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DEVELOPMENT OF NEW TECHNOLOGY FOR BENEFICIATION OF FLORIDA DOLOMITIC PHOSPHATE RESOURCES

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DEVELOPMENT OF NEW TECHNOLOGY FOR BENEFICIATION OF FLORIDA DOLOMITIC PHOSPHATE RESOURCES

FINAL REPORT

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DISCLAIMER

PERSPECTIVE

Patrick Zhang, Research Director - Beneficiation & Mining

With the depletion of the higher-grade, easy-to-process Bone Valley deposits, the central Florida phosphate industry has been forced to move into the lower grade, more contaminated ore bodies from the Southern Extension. The phosphate deposits in the Southern Extension may be divided into two zones: an upper zone and a lower zone. The upper zone is readily processable using the current technology, but the lower zone is highly contaminated by dolomite. Geological and mineralogical statistics show that about 50% of the phosphate resource would be wasted if the lower zone is bypassed in mining, and about 13% of the resource would be wasted if the dolomitic pebbles in the lower zone are discarded.

The dolomite flotation process developed under this project has three unique features: fine grinding, non-desliming flotation and a special dolomite collector. This process achieved, on bench scale, about 20 percent higher phosphate recovery than any previously developed processes by different organization, as is shown in the following table.

Summary of Flotation Results

Sample	Concentrate Analysis (%)		$\%P_2O_5$ Recovery
	BPL	MgO	
FLA-1	67.1	0.87	82.2
FLA-2	67.7	0.76	90.1
FLA-3	68.6	0.96	60.6
FLA-4	66.2	0.63	81.6
FLA-5	68.8	0.91	83.6

With this kind of performance in the lab, the next logical step is to conduct pilot testing. Indeed, the FIPR Board of Directors has approved funding for such a test program led by IMC-Agrico. Many hope that this process will eventually lead to the first industrial process for separating dolomite from phosphate in Florida.

ABSTRACT

Among the deleterious impurities in phosphate rock, dolomite is the most troublesome. Dolomite causes higher consumption of sulfuric acid, reduces filtration capacity, and lowers P_2O_5 recovery in the fertilizer manufacture process. Almost 50% of the future phosphate reserve in Florida are contaminated by dolomite. In 1994, FIPR conducted a comparative evaluation on five seemingly promising flotation processes for separating dolomite from phosphate. None of the processes evaluated proved to be a viable solution to the dolomite problem, with two failing to produce a concentrate of less than 1% MgO and all giving pool phosphate recoveries ranging from 30-60%. Building on their 30-year experience in dealing with dolomitic phosphate deposits in China, CLDRI (The Chinese Lianyungang Design and Research Institute) developed a complete processing flowsheet for the dolomitc pebbles in Florida. In this process, the sample is ground to liberate dolomite phosphate. The ground slurry is subject to dolomite flotation using a mixture of H_3PO_4 and H_2SO_4 as pH modifier and a fatty acid soup-based dolomite collector. The sink product of dolomite flotation can be beneficiated by either silica or phosphate flotation. Extensive laboratory testing demonstrated the technical and economic feasibility of the new process, achieving concentrates analyzing >31% P_2O_5 and <1% MgO with an overall phosphate recovery of more than 80%.

ACKNOWLEDGEMENTS

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The one-year study was jointly carried out by IMC-Agrico Company and China Lianyungang Design and Research Institute (CLDRI). We thank Ms. Xu Qin, Mr. Li Xuesheng, Mr. Shang Xianfu and Ms. Deng Hongxia for their hard work in the research project. Mr. Guy Casey deserves recognition for his full cooperation during the verification tests done in IMC-Agrico Company's laboratory. The authors also extend their appreciation to all engineers and technicians for their participation and assistance in undertaking mineralogical studies, conducting laboratory experiments, providing chemical analyses, etc.

Special thanks are given to Dr. Paul Clifford, Dr. Patrick Zhang, Dr. John R. Schmedeman and Dr. Ronald L. Wiegel for their helpful suggestions given during the field workshop and throughout the entire period of the research program.

FOREWORD

In October 1997, the Board of Directors of the Florida Institute of Phosphate Research (FIPR) approved a project which would permit the IMC-Agrico Company (IMCA), to subcontract with the Chinese Lianyungang Design and Research Institute (CLDRI), to investigate the separation of dolomite from high MgO Florida phosphate rock. CLDRI had previously demonstrated a capability in reducing the dolomite content of Mainland Chinese hard rock phosphate ores, and in fact have commercially implemented such separation processes in one or more Chinese phosphate mining and processing operations.

It is well known that the most predictable characteristic of the Florida phosphate deposit is its variability. The occurrence of dolomite in beneficiation products is associated with the particle size fractions which end up reporting to the pebble size range, that is, the plus 16 mesh or 1 mm size. The portion of matrix which reports to that pebble size is of itself quite variable, ranging from approximately 15% to about 40% of potential product. In addition to size variability, the pebble fraction ranges from being of a satisfactory chemical composition, with less than 1% MgO to extremely high dolomite contents with MgO in the 2% to 4% range.

This interpretation is not only supported by the results of exploratory drill hole separations, but also by the production trends experienced at several mining locations, where for periods of time the MgO content of pebble is acceptable, and at other times the MgO of the pebble is so high it must be separated and either discarded, or, blended into good product as a controlled diluent. There are also times when the resource area must be selectively mined, leaving as unmined, those areas known to have high dolomite contamination. In that case, not only is there a ton of high MgO pebble that is lost, but the flotation feed size fraction (1mm by 0.1mm), which if processed would make a satisfactory product (0.5-0.8%MgO) is also totally lost. It is against this backdrop that the CLDRI bench tests were begun on several samples selected to demonstrate the variability of Florida phosphate deposits.

This foreword, prepared by IMCA, is meant to attempt to summarize in capsule form, the results of the CLDRI bench scale testing. It includes some evaluations related to economic question regarding the commercial implementation of the process, and points out several additional areas of consideration when and if pilot demonstration of the process is planned.

