APPLIED NONMETALLIC MINERAL PROCESSING
RESEARCH AT THE NORTH CAROLINA
STATE UNIVERSITY MINERALS
RESEARCH LABORATORY
IN ASHEVILLE, NC, U.S.A.
BY
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NCSU MINERALS RESEARCH LABORATORY
APPLIED NONMETALLIC MINERAL PROCESSING RESEARCH
AT THE NORTH CAROLINA STATE UNIVERSITY
MINERALS RESEARCH LABORATORY IN ASHEVILLE, NC, U.S.A.
WITH EXAMPLES OF PROCESS DEVELOPMENT FOR FELDSPAR, MICA, QUARTZ,
SPODUMENE, PHOSPHATE AND GLASSAND

by: IMMO H. REDEKER
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In 1946, the State of North Carolina and the Tennessee Valley Authority (T.V.A.) established an applied mineral processing research facility in Asheville to assist with the development of the mineral resources of the South Eastern U.S.A. The Laboratory is now operated by the School of Engineering of North Carolina State University. North Carolina is blessed with an abundance of nonmetallic ores for ceramic, chemical, fertilizer and glass uses. The Laboratory has played a major role to bring new up-to-date mineral processing technology to existing feldspar, mica, quartz, spodumene and pyrophyllite producers, assisted in the evaluation of phosphate, olivine and sand deposits and developed flowsheets for the recovery of other industrial minerals. The Laboratory worked on the reduction of mineral and industrial waste. North Carolina is now the nation's leading State in feldspar, mica, spodumene, olivine, pyrophyllite, and ultra high purity quartz production. North Carolina has a large glassand flotation operation and probably the world's largest phosphate flotation plant. Talc processing, heavy mineral recovery and other processing techniques were developed, tested and applied to other industries. The process flowsheets developed for feldspar-quartz-mica separation in the Spruce Pine area of North Carolina, the development of spodumene flotation in the Kings Mountain area, the phosphate deposit evaluation and flotation plant flowsheet development on the East Coast of North Carolina, and glassand processing will be presented and discussed. During the last 40 years, the Laboratory has worked on nonmetallic mineral processing problems in North Carolina, the U.S.A. and every continent with the exception of the Arctic and Antarctic. During the last few years, the laboratory assisted with the exploration of spodumene on the Koralpe in Austria.
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INTRODUCTION

The North Carolina State University Minerals Research Laboratory (MRL) was established in 1946 in Asheville, North Carolina, U.S.A. through a cooperative agreement between the Tennessee Valley Authority (TVA) and Dept. of Conservation and Development. Dr. Jasper Stuckey, N. C. State Geologist and Professor at the then N. C. State College of Agriculture and Engineering, was instrumental in obtaining state support for the laboratory and we named the laboratory in his memory in 1981. Since 1954 the laboratory is solely operated by the School of Engineering of North Carolina State University in Raleigh as an off campus station. (Figure 1)

The main objective of the laboratory has been and still is to assist in the orderly development of mineral resources through applied mineral processing research in batch and pilot plant scale; and to encourage and assist the N. C. mineral producers. An active advisory committee consisting of 12 members from the North Carolina mineral industry, functions as a most valuable linkage between MRL and the different mining companies. The staff of MRL consists of 6 professionals, technicians, secretaries and utility men for a total of 14 people. The annual budget is $500,000, of which one-third has to be generated through industry requested projects with well defined objectives and time schedules. The key to the successful operation of MRL is this close cooperation with industry and the necessity to generate worked for funds, not granted funds (grants).

Feldspar - Quartz - Mica Processing

One of the first projects for MRL in 1946-47 was the application of the U. S. Bureau of Mines developed feldspar-quartz-mica flotation separation technique (1,2) to N. C. ores
Figure 1. North Carolina Industrial Mineral Locations
for the Feldspar Milling Company, the second largest feldspar operation in N. C. at that time. North Carolina has unlimited reserves of a coarse grained granite rock called alaskite in the Spruce Pine area (3,4). The alaskite is liberated at approximately 850 microns, and has following mineralogical and chemical composite:

