARATION AND ELECTRODEPOSITION .....	11
10. KYANITE ORE PROCESSING BY CARBOCHLORINATION .....	12
11. EFFECT OF FLUXING AGENTS ON THE SWELLING BEHAVIOR OF HEMATITE PELLETS .....	13
12. ASSESSMENT OF A MICROWAVE-ASSISTED RECYCLING PROCESS FOR THE RECOVERY OF HIGH-QUALITY AGGREGATES FROM CONCRETE WASTE .....	14
13. TECHNOLOGY OPPORTUNITIES TO DEVELOP LARGE-VOLUME PLATINUM ORE DEPOSITS IN DUNITE OF ZONED MAFIC–ULTRAMAFIC COMPLEXES .....	15
14. PARETO BASED OPTIMIZATION OF FLOTATION CELLS CONFIGURATION USING AN ORIENTED GENETIC ALGORITHM .....	16
15. INFLUENCE OF HUMIC ACIDS ON OIL SAND PROCESSING. PART I: DETECTION AND QUANTIFICATION OF HUMIC ACIDS IN OIL SAND ORES AND THEIR EFFECT ON BITUMEN WETTABILITY .....	17
16. INFLUENCE OF HUMIC ACIDS ON OIL SAND PROCESSING. PART II: RELATIONSHIP BETWEEN BITUMEN EXTRACTION, HUMIC ACIDS CONCENTRATION AND POWER DRAW MEASUREMENTS ON OIL SAND SLURRIES.....	18
17. INVESTIGATION OF DEPHOSPHORIZATION OF BROWN IRON ORE CONCENTRATES BY SINTERING AND MAGNETIC BENEFICIATION .....	19
18. A STUDY ON THE CHARACTERISTICS OF SPRAY ANGLE FORMATION IN A 2 INCH HYDROCYCLONE USING WATER ONLY .....	20

# 1. RECOVERY OF HIGH GRADE IRON COMPOUNDS FROM LD SLAG BY ENHANCED MAGNETIC SEPARATION TECHNIQUES

N. Menada<sup>a</sup>, N. Kanarib<sup>b</sup>, M. Savea<sup>b</sup>

*a BRGM, 3 av. C. Guillemin, BP 36009, 45060 Orléans Cedex 2, France*

*b Université de Lorraine, GeoRessources UMR CNRS 7359, 2 rue du Doyen Marcel Roubault, TSA 70605-54518 Vandoeuvre-les-Nancy Cedex, France*

## ABSTRACT

A representative sample of LD slag provided from French steel industry was treated by different physical separation techniques especially used in mineral processing. The results of characterisation study show that the LD slag may contain up to 35% of iron compounds. The X-ray diffraction reveals the presence of the following crystallized mineral phases: CaO, MgO, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, MnO, and P<sub>2</sub>O<sub>5</sub>. Larnite (Ca<sub>2</sub>SiO<sub>4</sub>), di-calcium aluminoferrite (brownmillerite: Ca<sub>2</sub>Fe<sub>2</sub> – xAl<sub>x</sub>O<sub>5</sub> with x = 0.6) and a solid solution (Fe, Mn, Mg)O structurally close to the wüstite (FeO) and some residual lime (CaO).

The results of grinding tests show that the manner of grinding is very important. Soft Grinding (SG) or sequential grinding is most suitable to reduce the particle size of LD slag avoiding the overproduction of fine particles, which hinder the effectiveness of magnetic separation techniques. The combination of Low and High Intensity Magnetic Separation (LIMS and HIMS) of ground LD slag at 63 µm in wet process may recover ferromagnetic particles (iron oxides) at LIMS and paramagnetic particles at HIMS, while the non-magnetic fraction contains mostly calcium silicates.

From this investigation, two flow-sheets of treatment of LD slag are suggested to recover high grade iron material for recycling in metallurgical processes. Calcium and silicon oxide rich product will be directed to the cement industry.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 2. MODELING VALIDATION OF CALICHE ORE LEACHING USING SEAWATER

Javier I. Ordóñez<sup>a</sup>, Luis Moreno<sup>a, b</sup>, Mario E. Mellado<sup>c</sup>, Luis A. Cisternas<sup>a, c</sup>

<sup>a</sup> *Department of Chemical and Mineral Process Engineering, Universidad de Antofagasta, Chile*

<sup>b</sup> *Department of Chemical Engineering and Technology, Royal Institute of Technology, Sweden*

<sup>c</sup> *Centro de Investigación Científico y Tecnológico para la Minería, Chile*

### ABSTRACT

Leaching column experiments of caliche were performed using seawater as the leaching agent because the caliche deposits are located in Northern Chile where water resources are scarce. The use of seawater without desalination is an attractive alternative for mining operations. The experimental recoveries of different ions were modeled using a hybrid model, which uses empirical information and fundamental principles. The following ions were considered: nitrate, iodine, sulfate, chloride, sodium, potassium and magnesium. The model explicitly considers different column heights, irrigation rates, and leaching agents. A sensitivity analysis showed that parameters associated with the particle level predominantly determined the calculated recoveries. The predictive capability was also tested, and the results were generally good, except for the sulfate ion, the dissolution of which was controlled by the presence of other ions.

Copyright © 2013 Elsevier B.V. All rights reserved.

### 3. AN IMPROVED ESTIMATION OF SHEAR RATE USING ROTATING COAXIAL-CYLINDER FANN VISCOMETER: A RHEOLOGICAL STUDY OF BENTONITE AND FLY ASH SUSPENSIONS

Rajesh Kumar, Vineet Kumar, Dilip K. Rajak, Chandan

*Department of Petroleum Engineering, Indian School of Mines, Dhanbad 826 004, India*

#### ABSTRACT

Bentonite is commonly used as a drilling fluid additive for viscosity control during drilling of oil wells. Rheological behavior of bentonite suspensions is very complex and it is very difficult to predict accurate shear rates from viscometer readings. This paper deals with the accurate estimation of shear rates of bentonite suspensions using rotating coaxial-cylinder Fann viscometer readings. Rotor rotations and bob deflections are the two important Fann viscometer readings which are used to predict shear rates with the help of generalized viscometer difference equation. Rheological behavior of bentonite suspensions are also compared with classified fly ash suspensions with varying particle size and solid loading. The proposed shear rate equation is quite general and can be applied to any fluid.

Copyright © 2013 Elsevier B.V. All rights reserved.

#### 4. INFLUENCE OF BACTERIA–COAL ELECTROSTATIC INTERACTION ON COAL CLEANING

A.A. El-Midanya<sup>a</sup>, M.A. Abdel-Khalek<sup>b</sup>

<sup>a</sup>*Mining, Petroleum, and Metallurgy Dept., Faculty of Engineering, Cairo University,*

*Egypt* <sup>b</sup>*Central Metallurgical R&D Institute (CMRDI), P.O. Box 87, Helwan, Egypt*

##### ABSTRACT

Bacteria have been studied in bioflotation and bioflocculation processes for impurity removal. Electrostatic forces are important for adsorption of bacteria onto mineral surfaces. In the present study, three strains of bacteria: *Bacillus subtilis*, *Paenibacillus polymyxa* and *Mycobacterium phlei*, are used to emphasize the importance of bacteria–coal electrostatic interaction on coal cleaning. They differ in their point of zero-charge (PZC). Flotation experiments were conducted using each bacterium individually to reduce coal impurities. Although promising results were observed for all studied bacteria, *B. subtilis* was the best. By correlating the PZC of coal particles as well as the bacteria, it was found that the superiority of *B. subtilis* is related to wider PZC difference between the coal and *B. subtilis*.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 5. A TECHNOLOGY OF PREPARING HONEYCOMB-LIKE STRUCTURE MgO FROM LOW GRADE MAGNESITE

Zhao Yuna<sup>a</sup>, Zhu Guocai

*Institute of Nuclear and New Energy Technology, Tsinghua University, Beijing 102201, China*

### ABSTRACT

Honeycomb-like structure MgO is prepared from low grade magnesite by a technology including four main steps: calcination of low grade magnesite, carbonization treatment of the calcined magnesite, thermal decomposition of Mg(HCO<sub>3</sub>)<sub>2</sub> and calcination of basic magnesium carbonate. In this study, the main experimental parameters of carbonization treatment and thermal decomposition were optimized. The carbonization conversion ratio of the calcined magnesite was 93% by controlling the ratio of solid and liquid (calcined ore/water) at 10 g/L, carbonization time at 45 min and CO<sub>2</sub> gas flow rate at 0.08 m<sup>3</sup>/h. Decomposition ratio of Mg(HCO<sub>3</sub>)<sub>2</sub> in solution can reach 88% at 50 °C for 60 min via fixing stirring speed at 600 r/min and the Mg<sup>2+</sup> content in filtrate at 8 g/L. Honeycomb-like structure MgO product with purity of 99% was eventually obtained by calcining basic magnesium carbonate at 800 °C for 2 h. The decomposition of basic magnesium carbonate was depicted by TGA. The MgO product was characterized by XRD and SEM.

Copyright © 2013 Published by Elsevier B.V.

## 6. PARTICLE SIZE DISTRIBUTION AND STRUCTURAL CHANGES IN LIMESTONE GROUND IN PLANETARY BALL MILL

Pedro L. Guzzo<sup>a</sup>, Juliano B. Santos<sup>b</sup>, Renato C. Davida<sup>b</sup>

<sup>a</sup>Department of Mining Engineering, Federal University of Pernambuco, 50740-530 Recife, PE, Brazil <sup>b</sup>National Department of Mineral Production, Ministry of Mines and Energy, 78030-150 Cuiabá, MT, Brazil

### ABSTRACT

The dry grinding of limestone in a planetary ball mill was investigated as a function of the diameter of the grinding balls and the revolution speed of the mill. The tests were carried out with the same feed size ( $75 \times 250 \mu\text{m}$ ) at variable times up to 240 min. The analyses of the mean particle size, the specific surface area and the dispersion in size distribution showed that the grinding rate slowdown when  $\sim 50\%$  of the particles became smaller than  $20 \mu\text{m}$ . Among the grinding conditions investigated here, that one set with 10 mm balls, 200 rpm and 60 min showed to be suitable to achieve mean particle size smaller than  $10 \mu\text{m}$ . The scanning electron microscopy was useful to explain the decrease in the grinding rate by the effect of agglomeration of fines lying on partially broken particles. The agglomeration was attributed to the increase in particle surface energy that was promptly associated with the creation surface defects and bulk distortions detected by electron paramagnetic resonance (EPR) spectroscopy. The X-ray diffracting peaks of dolomite and other accessory minerals (talc, quartz and biotite) present in the limestone decreased with prolonged grinding. Differential thermal analysis revealed that the activation energy related to  $\text{MgCO}_3$  decomposition in dolomite decreased with the grinding time increasing and a simple relation was found between the energy deposited by the mechanical action and the specific surface area of ground particles.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 7. RAPID ORE BREAKAGE PARAMETER ESTIMATION FROM A LABORATORY CRUSHING TEST

Fernando N. de Magalhães, Luís Marcelo Tavares Corresponding

*Department of Metallurgical and Materials Engineering, Universidade Federal do Rio de Janeiro, COPPE/UFRJ,  
Cx. Postal 68505, CEP 21941-972, Rio de Janeiro, RJ, Brazil*

### ABSTRACT

Drop weight testers have been widely and successfully used to characterize ore breakage parameters for simulation of crushers and mills. In some cases, limitations of time, sample availability, and even cost, can prevent the conduction of these tests when preparing samples for chemical analysis and/or grindability and flotation/concentration testing. The present work demonstrates how a laboratory cone crusher equipped with a power meter, in conjunction with Whiten–Awachie crusher model, has been used to quickly estimate the  $A^* b$  breakage parameters. With an average absolute error of 36% when applied to a variety of materials, it is demonstrated that the method is not a priori restricted to the particular crusher used, neither to the crushing conditions employed. It is discussed that the method, when part of a variability study, can be used as a convenient tool for geometallurgical mapping of ore deposits regarding ore breakage response.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 8. RECOVERY OF MANGANESE AND NICKEL FROM POLYMETALLIC MANGANESE NODULE USING COMMERCIAL EXTRACTANTS

E. Padhan<sup>a</sup>, K. Sarangi<sup>a</sup>, T. Subbaiah<sup>b</sup>

<sup>a</sup>Academy of Scientific and Innovative Research, India

<sup>b</sup>CSIR-Institute of Minerals and Materials Technology, Bhubaneswar, Odisha, India

### ABSTRACT

The paper describes a process for the recovery of manganese and nickel from the sulfuric acid leach liquor of Indian Ocean manganese nodules using starch as the reductant. The leach liquor contained 22.85 g/L manganese, 6.38 g/L iron, 1.01 g/L copper, 0.023 g/L zinc, 0.09 g/L cobalt and 1.44 g/L nickel. From this leach liquor, Fe was first precipitated out with Ca(OH)<sub>2</sub> at pH 3.8 followed by the extraction of copper using LIX 84I. From the almost Fe- and Cu-free leach liquor, Zn was extracted out as an impurity with D2EHPA in kerosene, after which the extraction of Mn was carried out with NaD2EHPA. The extraction efficiency of manganese using NaD2EHPA was 99.93% in 2-stages at A:O ratio of 3:4. The co-extraction of cobalt with manganese was 43 mg/L which was subsequently removed by scrubbing in two stages with MnSO<sub>4</sub> solution. The stripping of manganese was achieved with 4% H<sub>2</sub>SO<sub>4</sub> in 2-stages at A:O ratio of 1:1. After extraction of Mn, Ni was extracted with NaD2EHPA. The extraction isotherm of nickel reads 2-stages at A:O ratio of 3:2. The co-extraction of cobalt with nickel was 47 mg/L for which the Ni-LO was scrubbed out with NiSO<sub>4</sub> solution. The stripping efficiency of nickel was 99.93% with 0.5% H<sub>2</sub>SO<sub>4</sub> in 2-stages at A:O ratio of 1:1.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 9. RECOVERY OF RARE EARTH METALS FROM VOICE COIL MOTORS USING BIS(TRIFLUOROMETHYLSULFONYL)AMIDE MELTS BY WET SEPARATION AND ELECTRODEPOSITION

Masahiko Matsumiyaa<sup>a</sup>, Kota Ishiokaa<sup>b</sup>, Takahiro Yamadaa<sup>b</sup>, Mai Ishiia<sup>b</sup>, Satoshi Kawakamib<sup>b</sup>  
*a Graduate School of Environment and Information Sciences, Yokohama National University, 79-2 Tokiwadai, Hodogaya-ku, Yokohama 240-8501, Japan*  
*b Environmental Protection Laboratory, DOWA ECO-SYSTEM Co., Ltd., 65-1 Omoriyama-shita, Hanaoka, Odate, Akita 017-0005, Japan*

### ABSTRACT

In this study, a new recycling process for rare earth metals was developed for voice coil motors (VCMs) from hard disk drives. For the wet separation process, the leaching behavior of finely ground oxidized VCM powder in 1,1,1-trifluoro-N-[(trifluoromethyl)sulfonyl]methane sulfonamide; HN(SO<sub>2</sub>CF<sub>3</sub>)<sub>2</sub>, (HTFSA) aqueous solution was studied. The kinetics of the leaching process for the rare earth metals was explained using the shrinking core model. Then, the selective titration of Fe(OH)<sub>3</sub> precipitates was determined from the pH-log[Mn<sup>+</sup>] diagram. The ionic conductivity of the obtained metallic-TFSA melts obeyed the Arrhenius law and the Vogel–Tamman–Fulcher (VTF) parameters were estimated from empirical data. For the electrodeposition process, the reduction behavior of Nd in the metallic-TFSA melts was investigated using cyclic voltammetry, and it was also studied under the potentiostatic condition of – 3.2 V at 493 K. The deeper layer of the electrodeposits consisted mainly of Nd metal, which was confirmed by XPS. The oxygen content decreased to less than 0.1% at a depth of 1.0 μm. Thus, the series of recycling processes developed here enabled Fe in VCMs to be selectively removed by wet separation and metallic Nd to be efficiently recovered by electrodeposition.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 10. KYANITE ORE PROCESSING BY CARBOCHLORINATION

Andrey D., Oleg G. Parfenov, Leonid A. Solovyov, Sergey N. Vereshchagin

*Institute of Chemistry and Chemical Technology of SB RAS, Akademgorodok, 50/24, Krasnoyarsk, Russia*