CLDRI BENCH SCALE TESTING RESULTS

Five samples of high MgO pebble were collected by IMCA and sent to China for bench scale processing studies at the CLDRI laboratories located in Lianyungang, China. Several processing alternatives were evaluated, which are detailed in the CLDRI Final

Report. It has been concluded that the most likely process to be applicable to Florida's high MgO pebble is one which consists of the following steps:

1. Grinding the pebble to about 50% passing 200 mesh
2. Conditioning with a proprietary modified fatty acid reagent, PA-31 in the presence of phosphoric and sulfuric acids at 30 to 60% solids
3. Floating the dolomite from the ground phosphate rock and sand mixture without desliming to remove fines
4. Diluting the dolomite flotation concentrate and sizing to remove the concentrates finer than 400 mesh
5. Using an amine with kerosene as an extender, and soda ash to adjust pH, to float the sand in the oversize dolomite flotation concentrate to reduce the SiO_2 or insol content.
6. Combining the amine concentrate and the dolomite flotation concentrate which was finer that 400 mesh, to obtain a final concentrate with both a reduced MgO and SiO_2 composition

Table I summarizes the metallurgical results of treating each of the five Florida samples by this process. In the treatment of sample FLA-4 only the dolomite flotation step was necessary because of its high clay content which made the sample unresponsive to amine silica flotation. Its high content of clay was also the reason why FLA-4 was scrubbed and washed prior to grinding. With FLA-5, it was also necessary to scrub and wash the feed sample prior to using the process described above, because of the high initial MgO content. A set of data is also presented for sample FLA-5 to demonstrate that the use of phosphoric acid is not always necessary.

An examination of Table I indicates that in all samples except FLA-3, it was possible to obtain a concentrate with less than 1% MgO and more than 80% BPL recovery, which was the some what arbitrary target given for this research. Sample FLA-3 could not be expected to achieve that goal, because of its initial 9.7% MgO content, indicating a dolomite level of 35 to 40% in flotation feed. A flow sheet for this CLDRI process is shown in Figure I, which attempts to include those operating steps necessary to commercially produce a low MgO (<1.0%MgO) concentrate from a high MgO pebble or feed material.

COMMERCIAL IMPLEMENTATION AND ECONOMIC CONSIDERATIONS

Table II summarizes the estimated economic effects, on commercial implementation, of the required grinding (energy and media wear) and the consumption of various flotation reagents for the tests summarized in Table I, except for sample FLA-4 which was not responsive to cationic silica flotation. The grinding cost estimate is based

TABLE I: SUMMARY OF CLDRI METALLURGICAL DATA

SAMPLE		FLA - 1	FLA - 2	FLA - 3	FLA - 4	FLA - 5	FLA - 5
				*	**		***
FEED	%Wt.	100.0	100.0	100.0	100.0	100.0	100.0
	%BPL	54.1	55.3	30.7	52.5	56.9	56.9
	%MgO	1.77	1.08	9.66	2.00	2.52	2.52
SCRUB/WASH CONC	%WT				83.8	93.2	93.2
	%BPL				58.7	59.2	59.0
	%BPL Distr.				93.7	96.7	96.7
	%MgO				1.15	2.06	2.08
	%MgO Distr.				48.2	72.2	72.2
FLOT. FEED SIZE	% - 200 Mesh	44	29	70	42	44	44
DOLOMITE FLOT. TAIL	%Wt.	19.9	8.1	64.4	8.7	17.7	18.3
	%BPL	45.8	44.8	17.7	48.7	43.2	44.4
	%BPL Distr.	16.8	6.6	37.2	8.6	13.4	14.4
	%MgO	5.9	5.64	14.57	4.67	7.66	7.02
	%MgO Distr.	66.1	42.6	97.1	20.3	51.2	47.9
AMINE FLOT. TAIL	%Wt.	15.6	16.1	8.5		8.6	7.6
	%BPL	6.4	4.8	7.7		9.5	7.2
	%BPL Distr.	1.9	1.4	2.1		1.4	0.9
	%MgO	0.19	0.10	0.22		0.10	0.31
	%MgO Distr.	1.7	1.8	0.2		0.4	1.0
MINUS 400 MESH CONC.	%Wt.	9.8	11.7			11.6	11.9
	%BPL	61.6	62.3			65.9	64.5
	%BPL Distr.	11.1	13.2			13.4	13.5
	%MgO	1.18	1.10			1.08	0.63
	%MgO Distr.	6.8	12.0			4.6	2.8
AMINE FLOT. CONC.	%Wt.	54.8	64.0	27.2		55.3	55.5
	%BPL	69.3	68.0	68.6		70.7	69.0
	%BPL Distr.	70.2	78.8	60.6		68.5	67.8
	%MgO	0.83	0.74	0.96		0.78	0.99
	%MgO Distr.	25.4	43.5	2.7		16.1	20.5
COMBINED CONC.	%Wt.	64.5	75.7	27.2	75.1	66.9	67.4
	%BPL	68.2	67.2	68.6	59.5	69.9	68.2
	%BPL Distr.	81.3	92.0	60.6	85.1	81.9	81.3
	%MgO	0.88	0.79	0.96	0.7	0.82	0.93
	%MgO Distr.	32.2	55.5	2.7	27.9	20.7	23.3

NOTES

*Second flotation done without sizing @ 400 mesh.
** Scrub/wash and dolomite flotation data only. Pulp was not responsive to amine flot.
***Used only sulfuric acid in the dolomite flotation step. No phosphoric acid.

on an assumed 10KWH/t Bond Work Index for the phosphate pebble and a $0.04/KWH energy cost and a $0.034/KWH media wear cost. The reagent cost is based on accepted market rates for all chemicals currently used in the industry except for the modified fatty acid dolomite collector, PA-31, for which a $0.25/lb. rate was estimated. In addition, to account for the practical experience that significantly more reagent is required for commercial flotation operations than for bench scale separations, all bench test reagent consumption values were inflated by an arbitrary 50% effectiveness factor. Thus a 1 lb./t lab test would lead to a 1.5 lb./t estimate for commercial use.

TABLE II: COST ESTIMATES FOR REAGENT CONSUMPTION AND GRINDING FOR TEST SAMPLES

SAMPLE	FLA - 1	FLA - 2	FLA - 3 *	FLA - 5	FLA - 5 **
FLOTATION FEED					
% Minus 200 mesh	44	29	70	44	44
COMBINED CONCENTRATE					
% Wt. of Feed	64.5	75.7	27.2	66.9	67.4
BPL Analysis,%	68.2	67.2	68.6	69.9	68.2
MgO Analysis,%	0.88	0.79	0.96	0.82	0.93
REAGENT USAGE #/t FEED					
Phosphoric Acid	5.6	4.0	8.0	5	0
Sulfuric Acid	4.0	3	0.0	4	12
PA - 31	3.2	2.2	4	3.6	3.6
Soda Ash	0.6	0.6	0.5	0.6	0.6
Amine	0.8	0.8	0.5	0.8	0.8
Kerosine	0.3	0.25	0.125	0.25	0.25
REAGENT COST* **					
$/t of Test Feed	2.02	1.50	2.44	2.02	1.35
$/t of Test Conc.	3.14	1.98	8.97	3.02	2.01
$/t of Commercial Conc****	4.70	2.97	13.45	4.53	3.01
GRINDING COST					
$/t of Commercial Conc*****	0.61	0.39	1.47	0.60	0.59

NOTES

* Second flotation done without sizing @400 mesh

**Used only sulfuric acid in the dolomite flotation step. No phosphoric acid.

***REAGENT PRICE, $/lb

Phos. Acid=0.158; Sulfuric Acid=0.015; PA-31=0.25

S.Ash=0.07; Amine=0.25; Kerosene=0.12.