**TABLE 1. SPRUCE PINE ALASKITE FELDSPAR ORE**

<table>
<thead>
<tr>
<th>CHEMICAL ANALYSIS (% WT.)</th>
<th>MINERAL CONTENT (% WT.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na$_2$O 5.1</td>
<td>Soda Feldspar 42.0</td>
</tr>
<tr>
<td>K$_2$O 3.4</td>
<td>Potash Feldspar 14.7</td>
</tr>
<tr>
<td>CaO 0.9</td>
<td>Lime Feldspar 6.4</td>
</tr>
<tr>
<td>Al$_2$O$_3$ 15.4</td>
<td>Quartz 28.0</td>
</tr>
<tr>
<td>SiO$_2$ 74.4</td>
<td>Muscovite 7.5</td>
</tr>
<tr>
<td>Fe$_2$O$_3$ 0.4</td>
<td>Iron Minerals, Garnet 0.5</td>
</tr>
<tr>
<td>Ignition Loss 0.4</td>
<td>Clay very low</td>
</tr>
</tbody>
</table>

The alaskite ore is mined in open pits, trucked to the mills and crushed and ground to flotation size of minimum 850 microns (-20 mesh Tyler). After desliming at 75 microns (200 mesh, Tyler), a three step flotation process is applied.

First: removal of muscovite mica with cationic reagents in acid circuit, second: removal of garnet and other iron minerals with anionic petroleum sulfonate collectors in acid circuit and third: feldspar separation from quartz with cationic tallow amines in acid HF circuit. See the Schematic Flowsheet. (Figure 2)

Flotation is accomplished in acid proof open trough, Denver DR-type, supercharged machines. The feldspar is sold after drying as glass grade feldspar or is fine ground in Jasper lined pebble mills after drying and magnetic separation to ceramic or filler grade feldspar. In the early years of flotation the grade of feldspar produced was most important. Later, the recovery was improved and it was found that the final quartz, if cleaned of feldspar, would make an ideal feedstock for production of high purity and ultrahigh purity quartz for the semiconductor,
Figure 2 - Feldspar Flotation Flowsheet

Alaskite Ore
\[\downarrow\]
Crushing
\[\downarrow\]
Grinding
\[\downarrow\]
Desliming $\rightarrow$ Slimes
\[\downarrow\]
Amine, $\text{H}_2\text{SO}_4$ $\rightarrow$ Conditioning
\[\downarrow\]
Flotation $\rightarrow$ Mica $5\%$ of Ore
\[\downarrow\]
Dewatering
Petroleum-Sulfonate, $\text{H}_2\text{SO}_4$ $\rightarrow$ Conditioning
\[\downarrow\]
Flotation $\rightarrow$ Iron Waste
\[\downarrow\]
Dewatering
\[\downarrow\]
Conditioning
Amine, HF $\rightarrow$ Flotation $\rightarrow$ Quartz $20\%$ of Ore
\[\downarrow\]
Feldspar

55$\%$ of Ore
lightening fixture, optical glass, and fiber optical high technology markets. International Minerals Company started the high purity quartz production in 1965 with Quintus, and Iotagrade high grade and ultrahigh grade quartz. The Feldspar Corporation, successor of the Feldspar Milling Company committed 7.6 million to put a high purity silica facility into production in 1986. What was formerly a waste product is now a valuable high tech raw material (5.6).

The MRL also developed a flotation process to separate feldspar and quartz without the use of hydrofluoric acid (7.8).

PHOSPHATE PROCESSING

Our largest mineral resource, the N. C. Phosphates, are located in the eastern coastal plains of N. C. not far from the famous Cape Hatteras, close to the original town of Washington in N. C. Through gamma-ray logging and core drilling, about 10 billion \(10 \times 10^9\) tons of phosphate were delineated, of which about 3 billion \(3 \times 10^9\) tons can be mined by current technology. The MRL evaluated drill cores for Texasgulf, defined selective mining and tested the flotation and calcining process to maximize recovery and phosphate grade. (9,10) (Figure 3)

The high grade ore is mined and pumped to the mill. By sizing on 1 mm and desliming after scrubbing on 0.1 mm, the rest of the fine CaCO\(_2\) is removed and a flotation feed containing more than 50% phosphate pebble is obtained. The flotation process is conducted in two stages. First: single flotation with fatty acid and fuel oil for high recovery and fair grade. 90% phosphate, 10% quartz mainly for internal consumption in the phosphoric acid plant. Second: amine flotation after deoiling of the single flotation concentrate to remove the remaining fine silica and phosphate stained silica to obtain a high grade; 98% phosphate, 2% insol for triple superphosphate production and for overseas sales. A drying and calcining step to remove water,
Figure 3 - North Carolina Phosphate Mine Section

1.5 Meter Tidewater Land
↓
******************Sea Level******
↑
28.5 Meter Yorktown Overburden

4 Meter

Pungo River

12 Meter Phosphate Formation

HI GRADE ORE 87% of Phosphate 25% of Free Carbonate

3 Meter

LOWER LOW GRADE 6% of Phosphate 22% of Free Carbonate

Castle Hayne (Aquifer)
hydrocarbons and CO₂ from the high grade concentrate is also employed to obtain a high grade phosphate concentrate for super phosphoric acid production (11,12). (Figure 4)

The flotation plant was started by Texasgulf, Inc. in 1965 and can now produce 25,000 tons/day or more than 6 million tons/year of phosphate flotation concentrate, all of it floated, raised to the top of the flotation machines by small air bubbles (13,14,15).