### ABSTRACT

Kyanite, mullite and model kyanite ore carbochlorination processing was investigated in laboratory experiments and thermodynamics' calculations. The extraction of 94% alumina and silica takes place as a result of calcined kyanite carbochlorination at 1300 °C for 90 min at normal pressure. Kyanite processing into  $AlCl_3 + SiCl_4$  gas mixture is proposed for high purity alumina, nanodispersed silica and solar-grade silicon production.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 11. EFFECT OF FLUXING AGENTS ON THE SWELLING BEHAVIOR OF HEMATITE PELLETS

Srinivas Dwarapudi<sup>a</sup>, P.K. Banerjee<sup>a</sup>, Pradeep Chaudhary<sup>a</sup>, Surajit Sinha<sup>a</sup>, Ujjal Chakraborty<sup>a</sup>,  
Chandra Sekhar<sup>a</sup>, T. Venugopalan<sup>a</sup>, R. Venugopal<sup>b</sup>

<sup>a</sup> Tata Steel Ltd, Jamshedpur, India

<sup>b</sup> FME, ISM University, Dhanbad, India

### ABSTRACT

Pellet properties are largely governed by the form and degree of bonding achieved between ore particles during induration and the stability of these bonding phases during reduction. Fluxing agents play an important role in forming these bonds by forming different phases depending on the type of flux. In the present study, effect of different fluxing agents, viz., limestone, magnesite and pyroxenite, on melt formation & microstructure during the induration and on swelling behavior during reduction, was examined. Optical microstructural studies with image analysis were carried out to estimate the amount of different phases. SEM-EDS analysis was done to measure the chemical analysis of oxide and slag phases. X-ray mapping was also carried out to understand the distribution of CaO, MgO, SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> in different phases. From the results, it was observed that limestone addition decreased the swelling at lower basicity but exhibited highest swelling at 0.6 basicity and decreased thereafter. Formation of low melting point calcium olivines between Fe<sub>2</sub>SiO<sub>4</sub> and Ca<sub>2</sub>SiO<sub>4</sub> can be attributed to the highest swelling at 0.6 basicity. With increasing addition of magnesite and pyroxenite, pellet swelling found to be decreased considerably. Formation of magnesioferrite phase and high melting point slag formed during induration could be attributed to the improved swelling of magnesite and pyroxenite fluxed pellets. Limestone fluxed pellets at 0.8 basicity, pyroxenite fluxed pellets at 1.5% MgO and magnesite fluxed pellets at 1.0% MgO exhibited low swelling behavior among all the pellets studied.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 12. ASSESSMENT OF A MICROWAVE-ASSISTED RECYCLING PROCESS FOR THE RECOVERY OF HIGH-QUALITY AGGREGATES FROM CONCRETE WASTE

Kathy Bru<sup>a</sup>, Corresponding, Solène Touzé<sup>a</sup>, Florent Bourgeois<sup>b</sup>, Nicholas Lippiatt<sup>b</sup>, Yannick Ménard<sup>a</sup>

<sup>a</sup>BRGM, Water, Environment and Ecotechnologies Division — Waste and Raw Materials & Recycling Unit, 3 Avenue Claude Guillemin, BP 36009, Orléans 45060 Cedex 2, France

<sup>b</sup>Laboratoire de Génie Chimique, UMR CNRS 5503, Université de Toulouse, 4 Allée Emile Monso, BP 44362, Toulouse 31432 Cedex 4, France

### ABSTRACT

This study presents an innovative method for concrete waste up-cycling based on concrete weakening through microwave heating before impact crushing. Two series of tests were conducted in order to assess the influence of the aggregate properties (size distribution, mineralogical nature) and the influence of the operating conditions of the microwave heating pretreatment on concrete fragmentation; and thus to evaluate the feasibility and the robustness of this process. Experiments were carried out on lab-made, cylindrical concrete specimens and on no-slump concrete waste with a multimode cavity microwave equipment (2.45 GHz, 6 kW) and an impact crusher. Results showed that microwave heating always induced an embrittlement of concrete samples which resulted in lower fracture energy, higher fragmentation of samples and higher liberation of aggregates (i.e. free of cement paste). A microwave-assisted comminution process is therefore an effective recycling technique for the recovery of high-quality aggregates from concrete waste.

Copyright © 2013 Elsevier B.V. All rights reserved.

### 13. TECHNOLOGY OPPORTUNITIES TO DEVELOP LARGE- VOLUME PLATINUM ORE DEPOSITS IN DUNITE OF ZONED MAFIC–ULTRAMAFIC COMPLEXES

A.P. Kozlov, V.A. Chanturiya

*Institute of Comprehensive Exploitation of Mineral Resources Russian Academy of Sciences, ICEMR RAS, Russian Federation*

#### ABSTRACT

The results of mineralogical and technological studies of PGM mineralization in zoned mafic – ultramafic complexes of the Ural–Alaskan type are given. Technological properties, grain size, and good opening of PGM ore in grain size class + 80  $\mu\text{m}$  make it possible to concentrate ore using gravity methods with stadial comminution and between cycle separation of relatively large grains and nuggets into high-grade concentrates at the early stage of disintegration. The elaborated technological flow chart approved under semi-industrial conditions allows recovery of as much as 94% Pt.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 14. PARETO BASED OPTIMIZATION OF FLOTATION CELLS CONFIGURATION USING AN ORIENTED GENETIC ALGORITHM

D. Pirouzan, M. Yahyaei, S. Shahid Bahonar

*University of Kerman, Mining Engineering Group, Engineering Faculty, Islamic Republic Blvd., Kerman, P.O. Box 761175-133, Iran*

### ABSTRACT

It is customary to use more than one stage of flotation to obtain an acceptable level of separation of valuables. Flotation circuit design is usually accomplished by applying empirical rules or by using expertise of practitioners which may result in operations not working at their optimum conditions. Given the capabilities of genetic algorithms (GA) in finding an optimum solution in very disturbed search spaces, they can be used to obtain the desired flotation circuit configuration. In flotation circuit configuration optimization problem, a combination of metallurgical parameters such as yield and concentrate ash content could be used as the fitness function for the genetic algorithm. The multi-objective nature of the problem justified the use of the Pareto method to arrive at a set of solutions. The appropriate configuration based on technical or economic considerations could then be chosen. To obtain the fitness function for any given configuration, it is necessary to model every flotation stage. The proposed method was used to find the optimum circuit configuration for a coal washing plant. The objective was to arrive at the highest yield while producing a concentrate with a certain ash content (11.2%). The feed to the flotation circuit was characterized based on the size fractions and their flotation rate constants. Results showed that with a 95% confidence the absolute difference between the modeled and measured values were 2.9–5.5% for yield and 0.4–1.1% for concentrate ash content. When the proposed GA-based circuit configuration was implemented in the plant, the yield increased from the original value of 57.6% to 64.3% while producing concentrate ash content (10.9%) within acceptable limits. By adding one stage to the current three-stage circuit, it was predicted that the yield could be further increased by 3.8% while keeping the quality of the concentrate within the appropriate level.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 15. INFLUENCE OF HUMIC ACIDS ON OIL SAND PROCESSING. PART I: DETECTION AND QUANTIFICATION OF HUMIC ACIDS IN OIL SAND ORES AND THEIR EFFECT ON BITUMEN WETTABILITY

Leopoldo Gutierrez, Marek Pawlik

*The University of British Columbia, Norman B. Keevil Institute of Mining Engineering, 517-6350 Stores Road,  
Vancouver, BC V6T 1Z4, Canada*

### ABSTRACT

Eight oil sands ores were tested in order to quantify the levels of humic acids in these samples through the alkali extraction test originally developed to determine the oxidation of bituminous metallurgical coals. The test gives a concentration of humic acids released from ores, which in combination with the measurement of the total organic carbon (TOC) content in the alkali extracts provides a measure of ore/bitumen weathering. It was found that poor ores (low bitumen and high fines contents) exhibited the highest tendency to leach large amounts of humic acids per gram of bitumen in the samples. It was also shown that the absorbance at 520 nm obtained from the UV/visible spectra of the alkali extracted solutions correlated very well with the quality of the ores, with the highest absorbance obtained from the extracts produced by the poor ores. A comparison between the UV/Visible spectra of solutions obtained from the alkali-extraction tests and spectra of solutions of commercial humic acids also indicated that the total organic carbon content of the extracts of good ores primarily originated from compounds other than humic acids while the organic matter released by poor ores was dominated by humic acids.

The results of contact angle measurements on fresh and oxidized bitumen samples indicated that contact angles decreased and bitumen became more hydrophilic as pH increased. It was also shown that the hydrophobicity of bitumen drastically decreased when the sample was artificially oxidized, although the amount of humic acids generated in bitumen during the oxidation process was small. Additionally, the effect of added humic acids on the wettability of bitumen was also evaluated. It was found that the contact angle of bitumen significantly decreased in the presence of humic acids at pH 3, but the effect of humic acids was not significant at higher pH values (7 and 10). At pH 7 the effect of humic acids on bitumen wettability was minor compared to the effect of artificial oxidation of bitumen. These results suggest that humic acids make bitumen hydrophilic if they are part of internal/surface bitumen structure. However, if they are added as free chemicals their effect is minor, particularly under neutral and alkaline pH conditions, which also indicates that adsorption of humic acids from solution onto bitumen is low.

Copyright © 2013 Elsevier B.V. All rights reserved.

16. INFLUENCE OF HUMIC ACIDS ON OIL SAND PROCESSING. PART  
II: RELATIONSHIP BETWEEN BITUMEN EXTRACTION,  
HUMIC ACIDS CONCENTRATION AND POWER DRAW  
MEASUREMENTS ON OIL SAND SLURRIES

Leopoldo Gutierrez, Marek Pawlik

*University of British Columbia, Norman B. Keevil Institute of Mining Engineering, 517-6350 Stores Road, Vancouver, BC, Canada*

**ABSTRACT**

The processability of four oil sand ores was evaluated through batch flotation experiments under different conditions of pH and temperature. The data of bitumen recovery were accurately modeled using a first-order kinetic equation assuming that the total amount of bitumen in the system consisted of two bitumen components, i.e., a fully-liberated bitumen fraction,  $f$ , characterized by a high flotation rate constant ( $k_f$ ), and poorly-liberated bitumen fraction,  $s$ , of a low flotation rate constant ( $k_s$ ) so that  $f + s = 1$ . The role of humic acids naturally present in the ores was basically that of a depressant of bitumen since poor ores contained the highest proportion of humic acids per gram of bitumen. The recovery of bitumen from oil sand ores correlated very well with the absorbance at 520 nm of the alkali extracts produced from the ores. High bitumen recovery was achieved for ores characterized by a low absorbance value of the extract, while low bitumen recoveries were obtained for ores producing a high absorbance value in the alkali extraction test.

Power draw measurements ahead of the bitumen flotation/extraction stage showed that power drawn by slurries of poor processing ores was not affected by pH and temperature, which was attributed to the low amount of bitumen and low degree of bitumen liberation in these ores. In contrast, good ores required more power for mixing under conditions of low pH and low temperature than under conditions of high pH and high temperature. Gradual bitumen liberation at higher temperature and pH improved the dispersion of the slurries and led to lower power requirements for mixing. Additionally, extraction data indicated that bitumen recovery was proportional to the power draw measured during feed conditioning. Poor ores produced slurries of lower viscosities than slurries prepared from good ores. As a result, higher power consumption was required during conditioning of good ores to promote bitumen liberation and to increase bitumen recovery.

Overall, it is possible to assess the processability of oil sand ores by quantifying the occurrence of humic acids in the ores, and to correlate ore processability with the rheology of ore slurries. Although poor ores are characterized by lower viscosities and lower power requirements during mixing, the presence of humic acids in these ores and their depressing action also contribute to lower bitumen recoveries.

Copyright © 2013 Elsevier B.V. All rights reserved.

Provided by  
DEPARTMENT OF FUEL AND MINERAL ENGINEERING  
INDIAN SCHOOL OF MINES, DHANBAD

## 17. INVESTIGATION OF DEPHOSPHORIZATION OF BROWN IRON ORE CONCENTRATES BY SINTERING AND MAGNETIC BENEFICIATION

B. Khassen<sup>a</sup>, N. Baltynova<sup>a</sup>, L. Dakhno<sup>b</sup>

<sup>a</sup>*The Institute of Problems of Complex Development of Mineral Resources, Karaganda,  
Kazakhstan* <sup>b</sup>*Karaganda State Technical University, Karaganda, Kazakhstan*

### ABSTRACT

The results of investigation of sintering and magnetic beneficiation of brown iron ore concentrate are presented. The investigations are based on using a possibility of redistribution of phosphorus from the ore constituents into slag-forming ones in the process of sinter roasting.

Copyright © 2013 Elsevier B.V. All rights reserved.

## 18. A STUDY ON THE CHARACTERISTICS OF SPRAY ANGLE FORMATION IN A 2 INCH HYDROCYCLONE USING WATER ONLY

A. Mazumdar<sup>a</sup>, R.K. Dubey<sup>a</sup>, C. Banerjee<sup>a</sup>, K. Sengupta<sup>b</sup>, A.K. Majumder<sup>a</sup>  
*a Department of Mining Engineering, Indian Institute of Technology, Kharagpur, 721302, India b Tega Industries Limited, Kolkata, 700053, India*

### ABSTRACT

It has been understood that the nature of slurry discharge through a hydrocyclone underflow provides an indication of its performance. Although a few attempts have already been made to correlate the discharge profile with hydrocyclone performance in quantifiable terms, a user friendly, robust tool is yet to be developed. In this article an attempt has been made to measure the spray angle at various operating conditions using a simple photographic technique. The study is restricted to water only. The measured data thus generated has been correlated well with inlet Reynolds number and with two exit diameters. Good reproducibility of the measured data over a prolonged duration of time and the developed correlation reaffirms that the spray angle could possibly be used as a useful tool to monitor hydrocyclone performance.

Copyright © 2013 Elsevier B.V. All rights reserved.