****Reagent consumption for commercial operation arbitrarily set at 1.5*($/t test conc.).

*****Based on: power consumption = $0.04/ KWH and $0.034/KWH for media.

With the exception of sample FLA-3 which has extraordinary feed MgO content, the grinding costs are estimated to be in the range of $0.40 to $0.60 per ton of concentrate. The commercial reagent costs are estimated to be in the $3.00 to $5.00 per ton of concentrate with exception again of sample FLA-3.

Concentrate thickening and filtering steps have been included in the commercial CLDRI process flowsheet, shown in Figure I, to permit handling and transportation of the much finer concentrate than is currently encountered in the flotation plant concentrate which is about a 20 mesh topsize. The filter cake, containing about 20% moisture, would not have excessive water content nor be dry enough to allow a free flow of fine slurry or dry dust through cracks or holes in either rail cars or trucks that are used to transport from flotation plants to chemical plants. Meanwhile this finer concentrate is likely to be sufficiently fine to permit acidulation without further grinding, providing a partially offsetting cost saving for the flotation feed grinding step.

Based on the above metallurgical data and the magnitude of the estimated operating costs for implementing this process, it is the opinion of IMCA that the target for bench scale testing has been attained and due consideration should be given to a pilot demonstration.

Given the degree of variability in phosphate deposits here in Florida, it would seem important that any dolomite separation process for Florida matrix must be capable of being operated independently of the rest of the conventional processing steps. In the past, this has been viewed as requiring the stocking and depletion of a high dolomite pebble surge pile. The withdrawal from the surge pile would then feed a separate part of the beneficiation plant, where operating rates and schedules could be manipulated to keep the inventory and composition of high dolomite pebble at some reasonable level. Engineering studies have been made in the past to evaluate this approach. One such study conducted internally by IMCA, evaluated IMCA's cationic flotation process, which uses a primary amine to float phosphates from dolomite, but there was insufficient economic return to justify the investment of risk capital for such a project.

The economics for the implementation of the CLDRI dolomite flotation process are expected to be considerably better. This is due to the bench scale testing results, which indicate that although a finer grind of the high dolomite pebble may be necessary for the CLDRI process, this expense may be more than offset by the fact that the flotation separation can be done without significant phosphate desliming losses, which were of the order of 20-30% in IMCA's cationic flotation process.

COMMENTS REGARDING THE DOLOMITE COLLECTOR

In the CLDRI process, questions exist regarding the economic and perhaps environmental consequences of the use of the dolomite collector, PA-31. To date, details of this reagent are covered by a confidentiality agreement between CLDRI, FIPR and IMCA. It is the considered opinion of all parties to that agreement, that the quantity of PA-31 raw materials needed for its domestic manufacture is readily available at a reasonable price in the US market, that the simplicity and cost of manufacture is such that a reasonably priced product ($0.20 - $0.25 per pound) could be supplied to the phosphate industry, and there is no reason to believe there will be significant environmental effects of this reagent. The lack of an environmental problem is supported by the commercial use of this reagent in similar application in China.

Despite all of these considerations, it is important to quickly determine that this is indeed the situation, before too much more time, effort and funds are spent in the further development of the CLDRI process for Florida phosphate. In order to reach an acceptable confidence level with respect to these issues, it would be wise to get a reputable reagent supplier involved in such an evaluation early in proposed pilot testing. This would in all probability require that this reagent supplier become a participant in the confidentiality agreement with IMCA, FIPR and CLDRI. It may also require some provision that said reagent supplier would at their option become licensed for the production of PA-31, with some form of payment to CLDRI and FIPR.

THE CLDRI FINAL REPORT

The project final report from CLDRI can be found in the pages following this foreword. The entire report is being provided to the reader as it was received from CLDRI, and serves as a comprehensive documentation of the work done on all of the five Florida phosphate pebble samples, by CLDRI researchers.