MICA-OLIVINE

MRL pioneered the use of Humphrey Spirals for recovery of flaky mica from weathered pegmatite or weathered mica schist ores. The separation is based on shape factor instead of gravity. The flaky mica goes to the outside of the spirals with slimes and waste. The granular quartz and feldspar material is removed on the inside of the spirals where normally the heavy minerals are concentrated and removed (16).

The use of the Humphrey Spirals for the separation of serpentine and other hydrated magnesium silicates from olivine was also first demonstrated by MRL. This separation is based on the difference in gravity. Spirals are now extensively used for the recovery of mica here in North Carolina, and spirals are used for the production of olivine foundry sand by one company (17). Calcining of olivine to produce superiority foundry sand was also studied and proposed (18).

SPODUMENE FLOTATION PROCESSING

MRL assisted in the development of the lithium industry in North Carolina where large reserves of spodumene pegmatites were discovered while exploring for tin (19,20). At first, a reserve
Figure 4 - Phosphate Flotation

Hi-Grade Ore Pumped From Mine to Mill
  ↓
  Sizing → + 1 mm Waste
  ↓
  Desliming → + 0.1 mm Waste
  ↓
Fatty Acid, Fuel Oil → Conditioning
  ↓
  Flotation → Tailings Waste
  ↓
  Single Flot. Concentrate → Internal Use
  ↓
  H₂SO₄ → Deoiling
  ↓
  Washing
  ↓
  Amine, Kerosene → Conditioning
  ↓
  Flotation → Tailings Waste
  ↓
  Double Flot. Concentrate
  ↓
  Drying → Internal Use & Sales
  ↓
  Calcining
  ↓
Calcined Double Flot. Conc.
  ↓
Sales
Figure 4 - Phosphate Flotation

Pumped Ore → Screen → +1 mm → -0.1 mm → Desliming
-1 mm → +0.1 mm → Dewatering Screw
Fatty Acid Fuel Oil
Conditioner → Fatty Acid Flotation

Single Float Concentrate For Internal Use

H₂SO₄ → Wash Cyclone

Deoiling

Amine Flotation

Drier - Calciner

Calcined Double Float Conc. For Sale

Waste Pond
flotation procedure for spodumene was developed and tested in pilot plant at MRL using cationic amine to remove gangue minerals such as feldspar, quartz, mica and amphibolite from the coarse rod mill ground spodumene with the so-called gangue flotation procedure (21). Later MRL improved upon the flotation procedure and developed the anionic fatty acid flotation process after a fine ball mill grind. The recovery of mica, feldspar and quartz was also investigated and put into use by Lithium Corporation of America, the largest U. S. lithium producer (22,23). Figure 5 gives a schematic flowsheet of spodumene and byproduct, mica, feldspar and quartz processing. Since 1983, MRL has been working with Minrex here in Austria on the Koralpe spodumene deposit and we are now doing pilot plant flotation studies. The ore has similarities with N. C. spodumene pegmatites, but will be more diluted with amphibolite gangue, and presents an added challenge.

GLASSAND PROCESSING

In 1974, Libbey-Owens-Ford started a new float glass plant in Laurinburg, N. C. All major raw materials, silica sand, limestone, soda ash, and energy, had to be moved into N. C. from other states. MRL looked at the sandhills of N. C. and was able to produce a superior quality glasssand from drill core material and in continuous pilot plant (24,25,26) operation.