# MINERALS ENGINEERING

Volume 55

January 2014

Copyright © 2014 Elsevier Ltd. All rights reserved



Provided by:  
DEPARTMENT OF FUEL AND MINERAL ENGINEERING  
INDIAN SCHOOL OF MINES, DHANBAD

---

## TABLE OF CONTENTS

1. PURIFICATION OF IN SITU LEACHING SOLUTION FOR URANIUM MINING BY REMOVING SOLIDS FROM SUSPENSION .....	4
2. EXPERIMENTAL STUDY ON THE SHAPE–VELOCITY RELATIONSHIP OF AN ELLIPSOIDAL BUBBLE IN INORGANIC SALT SOLUTIONS .....	5
3. THE EXTRACTION OF Pt, Pd AND Au FROM AN ALKALINE CYANIDE SIMULATED HEAP LEACHATE BY GRANULAR ACTIVATED CARBON .....	6
4. THE EFFECT OF COLUMN DIMENSIONS AND PARTICLE SIZE ON THE RESULTS OF KINETIC COLUMN TEST USED FOR ACID MINE DRAINAGE (AMD) PREDICTION .....	7
5. COLUMN FLOTATION SIMULATION: A DYNAMIC FRAMEWORK .....	8
6. THE SAG GRINDABILITY INDEX TEST .....	9
7. ELECTROKINETIC REMEDIATION OF MINE TAILINGS BY APPLYING A PULSED VARIABLE ELECTRIC FIELD .....	10
8. SHOULD MAXIMUM PRESSURES IN ORE PIPELINES BE COMPUTED OUT OF SYSTEM STARTUPS OR POWER OUTAGES? .....	11
9. AN XPS INVESTIGATION OF SURFACE SPECIES FORMED BY ELECTROCHEMICALLY INDUCED SURFACE OXIDATION OF ENARGITE IN THE OXIDATIVE POTENTIAL RANGE .....	12
10. EVALUATION OF THE POTENTIAL FOR USING DIELECTROPHORESIS TO SEPARATE MINERALS	13
11. REFINEMENT OF THE ISOMORPHIC SUBSTITUTIONS IN GOETHITE AND HEMATITE BY THE RIETVELD METHOD, AND RELEVANCE TO BAUXITE CHARACTERISATION AND PROCESSING .....	14
12. THE INFLUENCE OF PYRITE CONTENT ON THE FLOTATION OF CHALCOPYRITE/PYRITE MIXTURES .....	15
13. A PARAMETRIC COST MODEL FOR MINERAL GRINDING MILLS .....	16
14. A NOVEL METHOD TO RECOVER ZINC AND IRON FROM ZINC LEACHING RESIDUE .....	17
15. AUTOMATED RELIEF-BASED DISCRIMINATION OF NON-OPAQUE MINERALS IN OPTICAL IMAGE ANALYSIS .....	18
16. AN INVESTIGATION INTO THE ROLE OF FROTH HEIGHT AND DEPRESSANT DOSAGE IN THE RECOVERY OF CHROMITE IN THE FLOTATION OF UG2 ORE USING A LABORATORY COLUMN .....	19
17. LIMITS OF THE CIL CIRCUIT IN COPPER–GOLD PLANTS .....	20
18. CAPTURE OF IMPACTING PARTICLES ON A CONFINED GAS–LIQUID INTERFACE .....	21
19. EFFECT OF CALCIUM, SULPHATE AND GYPSUM ON COPPER-ACTIVATED AND NON-ACTIVATED SPHALERITE SURFACE PROPERTIES .....	22
20. SELF-HEATING ACTIVATION ENERGY AND SPECIFIC HEAT CAPACITY OF SULPHIDE MIXTURES AT LOW TEMPERATURE .....	23
21. QUANTIFYING PARTICLE PICK UP AT A PENDANT BUBBLE: A STUDY OF NON-HYDROPHOBIC PARTICLE–BUBBLE INTERACTION .....	24

22.	PYROCHLORE SURFACE OXIDATION IN RELATION TO MATRIX FE COMPOSITION: A STUDY BY X-RAY PHOTOELECTRON SPECTROSCOPY .....	25
23.	IMPACT OF GYPSUM SUPERSATURATED PROCESS WATER ON THE INTERACTIONS BETWEEN SILICA AND ZINC SULPHIDE MINERALS .....	26
24.	RECOVERY OF GERMANIUM FROM WASTE SOLAR PANELS USING ION-EXCHANGE MEMBRANE AND SOLVENT EXTRACTION .....	27
25.	A NOVEL METHOD TO IMPROVE DEPRESSANTS ACTIONS ON CALCITE FLOTATION .....	28
26.	A PRELIMINARY RHEOLOGICAL CLASSIFICATION OF PHYLLOSILICATE GROUP MINERALS.....	29

# 1. PURIFICATION OF IN SITU LEACHING SOLUTION FOR URANIUM MINING BY REMOVING SOLIDS FROM SUSPENSION

A.M. Pastukhov<sup>a</sup>, V.N. Rychkov<sup>a</sup>, A.L.Smirnov<sup>a</sup>, S.Yu. Skripchenko<sup>a</sup>, N.A. Poponin<sup>b</sup>

*a: Department of Rare Metals and Nanomaterials, Institute of Physics and Technology, Ural Federal University, St. Mira, 19, 620002 Ekaterinburg, Russian Federation*

*b: JSC Dalur, St. Lenin, 42, Kurgan region, Dalmatovsky District, Uksyanskoye village 641750, Russian Federation*

## ABSTRACT

This study investigated the process of in situ leaching (ISL) method of uranium mining, and the removal of solid particles from the leaching solution. Investigations were carried out for 4 months. The content of firm suspensions in the productive solutions arriving from the well field was up to standard of 3–5 mg/l. After keeping in a settler of productive solutions within one hour concentration of suspensions decreases to 2–2.5 mg/l. To increase the life of the wells requires more fine purification of the ISL solutions. The best results can be obtained but using filtration. Bag filters were used in experiments carried out at the extraction site. All samples of polypropylene bag filter was produced by the Tamfelt Corporation. The best results were obtained for fabrics S-51M03-L2K4 (pore size 3  $\mu\text{m}$ ). After three month of trials following indicators of wells work were fixed: on the trial cell decrease in intake capacity did not occur; on the other cells of well field injectability of holes for the same period of time decreased for 15–40%. The results illustrated the high efficiency of this method, which allows injection wells to reach a constant intake capacity, making it possible for technological cells to achieve a constant productivity and balance. Purification of solutions allows to reduce acidulation term of new technological cells from 3–4 to 1.5–2 months

© 2013 Elsevier Ltd. All rights reserved.

## 2. EXPERIMENTAL STUDY ON THE SHAPE-VELOCITY RELATIONSHIP OF AN ELLIPSOIDAL BUBBLE IN INORGANIC SALT SOLUTIONS

J.J. Quinn , M. Maldonado<sup>1</sup>, C.O. Gomez, J.A. Finch

*Department of Mining and Materials Engineering, McGill University, 3610 University Street, Montreal, QC H3A 0C5, Canada*

### ABSTRACT

Individual bubbles ca. 2.3 mm in diameter were produced at a capillary in water containing an inorganic salt (NaClO<sub>4</sub>, KCl, NaCl, Na<sub>2</sub>SO<sub>4</sub>, or CaCl<sub>2</sub>). Using high speed photography and image analysis techniques, bubble aspect ratio and rise velocity were measured at 1 ms time intervals over a distance ca. 1.15–1.20 m above the capillary. All conditions showed oscillations in bubble aspect ratio and velocity that were related. Increasing concentration, on average, created more spherical bubbles that rose at lower velocities. The same observations were made in the presence of MIBC frother. Results suggest a unique relationship between bubble shape and rise velocity independent of solute type. The effect of inorganic salts on bubble behavior and gas dispersion in flotation systems is discussed.

© 2013 Elsevier Ltd. All rights reserved.

### 3. THE EXTRACTION OF Pt, Pd AND Au FROM AN ALKALINE CYANIDE SIMULATED HEAP LEACHATE BY GRANULAR ACTIVATED CARBON

C.N. Mpinga<sup>a</sup>, S.M. Bradshaw<sup>a</sup>, G. Akdogan<sup>a</sup>, C.A. Snyders<sup>a</sup>, J.J. Eksteen<sup>b</sup>

*a: Department of Processing Engineering, Stellenbosch University, Private Bag X1, Matieland 7602, South Africa*

*b: Department of Metallurgical Engineering, Western Australian School of Mines, Curtin University, GPO Box U1987, Perth, WA 6845, Australia*

#### ABSTRACT

Au(CN)<sub>2</sub>->Pt(CN)<sub>4</sub><sup>2-</sup>->Pd(CN)<sub>4</sub><sup>2-</sup>->Ni(CN)<sub>4</sub><sup>2-</sup>->Cu(CN)<sub>3</sub><sup>2-</sup>-It was shown that adsorption rates of precious metals within the first 60 min were very high, giving more than 90% extraction. Among the different adsorption parameters, nickel concentration had the most influential effect on the adsorption process followed by the adsorbent concentration. Adsorption of Ni was found to proceed at approximately the same rate as the precious metals, showing a recovery of approximately 90% in 2 h. The kinetics of Cu adsorption were slower, with less than 30% being recovered within the 120 min period. This suggests that the co-adsorption of Cu can be minimised by shortening the residence time. Adsorption of Fe was found to be less than 5%, while the recovery of Rh was negligibly small. The effect of thiocyanate ion concentration was not as important as the effect of free cyanide ion concentration but still had some influence. Under optimal (best) conditions, for a load cycle time of 2 h and 10 discontinuous loading cycles, the loading capacity of the activated carbon for PMs was observed to be 0.64, 0.66, 0.17 mg of Pt, Pd and Au/g of carbon, respectively.

© 2013 Elsevier Ltd. All rights reserved.

#### 4. THE EFFECT OF COLUMN DIMENSIONS AND PARTICLE SIZE ON THE RESULTS OF KINETIC COLUMN TEST USED FOR ACID MINE DRAINAGE (AMD) PREDICTION

Güzide Kalyoncu Erguler<sup>a</sup>, Zeynal Abiddin Erguler<sup>b</sup>, Hamdi Akcakoca<sup>c</sup>, Ali Ucar<sup>c</sup>

*a: Mineral Research & Exploration General Directorate (MTA), 06800 Çankaya, Ankara, Turkey*

*b: Dumlupınar University, Department of Geological Engineering, 43100 Kütahya, Turkey*

*c: Dumlupınar University, Department of Mining Engineering, 43100 Kütahya, Turkey*

#### ABSTRACT

Acid mine drainage (AMD) is known as one of the most important environmental problem of sulfide bearing rocks encountered worldwide. Several methods based on static and kinetic principles have been developed for estimation of AMD potential and determination of the contaminants concentration to the environment. Of these methods, kinetic column test is commonly performed due to its better representative of actual field conditions. However, the effect of main controlling factors such as column dimensions, the amount of material and its particle size on the results of kinetic column test were not investigated in details. Considering the importance of AMD and the limitations in conjunction with these main controlling factors, the objective of this study is to investigate the mechanism of the AMD generation and to perform this test by using samples in varied particle size and columns in different dimensions. For this purpose, columns were designed in various dimensions and samples were collected from Murgul Damar open-pit mining to be used in the kinetic column test. Several techniques were utilized to determine the mineralogical, physical and/or chemical composition of these samples before and during kinetic column tests. The variations of the pH and the concentration release rates of many elements and compounds were monitored during kinetic column tests. Statistically significant correlations were obtained between column dimensions and “lag time”. Similarly, it is determined that statistically significant correlations exist among column dimensions and cumulative mass release rates of SO<sub>4</sub> and the Ca + Mg + Mn.

© 2013 Elsevier Ltd. All rights reserved.

## 5. COLUMN FLOTATION SIMULATION: A DYNAMIC FRAMEWORK

Jocelyn Bouchard<sup>a, b</sup>, André Desbiens<sup>a, c</sup>, René del Villar<sup>a, b</sup>

*a: LOOP – Laboratoire d’observation et d’optimisation des procédés, Université Laval, Pavillon Adrien-Pouliot, Quebec City, Québec G1V 0A6, Canada*

*b: Département de génie des mines, de la métallurgie et des matériaux, Université Laval, Pavillon Adrien-Pouliot, Quebec City, Québec G1V 0A6, Canada*

*c: Département de génie électrique et de génie informatique, Université Laval, Pavillon Adrien-Pouliot, Quebec City, Québec G1V 0A6, Canada*

### ABSTRACT

Since the introduction of column flotation in mineral processing plants in the early 1980s, modelling of this process has become a prevailing field of investigation. Even if significant progresses have been made with every new attempt, most of the proposed models or simulators have been restricted to the steady-state behaviour. When dynamic mass balance equations were considered, a constant pulp level during the simulation was always assumed. The objective of this paper is to present a framework for the fully dynamic simulation of column flotation. The emphasis is placed on the simulation of water, solids and gas flows and their effect on the pulp level and output flow rates.

© 2013 Elsevier Ltd. All rights reserved.

## 6. THE SAG GRINDABILITY INDEX TEST

Peter Amelunxen<sup>a</sup>, Patricio Berríos<sup>b</sup>, Esteban Rodríguez<sup>b</sup>

*a: AME Ltda, Cerros de Camacho 440, Dep F-17, Santiago de Surco, Lima, Peru*

*b: Aminpro Chile SpA, Cerro San Cristobal 95111, Quilicura, Santiago, Chile*

### ABSTRACT

In this paper, the authors undertake a critical review of the Starkey test and the publicly available information related to the test equipment, procedures, and scale-up methodology. The following recommendations are proposed to improve the test method:

1. The test should be conducted for a fixed grinding time of 120 min, regardless of the time required to reach 80% passing Tyler #10 mesh.
2. The test should be conducted with constant time intervals of 15, 30, 60, and 120 min (cumulative) in order to facilitate the application of geostatistics to the resulting index values. This would also allow for multiple tests to be conducted in parallel (through the use of multiple mill rollers).
3. The feed size should be prepared using a more rigorous procedure to ensure constant mass in each of the course screen fractions.
4. The curve of finished product versus time should be modeled and the resulting index calculated from the model for a standard feed size distribution, so that errors attributable to the sample preparation step are minimized.

The improved feed preparation steps and the use of constant grinding intervals enables the development of a faster alternative to the standard test that is more cost effective for high volume geometallurgical programs.

In addition to the updated procedures, a new calibration equation is proposed, with calibration factors for pebble crushing, fine feed and autogenous grinding, based on information in the public literature. Detailed descriptions of the test equipment, procedures, and calibration are provided, and it is proposed that this become an open standard procedure for SAG mill hardness testing, particularly for soft to medium-hard ores, over which range the test is most effective.

© 2013 Elsevier Ltd. All rights reserved.

## 7. ELECTROKINETIC REMEDIATION OF MINE TAILINGS BY APPLYING A PULSED VARIABLE ELECTRIC FIELD

Adrián Rojo, Henrik K. Hansen, Omara Monárdez

*Departamento de Ingeniería Química y Ambiental, Universidad Técnica Federico Santa María, Casilla 110-V, Valparaíso, Chile*

### ABSTRACT

Following the flotation step of copper sulphide mineral processing, a considerable amount of ground ore in the form of a pulp containing heavy metals and other polluting compounds is discarded as waste, known as “mine tailings”. This waste is deposited behind dams, and unless it is treated, it represents a danger to the environment because the natural oxidation of heavy metals makes the waste chemically unstable.

Electrokinetic remediation (EKR) is a technology used to remove contaminants from soils. In recent years, the technology has been of research interest for stabilising mine tailings from the copper industry.

Nine EKR experiments with pulses of sinusoidal electric fields (by applying simultaneously DC and AC voltages) and an AC voltage frequency of the order of kHz were performed to improve conventional EKR and stabilise synthetic tailings samples. The synthetic tailings were prepared based on representative data for tailings from a combined Cu–Mo concentrator plant.

It was found that, in general, the use of a pulsed sinusoidal electric field favoured overall copper removal in the EKR cell, and particularly good results were observed when this type of electric field produced periodical polarity reversal in the electrodes.

The best results in terms of overall cell removal and specific energy consumption were obtained under the following conditions: (i) effective voltage of 14.6 V (VDC = 10 V and VAC = 15 V), (ii) AC voltage frequency 1000 Hz, (iii) electrical field applied in pulses with a time ratio of 25.

© 2013 Elsevier Ltd. All rights reserved.

## 8. SHOULD MAXIMUM PRESSURES IN ORE PIPELINES BE COMPUTED OUT OF SYSTEM STARTUPS OR POWER OUTAGES?

Christian F. Ihle

*Department of Mining Engineering, Universidad de Chile, Av. Tupper 2069, Santiago, Chile  
Advanced Mining Technology Center, Universidad de Chile, Av. Tupper 2007, Santiago, Chile*

### ABSTRACT

A key aspect of the design of long distance ore concentrate pipelines is the need to properly predict maximum pressures. This is traditionally done by means of transient analysis, thus predicting the possible impact of slurry hammers, which may occur during operation in a potentially uncontrolled manner in case of power outages. In this technical note, it is shown for typical ore slurry and pipeline characteristics, that in long distance systems with routes having inclined sections, the plug formation mechanism may become a dominant factor in system overpressures. A dimensionless number expressed as the ratio of the Joukowski and the plug overpressure value, suggests that a scale for the critical plug length above which maximum pressures are controlled by the plug mechanism rather than the transient flow is between about 150 m and 500 m, or a few percent points of the overall pipeline length in common long distance systems. A critical dependence on the solids initial concentration and the product of the static friction factor and the solids settled concentration is addressed.

© 2013 Elsevier Ltd. All rights reserved.