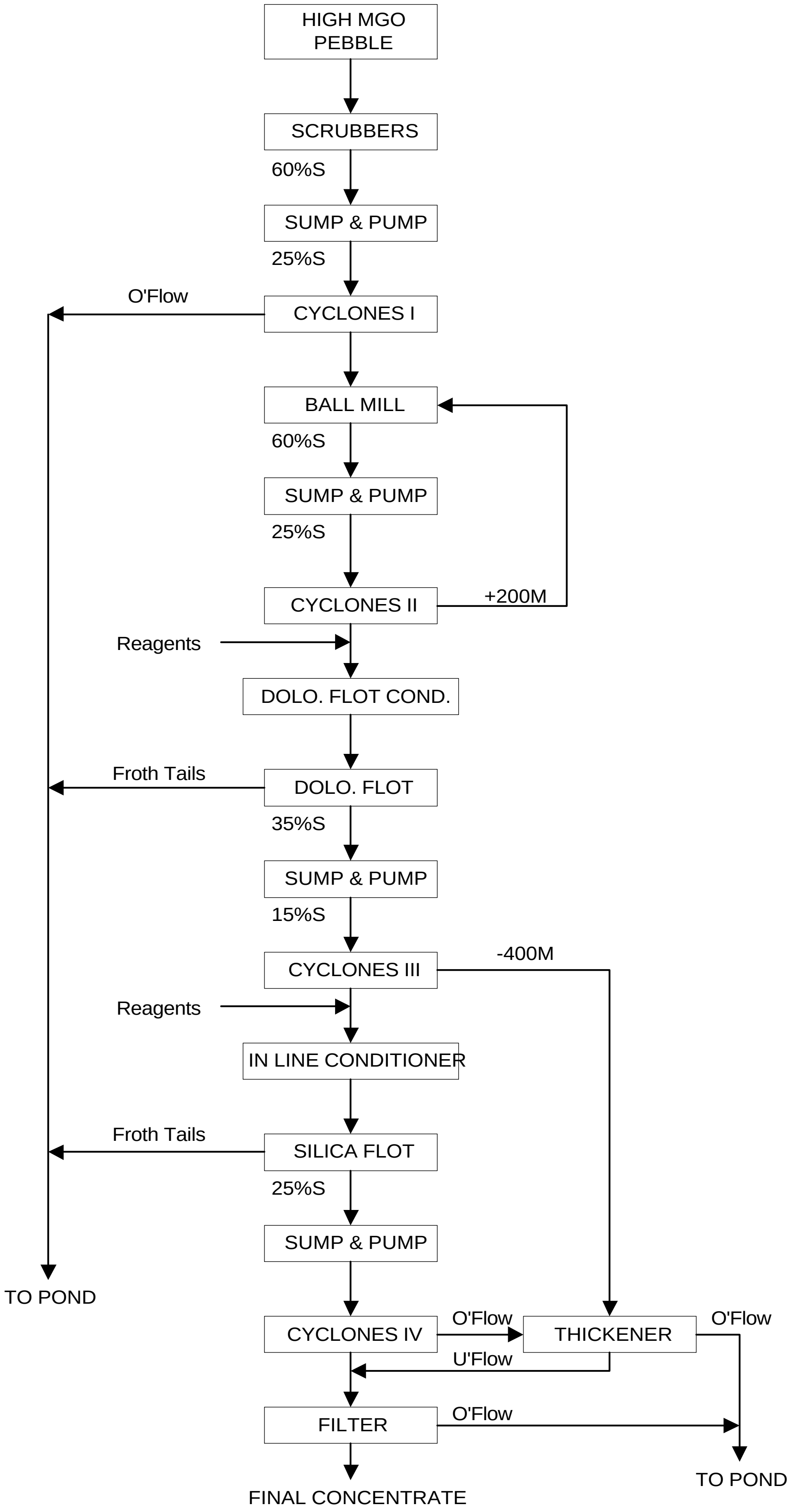

Figure I. IMCA/CLDRI Dolomite Flotation Flowsheet

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EXECUTIVE SUMMARY

The United States is one of the world's largest producers of phosphate rock. Its phosphate production represents approximately one third of the world's total, while about 70% of the U.S. production comes from Florida. Unfortunately the high-grade siliceous phosphate ore from the Bone Valley formation is being depleted rapidly. It is estimated that the Florida phosphate reserve that can be economically processed with the available technology can only last about 20 years at the current mining rate. It is well recognized that the development of new technology for effective recovery of phosphate values from high dolomitic phosphate resources thus becomes extremely urgent. Therefore, the separation of dolomite from phosphate has been considered as the priority research project in recent years. In October 1997, the FIPR Board of Directors approved funding a one-year research contract (97-02-129) designed to develop a flotation technology for beneficiating Florida high-dolomite phosphate pebble. In this project, China Lianyungang Design and Research Institute (CLDRI) worked as a subcontractor for laboratory development. The objective of the investigation was to develop a complete process for effective recovery of phosphate from Florida high dolomite pebble with the target result of obtaining a flotation concentrate analyzing over 65% BPL and less than 1% MgO at the overall BPL recovery over 80%.

The research project involved the following major tasks:

- Sampling and Sample Preparation
- Sample Characterization
- Bench Flotation Test
- Product Characterization
- Preliminary Economic Evaluation

SAMPLING AND SAMPLE PREPARATION

IMC-Agrico Company collected and shipped five high MgO pebble samples, of about 500 pounds in weight each, to CLDRI for conducting laboratory testing. The samples were collected from different phosphate mines in Florida with various chemical composition. The identification, sampling site and major components of the samples are shown as follows:

Sample ID	Sample Source	BPL%	MgO%	Insol.%
FLA-1	Fort Green pebble #1	54.17	1.85	19.48
FLA-2	Fort Green pebble #2	56.68	1.19	19.20
FLA-3	Fort Green pebble #3	32.67	9.40	11.06
FLA-4	Clear Springs pebble	52.13	2.04	20.20
FLA-5	Kingsford pebble	55.46	2.88	12.24

Each as-received sample was thoroughly mixed, and divided so that approximately half would be used for laboratory research while the remaining portion would be kept in storage. The test sample was air-dried, crushed to -1mm and split to batches for grinding and flotation tests. Because samples FLA-4 and FLA-5 were contaminated with a lot of fine clays, these two samples were washed and deslimed to remove primary slimes in order to minimize the effect of such high-impurity fines on the subsequent flotation process. The deslimed samples were further prepared by following the above procedures.

SAMPLE CHARACTERIZATION

The representative samples were taken from the as-received samples for mineral characterization studies with regards to size distribution, liberation, mineralogical and chemical compositions. Mineralogical and X-ray diffraction studies indicated that the principal phosphate mineral in the samples was francolite corresponded to carbonate-apatite. Impurity minerals consisted of quartz, dolomite, calcite, clays, feldspars and others.

Size and liberation studies showed that the samples should be ground quite finely before flotation in order to achieve good liberation of the dolomite impurity from the phosphate mineral. The liberation sizes of those samples vary in the range of 0.2 to 0.5 mm. Samples FLA-2 and FLA-4 should be ground to about 0.5mm, and sample FLA-1 to about 0.2 mm in order to obtain liberation sizes.

BENCH FLOTATION TEST

Laboratory flotation tests aimed at the development of a flotation process for beneficiating Florida dolomitic phosphate pebbles were carried out with mechanical cells. According to mineralogical characterization study results, the flotation process to be developed should have the functions of rejecting both dolomite and silica in order to achieve acceptable flotation concentrate for manufacturing wet phosphoric acid.

Although different flowsheets were developed in accordance with the sample characteristics, a core flowsheet has evolved and is expected to work for most of the Florida dolomite pebbles. The flowsheet consists of several operations. Firstly, the test sample was ground to liberation size. Then carbonate flotation was performed by adjusting the pH of the slurry to 4.5 to 5.5 using mixed acids of H_3PO_4 and H_2SO_4 and using PA-31 as a dolomite collector. After dolomite was floated, the underflow was sized at 400 mesh to obtain a -400 mesh fine product because the fine fraction contained low MgO and silica. The plus 400 mesh fraction was subject to either silica flotation with amine or phosphate flotation with fatty acid in alkaline solution for separation of silica from phosphate. The fine fraction and flotation concentrate were combined to obtain a final composite phosphate concentrate. The overall reagent consumption varied from sample to sample. There were also some differences in flowsheet structures for beneficiating different samples, such as no sizing step for sample FLA-3, no amine flotation to reject silica for sample FLA-4, etc. The flotation concentrate analyses of the five dolomite pebble samples are summarized as follows:

Sample ID	Flotation Process	BPL%	MgO%	BPL Rec. %
FLA-1	Grind-dolo flot-sizing-phos flot	67.1	0.87	82.2
FLA-1	Grind-dolo flot-sizing-silica flot	68.4	0.91	81.7
FLA-2	Grind-sizing-dolo flot-silica flot	67.7	0.76	90.1
FLA-2	Grind-dolo flot-sizing-silica flot	68.4	0.78	90.8
FLA-3	Grind-dolo flot-silica flot	68.6	0.96	60.6
FLA-4	Grind-dolo flot-sizing-phos flot	67.5	0.77	90.1
FLA-4	Grind-sizing-phosphate flot	67.7	0.63	81.5
FLA-5	Grind-dolo flot-sizing-silica flot	68.2	0.93	84.2

The beneficiation results obtained demonstrated that all the Florida dolomitic pebble samples were amenable to the CLDRI fine flotation technology. The target results could be achieved from those samples with the exception of low overall BPL recovery for sample FLA-3 due to its high MgO content, nearly 10%, much higher than that in the other samples.