The specifications for the glasssand were based by Libby-Owen-Ford on sand of very high purity, which was shipped 1000 Kilometers from Arkansas to North Carolina (26). A 500,000 Tpa. flotation operation was installed by Carolina Silica, Inc. in 1975 and had for five years a remarkable record of performance. The total heavy mineral content was consistently reduced from 0.5 to 1.0% in the flotation feed to less and 0.0005 to 0.0008% in the final glassand, with an efficiency of heavy minerals removal of over 99.9%. The key to such success was good flotation feed preparation by sizing, attritioning, and desliming followed by intensive conditioning at low pH with petroleum sulfonate, and froth flotation of heavy minerals using a glycol or alcohol type
Figure 5 - Spodumene Flotation

Pegmatite Ore
  ▼
  Crushing
  ▼
  Grinding
  ▼
  Desliming ▶ Slimes
  ▼
  Scrubbing
  ▼
  Desliming ▶ Slimes
  ▼
  Pasty Acid Conditioning
  ▼
  Flotation
  ▼
  Cleaner Conditioning ▶ Amine
  ◀
  Recleaner Conditioning ▶ Mica
  ◀
  Spodumene ▶ Mica Floation
  ▼
  Conditioning ▶ Petroleum Sulfonate Sulfuric Acid
  ◀
  Iron Floation ◀ Waste
  ▼
  Amine, HF Conditioning ▶ Feldspathic Sand
  ▼
  Flotation ▶ Feldspar
  ▼
  Quartz
Figure 5 - Spodumene Flotation

Dre from Pit

Jaw Crusher

Cone Crusher

Ball Mill

Scrubber

Conditioner

Fatty Acid

Belt Filter

Spodumene

Petroleum Sulfonate

Sulfuric Acid

Spodumene Cleaner Flotation

Spodumene Rougher Flotation

Dewatering Screw

Amine, HF

Tailings

Mica Iron Flotation

Quartz, Feldspar Flotation

Quartz

Feldspar

Mica Cleaner Flotation

Mica
frother to separate the heavy minerals from the glasssand. See Figure 5. Carolina Silica's operation was bought by Unimin Corporation in 1982. Unimin is partly owned by Sibelco, a Belgian sand producer and Libbey-Owens-Ford is now partly owned by Pilkington Brothers, Ltd. the inventor of the flat-glass process which revolutionized float glass making.

FOUNDARY SAND FLOTATION

MRL developed and piloted a foundry sand flotation procedure which was put into plant production by Manito Investment Company, of Peoria, IL. The plant supplies high grade foundry sand to the automated Caterpillar Foundry in Mapleton, IL. Flotation of dune sand after sizing, scrubbing, desliming and flotation produces a foundry sand with very low ADV (Acid Demand Value) which is bonded with resins in the foundry process. The plant can produce up to 300,000 tons of foundry sand per year.

WEATHERED-PEGMATITE PROCESSING

A number of projects for the former Kings Mountain Mica Company, now Kings Mountain Group, resulted in nearly 100% conversion of a weathered pegmatite ore into saleable products. The results of the different projects are demonstrated in Figure 7. In the early years of scrap mica production, so-called "mica jigs," a combination of rolls crushers and Trommel screens recovered about 5% of the weathered pegmatite ores in the form of coarse scrap mica. The use of the Humphreys Spiral increased mica recovery to between 8 - 10% of the ore. Further mica recovery was achieved by use of flotation with amines in acid circuit or with a combination of fatty acid and amine in alkaline circuit (27). Testwork on the clay slimes by MRL and the Ceramic Department of North Carolina State University showed that very good white bricks could be produced from dewatered clay with
FIGURE 6 - GLASSAND FLOTATION

Ore
↓
Desliming \rightarrow - 0.1 mm Waste
↓
Hydrosizing \rightarrow + 0.5 mm Waste
↓
Dewatering
↓
Scrubbing
↓
Desliming \rightarrow - 0.1 mm Waste
↓
Scrubbing
↓
Desliming \rightarrow - 0.1 mm Waste
↓
Dewatering
\(\text{(Fatty Acid)}\)
Petroleum Sulfonate \rightarrow Conditioning
Sulfuric Acid
↓
Flotation \rightarrow Heavy Minerals Waste
↓
Filtering
↓
Drying
↓
Screening
↓
Shipping
↓
Glassand
admixtures of bentonitic clay from Alabama. Further testwork on the coarser waste sand resulted in high grade potash feldspar recovery for color television tubes, and the residual quartz is now used to make glass containers for baby foods (28,29,30). The results show a nearly 100% use of an industrial mineral resource. It is to be noted that all process water in this quite complex operation is recirculated through settling ponds, and zero discharge into the surface streams is achieved. (Figure 7)
Figure 7. Reduction of Waste by Improved Processing
Of Weathered Pegmatite and Shist Ore.

Spirals
Spirals & Flotation
Spirals & Flotation & Thickening
Spirals & Flotation & Thickening & Scrubbing & Feldspar Flotation
REFERENCES