## 9. AN XPS INVESTIGATION OF SURFACE SPECIES FORMED BY ELECTROCHEMICALLY INDUCED SURFACE OXIDATION OF ENARGITE IN THE OXIDATIVE POTENTIAL RANGE

Chris Plackowski<sup>a</sup>, Marc A. Hampton<sup>a</sup>, Warren J. Bruckard<sup>b</sup>, Anh V. Nguyen<sup>a</sup>

*a: School of Chemical Engineering, The University of Queensland, Brisbane, Queensland 4072, Australia*

*b: CSIRO Process Science and Engineering, Box 312, Clayton, Victoria, Australia*

### ABSTRACT

Oxidation of the surface of natural enargite ( $\text{Cu}_3\text{AsS}_4$ ) under potentiostatic control and the formation of oxidation species at the mineral surface has been investigated at selected applied potentials in the oxidative range. Potentials at which oxidation reactions were found to occur were identified by cyclic voltammetry as +347, +516, +705, +869 and +1100 mV SHE (versus Standard Hydrogen Electrode). The mineral surface was oxidized by application of these potentials in pH 10 buffer solution, and then analyzed by X-ray photoelectron spectroscopy (XPS). Liquid nitrogen cooling was used during XPS analysis to minimize the effects of exposure to X-rays, thermal degradation, and ultra-high vacuum conditions. The surface speciation of electrochemically oxidized natural enargite obtained for these anodic potentials at pH 10 demonstrated a progressively increasing level of oxidation as applied potential increases. Surface layer deposition was linked to potential, with limited evidence of oxidation products at the surface found after treatment at applied potentials of +347 and +516 mV. At these potentials no evidence of Cu(II) compounds was found, while a decrease in the proportion of copper at the surface suggests dissolution as the primary reaction mechanism. At a treatment potential of +705 mV, Cu(II) species identified as  $\text{CuSO}_4$  and  $\text{Cu(OH)}_2$  were found, although the presence of arsenic oxides or sulfides was not found. After treatment at +869 and +1100 mV significant evidence of oxidation was found, with Cu(II) species of  $\text{CuSO}_4$  and  $\text{Cu(OH)}_2$  found. Additional sulfur and arsenic species of  $\text{CuS}$  and  $\text{As}_2\text{O}_3$  were identified that were not present after treatment at lower potentials. Comparison of the XPS findings with previously published proposed reaction mechanisms for similar treatment potentials showed that they did not account for all species identified in the XPS data. Analysis of buffer solutions post-treatment by ICP (Inductively Coupled Plasma spectroscopy) showed a pattern of change in concentrations of Cu, As and S characterized by a step-change increase in dissolution between the +516 and +705 mV treatment conditions, which correlates with the formation of Cu(II) identified on the mineral surface.

© 2013 Elsevier Ltd. All rights reserved.

## 10. EVALUATION OF THE POTENTIAL FOR USING DIELECTROPHORESIS TO SEPARATE MINERALS

G.R. Ballantyne, P.N. Holtham

*The University of Queensland, Sustainable Minerals Institute, Julius Kruttschnitt Mineral Research Centre, 40 Isles Road, Indooroopilly, Brisbane, QLD 4068, Australia*

### ABSTRACT

The mineral processing industry requires alternative separation techniques to help deal with rising energy costs and decreasing ore grades. Dielectrophoresis is a little known mechanism potentially able to separate mineral particles based on differences in electrical properties. There are many areas of minerals processing that would benefit from the application of dielectrophoresis. These include early removal of gangue, recovery of ultra-fine particles, upgrading of flotation concentrates or simply as a laboratory analysis technique. However, a more thorough understanding of the dielectrophoretic force is required to effectively design a practical separator.

The aim of the work described here was to measure the dielectrophoretic force on single particles of chalcopyrite and quartz to determine the effect of particle size, shape and position in an electric field. The force was determined by measuring the deflection of a particle attached to a glass fibre positioned between a pin and plate electrode. The dielectrophoretic force was found to be proportional to particle size cubed and inversely proportional to the distance between the particle and the pin cubed. Particle shape also affects the experienced force, and strategies were investigated to minimise this source of variation. With the effect of these parameters fully characterised, the single particle test cell can be used to determine the permittivity of single particles of various composition.

© 2013 Elsevier Ltd. All rights reserved.

## 11. REFINEMENT OF THE ISOMORPHIC SUBSTITUTIONS IN GOETHITE AND HEMATITE BY THE RIETVELD METHOD, AND RELEVANCE TO BAUXITE CHARACTERISATION AND PROCESSING

Reiner Neumann<sup>a</sup>, Angela Nair Avelar<sup>b</sup>, Geraldo Magela da Costa<sup>c</sup>

*a: CETEM – Centre for Mineral Technology, Division for Technological Characterisation, Avenida Pedro Calmon, 900, 21941-908 Rio de Janeiro, RJ, Brazil*

*b: Vale SA, Process Development Americas, Santa Luzia, MG, Brazil*

*c: Chemistry Department, Universidade Federal de Ouro Preto, Ouro Preto, MG, Brazil*

### ABSTRACT

Although bauxites usually have a quite simple mineralogy – gibbsite (+boehmite), quartz, kaolinite, hematite, goethite, anatase (+rutile) and minor or less common phases, fine particle size, low crystallinity and variable compositions of the iron minerals might render phase quantification difficult, as well as impairing bauxite processing. A reliable and complete characterisation is therefore necessary in order to predict processing performance and ensure compliance to plant specifications.

X-ray diffraction is the most important single tool for bauxite characterization, and the constrained refinement of the Al-for-Fe substitution in goethite during one-step phase quantification by fundamental parameters Rietveld method has been successfully used. The same method was developed to analyze the coupled Al-for-Fe and OH<sup>-</sup>-for-O<sup>2-</sup> substitutions in hematite. The method was tested against Mössbauer spectroscopy iron distribution on bauxite samples with a large compositional range, and on bauxite Certified Reference Materials from the main Brazilian mines, with improved results and widened range of conclusions that can be drawn related to bauxite processing.

© 2013 Elsevier Ltd. All rights reserved.

## 12. THE INFLUENCE OF PYRITE CONTENT ON THE FLOTATION OF CHALCOPYRITE/PYRITE MIXTURES

Clement Owusu, Susana Brito e Abreu, William Skinner, Jonas Addai-

Mensah, Massimiliano Zanin

*Ian Wark Research Institute, The ARC Special Research Centre for Particle and Material Interfaces, University of South Australia, Mawson Lakes, Adelaide, SA 5095, Australia*

### ABSTRACT

In the flotation of copper ores, several processing plants report that copper recovery is affected by the proportion and reactivity of pyrite in the ore, with the effect becoming more intense when the feed particles are finer as a result of regrinding. In this work, a mixed model mineral system consisting of chalcopyrite ( $\text{CuFeS}_2$ ) and pyrite ( $\text{FeS}_2$ ) with varying pyrite content (20–80 wt. %) was used to investigate the effect of pyrite on the pulp chemistry and chalcopyrite recovery. Flotation tests showed that chalcopyrite flotation rate, recovery and grade, as well as the pulp oxidation potential, decreased with increasing pyrite content whilst pyrite recovery increased. Surface analysis (XPS, ToF-SIMS and EDTA) indicated that copper activation of pyrite increased with increasing pyrite content, facilitating pyrite recovery. The decrease in chalcopyrite recovery can be attributed to increased surface oxidation.

© 2013 Elsevier Ltd. All rights reserved.

### 13. A PARAMETRIC COST MODEL FOR MINERAL GRINDING MILLS

Ahmad Reza Sayadi, Mohammad Reza Khalesi, Mahsa Khoshfarman Borji

*Mining Department, Tarbiat Modares University, Tehran, Iran*

#### ABSTRACT

The adequate cost estimation of mill plants plays a crucial role in the success of feasibility studies of mining projects. Grinding is one of the most important operations in mineral processing plants and assumes a substantial share of the total milling costs. The objective of this work was to develop a set of cost functions for major grinding mill equipment. These cost models were developed using two relatively different techniques: uni-variate regression (UVR) as well as multivariate regression (MVR) based on principal component analysis (PCA). The first is appropriate for the quick estimation of costs in the early stages of project evaluation, while the second method can be helpful in the feasibility study stage. The explanatory variable in UVR was power ( $P$ ), while in MVR the power and some other variables depending on the type of mill were used. The PCA technique was employed in order to omit the correlation between the independent variables in the multivariate regression. Furthermore, the scale-up factor for all mills has been calculated. The result of the evaluation of the models showed that the mean absolute error rates were less than 9.84% and 11.36% on average for the capital and operating costs of the uni-variate model, and 5.82% and 4.9% for the multivariate model, respectively.

© 2013 Elsevier Ltd. All rights reserved.

## 14. A NOVEL METHOD TO RECOVER ZINC AND IRON FROM ZINC LEACHING RESIDUE

Huan Yan<sup>a</sup>, Li-yuan Chai<sup>a, b</sup>, Bing Peng<sup>a, b</sup>, Mi Li<sup>a</sup>, Ning Peng<sup>a</sup>, Dong-ke Hou<sup>a</sup>

*a: Institute of Environmental Science and Engineering, School of Metallurgy and Environment, Central South University, 410083 Changsha, Hunan, China*

*b: Chinese National Engineering Research Center for Control & Treatment of Heavy Metal Pollution, 410083 Changsha, Hunan, China*

### ABSTRACT

A novel method to recover zinc and iron from zinc leaching residue (ZLR) by the combination of reduction roasting, acid leaching and magnetic separation was proposed. Zinc ferrite in the ZLR was selectively transformed to ZnO and Fe<sub>3</sub>O<sub>4</sub> under CO, CO<sub>2</sub> and Ar atmosphere. Subsequently, acid leaching was carried out to dissolve zinc from reduced ZLR while iron was left in the residue and recovered by magnetic separation. The mineralogical changes of ZLR during the processes were characterized by XRF, TG, XRD, SEM-EDS and VSM. The effects of roasting and leaching conditions were investigated with the optimum conditions obtained as follows: roasted at 750 °C for 90 min with 8% CO and CO/CO + CO<sub>2</sub> ratio at 30%; leached at 35 °C for 60 min with 90 g/l sulfuric acid and liquid to solid ratio at 10:1. The iron was recovered by magnetic separation with magnetic intensity at 1160 G for 20 min. Under the optimum operation, 61.38% of zinc was recovered and 80.9% of iron recovery was achieved. This novel method not only realized the simultaneous recovery of zinc and iron but also solved the environmental problem caused by the storage of massive ZLR.

© 2013 Elsevier Ltd. All rights reserved.

## 15. AUTOMATED RELIEF-BASED DISCRIMINATION OF NON-OPAQUE MINERALS IN OPTICAL IMAGE ANALYSIS

A. Poliakov, E. Donskoi

*Processing and Agglomeration, CSIRO CPSE, PO Box 883, Kenmore, QLD 4069, Australia*

### ABSTRACT

Ore characterisation is important in order to understand the quality of ores and their behaviour during downstream processing. Many significant ore characteristics can only be determined through the use of various imaging techniques. Optical Image Analysis (OIA) is one such technique and is particularly attractive for many applications due to its low cost and high resolution. However OIA also has some limitations, one of which is the difficulty with discriminating non-opaque minerals. Some non-opaque minerals, such as quartz, are typical gangue minerals in certain types of iron ores. Even though in many cases quartz particles can be easily seen and attributed by mineralogists in polished sections, their automated discrimination has always been an issue, the reasons for which are discussed in this article. The ability to automatically discriminate quartz and other non-opaque minerals would significantly increase the value of OIA for the mineral industry.

This paper describes a novel method of discriminating non-opaque minerals in the sample by their optical relief, which results in visible borders between the mineral and the epoxy resin mounting medium. An algorithm for such discrimination that has been developed for the CSIRO Mineral4/Recognition4 OIA software package is described. The algorithm is based on dynamic thresholding of the image with subsequent cleanup and enhancement to reliably determine borders between non-opaque particles and epoxy and on subsequent attribution of image areas created by these borders to either the non-opaque mineral or the epoxy resin. Further, this article discusses difficulties that may arise when applying this algorithm due to sample peculiarities and describes algorithm enhancements incorporated in Mineral4 in order to overcome these issues. The resulting software is capable of reliably discriminating non-opaque minerals in a variety of samples, including iron and manganese ores.

© 2013 Elsevier Ltd. All rights reserved.

## 16. AN INVESTIGATION INTO THE ROLE OF FROTH HEIGHT AND DEPRESSANT DOSAGE IN THE RECOVERY OF CHROMITE IN THE FLOTATION OF UG2 ORE USING A LABORATORY COLUMN

M. Alvarez-Silva<sup>a, b</sup>, J. Wiese<sup>b</sup>, C.T. O'Connor<sup>b</sup>

*a: Department of Chemical and Environmental Engineering, Universidad Técnica Federico Santa María, Av. España 1680, Valparaíso, V de Valparaíso 2390123, Chile*

*b: Centre for Minerals Research, Department of Chemical Engineering, Private Bag X3, University of Cape Town, Rondebosch 7701, Cape Town, Western Cape, South Africa*

### ABSTRACT

The control of chromite during the flotation of UG2 ore is of critical importance since its presence affects the downstream pyrometallurgical treatment. It has been reported that the main mechanism for chromite recovery is entrainment ( ) and hence monitoring the mass of solids and water recovered is important in such an investigation. It is also well known that the froth phase plays a key role in the control of entrained gangue ( ). In the present investigation a laboratory scale column cell was used to determine the mass of solids and water recovered in the concentrate as a function of different froth heights, superficial gas velocity and depressant dosages. Chromite assays were also carried out on all streams. Tests were carried out at two different froth heights (viz., 30 cm and 60 cm) and in the absence of depressant, using 100 g/t and 500 g/t of guar. Superficial gas velocity was set at three different values, 1 cm/s, 1.5 cm/s and 2 cm/s. The collector was SIBX (80 g/t) and the frother was Dow 200 (100 g/t). The column attained operational steady state. Samples of feed, concentrate and tailings were taken to determine solids and water recoveries, particle size distributions of all streams and chromite grades of the bulk feed material as well as of each stream on a size by size basis. Also, mineralogical examination using QEMSCAN were performed on selected concentrates samples. It was found that froth height had no significant effect on the water, solids recovered or on the chromite grade of the concentrate. However the higher depressant dosage resulted in an increase in chromite grade in the concentrate which is ascribed to the effect of the guar in depressing the recovery of gangue minerals. The paper will present the detailed results of the investigation.

© 2013 Elsevier Ltd. All rights reserved.

## 17. LIMITS OF THE CIL CIRCUIT IN COPPER–GOLD PLANTS

Fraser Burns<sup>a, b</sup>, Yongjun Peng<sup>a, c</sup>, David Seaman<sup>b</sup>, Dee Bradshaw<sup>a</sup>

*a: Julius Kruttschnitt Mineral Research Centre, University of Queensland, Isles Road, Indooroopilly, Brisbane, QLD 4068, Australia*

*b: Newcrest Mining Limited, 193 Great Eastern Highway, Belmont, WA 6104, Australia*

*c: School of Chemical Engineering, University of Queensland, St. Lucia, Brisbane, QLD 4072, Australia*

### ABSTRACT

This study examined the performance of the CIL (Carbon-in-Leach) circuit at Telfer, a copper–gold plant treating porphyry copper deposits containing gold associated with both copper and iron sulphides, with an objective to identify factors normally limiting the gold recovery in the CIL circuit in the presence of a small amount of copper after copper flotation, and then propose a means to improve it. Diagnostic leaching assessment and mineralogical analysis by MLA revealed that the occlusion of gold by other minerals and the fine grain size of gold associated with them may be the contributing factors to the low gold recovery in the CIL circuit. Fine grinding of the CIL feed increased gold recovery significantly from the leaching process. However, it is interesting to find that fine grinding increased the amount of released copper ions which complex with cyanide resulting in significantly higher cyanide consumption. It is therefore proposed that regrinding of the CIL feed followed by copper flotation is an appropriate pre-treatment method for the CIL circuit.

© 2013 Elsevier Ltd. All rights reserved.

## 18. CAPTURE OF IMPACTING PARTICLES ON A CONFINED GAS– LIQUID INTERFACE

Dongmei Liu, Qinglin He, Geoffrey Evans

*School of Engineering, The University of Newcastle, Callaghan, NSW 2308, Australia*

### ABSTRACT

Film flotation is highly dependent upon how well wanted and unwanted particles are separated at the gas–liquid free surface. Single particle experiments and modelling analysis can be undertaken to determine a critical impact velocity at which the optimum separation is likely to occur. However, in commercial film flotation systems the higher loading density of the free surface can result in interactions that change the critical impact velocity. To investigate this influence, film flotation experiments have been undertaken with 4, 5 and 6 mm diameter spherical polypropylene particles with water, sucrose and surfactant (CTAB) solutions contained within vessels of varying dimensions and wettability (static contact angle). Experimentally it was observed that for a given particle diameter the critical impact velocity was found to decrease with decreasing vessel diameter, especially when the particle-to-vessel diameter ratio increased beyond about 0.2. Conversely, the critical impact velocity was found to be relatively independent of the liquid depth; but did decrease with decreasing static contact angle in the region where the vessel wall had an influence. The experimental system was also modelled using the Young–Laplace equation using both static and advancing contact angle measurements for both the particle and vessel surfaces. The model predictions were generally in good agreement with the experimental observations, including showing an increase in particle penetration depth with increasing vessel diameter and meniscus profiles, both at the particle impact point and the wall of the vessel. The predictions were improved when the advancing contact angle was used, especially for the smaller diameter vessels where there was more liquid motion. Finally, a model to determine the critical (minimum) diameter of vessel required so that the cavity profile generated by the impacting particle is unlikely to be influenced by the vessel walls is presented.