PRODUCT CHARACTERIZATION

The phosphate concentrates from those samples were analyzed for chemical compositions and size distribution. In flotation feed, the fine size fractions contained much higher MgO than in the coarse sizes. Low MgO content in the fine fractions of the phosphate concentrate indicated that the CLDRI fine particle flotation technology was very effective in separating dolomite from phosphate. Because the phosphate concentrate had been finely ground for mineral liberation prior to flotation, it could be directly used for phosphoric acid production with no need for further grinding. The chemical analysis showed that all the phosphate concentrates contained over 65% BPL, less than 1% MgO and 5% insolubles, which could meet the requirements of the wet acid manufacturing process.

PRELIMINARY ECONOMIC EVALUATION

The overall reagent costs were between \$1 to \$3 per ton of concentrate for most of the samples, but were much higher for sample FLA-3. The detailed evaluation will be conducted by IMC-Agrico Company.

INTRODUCTION

Phosphate rock, as a strategic commodity for the fertilizer industry, plays an important role in agriculture. It is estimated that over 80% of the phosphate rock output in the world is used for fertilizer production. The United States is one of the world's largest producers of phosphate rock and its phosphate production represents approximately one third of the world total, while about 70% of the U.S. production comes from Florida.

Unfortunately the easily beneficiated and high-grade siliceous phosphate ore from the Bone Valley formation is being depleted rapidly. It has been estimated that the Florida phosphate reserve that could be economically processed with the available technology could last only about 20 years at the current mining rate. In order to meet the domestic demands on phosphate rock for fertilizer production, the phosphate rock export has been limited since 1997. As the phosphate mining moves further south and west, the phosphate matrix will be leaner in grade and higher in dolomite content. For the purposes of extending the phosphate resource availability in the Florida area and maintaining its dominant position in phosphate industry, the development and utilization of dolomitic phosphate resources must be taken into consideration.

Since the 1970s, many investigators and researchers have been actively engaged in studies on the beneficiation of dolomitic phosphate resources, and some encouraging results have been achieved. In 1994, FIPR conducted a comparative evaluation on five seemingly promising flotation processes for separating dolomite from phosphate, utilizing the same flotation feed. These processes included the University of Florida two-stage flotation process, the U.S. Bureau of Mines fluosilicic acid process, the IMCF cationic process, and the TVA diphosphonic acid process. Three of the processes could not even reduce the concentrate MgO to below 1% and all the processes gave very poor overall recovery, ranging from 30 to 60%. The cause of lower BPL recovery in the concentrate is mainly attributed to the desliming operation after grinding the flotation feed. About 30% of the overall BPL was lost in the deslimed fines. Flotation reagent costs were also high, at over $2.00 per ton of concentrate in most cases.

Based on the characteristics of the indigenous phosphate resources, CLDRI has developed a series of fine particle flotation technologies for effective beneficiation of fine-grained siliceous and carbonated phosphate ores. Some of those fine particle flotation technologies have been successfully applied on commercial scale in China. One very important feature of those beneficiation technologies is that no desliming operation is required before flotation, even though the ore must be ground to very fine sizes for liberating impurities from phosphate. Besides achieving the selective separation of dolomite from phosphate, the CLDRI technologies effectively recover the BPL in fines, which leads to a high overall BPL recovery. The lack of desliming also simplifies, to some extent, the processing flowsheet.

In order to apply CLDRI fine particle flotation technology to the beneficiation of Florida dolomitic phosphate resources, IMC-Agrico Company and CLDRI jointly submitted the proposal “Development of New Beneficiation Technology for Florida Dolomitic Phosphate Resources” for review by FIPR’s Technical Advisory Committee in May 1997, and it was approved by the FIPR Board of Directors in October of the same year (Project No. 97-02-129).

According to the tasks specified in the proposal, IMC-Agrico Company was to collect five dolomitic phosphate pebble samples from the Florida area and ship them to CLDRI in China. CLDRI would carry out laboratory bench tests on those samples with the principal objective of demonstrating the applicability of CLDRI fine particle flotation technology for processing the Florida dolomitic phosphate pebbles. The target results are to obtain phosphate concentrate analyzing 65%BPL, less than 1% MgO, at an overall BPL recovery of over 80%.

Bench testwork was conducted in CLDRI’s laboratory aimed at developing and optimizing the beneficiation process for the high-dolomite samples received. Firstly, all the samples were characterized in regards to their mineral composition, size distribution, liberation size, interlocking property of various minerals, etc. Then each sample was crushed and ground to liberation size, particularly for liberating dolomite from phosphate. The ground material was subject to flotation processes for rejecting both dolomite and silica impurities and producing a phosphate concentrate. With the CLDRI’s fine particle flotation process, most of the dolomitic phosphate pebble samples received could produce the target results with the exception of low BPL recovery, about 60%, for sample FLA-3 due to its extremely high MgO content.

The testwork indicated that CLDRI fine particle flotation process could be successfully used for effective recovery of phosphate from Florida dolomitic phosphate resources. Based on the CLDRI’s experience in phosphate beneficiation, the process could be run at large scale with little problem.

RESEARCH SAMPLES

SAMPLE COLLECTION

The five dolomitic phosphate pebble samples, each weighing about 500 pounds, were collected from the Florida area by IMC-Agrico Company. The samples were put into five drums and identified as FLA-1, FLA-2, FLA-3, FLA-4 and FLA-5. The five drums were then air-shipped to Shanghai on January 16, 1998, and were delivered to CLDRI on February 4. The sample identification and the sources are given in Table 1.

Table 1. Sample Sources and Descriptions.

Sample ID	Sample Source	Description
FLA-1	Fort Green	From pebble storage, net wt. 500 lbs.
FLA-2	Fort Green	From pebble storage, net wt. 500 lbs.
FLA-3	Fort Green	From dragline #16 pit, net wt. 500 lbs.
FLA-4	Clear Springs	From pebble storage, net wt. 463 lbs. with high slimes, high moisture
FLA-5	Kingsford	From pebble storage, net wt. 500 lbs. Contaminated with primary slimes

SAMPLE PREPARATION

After the sample drums were received, each sample was taken out of its drum and thoroughly mixed. A two-kilogram split was made from each sample for mineralogical study, analyses of size distribution, and chemical composition. Then each sample was cone-quarterly split into two portions, one for test work and the other for storage in the original drum.

The test samples were air-dried and roll-crushed to –1 mm size. The crushed samples were fully mixed. A sample weighing about 200 grams was taken for analyzing P_2O_5, MgO, CaO, CO_2, SiO_2, Al_2O_3 and other components, and a 500-gram sample was taken for size analysis. The remaining sample was split into batches for laboratory flotation testing. Figure 1 shows the sample preparation procedures.

Due to the high slime content in samples FLA-4 and FLA-5, those two samples were scrubbed and washed to remove primary slimes before being dried and crushed. The objective of this procedure was to minimize the negative effects of the clay minerals on flotation.

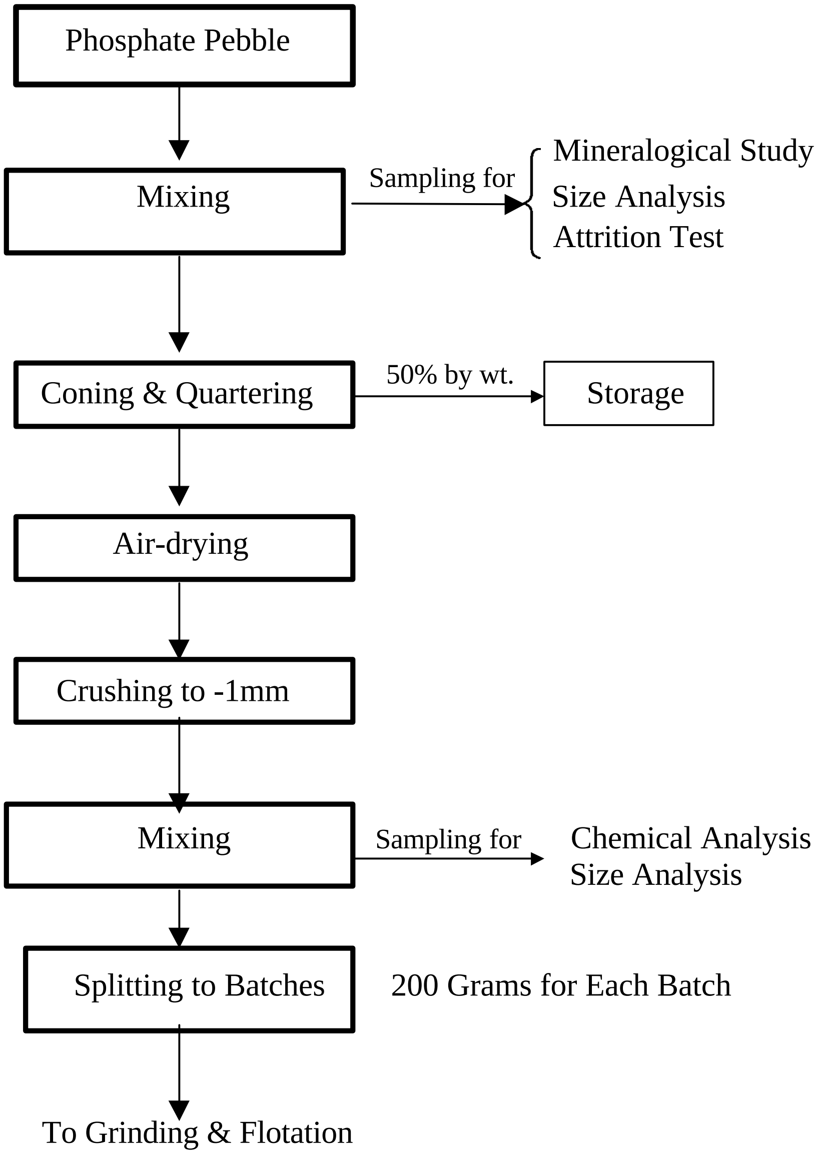

Figure 1. Sample Preparation Procedure.

SAMPLE CHARACTERIZATION

CHEMICAL ANALYSIS

Representative samples were taken and dried at 105 degrees Centigrade for 8 hours, then ground to pass 100 mesh. The ground samples were chemically analyzed. The analysis results of run-of-mine samples are listed in Table 2. The sample FLA-3 contains 32.05% BPL with very high MgO content, up to 9.6%. The BPL grade in other samples ranged from 52.68% to 56.02%, and MgO from 1.12% to 2.88%. It can been seen from chemical analysis that both dolomite and silica impurities should be rejected in order to obtain the acceptable concentrates from those samples.

Table 2. Chemical Analyses of As-Received Dolomitic Pebble Samples (%).

Component	FLA-1	FLA-2	FLA-3	FLA-4	FLA-5
BPL	53.20	56.02	32.05	52.68	55.30
P_2O_5	24.35	25.64	14.67	24.11	25.31
MgO	1.69	1.12	9.59	2.04	2.88
CaO	36.70	37.54	34.77	38.25	43.22
Fe_2O_3	1.43	1.43	1.32	1.94	1.42
Al_2O_3	1.40	1.40	0.88	1.91	0.96
SiO_2	18.38	18.24	9.85	18.55	10.74
CO_2	6.80	6.04	23.98	4.42	8.54
F^-	3.48	3.68	3.26	3.80	3.86
A.I.	19.48	19.20	11.30	20.20	12.24
I.L	8.78	7.86	24.19	9.22	10.76

SIZE ANALYSIS

The size and chemical distribution of the five as-received samples are listed in Tables 3 through 7. For understanding the effects of scrubbing on size and chemical distribution, scrubbing tests were made on all the samples with a Denver attrition box, and the scrubbed materials were analyzed for size and chemical distribution. The analysis results are given in Tables 8 through 12. Prior to grinding and flotation, the samples were roll-crushed to –1 mm to obtain a homogeneous grinding size in a small capacity laboratory mill. Tables 13 to 17 show size and chemical analyses after the samples were roll-crushed to - mm. From the results of size and chemical analyses listed in Tables 3