© 2013 Elsevier Ltd. All rights reserved.

## 19. EFFECT OF CALCIUM, SULPHATE AND GYPSUM ON COPPER-ACTIVATED AND NON-ACTIVATED SPHALERITE SURFACE PROPERTIES

G.I. Dávila-Pulido, A. Uribe-Salas

*Centro de Investigación y de Estudios Avanzados del IPN, Unidad Saltillo, Ramos Arizpe, Coahuila, Mexico*

### ABSTRACT

This work presents results of an experimental study on the depression of sphalerite by the precipitation of calcium sulphate dihydrate (gypsum,  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ ), and the role of the addition of sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) in restoring hydrophobicity. Zeta potential, microflotation, contact angle measurement and scanning electron microscopy were used. The zeta potential results show that sphalerite and gypsum were negatively charged over the pH range studied (5–10), and therefore no interaction between the two species was observed; however, when sphalerite was activated with 5 mg/L of Cu at pH 9, the mineral developed a positive charge, thus enabling the electrostatic interaction with gypsum particles which resulted in gypsum covering a significant fraction of the mineral surface. The contact angle measurements show that the simultaneous presence of  $\text{Ca}^{2+}$  and  $\text{SO}_4^{2-}$  ions decreased the hydrophobicity that the mineral had developed due to copper activation in the absence of these two species ( $19.6^\circ$  and  $28.7^\circ$ , respectively). The presence of 10 mg/L of  $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$  in equilibrium with the saturated solution (0.0205 mol/L  $\text{CaSO}_4$ ), further decreased the contact angle to  $9.7^\circ$ . In turn, the addition of 1 g/L of  $\text{Na}_2\text{CO}_3$  and 15 min of conditioning substantially restored the hydrophobicity of the sphalerite by a mechanism consisting of gypsum dissolution and calcium consumption via calcium carbonate precipitation, which disperses in the bulk solution. This last practice resulted in a contact angle of  $23.4^\circ$ .

© 2013 Elsevier Ltd. All rights reserved.

## 20. SELF-HEATING ACTIVATION ENERGY AND SPECIFIC HEAT CAPACITY OF SULPHIDE MIXTURES AT LOW TEMPERATURE

Barnabe Ngabe, James A. Finch

*McGill University, Mining and Materials Engineering Department, Montreal, QC H3A 2B2, Canada*

### ABSTRACT

Energy of activation ( $E_a$ ) and specific heat capacity ( $C_p$ ) for mixtures of sulphide minerals that on their own do not self-heat (SH), sphalerite/pyrite, pyrite/galena, chalcopyrite/galena and sphalerite/galena, were determined using a self-heating apparatus at temperatures below 100 °C in the presence of moisture. The mixtures all gave  $E_a$  ranging from 22.0 to 27.8 kJ mol<sup>-1</sup>, similar to the range reported for Ni- and Cu-concentrates. The  $E_a$  is close to that for partial oxidation of H<sub>2</sub>S which adds to the contention that the partial oxidation of H<sub>2</sub>S contributes to SH of sulphides at low temperature. The  $C_p$  values ranged from 0.152 to 1.071 JK<sup>-1</sup> g<sup>-1</sup> as temperature rose from 50 °C to 80 °C, similar to the reported findings on Ni- and Cu-concentrates. The role of galvanic interaction in promoting SH is tested by examining correlations with the rest potential difference of the sulphides in the mixture.

© 2013 Elsevier Ltd. All rights reserved.

21. QUANTIFYING PARTICLE PICK UP AT A PENDANT BUBBLE:  
A STUDY OF NON-HYDROPHOBIC PARTICLE-BUBBLE  
INTERACTION

P. Chu, M. Mirnezami, J.A. Finch

*Department of Mining and Materials Engineering, McGill University, 3610 University St., Montreal, QC  
H3A 0C5, Canada*

ABSTRACT

To study bubble interaction with non-hydrophobic particles an imaging technique has been developed to quantify particle pick up at a pendant bubble by measuring the bubble-particle attachment angle (BPA) made by the particle bed on the bubble. The technique was verified by correlating pick up mass against BPA. Pick up of alumina was shown to correlate with difference in alumina and bubble zeta potential supporting an electrostatic model of interaction with non-hydrophobic particles. Pick up also correlated with contact angle (Washburn method) indicating the electrostatic force is sufficient to establish a solid-air interface.

© 2013 Elsevier Ltd. All rights reserved.

## 22. PYROCHLORE SURFACE OXIDATION IN RELATION TO MATRIX FE COMPOSITION: A STUDY BY X-RAY PHOTOELECTRON SPECTROSCOPY

S. Chehreh Chelgani<sup>a</sup>, B. Hart<sup>a</sup>, M. Biesinger<sup>a</sup>, J. Marois<sup>b</sup>, M. Ourriban<sup>b</sup>

*a: Surface Science Western, Research Park, University of Western Ontario, London, Ont. N6G0J3, Canada*

*b: Niobec Inc. 3400 ch. du Columbium, St-Honoré-de-Chicoutimi, Québec G0V 1L0, Canada*

### ABSTRACT

The flotation recovery of pyrochlore from the Saint-Honore Carbonatite ore has been related to the variability in the mineral matrix Fe content; high Fe pyrochlore grains consistently report to the tails. Previous research has tentatively suggested that surface oxidation of high Fe pyrochlore grains may be driving partitioning of the pyrochlore grains to the tails. X-ray photoelectron spectroscopy (XPS) was used to examine the relationship between matrix Fe content and surface oxidation in high Fe pyrochlore grains and high Fe zoned regions in pyrochlore grains from the Carbonatite ore. XPS analyses of pyrochlore grains showed that a greater proportion of surface oxidation species corresponded to the zones with high matrix Fe content. The XPS data along with previously presented Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS) data demonstrates a relationship between matrix Fe content and collector attachment. The Fe rich pyrochlore grains or zones show a higher degree of oxidation relative to the Fe poor grains or zones. The data reveal that collector attachment favors low Fe grains or zones, likely in response to a lower degree of surface oxidation.

© 2013 Elsevier Ltd. All rights reserved.

## 23. IMPACT OF GYPSUM SUPERSATURATED PROCESS WATER ON THE INTERACTIONS BETWEEN SILICA AND ZINC SULPHIDE MINERALS

Meijiao Deng, Zhenghe Xu, Qingxia Liu

*Department of Chemical and Materials Engineering, University of Alberta, Edmonton, Alberta T6G 2G6, Canada*

### ABSTRACT

Flotation recovery and selectivity problems have been reported in the flotation of fine sulphide minerals in gypsum supersaturated process water. In this study, the effect of gypsum supersaturated solution on the interactions between silica and sphalerite (ZnS) minerals was examined by observing deposition behaviour of silica nanoparticles on sphalerite surface using a quartz crystal microbalance with dissipation (QCM-D). Significant deposition of silica nanoparticles on ZnS coated sensor surface was observed in the gypsum supersaturated solution, indicating consequential slime coating of silica fines on sphalerite mineral surface. Substantial deposition of silica nanoparticles on SiO<sub>2</sub> coated surface was also observed suggesting strong homo-aggregation of silica fines in the gypsum supersaturated solution. The interaction behaviour between silica–sphalerite and silica–silica is mainly attributed to the high calcium concentration of the gypsum supersaturated solution. Similar deposition behaviour of silica nanoparticles onto ZnS or SiO<sub>2</sub> coated sensor surface was observed in 800 ppm calcium solution, which is similar to the calcium concentration of the gypsum supersaturated solution. Colloidal force measurement between a silica particle and a fractured sphalerite surface or a silica wafer surface by an atomic force microscopy (AFM) revealed attractive van der Waals force between the mineral particles in both gypsum supersaturated solution and 800 ppm calcium solution. The high calcium concentration of the gypsum supersaturated solution induced the hetero-aggregation between silica and sphalerite, accounting for the observed decrease in flotation selectivity.

© 2013 Elsevier Ltd. All rights reserved.

## 24. RECOVERY OF GERMANIUM FROM WASTE SOLAR PANELS USING ION-EXCHANGE MEMBRANE AND SOLVENT EXTRACTION

Keisuke Kuroiwa<sup>a</sup>, Shin-ichiro Ohura<sup>a</sup>, Shintaro Morisada<sup>a</sup>, Keisuke Ohto<sup>a</sup>, Hidetaka

Kawakita<sup>a</sup>, Yoshiyasu Matsuo<sup>b</sup>, Daisuke Fukuda<sup>b</sup>

*a: Department of Applied Chemistry, Saga University, Saga 840-8502, Japan*

*b: NT Corporation, 1542-2 Tachibana, Imari 848-0027, Japan*

### ABSTRACT

Germanium was recovered from the waste solar panels using the processes of selective catechol complexation, membrane adsorption and elution, and solvent extraction. Because the solar panels included a high concentration of germanium, the key technique used was high selectivity against silicate ion using hydrometallurgy. Due to the selective complexation of germanium ions with catechol, the operation at low pH enhanced the selectivity. 0.1 M hydrochloric acid was used to elute the germanium ions. To remove the catechol from the germanium containing solution, solvent extraction using trioctylphosphine oxide was performed, demonstrating that 81% of catechol was removed. Compared with the initial percentage of germanium to silica, the concentration of silicate was 39 times higher in the germanium-concentrated solution, indicating that the proposed sequential process was very a powerful technique for the recovering germanium.

© 2013 Elsevier Ltd. All rights reserved.

## 25. A NOVEL METHOD TO IMPROVE DEPRESSANTS ACTIONS ON CALCITE FLOTATION

Qing Shi, Qiming Feng, Guofan Zhang, Hong Deng

*Department of Minerals Engineering, School of Minerals Processing and Bioengineering, Central South University, Changsha 410083, China*

### ABSTRACT

In this study, the flotation behaviour, contact angle and surface adsorption of calcite were investigated in aqueous solution containing excess lattice ions ( $\text{Ca}^{2+}$  and  $\text{CO}_3^{2-}$ ). Flotation results show that adding lattice ions makes three kinds of depressants more effective. The contact angle test suggests that the use of both  $\text{Ca}^{2+}$  and  $\text{CO}_3^{2-}$  results in a remarkable decrease in calcite surface hydrophobicity with sodium oleate and depressants, while it has little influence on calcite surface hydrophobicity without sodium oleate. Adsorption data indicates calcium carbonate coating on calcite surfaces may change the adsorption mechanism of depressants. The study presented that added lattice ions change reactivity of the calcite surface and improve depressants actions on calcite flotation.

© 2013 Elsevier Ltd. All rights reserved.

## 26. A PRELIMINARY RHEOLOGICAL CLASSIFICATION OF PHYLLOSILICATE GROUP MINERALS

Bulelwa Ndlovu<sup>a, b</sup>, Elizaveta Forbes<sup>c</sup>, Saeed Farrokhpay<sup>a</sup>, Megan Becker<sup>b</sup>, Dee

Bradshaw<sup>a</sup>, David Deglon<sup>b</sup>

*a: Julius Kruttschnitt Mineral Research Centre, University of Queensland, 40 Isles Road, Indooroopilly, 4068 QLD, Australia*

*b: Minerals to Metals Research Initiative, Department of Chemical Engineering, University of Cape Town, South Africa*

*c: CSIRO Process Science and Engineering, Clayton, Victoria, Australia*

### ABSTRACT

With the increased exposure to progressively complex ores, there is growing concern over the effects of phyllosilicate gangue minerals. These minerals present challenges during ore beneficiation, with issues such as reduced flotation performance and complex tailings treatment arising. Often broadly classified as 'clays', the understanding of the distinct behaviour of phyllosilicates remains poor. This work focusses on talc, illite and kaolinite, and forms part of an ongoing study which aims at investigating the entire phyllosilicate group, categorised as serpentine, micas; talc/pyrophyllite, kaolinites, illites, smectites and vermiculite. Using pure minerals belonging to each phyllosilicate type, a comprehensive surface charge and rheological analysis was conducted, incorporating their charge anisotropy and non-spherical morphology. The mineralogy was discussed, based on pre-existing knowledge of the minerals. Talc, kaolinite and illite suspensions are characterised by high yield stresses and low viscosities, with differences in their behaviour attributed to variations in charge anisotropy, aspect ratio and surface morphology. A comparison with other phyllosilicates showed that muscovite (mica) results in the least rheologically complex suspensions, while the fibrous nature of chrysotile (serpentine) leads to suspensions with the highest yield stresses and viscosities. The other minerals demonstrate intermediate rheological behaviour. Such a classification may be foundational to geometallurgical advances which can enable process performance predictions based on mineralogy.

© 2013 Elsevier Ltd. All rights reserved.

# FUEL

Volume 127

July 2014

Copyright © 2014 Elsevier Ltd. All rights reserved



Provided by:  
DEPARTMENT OF FUEL AND MINERAL ENGINEERING  
INDIAN SCHOOL OF MINES, DHANBAD

---

## TABLE OF CONTENTS

1.	A SYSTEMATIC APPROACH FOR HIGH TEMPERATURE LOOPING CYCLES INTEGRATION .....	4
2.	CARBONATE LOOPING EXPERIMENTS IN A 1 MWTH PILOT PLANT AND MODEL VALIDATION .	5
3.	DEVELOPMENT OF THE CALCIUM LOOPING CO <sub>2</sub> CAPTURE TECHNOLOGY FROM LAB TO PILOT SCALE AT IFK, UNIVERSITY OF STUTTGART.....	6
4.	COAL COMBUSTION UNDER CALCIUM LOOPING PROCESS CONDITIONS .....	7
5.	COAL COMBUSTION CHARACTERISTICS ON AN OXY-FUEL CIRCULATING FLUIDIZED BED COMBUSTOR WITH WARM FLUE GAS RECYCLE.....	8
6.	UNDESIRE EFFECTS IN THE DETERMINATION OF CO <sub>2</sub> CARRYING CAPACITIES OF CAO DURING TG TESTING .....	9
7.	DEVELOPMENT OF A NOVEL-SYNTHESIZED CA-BASED CO <sub>2</sub> SORBENT FOR MULTICYCLE OPERATION: PARAMETRIC STUDY OF SORPTION .....	10
8.	INVESTIGATION OF THE FRIABILITY OF CA LOOPING SORBENTS DURING AND AFTER HYDRATION BASED REACTIVATION.....	11
9.	KINETICS OF CO <sub>2</sub> ABSORPTION BY CAO THROUGH PRESSURE SWING CYCLING .....	12
10.	UNDERSTANDING THE ENHANCEMENT EFFECT OF HIGH-TEMPERATURE STEAM ON THE CARBONATION REACTION OF CAO WITH CO <sub>2</sub> .....	13
11.	SOL-GEL-DERIVED, CAO-BASED, ZRO <sub>2</sub> -STABILIZED CO <sub>2</sub> SORBENTS.....	14
12.	NOVEL SYNTHETIC SOL-GEL CAO BASED PELLETS USING POROUS MESOSTRUCTURED SILICA IN CYCLIC CO <sub>2</sub> CAPTURE PROCESS .....	15
13.	REACTIVATION BY WATER HYDRATION OF THE CO <sub>2</sub> CAPTURE CAPACITY OF A CALCIUM LOOPING SORBENT.....	16
14.	AN INTERNALLY CIRCULATING FLUID BED FOR ATTRITION TESTING OF CA LOOPING SORBENTS.....	17
15.	EFFECT OF SULFATION ON CO <sub>2</sub> CAPTURE OF CAO-BASED SORBENTS DURING CALCIUM LOOPING CYCLE.....	18
16.	MULTICYCLIC CONVERSION OF LIMESTONE AT CA-LOOPING CONDITIONS: THE ROLE OF SOLID-SATE DIFFUSION CONTROLLED CARBONATION.....	19
17.	MODELING THE BEHAVIOR OF LIMESTONE PARTICLES IN OXY-FUEL CFB PROCESSES .....	20
18.	COLD MODEL STUDY OF A DUAL FLUIDIZED BED SYSTEM FOR THE GASIFICATION OF SOLID FUELS.....	21
19.	DEFLUIDIZATION OF A QUARTZ BED – LABORATORY EXPERIMENTS WITH POTASSIUM SALTS .....	22
20.	THE COMBUSTION OF A FUEL-RICH MIXTURE OF METHANE AND AIR IN A BUBBLING FLUIDISED BED OF SILICA SAND AT 700 °C AND ALSO WITH PARTICLES OF Fe <sub>2</sub> O <sub>3</sub> OR Fe PRESENT ....	23
21.	CHEMICAL-LOOPING COMBUSTION OF RAW SYNGAS FROM BIOMASS STEAM GASIFICATION – COUPLED OPERATION OF TWO DUAL FLUIDIZED BED PILOT PLANTS.....	24