Table 3. Size Distribution and Chemical Analysis of Sample FLA-1.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.0	4.68	46.32	21.20	2.98	22.99	3.95	8.38	5.44
8.0~5.0	5.70	52.22	23.90	3.44	14.40	5.42	11.98	4.13
5.0~3.15	10.30	58.78	26.90	2.30	10.47	11.04	14.37	9.57
3.15~2.0	17.71	60.39	27.64	1.77	9.92	19.54	18.56	8.86
2.0~1.0	33.15	61.40	28.10	1.34	12.94	37.16	26.35	21.60
1.0~0.5	22.76	48.07	22.00	0.99	31.38	19.98	13.77	35.95
0.5~0.15	3.75	21.06	9.64	0.91	66.18	1.44	1.80	12.49
-0.15	1.95	41.12	18.82	4.12	19.75	1.47	4.79	1.96
Total	100.00	54.80	25.08	1.67	19.86	100.00	100.00	100.00

Table 4. Size Distribution and Chemical Analysis of Sample FLA-2.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.0	1.17	30.15	13.80	6.08	22.84	0.60	5.30	1.43
8.0~5.0	4.78	51.87	23.74	2.74	17.04	4.31	9.85	4.30
5.0~3.15	11.66	60.92	27.88	1.54	12.49	12.28	13.64	7.76
3.15~2.0	19.12	62.93	28.80	1.22	11.76	20.82	17.42	11.96
2.0~1.0	37.57	64.76	29.64	1.20	11.27	42.10	34.09	22.48
1.0~0.5	20.99	51.04	23.36	0.98	32.43	18.53	15.91	36.13
-0.5	4.71	16.61	7.60	1.02	63.63	1.36	3.79	15.94
Total	100.00	57.80	26.46	1.32	18.82	100.00	100.00	100.00

Table 5. Size Distribution and Chemical Analysis of Sample FLA-3.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.0	13.16	15.82	7.24	14.27	9.12	6.18	20.39	10.90
8.0~5.0	12.12	21.59	9.88	12.80	8.95	7.81	16.81	9.81
5.0~3.15	17.36	30.02	13.74	10.72	8.68	15.56	20.17	13.71
3.15~2.0	19.01	40.34	18.46	7.66	9.26	22.85	15.84	15.99
2.0~1.0	26.01	44.05	20.16	6.36	11.64	34.12	17.90	27.52
1.0~0.5	11.79	37.49	17.16	6.64	18.68	13.15	8.46	19.98
-0.5	0.55	18.40	8.42	7.08	42.06	0.33	0.43	2.09
Total	100.00	33.56	15.36	9.22	11.01	100.00	100.00	100.00

Table 6. Size Distribution and Chemical Analysis of Sample FLA-4.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.0	8.82	46.28	21.18	3.03	22.24	7.73	12.33	9.68
8.0~5.0	9.43	58.60	26.82	1.68	13.32	10.46	7.30	6.23
5.0~3.15	13.91	63.06	28.86	1.37	10.78	16.57	8.68	7.41
3.15~2.0	18.10	64.17	29.37	1.17	10.59	21.98	9.59	9.49
2.0~1.0	21.40	64.15	29.36	1.10	10.76	25.95	10.96	11.36
1.0~0.5	8.19	54.28	24.84	1.39	23.18	8.39	5.02	9.39
0.5~0.15	6.92	29.10	13.32	1.66	50.21	3.80	5.48	17.14
-0.15	13.23	20.41	9.34	6.73	44.79	5.12	40.64	29.30
Total	100.00	52.88	24.20	2.19	20.24	100.00	100.00	100.00

Table 7. Size Distribution and Chemical Analysis of Sample FLA-5.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.0	1.98	41.60	19.04	8.02	13.86	1.43	5.99	2.42
8.0~5.0	6.64	49.60	22.70	5.12	9.57	5.69	12.74	5.75
5.0~3.15	13.49	60.52	27.70	2.78	6.91	14.10	14.23	8.36
3.15~2.0	23.14	62.84	28.76	1.92	9.23	25.10	16.48	19.23
2.0~1.0	34.95	62.80	28.74	1.46	10.58	37.85	19.10	33.24
1.0~0.5	11.01	58.12	26.60	1.61	17.23	11.05	6.74	17.07
0.5~0.15	2.65	45.05	20.62	1.92	33.52	2.07	1.87	8.00
-0.15	6.14	25.70	11.76	9.90	10.69	2.71	22.85	5.93
Total	100.00	57.97	26.53	2.67	11.13	100.00	100.00	100.00

Table 8. Size Distribution and Chemical Analysis of Sample FLA-1 after Being Scrubbed at 50% Solids and 1000 rpm for 10 Minutes.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.00	7.08	49.93	22.85	3.04	18.43	6.42	12.16	7.10
8.00~5.00	5.35	57.03	26.10	2.02	13.32	5.55	6.08	3.88
5.00~3.15	11.16	59.17	27.08	2.30	10.18	11.97	14.36	6.23
3.15~2.00	16.21	61.75	28.26	1.78	9.95	18.16	16.02	8.80
2.00~1.00	29.83	61.53	28.16	1.40	12.45	33.31	23.20	20.27
1.00~0.50	21.01	49.16	22.50	0.95	30.96	18.76	11.05	35.52
0.50~0.10	4.87	26.39	12.08	1.56	57.59	2.34	4.42	15.30
-0.10	4.49	42.83	19.60	5.14	11.83	3.49	12.71	2.90
Total	100.00	55.11	25.22	1.81	18.30	100.00	100.00	100.00

Table 9. Size Distribution and Chemical Analysis of Sample FLA-2 after Being Scrubbed at 50% Solids and 1000 rpm for 10 Minutes.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.00	4.15	58.08	26.58	1.16	18.69	4.21	4.46	4.20
8.00~5.00	3.86	56.11	25.68	2.01	13.24	3.79	7.14	2.75
5.00~3.15	9.33	60.66	27.76	1.12	13.94	9.90	8.93	7.00
3.15~2.00	18.63	63.23	28.94	1.02	10.68	20.61	16.97	10.72
2.00~1.00	35.81	63.45	29.04	0.97	11.60	39.77	31.25	22.36
1.00~0.50	20.35	51.09	23.38	0.72	28.70	18.20	13.39	31.47
0.50~0.10	5.30	18.40	8.42	0.71	70.56	1.72	3.57	20.15
-0.10	2.57	40.25	18.42	6.07	9.84	1.80	14.29	1.35
Total	100.00	57.14	26.15	1.12	18.56	100.00	100.00	100.00