22.	CO <sub>2</sub> -GASIFICATION OF A LIGNITE COAL IN THE PRESENCE OF AN IRON-BASED OXYGEN CARRIER FOR CHEMICAL-LOOPING COMBUSTION .....	25
23.	ENTROPY GENERATION ANALYSES OF ENDEX AND CONVENTIONAL CALCIUM LOOPING PROCESSES FOR CO <sub>2</sub> CAPTURE .....	26
24.	DIRECT CO <sub>2</sub> CAPTURE FROM AMBIENT AIR USING K <sub>2</sub> CO <sub>3</sub> /Y <sub>2</sub> O <sub>3</sub> COMPOSITE SORBENT .....	27
25.	PARTITIONING OF INORGANIC ELEMENTS IN PILOT-SCALE AND DEMONSTRATION-SCALE ENTRAINED-FLOW GASIFIERS .....	28

# 1. A SYSTEMATIC APPROACH FOR HIGH TEMPERATURE LOOPING CYCLES INTEGRATION

Yolanda Lara, Pilar Lisbona, Ana Martínez, Luis M. Romeo

*CIRCE (Research Centre for Energy Resources and Consumption), Universidad de Zaragoza, C/Mariano Esquillor 15, 50018 Zaragoza, Spain*

## ABSTRACT

High temperature solid cycles are promising technologies for implementing large-scale CO<sub>2</sub> capture facilities in the mid-term. Energy integration plays a major role in the development of these cycles. Waste energy from the capture process may be used to retrofit an existing power plant or to power a new steam cycle, thus diminishing the energy penalty that the process entails. A procedure based on pinch analysis for the integration of these systems, is proposed and tested using limestone as CO<sub>2</sub> sorbent. Although Li<sub>4</sub>SiO<sub>4</sub> has been already dismissed for this purpose, it is used to validate the systematic procedure under a very different problem in terms of available heat and initial temperatures of hot streams. In both scenarios the integration was designed to take advantage of all available heat of the process and to power a supercritical steam cycle, quantifying their minimum energy penalty. A sensitivity analysis for each process was also developed, and the heat exchanger network achieved by the proposed algorithm obtained the best results in costs terms.

© 2013 Elsevier Ltd. All rights reserved.

## 2. CARBONATE LOOPING EXPERIMENTS IN A 1 MWTH PILOT PLANT AND MODEL VALIDATION

Jochen Ströhle , Markus Junk, Johannes Kremer, Alexander Galloy, Bernd Epple

*Energy Systems and Technology, Technische Universität Darmstadt, Otto-Berndt-Str. 2, 64287 Darmstadt, Germany*

### ABSTRACT

Carbonate looping is an efficient post-combustion CO<sub>2</sub> capture technology using limestone based sorbents. A carbonate looping pilot plant consisting of two interconnected circulating fluidized bed (CFB) reactors with a thermal capacity of 1 MWth has been designed and erected at Technische Universität Darmstadt. The pilot plant has been operated for >1500 h in fluidized bed mode, thereof >400 h with CO<sub>2</sub> capture. The heat for the endothermic regeneration of CaO in the calciner was provided by combustion of either propane or pulverised coal with O<sub>2</sub> enriched air. High CO<sub>2</sub> absorption efficiencies of up to 85% in the carbonator were achieved for long periods. Taking the CO<sub>2</sub> produced by oxyfuel-combustion in the calciner into account, the pilot plant was operated with total CO<sub>2</sub> capture rates above 90%. A process model for the carbonate looping pilot plant has been developed with ASPEN PLUS™. A 1D CFB model has been implemented in the process model to determine the effect of hydrodynamics within a fast fluidized bed on the CO<sub>2</sub> absorption rate in the carbonator. Operating conditions of a selected test campaign were used as boundary conditions. The results of process simulations show good agreement of calculated CO<sub>2</sub> Absorption rate with experimental data. Hence, this process model can be considered as a reliable tool for scale-up of the process.

© 2013 Elsevier Ltd. All rights reserved.

### 3. DEVELOPMENT OF THE CALCIUM LOOPING CO<sub>2</sub> CAPTURE TECHNOLOGY FROM LAB TO PILOT SCALE AT IFK, UNIVERSITY OF STUTTGART

Heiko Dieter, Ajay R. Bidwe, Glykeria Varela- Duelli, Alexander Charitos,

Craig Hawthorne, Günter Scheffknecht

*Institute of Combustion and Power Plant Technology (IFK), University of Stuttgart, Pfaffenwaldring 23, 70569 Stuttgart, Germany*

#### ABSTRACT

Calcium Looping CO<sub>2</sub> capture technology has made vast progress within the last 10 years and has become a viable option for efficient CO<sub>2</sub> capture from power plant flue gases. Along with Prof. Ben Anthony many groups have contributed to the success story of Calcium Looping within Europe and worldwide. Development of Calcium Looping was initiated in the year 1999 with the process idea, and has continuously proceeded, starting from lab investigations at sorbent level, feasibility studies including process efficiency and economic analyses, process demonstration in the first electrically heated test rigs and finally making the step into pilot scale. Inspired by the sorption-enhanced reforming (SER) of biomass using limestone for in situ CO<sub>2</sub> capture, the first work on Calcium Looping done at the Institute of Combustion and Power Plant Technology, IFK of the University of Stuttgart was in 2003. From that point IFK has been one of the groups pushing Calcium Looping from basic research towards successful process demonstration at pilot scale. This paper will give an overview over the research work carried out at IFK in the years 2003–2013 and shows the major steps of development and achievements of different research periods.

© 2014 Elsevier Ltd. All rights reserved.

## 4. COAL COMBUSTION UNDER CALCIUM LOOPING PROCESS CONDITIONS

Caiyun Gao<sup>a</sup>, Takayuki Takahashi<sup>a</sup>, Hiroko Narisawa<sup>a</sup>, Ayato Yoshizawa<sup>b</sup>, Tadaaki

Shimizu<sup>b</sup>, Heejoon Kim<sup>b</sup>, Liuyun Li<sup>b</sup>

*a: Graduate School of Science and Technology, Niigata University, Japan*

*b: Department of Chemistry and Chemical Engineering, Niigata University, Japan*

### ABSTRACT

Coal of three kinds was burned in an oxygen-enriched atmosphere using a twin-fluidized bed solid circulation system under conditions of the Calcium Looping Process. This twin-fluidized bed system comprised a fast bed regenerator (calciner), into which fuel and oxygen-enriched gas were fed, and a bubbling bed absorber (carbonator), into which air was fed. Inert quartz sand was used as the bed material to evaluate the coal combustion behavior, including char transportation from regenerator to absorber and formation of CO and CO<sub>2</sub> there. First, the circulation rate and the residence time of solids in the regenerator (calciner) were measured to determine the suitable operation conditions. The effect of gas feed staging to the regenerator on the solid residence time was evaluated. By reducing the ratio of the primary gas feed rate to total gas feed rate to 0.5, average solid residence time of about 40 s was attained. Under this gas-feed condition, coal combustion experiments were conducted. Effects of volatile matter content of coal on CO and CO<sub>2</sub> formation in the absorber and NO<sub>x</sub> emissions from the regenerator were investigated. High-volatile matter coal was found to be favorable to reduce CO and CO<sub>2</sub> formation in the absorber, but conversion of the fuel-N to NO<sub>x</sub> of high-volatile matter coal was higher than that from low-volatile coal.

© 2013 Elsevier Ltd. All rights reserved.

## 5. COAL COMBUSTION CHARACTERISTICS ON AN OXY-FUEL CIRCULATING FLUIDIZED BED COMBUSTOR WITH WARM FLUE GAS RECYCLE

Lunbo Duan, Haicheng Sun, Changsui Zhao, Wu Zhou, Xiaoping Chen

*Key Laboratory of Energy Thermal Conversion and Control of Ministry of Education, School of Energy and Environment, Southeast University, Nanjing 210096, China*

### ABSTRACT

More than 100-h steady warm flue gas recycle operation was carried out on a 50 kWth oxy-fuel circulating fluidized bed (CFB) combustor burning three kinds of fuel. The results demonstrate the good safety benefit of oxy-CFB operation, especially in the oxy-transition process. A slightly higher oxygen concentration, ranging from 22.2% to 23.4% for different fuels in oxy-fuel operation, can bring equivalent or higher carbon burnout than air combustion. SO<sub>2</sub> concentration in ppm unit is higher in flue gas while the SO<sub>2</sub> emission in mg/MJ unit is lower than air combustion. The desulfurization efficiency of limestone can reach 80% in oxy-fuel combustion in this test. The higher Ca utilization rate burning coal in oxy-fuel combustion than that in air combustion may be associated with the high moisture content in the flue gas. Fuel nitrogen conversion ratio in oxy-fuel is much lower than in air combustion, and it looks like higher volatile content in fuel leads to a bigger reduction of NO in the recycle flue gas.

© 2013 Elsevier Ltd. All rights reserved.

## 6. UNDESIREE EFFECTS IN THE DETERMINATION OF CO<sub>2</sub> CARRYING CAPACITIES OF CAO DURING TG TESTING

M. Alonso<sup>a</sup>, Y.A. Criado<sup>a</sup>, J.C. Abanades<sup>a</sup>, G. Grasa<sup>b</sup>

*a: Instituto Nacional del Carbón, INCAR-CSIC, C/Francisco Pintado Fe, 26, 33011 Oviedo, Spain*

*b: Instituto de Carboquímica, ICB-CSIC, C/Miguel Luesma Castán, 4, 50018 Zaragoza, Spain*

### ABSTRACT

Calcium looping CO<sub>2</sub> capture systems use CaO as a reversible sorbent of CO<sub>2</sub>. Therefore, the evolution of the CO<sub>2</sub> carrying capacity of CaO-materials at increasing number of carbonation–calcination needs to be determined to assess sorbent performance. Thermogravimetric analyzers (TGA) are commonly used for this purpose, by simulating around a small batch of material the average cyclic conditions expected in the real system. Many variables have been reported to influence the results and we review in this paper the main observations and trends, which can at times be conflicting when diffusional effects are not ruled out from the experiments. Furthermore, in a selected number of tests on a typical limestone using four different TG equipment, we have detected that some design characteristics of the TGA apparatus can strongly affect the determination of the CO<sub>2</sub> carrying capacities of the material. In particular, we note that the decay in CO<sub>2</sub> carrying capacity is accelerated as the power density of the TGA oven increases. This effect is most pronounced in the first calcination cycle, and it seems to be linked to an additional shrinking of the particles taking place in the TG apparatus with the highest heating rates. The use of larger sample masses and/or larger particle sizes tends to reduce the error in the determination of CO<sub>2</sub> carrying capacity curves at the expense of departing from differential conditions that are required to obtain kinetic information on the sample.

© 2013 Elsevier Ltd. All rights reserved.

## 7. DEVELOPMENT OF A NOVEL-SYNTHESIZED CA-BASED CO<sub>2</sub> SORBENT FOR MULTICYCLE OPERATION: PARAMETRIC STUDY OF SORPTION

Sofia D. Angeli, Christina S. Martavaltzi, Angeliki A. Lemonidou

*Department of Chemical Engineering, Aristotle University of Thessaloniki, University Campus, GR-54124 Thessaloniki, Greece*

### ABSTRACT

One of the most promising technologies for CO<sub>2</sub> capture is the Calcium Looping Cycle, which is based on the reversible carbonation reaction of CaO. The main challenge for CaL technology is the deterioration of CO<sub>2</sub> capture capacity during multicyclic operation. In this work, the triethanolamine (TEA) was used as a complexing agent in a modified sol–gel method for the preparation of sintering resistant CaO–Ca<sub>3</sub>Al<sub>2</sub>O<sub>6</sub>. The sorbent showed high capture capacity (0.45 g/g sorb. or 84% carbonation conversion) which was retained after 45 cycles. Parametric study of sorption was realized by varying the carbonation and calcination conditions (temperature and CO<sub>2</sub> concentration). Under severe conditions of calcination temperature at 950 °C in pure CO<sub>2</sub> flow after 100 cycles the sorbent still retained almost 40% of the initial sorption capacity corresponding to 30% carbonation conversion.

© 2013 Elsevier Ltd. All rights reserved.

## 8. INVESTIGATION OF THE FRIABILITY OF CA LOOPING SORBENTS DURING AND AFTER HYDRATION BASED REACTIVATION

Vlatko Materic<sup>a</sup>, Margaret Hyland<sup>b</sup>, Mark Ian Jones<sup>b</sup>, Robert Holt<sup>a</sup>

*a: Callaghan Innovation, 69 Gracefield Road, 5040 Lower Hutt, New Zealand*

*b: University of Auckland, 20 Symonds Street, 1142 Auckland, New Zealand*

### ABSTRACT

The progressive decay in the CO<sub>2</sub> carrying capacity of limestone is one of the major limitations of the CaCO<sub>3</sub>–CaO thermochemical cycle (Ca looping) which forms the basis of a number of emerging energy conversion technologies. A reactivation method consisting of hydrating and dehydrating the spent lime was reported to restore its CO<sub>2</sub> carrying capacity but also to considerably increase sorbent friability thus limiting the applicability of this method in an industrial setting. However there is little experimental information about the mechanism of this friability increase. This work aimed at collecting experimental data on the evolution of sorbent friability and the dominant attrition mechanisms during and after reactivation. The dehydration step was identified as the point at which the weakness of the sorbent was first detected. This weakness manifested as an increased tendency for fragmentation by chipping of the dehydrated sorbent and all sorbents deriving from it. A hypothesis was postulated to explain the results.

© 2013 Elsevier Ltd. All rights reserved.

## 9. KINETICS OF CO<sub>2</sub> ABSORPTION BY CaO THROUGH PRESSURE SWING CYCLING

James W. Butler, C. Jim Lim, John R. Grace

*Department of Chemical and Biological Engineering, University of British Columbia, Vancouver V6T 1Z3, Canada*

### ABSTRACT

CO<sub>2</sub> capture by means of CaO cycling represents a cost effective, immediate solution to rising CO<sub>2</sub> emissions. The mechanism of loss of utilization efficiency and associated change in carbonation kinetics of CaO particles was examined by conducting calcination/carbonation cycling of 150–250  $\mu$ m Strasburg limestone precursor, by swinging the pressure from atmospheric (for calcination) to 5, 10 and 20 barg (for carbonation) at constant temperatures of 975–1025°C and a flow of pure CO<sub>2</sub>. Increased carbonation pressure led to an increase in utilization over 100 cycles from  $0.128 \pm 0.005$  to  $0.271 \pm 0.035$  for 5 and 20 barg respectively. Samples were examined by scanning electron microscopy and BET. The carbonation kinetics were determined by plotting the rates of reaction for both the kinetically controlled and diffusion controlled carbonation stages. Sintering, grain boundary formation and elimination, and changes in particle surface area are found to all play important roles in causing the initial rapid loss in surface reaction-controlled rate, followed by partial recovery over multiple cycles. The loss of grain boundaries due to sintering caused an increase in the reaction-controlled rate and residual calcium utilization due to a shift in the dynamics of carbonation/calcination nucleation and molecular volume contraction. The rate of the reaction-controlled regime was found to be a function of carbonation pressure. The diffusion-controlled rate is independent of pressure and temperature.

© 2013 Elsevier Ltd. All rights reserved.

## 10. UNDERSTANDING THE ENHANCEMENT EFFECT OF HIGH-TEMPERATURE STEAM ON THE CARBONATION REACTION OF CAO WITH CO<sub>2</sub>

Zhenshan Li<sup>a</sup>, Yang Liu<sup>b</sup>, Ningsheng Cai<sup>a</sup>

*a Key Laboratory for Thermal Science and Power Engineering of Ministry of Education, Department of Thermal Engineering, Tsinghua University, Beijing 100084, China*

*b School of Energy Power and Mechanical Engineering, North China Electric Power University, Beijing 102206, China*

### ABSTRACT

The steam-enhanced carbonation reaction of CaO with CO<sub>2</sub> is a widely observed phenomenon, but its mechanism is unclear. This study proposed that OH<sup>-</sup> formation caused by the dissociation of H<sub>2</sub>O molecules may explain enhanced carbonation. The relationship of oxygen vacancies with OH<sup>-</sup> at different temperatures and steam fractions was established and integrated into a new carbonation model. This new model included a simplified rate equation model to describe product island formation and a multi-ion diffusion model that describes product layer growth. Experiments on carbonation and sorption-enhanced water gas shift reactions in a laboratory-scale fluidized bed reactor were conducted to validate the idea of OH<sup>-</sup> formation. The new developed models were validated by thermogravimetric analysis.

© 2013 Elsevier Ltd. All rights reserved.

## 11. SOL-GEL-DERIVED, CAO-BASED, ZRO2-STABILIZED CO2 SORBENTS

Marcin Broda, Christoph R. Müller

*Laboratory of Energy Science and Engineering, ETH Zurich, Leonhardstrasse 27, 8092 Zurich, Switzerland*

### ABSTRACT

We report the synthesis of CaO-based, ZrO<sub>2</sub>-stabilized CO<sub>2</sub> sorbents using a sol-gel technique. In addition, the influence of different synthesis parameters, i.e. the calcium precursor, the condensation time and the ratio of Ca<sup>2+</sup> to Zr<sup>4+</sup> on the morphology and the CO<sub>2</sub> uptake of the materials was evaluated. The morphology and the chemical composition of the materials were characterized using X-ray diffraction, N<sub>2</sub> physisorption and scanning electron microscopy whereas the CO<sub>2</sub> uptake of the materials was determined using a thermogravimetric analyzer and a packed bed reactor. After 90 cycles of the repeated calcination and carbonation reactions, the CO<sub>2</sub> uptake of the best sorbent was 0.34 g CO<sub>2</sub>/g sorbent exceeding the performance of the reference limestone by 160%. The favorable CO<sub>2</sub> capture characteristics of the synthetic CO<sub>2</sub> sorbent were attributed to the formation of a finely dispersed, high Tammann temperature calcium zirconate framework, which minimized thermal sintering.

© 2013 Elsevier Ltd. All rights reserved.

## 12. NOVEL SYNTHETIC SOL–GEL CAO BASED PELLETS USING POROUS MESOSTRUCTURED SILICA IN CYCLIC CO<sub>2</sub> CAPTURE PROCESS

Mohammad Hashem Sedghkerdar<sup>a</sup>, Nader Mahinpey<sup>a</sup>, Zhenkun Sun<sup>b</sup>, Serge Kaliaguine<sup>b</sup>

*a: Department of Chemical and Petroleum Engineering, University of Calgary, Calgary T2N 1N4, Canada*

*b: Department of Chemical Engineering, Université Laval, Ste-Foy, Qué. G1V 0A6, Canada*

### ABSTRACT

In this study, four CO<sub>2</sub> carriers were examined through 31 carbonation–calcination cycles. The carriers included one natural limestone (Cadomin) and three modified calcium oxide-based sorbents, Cadomin–calcium aluminate cement pellets (CD–CA-14), Cadomin–silica-sol pellets (CD–Si), and Cadominmesostructured silica core/shell pellets (CD–CS). The modified sorbents were not only prepared using two different binders (calcium aluminate cement, CA-14, and silica-sol, HS-40), but the CD–CS sample was provided with a protective porous shell (using a mesoporous silica layer). Natural Cadomin limestone was the base common to the modified sorbents. The surface morphology and porosity of these sorbents before and after different cycles were investigated by scanning electron microscopy (SEM) and nitrogen physisorption (BET/BJH). X-ray diffraction (XRD) was also used to identify the crystal phase composition of the sorbents before and after calcination. The results showed that the presence of the Mayenite phase (Ca<sub>12</sub>Al<sub>14</sub>O<sub>33</sub>) in the CD–CA-14 pellets led to slower decay in sorbent activity with increased number of cycles. Although the CD–CS pellets showed the best performance in the retention of CO<sub>2</sub> uptake activity over 31 cycles, with an activity loss of only 46%, the pellet shells experienced severe disintegration during the first few cycles.

© 2013 Elsevier Ltd. All rights reserved.

### 13. REACTIVATION BY WATER HYDRATION OF THE CO<sub>2</sub> CAPTURE CAPACITY OF A CALCIUM LOOPING SORBENT

Antonio Coppola<sup>a,b</sup>, Piero Salatino<sup>a</sup>, Fabio Montagnaro<sup>c</sup>, Fabrizio Scala<sup>b</sup>

*a: Dipartimento di Ingegneria Chimica, dei Materiali e della Produzione Industriale, Università degli Studi di Napoli Federico II, Piazzale Vincenzo Tecchio 80, 80125 Napoli, Italy*

*b: Istituto di Ricerche sulla Combustione, Consiglio Nazionale delle Ricerche, Piazzale Vincenzo Tecchio 80, 80125 Napoli, Italy*

*c: Dipartimento di Scienze Chimiche, Università degli Studi di Napoli Federico II, Complesso Universitario di Monte Sant'Angelo, 80126 Napoli, Italy*

#### ABSTRACT

The potential of water hydration as a mean to reactivate a spent reactive limestone-based sorbent used in a fluidized bed calcium looping process for CO<sub>2</sub> capture is demonstrated. The study has been specifically targeted at investigating the changes of sorbent properties induced by hydration, and the influence that this treatment had on the CO<sub>2</sub> capture capacity and on the attrition tendency of the reactivated material. To this end, the spent sorbent obtained from calcium looping tests in a lab-scale fluidized bed reactor (calcination at 940 °C in 70% CO<sub>2</sub>, carbonation at 650 °C in 15% CO<sub>2</sub>) was hydrated at 25 °C for times ranging from 10 to 60 min, and characterized by TG, SEM and porosimetric analyses. The hydrated materials were further subjected to calcium looping cycles under the same operating conditions, and the CO<sub>2</sub> capture capacities, the elutriation rates and the particle size distributions after each stage were measured and compared with those obtained in the tests before reactivation. It is demonstrated that hydration is effective in reactivating the spent sorbent: the CO<sub>2</sub> capture capacity increased from 0.04 g g<sup>-1</sup> (last carbonation before hydration) to 0.32–0.37 g g<sup>-1</sup> (first carbonation after hydration). The capacity, however, rapidly decayed along with the cycles. Results suggested that the CO<sub>2</sub> capture capacity, the sorbent properties and the attrition tendency are linked one to the other. The sorbent hydrated for the longest time (60 min) not only developed an enhanced active porosity, but was also characterized by a limited attrition tendency (with respect to the material hydrated for 10 min). Notably, results obtained in the present study differed from those obtained in a previous study on a different limestone, highlighting the importance of sorbent reactivity for an optimally tailored sorbent regeneration process.

© 2013 Elsevier Ltd. All rights reserved.

Graphical representation

## 14. AN INTERNALLY CIRCULATING FLUID BED FOR ATTRITION TESTING OF CA LOOPING SORBENTS

Vlatko Materic<sup>a</sup>, Robert Holt<sup>a</sup>, Margaret Hyland<sup>b</sup>, Mark I. Jones<sup>b</sup>

*a: Callaghan Innovation, 69 Gracefield Road, 5040 Lower Hutt, New Zealand*

*b: University of Auckland, 20 Symonds Street, 1142 Auckland, New Zealand*

### ABSTRACT

Particle breakage, i.e. attrition, inevitably occurs in most processes involving the transport of solid particles, such as Ca looping processes. Attrition can have nefarious effects on the operation of such systems as it affects the hydrodynamics of the system and increases the need for gas clean-up. Therefore characterizing the particles' tendency to break, i.e. their friability, is essential for the design and development of Ca looping processes. Although a number of friability testing methods exist and have been used to produce satisfying results, it is argued here that existing methods cannot easily provide a full characterization of the friability of Ca looping sorbents. A new friability testing method is proposed in this work to overcome this difficulty, based on the use of an internally circulating fluid bed (ICFB). A prototype apparatus was built and the feasibility of this method evaluated.

© 2013 Elsevier Ltd. All rights reserved.

## 15. EFFECT OF SULFATION ON CO<sub>2</sub> CAPTURE OF CAO-BASED SORBENTS DURING CALCIUM LOOPING CYCLE

Cong Luo<sup>a,c</sup>, Ying Zheng<sup>a</sup>, Jia Guo<sup>c</sup>, Bo Feng<sup>b</sup>

*a: State Key Laboratory of Coal Combustion, Huazhong University of Science and Technology, Hubei 430074, China*

*b: School of Mechanical and Mining Engineering, The University of Queensland, St. Lucia, Queensland 4072, Australia*

*c :Key Laboratory for Green Chemical Process of Ministry of Education, Wuhan Institute of Technology, Hubei 430073, China*

### ABSTRACT

Calcium looping cycle is considered as one of the most promising post combustion CO<sub>2</sub> capture technologies that can curb CO<sub>2</sub> emissions from power plants fired with fossil fuel. However the presence of SO<sub>2</sub> and steam in the flue gas leads to the sulfation of CaO-based sorbents, and thus reduces the sorbents' capacity for CO<sub>2</sub> capture. In this study, the effect of sorbent sulfation during both carbonation and calcination stage on the cyclic CO<sub>2</sub> capture performance was investigated. The results showed that the reaction time, SO<sub>2</sub> concentrations and steam greatly influenced the capacity of CaO-based sorbent. The sulfation degree of the sorbent decreased with the reduction of carbonation and calcination time per cycle. High SO<sub>2</sub> concentration present with steam led to a rapid loss in capacity of the sorbent within a few cycles. Furthermore, the synthetic sorbent derived from a sol-gel process had much higher cyclic CO<sub>2</sub> capture capacities than natural limestone sorbent whether in conditions without SO<sub>2</sub> or with SO<sub>2</sub> and steam. Microscopic images showed that after multiple cycles in the presence of SO<sub>2</sub> and steam, the porous structure of the synthetic sorbent could be retained while limestone experienced serious pore blockage and agglomeration of grains.

© 2013 Elsevier Ltd. All rights reserved.

## 16. MULTICYCLIC CONVERSION OF LIMESTONE AT CA-LOOPING CONDITIONS: THE ROLE OF SOLID-STATE DIFFUSION CONTROLLED CARBONATION

Pedro E. Sanchez-Jimenez<sup>a</sup>, Jose M. Valverde<sup>b</sup>, Luis A. Perez-Maqueda<sup>a</sup>

*a: Instituto de Ciencia de Materiales de Sevilla (C.S.I.C.- Univ. Sevilla), Americo Vespucio 49, 41092 Sevilla, Spain*

*b: Faculty of Physics, University of Seville, Avenida Reina Mercedes s/n, 41012 Sevilla, Spain*

### ABSTRACT

Limestone derived CaO conversion when subjected to multiple carbonation/calcination cycles is a subject of interest currently fueled by several industrial applications of the so-called Ca-looping (CaL) technology. The multicyclic CaO conversion at Ca-looping conditions exhibits two main features as demonstrated by thermogravimetric analysis (TGA). On one hand, carbonation occurs by two well differentiated phases: a first kinetically-driven fast phase and a subsequent much slower solid-state diffusion controlled phase. On the other, carbonation in the fast phase usually shows a drastic decay with the cycle number along the first carbonation/calcination cycles. This trend can be reversed by means of heat pretreatment, which induces a marked loss of fast conversion in the first carbonation but enhances diffusion of CO<sub>2</sub> in the solid. Upon decarbonation the regenerated CaO skeleton displays an increased conversion in the fast carbonation phase of the next cycle, a phenomenon which has been referred to as reactivation. Nonetheless, sorbent reactivation is hampered by looping carbonation/calcination conditions as those to be likely found in practice such as carbonation stages characterized by low CO<sub>2</sub> concentrations and short duration and calcination stages at high temperatures in a CO<sub>2</sub> enriched atmosphere, which causes a sintering and loss of activity of the regenerated CaO skeleton. We analyze in this work sorbent reactivation as affected by heat pretreatment and carbonation/calcination conditions. Aimed at shedding light on the role played by these conditions on reactivation we look separately at the multicyclic evolution of conversion in the kinetic and diffusive phases. Generally, the evolution of multicyclic conversion after the first cycle can be described by a balance between the surface area gain due to diffusive carbonation and the surface area loss as caused by sintering in the calcination stage. A significant gain of relative surface area after the first cycle, which is favored by harshening the heat pretreatment conditions, leads however to a marked decay of it during subsequent cycles, which precludes reactivation for an extended interval of cycles. On the other hand, sorbent grinding, if performed before heat pretreatment, leads to a less marked but more sustainable reactivation along the cycles. A novel observation reported in our work is that pretreatment of limestone in a CO<sub>2</sub> atmosphere leads upon a subsequent quick decarbonation to a CaO skeleton with extraordinarily enhanced reactivity in the kinetically-driven carbonation phase and with a high resistance to solid-state diffusion, which can be attributed to annealing of the crystal structure as reported by independent studies.

© 2013 Elsevier Ltd. All rights reserved.

## 17. MODELING THE BEHAVIOR OF LIMESTONE PARTICLES IN OXY-FUEL CFB PROCESSES

Sirpa Rahiala, Kari Myöhänen, Timo Hyppänen

*Lappeenranta University of Technology, P.O. Box 20, FI-53851 Lappeenranta, Finland*

### ABSTRACT

The limestone behavior in an oxy-fuel CFB process was studied by means of modeling. Different modelling methods were combined: the calculation of particle trajectories in a three-dimensional steady state CFB furnace model was utilized to investigate the real process environment which the particles experience and a particle model was used to study the effects of the transient environment on limestone reactions. The studied cases included the air-fired and oxygen-fired combustion and conditions on both sides of the equilibrium curve for calcination, which enabled both indirect and direct sulfation mechanisms. Also the effect of the return leg system was studied; it was shown that depending on the conditions in the return leg, a complex behavior consisting of simultaneous reactions (calcination–carbonation–sulfation–direct sulfation) is conceivable. Moreover, the modeling approach was used to evaluate whether the transient environment has an effect on the limestone behavior compared to the averaged conditions and in which conditions the effect is the largest. The difference between the averaged and transient conditions was notable only in the conditions which were close to the equilibrium curve.

© 2013 Elsevier Ltd. All rights reserved.

## 18. COLD MODEL STUDY OF A DUAL FLUIDIZED BED SYSTEM FOR THE GASIFICATION OF SOLID FUELS

Ajay R. Bidwe, Craig Hawthorne, Yu Xizhi, Heiko Dieter, Günter Scheffknecht

*Institute of Combustion and Power Plant Technology (IFK), University of Stuttgart, Pfaffenwaldring 23, 70569 Stuttgart, Germany*

### ABSTRACT

Sorption-enhanced reforming (SER) is a process that couples steam gasification with carbonation/calcination reaction of limestone to enhance the hydrogen concentration in a product gas. Inspired from lab scale results a 200 kWth pilot plant is constructed at IFK, University of Stuttgart. This pilot plant is a dual fluidized bed (DFB) system comprised of a bubbling fluidized bed (BFB) gasifier and a circulating fluidized bed (CFB) regenerator. These two fluidized beds are coupled with an L valve and a lower loop seal located at the bottom of the gasifier. This paper delineates the cold model studies of this pilot plant carried out during the design phase. The cold model is based on the Glicksman's simplified scaling ratios, which

determine the particle properties and operating conditions of the cold model. Results of the cold model can be extrapolated to the pilot plant using the scaling ratios. The results validate long term stable operation is possible with the planned DFB system. The solid circulation patterns in the BFB are studied and results show that the proposed BFB design is suitable for the gasifier. The parameters such as gasifier inventory, riser velocity and riser entrainment rates are tested under scaled conditions to prove the feasibility of the pilot plant. The results show that the required design criteria of the 200 kWth DFB pilot plant are fulfilled and the proposed DFB pilot plant is feasible.

© 2013 Elsevier Ltd. All rights reserved

## 19. DEFLUIDIZATION OF A QUARTZ BED – LABORATORY EXPERIMENTS WITH POTASSIUM SALTS

Christoffer Sevonius, Patrik Yrjas, Mikko Hupa

*Åbo Akademi University, Process Chemistry Centre (PCC), Piispankatu 8, FI-20500 Turku, Finland*

### ABSTRACT

As the need for CO<sub>2</sub> neutral and renewable fuels increase, the use of biomass in combustion for heat and power becomes more and more important. Biomass, however, has a tendency to cause problems for the boilers such as fouling and corrosion of super heaters as well as agglomeration of the bed material in the case of fluidized bed boilers. The problems arise because of the reactive alkali in biomass. Previous agglomeration studies have been done with laboratory scale fluidized beds but usually by burning biomass or by using biomass ash. This study focused on the separate alkali components in biomass to get a better understanding of the agglomeration mechanisms. Pure salts were used which in this case were potassium chloride, potassium carbonate and potassium sulfate. An electrically heated laboratory scale fluidized bed reactor was used for the defluidization experiments and quartz sand was used as bed material. The bed was heated to the set temperature and salt was added until defluidization occurred. The experiments were done in a temperature range of 750–900 °C under both dry and wet conditions. The pressure drop and the bed temperatures were used to detect the onset of agglomeration and defluidization. The bed was then analyzed with SEM/EDX and ion chromatography to determine the bed composition. It was found that potassium chloride did not react with the quartz bed but in fact only acted as glue between the sand particles. Potassium carbonate on the other hand reacted with the bed particles forming potassium silicates. Potassium sulfate did not cause defluidization as was expected.

© 2013 Elsevier Ltd. All rights reserved.

## 20. THE COMBUSTION OF A FUEL-RICH MIXTURE OF METHANE AND AIR IN A BUBBLING FLUIDISED BED OF SILICA SAND AT 700 C AND ALSO WITH PARTICLES OF Fe<sub>2</sub>O<sub>3</sub> OR Fe PRESENT

D.R. Chadeesingh, A.N. Hayhurst

*Department of Chemical Engineering and Biotechnology, University of Cambridge, Pembroke Street, Cambridge CB2 3RA, UK*

### ABSTRACT

A cylindrical bed of quartz sand was electrically heated and held at 700 °C, whilst being fluidised by a slightly fuel-rich mixture of methane and air. The temperature was measured along the bed's axis, both in and above the sand. Likewise, gas was sampled through a special probe, which, when immersed in the bed, sampled only gas flowing interstitially between particles of sand, and not gas in bubbles. It was confirmed that nothing much occurs inside the bed, but above the bed, CO, CO<sub>2</sub> and C<sub>2</sub>H<sub>4</sub> were detected (and also CH<sub>4</sub> disappeared), where large gaseous bubbles were separating (i.e. disengaging) from sand ejected up into the freeboard. Hence quartz sand inhibits combustion in this case, since O<sub>2</sub> hardly reacted with CH<sub>4</sub> within the bed. When a small quantity (<0.1 wt.%) of particles of Fe<sub>2</sub>O<sub>3</sub> was added to the sand, CH<sub>4</sub> was oxidised in the bed by the Fe<sub>2</sub>O<sub>3</sub>, which in turn was reduced to Fe<sub>3</sub>O<sub>4</sub>. The O<sub>2</sub> in the gas fluidising the bed then oxidised the Fe<sub>3</sub>O<sub>4</sub> quickly back to Fe<sub>2</sub>O<sub>3</sub>; that O<sub>2</sub> reacts extremely rapidly with Fe<sub>3</sub>O<sub>4</sub> created a steady state concentration for Fe<sub>3</sub>O<sub>4</sub> on the particles, which initially were entirely Fe<sub>2</sub>O<sub>3</sub>. One outcome is that the overall rate of oxidation of CH<sub>4</sub> in the bed is non-linear in the mass of Fe<sub>2</sub>O<sub>3</sub> originally added to the bed. After a certain amount of Fe<sub>2</sub>O<sub>3</sub> had been added, combustion above the fluidised sand ceased, although some C<sub>2</sub>H<sub>4</sub> was still produced there. Interestingly, this fluidised bed of sand, in fact, with either Fe<sub>2</sub>O<sub>3</sub> or metallic Fe added, achieved the chemical-looping combustion of CH<sub>4</sub> without any need for two separate reactors. It appeared that Fe<sub>2</sub>O<sub>3</sub> is a more reactive oxidising agent of CH<sub>4</sub> than is Fe<sub>3</sub>O<sub>4</sub> or FeO. Likewise, Fe<sub>2</sub>O<sub>3</sub> oxidises CO and H<sub>2</sub> faster than it oxidises CH<sub>4</sub>. The addition of particles of metallic iron to this bed, fluidised by CH<sub>4</sub> and O<sub>2</sub> at 700 °C, resulted in the rapid production of Fe<sub>2</sub>O<sub>3</sub>, so in order to promote the combustion of CH<sub>4</sub> it is unimportant in which form the iron is added to the sand.

© 2013 Elsevier Ltd. All rights reserved.

## 21. CHEMICAL-LOOPING COMBUSTION OF RAW SYNGAS FROM BIOMASS STEAM GASIFICATION – COUPLED OPERATION OF TWO DUAL FLUIDIZED BED PILOT PLANTS

Stefan Penthor<sup>a</sup>, Karl Mayer<sup>a</sup>, Stefan Kern<sup>a</sup>, Hannes Kitzler<sup>a</sup>, David Wöss<sup>b</sup>, Tobias Pröll<sup>b</sup>, Hermann Hofbauer<sup>a</sup>

*a Vienna University of Technology, Institute of Chemical Engineering, Getreidemarkt 9/166, 1060 Vienna, Austria*

*b University of Natural Resources and Life Sciences, Institute for Chemical and Energy Engineering, Peter-Jordan-Strasse 82, 1190 Vienna, Austria*

### ABSTRACT

Product gas from a 100 kW fuel power dual fluidized bed (DFB) steam gasifier for solid biomass was used as fuel for chemical looping combustion in a continuously operated dual circulating fluidized bed (DCFB) 120 kW chemical looping combustion (CLC) pilot plant. The two pilot units were coupled through a hot product gas fan. Olivine was used as bed material in the gasifier and a synthetic oxygen carrier containing 9 wt% CuO was used as oxygen carrier in the CLC unit. Standard wood pellets with a water content of 6 wt% were used as primary fuel. In the gasifier, conditions were kept constant at 850 °C and a steam/fuel ratio of 1.6 kg/kg dry biomass. Two operating conditions were employed at 850 °C and 900 °C in the fuel

reactor of the CLC unit. The composition was monitored with respect to CO, CO<sub>2</sub>, H<sub>2</sub>, and CH<sub>4</sub> for product gas, fuel reactor exhaust gas and air reactor exhaust gas. Also, H<sub>2</sub>S and SO<sub>2</sub> were measured in the product gas and CLC off gas streams. A product gas with 41.9 vol% (db) H<sub>2</sub>, 31.2 vol% (db) CO, 17 vol% (db) CO<sub>2</sub>, and 7.8 vol% (db) CH<sub>4</sub> was obtained from the gasifier. While CH<sub>4</sub> conversion in CLC was between 53% and 64%, almost full conversion of the CO and H<sub>2</sub> contained in the product gas were achieved. The effect of temperature was significant only with respect to CH<sub>4</sub> conversion. Analysis of particulate matter in product gas and CLC off gas streams indicate that fines contained in the product gas tend to pass through the CLC unit. No performance drop was evident during the experiment, which lasted for 10 h. It can be concluded that it is possible to couple two DFB systems in a robust way when using a high temperature fan and that biomass gasifier product gas can be effectively converted to CO<sub>2</sub> and H<sub>2</sub>O in CLC using copper-based oxygen carriers.

© 2014 Elsevier Ltd. All rights reserved.

## 22. CO<sub>2</sub>-GASIFICATION OF A LIGNITE COAL IN THE PRESENCE OF AN IRON-BASED OXYGEN CARRIER FOR CHEMICAL-LOOPING COMBUSTION

Marco A. Saucedo<sup>a</sup>, Jin Yang, Lim<sup>a</sup>, John S. Dennis<sup>a</sup>, Stuart A. Scott<sup>b</sup>

*a: University of Cambridge, Department of Chemical Engineering and Biotechnology, New Museums Site, Pembroke Street, Cambridge CB2 3RA, United Kingdom*

*b: University of Cambridge, Department of Engineering, Trumpington Street, Cambridge CB2 1PZ, United Kingdom*

### ABSTRACT

Chemical-looping combustion (CLC) has the inherent property of separating the product CO<sub>2</sub> from flue gases. Instead of air, it uses an oxygen carrier, usually in the form of a metal oxide, to provide oxygen for combustion. All techniques so far proposed for chemical looping with solid fuels involve initially the gasification of the solid fuel in order for the gaseous products to react with the oxygen carrier. Here, the rates of gasification of coal were compared when gasification was undertaken in a fluidised bed of either (i) an active Fe-based oxygen carrier used for chemical looping or (ii) inert sand. This enabled an examination of the ability of chemical looping materials to enhance the rate of gasification of solid fuels. Batch gasification and chemical-looping combustion experiments with a German lignite and its char are reported, using an electrically-heated fluidised bed reactor at temperatures from 1073 to 1223 K. The fluidising gas was CO<sub>2</sub> in nitrogen. The kinetics of the gasification were found to be significantly faster in the presence of the oxygen carrier, especially at temperatures above 1123 K. A numerical model was developed to account for external and internal mass transfer and for the effect of the looping agent. The model also included the effects of the evolution of the pore structure at different conversions. The presence of Fe<sub>2</sub>O<sub>3</sub> led to an increase in the rate of gasification because of the rapid oxidation of CO by the oxygen carrier to CO<sub>2</sub>. This resulted in the removal of CO and maintained a higher mole fraction of CO<sub>2</sub> in the mixture of gas around the particle of char, i.e. within the mass transfer boundary layer surrounding the particle. This effect was most prominent at about 20% conversion when (i) the surface area for reaction was at its maximum and (ii) because of the accompanying increase in porosity and pore size, intraparticle resistance to gas mass transfer within the particle of char had fallen, compared with that in the initial particle. Excellent agreement was observed between the rates predicted by the numerical model and those observed experimentally.

© 2013 Elsevier Ltd. All rights reserved.

## 23. ENTROPY GENERATION ANALYSES OF ENDEX AND CONVENTIONAL CALCIUM LOOPING PROCESSES FOR CO<sub>2</sub> CAPTURE

Rowena Ball

*College of Physical and Mathematical Sciences, The Australian National University, Canberra, ACT 0200,  
Australia*

### ABSTRACT

Endex thermoreactive processes, in which an exothermic reaction is coupled thermokinetically to an endothermic reaction, in principle are more efficient than conventional processes. The purpose of this research is to quantify rigorously and in detail the thermodynamic efficiency advantage of an Endex calcium looping process for CO<sub>2</sub> capture from flue gas streams, relative to a comparable conventional process, by entropy generation analysis. The theoretical background of second law analysis is reviewed, control volumes are defined and modelled, and the entropy generation rates are computed of all identified subprocesses. The Endex process is found to have superior relative second law efficiency by a factor of approximately 1.8, achieved mainly by a large reduction in the entropy penalty of regeneration at the expense of a modest increase in the entropy penalty of separation. The subprocesses most worthy (and unworthy) of optimization are identified. Results indicate that the Endex process is a promising candidate for thermal engineering optimization, because entropy generation is distributed relatively evenly across many subprocesses rather than concentrated in one or two.

© 2014 Elsevier Ltd. All rights reserved.

## 24. DIRECT CO<sub>2</sub> CAPTURE FROM AMBIENT AIR USING K<sub>2</sub>CO<sub>3</sub>/Y<sub>2</sub>O<sub>3</sub> COMPOSITE SORBENT

Vladimir S. Derevschikov<sup>a,b</sup>, Janna V. Veselovskaya<sup>a,b</sup>, Tatyana Yu. Kardash<sup>a,c</sup>, Dmitry

A. Trubitsyn<sup>d</sup>, Aleksey G. Okunev<sup>a,b</sup>

*a: Boreskov Institute of Catalysis SB RAS, Akademika Lavrentieva av. 5, Novosibirsk 630090, Russia*

*b: Center for Energy Efficient Technologies, Novosibirsk State University, Pirogova str. 2, Novosibirsk 630090, Russia*

*c: Department of Physics, Novosibirsk State University, Pirogova str. 2, Novosibirsk 630090, Russia*

*d: Aeroservice Ltd., Inzhenernaya str. 20, Novosibirsk 630090, Russia*

### ABSTRACT

Carbonate–bicarbonate looping was tested for direct CO<sub>2</sub> capture from air using a composite sorbent K<sub>2</sub>CO<sub>3</sub>/Y<sub>2</sub>O<sub>3</sub>. The phase composition, the porous structure and the texture of the composite sorbent were characterized by means of X-ray diffraction, mercury intrusion and scanning electron microscopy methods. The thermal properties of the sorbent were additionally studied by X-ray diffraction in situ and thermogravimetry methods. CO<sub>2</sub> absorption from air and desorption experiments were performed in a continuous-flow system. The effect of regeneration temperature on CO<sub>2</sub> uptake was investigated. It was shown that CO<sub>2</sub> absorption uptake from air is about 28 mg (CO<sub>2</sub>)/g in temperature swing absorption cycles within regeneration temperature range of 150–250 °C. However, the increase of the regeneration temperature up to 300 °C results in gradual decrease of the absorption uptake down to 10 mg (CO<sub>2</sub>)/g. The XRD pattern of the cycled sample contains a set of reflections that cannot be assigned to any known potassium- or yttrium-containing crystalline phase. The new phase, which is thermally stable up to 460 °C, accumulates potassium ions and is, probably, responsible for the sorbent capacity decay.

© 2013 Elsevier Ltd. All rights reserved.

## 25. PARTITIONING OF INORGANIC ELEMENTS IN PILOT-SCALE AND DEMONSTRATION-SCALE ENTRAINED-FLOW GASIFIERS

Marc A. Duchesne, Robin W. Hughes

*Natural Resources Canada, CanmetENERGY, 1 Haanel Drive, Ottawa, Ontario K1A 1M1, Canada*

### ABSTRACT

The integrated gasification combined cycle (IGCC) has been recognized as one of the leading methods of power generation with near zero CO<sub>2</sub> emissions from fossil fuels via carbon capture and storage. A suite of emerging IGCC technologies provide the promise of both high efficiency and reduced capital costs. Many of these operate at elevated temperature and hence a number of inorganic elements (i.e. elements other than C, H, O, N and S) may be present in the syngas at later stages of processing than is typical of conventional processing arrangements. Experimental results are presented for inorganic element distribution in slag and fly ash from seven entrained-flow slagging gasification plants. Data for the Siemens, Louisiana Gasification Technology Inc. (LGTI), Wabash River, ELCOGAS and Shell gasification systems were taken from literature. Data for the CanmetENERGY and Pratt and Whitney Rocketdyne (PWR) systems are presented for the first time. Mass balances and enrichment factors are calculated. All values are available in supplementary data tables. Challenges in data interpretation and general trends are highlighted. Mass balance closures for low volatility elements are within the range of 80–120% for the PWR, LGTI and Shell systems. Closures for the CanmetENERGY, Wabash River and ELCOGAS systems are further from 100%.

Accumulation, unaccounted streams, measurement inaccuracy and sampling imperfections can cause poor mass balance closures. Comparison of enrichment factors for slag and fly ash demonstrate that many elements have similar fates in gasification systems as they do in combustion systems, although several elements are less volatile in gasification systems. Partitioning can vary for a given element when comparing different gasification systems and different operating conditions. The assessments of several elements which are of environmental or technological concern are provided as examples.

© 2013 Elsevier Ltd. All rights reserved.