Table 10. Size Distribution and Chemical Analysis of Sample FLA-3 after Being Scrubbed at 50% Solids and 1000 rpm for 10 Minutes.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.00	11.36	14.90	6.82	13.92	9.58	4.94	17.25	8.41
8.00~5.00	7.70	24.78	11.34	11.64	8.34	5.58	9.83	4.94
5.00~3.15	11.25	34.13	15.62	9.58	7.64	11.29	19.21	6.64
3.15~2.00	14.93	45.89	21.00	5.08	8.38	20.14	8.30	9.64
2.00~1.00	25.67	47.59	21.78	5.20	16.64	35.86	14.63	32.95
1.00~0.05	15.84	38.28	17.52	6.04	18.80	17.83	10.48	22.99
0.50~0.16	3.73	17.74	8.12	7.93	33.94	1.92	3.27	9.80
-0.16	9.52	8.78	4.02	16.36	6.30	2.44	17.03	4.63
Total	100.00	34.06	15.50	9.16	12.96	100.00	100.00	100.00

Table 11. Size Distribution and Chemical Analysis of Sample FLA-4 after Being Scrubbed at 40% Solids and 1000 rpm for 10 Minutes.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.00	10.19	56.98	26.08	1.39	16.39	11.04	7.14	8.06
8.00~5.00	8.53	62.84	28.76	0.80	12.78	10.16	3.57	5.26
5.00~3.15	11.70	64.46	29.50	1.12	10.75	14.32	6.63	6.09
3.15~2.00	14.08	66.34	30.36	0.91	8.33	17.76	6.63	5.65
2.00~1.00	18.38	65.62	30.03	0.48	8.50	22.90	4.59	7.53
1.00~0.05	7.50	57.42	26.28	0.62	19.92	8.17	2.56	7.20
0.50~0.16	7.77	35.31	16.16	1.07	52.66	5.23	4.08	19.75
0.16~0.036	4.45	38.24	17.50	1.88	39.40	3.24	4.08	8.45
-0.036	17.40	21.72	9.94	6.83	38.10	7.18	60.72	32.01
Total	100.00	52.66	24.10	1.96	20.71	100.00	100.00	100.00

Table 12. Size Distribution and Chemical Analysis of Sample FLA-5 after Being Scrubbed at 50% Solids and 1000 rpm for 10 Minutes.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+8.00	1.17	51.13	23.40	2.75	16.62	1.03	1.09	1.61
8.00~5.00	5.44	51.26	23.46	4.86	9.43	4.90	9.45	4.32
5.00~3.15	12.95	57.79	26.45	3.09	8.88	13.14	14.55	9.74
3.15~2.00	21.69	62.45	28.58	2.12	8.67	23.75	16.73	15.92
2.00~1.00	34.92	63.26	28.95	1.28	10.66	38.72	16.36	31.50
1.00~0.50	11.09	59.00	27.00	1.34	17.48	11.45	5.45	16.43
0.50~0.10	3.55	41.86	19.16	1.32	39.93	2.61	1.82	11.85
0.10~0.036	0.78	45.89	21.00	1.96	30.26	0.61	0.73	2.03
-0.036	8.41	25.61	11.72	11.10	9.23	3.79	33.82	6.60
Total	100.00	57.05	26.11	2.75	11.81	100.00	100.00	100.00

Table 13. Size Distribution and Chemical Analysis of Sample FLA-1 after Being Crushed to -1 mm.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.50	39.12	55.06	25.20	1.52	19.79	39.82	32.78	39.96
0.50~0.315	19.73	53.36	24.42	1.60	22.21	19.47	17.78	22.61
0.315~0.16	17.47	52.92	24.22	1.53	23.22	17.08	15.00	20.96
0.16~0.10	6.24	53.97	24.70	1.50	20.70	6.22	5.00	6.66
0.10~0.071	3.79	57.55	26.34	1.66	15.35	4.04	3.33	2.99
0.071~0.056	1.59	58.16	26.62	1.68	13.79	1.70	1.67	1.14
0.056~0.036	2.04	59.13	27.06	2.03	12.02	2.22	2.22	1.29
-0.036	10.02	51.02	23.35	3.98	8.45	9.45	22.22	4.39
Total	100.00	54.10	24.76	1.80	19.37	100.00	100.00	100.00

Table 14. Size Distribution and Chemical Analysis of Sample FLA-2 After Being Crushed to –1 mm.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.50	35.68	57.38	26.26	0.99	19.14	36.67	27.91	35.21
0.50~0.315	21.80	53.02	24.54	1.26	24.19	20.83	20.93	27.05
0.315~0.16	18.79	53.40	24.44	1.10	23.86	17.87	16.28	23.00
0.16~0.10	7.14	58.43	26.74	1.12	17.55	7.43	6.20	6.42
0.10~0.071	4.25	61.31	28.06	0.99	13.40	4.63	3.10	2.93
0.071~0.056	1.62	62.71	28.70	1.23	11.39	1.79	1.55	0.92
0.056~0.036	2.03	63.41	29.02	2.02	9.75	2.30	3.10	1.03
-0.036	8.51	56.02	25.64	3.14	7.91	8.48	20.93	3.44
Total	100.00	56.13	25.69	1.29	19.48	100.00	100.00	100.00

Table 15. Size Distribution and Chemical Analysis of Sample FLA-3 After Being Crushed to –1 mm.

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.50	30.67	36.56	16.73	8.22	11.38	35.21	26.47	31.16
0.50~0.315	16.95	34.54	15.81	8.74	12.43	18.39	15.55	18.84
0.315~0.16	18.15	34.54	15.81	7.94	16.40	19.70	15.12	26.61
0.16~0.10	6.18	37.10	16.98	7.24	15.82	7.21	4.73	8.75
0.10~0.071	3.66	39.33	18.00	7.42	11.92	4.53	2.84	3.93
0.071~0.056	2.10	39.59	18.12	8.18	9.14	2.61	1.78	1.69
0.056~0.036	2.39	33.30	15.24	10.23	7.30	2.47	2.52	1.52
-0.036	19.90	15.82	7.24	14.84	4.20	9.88	30.99	7.50
Total	100.00	31.84	14.57	8.52	11.20	100.00	100.00	100.00

Table 16. Size Distribution and Chemical Analysis of Sample FLA-4 After Being Crushed to –1 mm.*

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.50	47.05	64.89	29.70	1.00	10.48	51.27	40.17	31.16
0.50~0.315	14.77	57.95	26.52	0.91	18.50	14.39	11.11	17.26
0.315~0.16	17.19	50.01	22.89	1.22	29.35	14.42	17.95	31.92
0.16~0.10	6.07	52.88	24.20	0.99	23.38	5.39	5.13	8.98
0.10~0.071	2.83	60.50	27.69	1.10	13.74	2.86	2.57	2.47
0.071~0.036	2.91	63.08	28.87	1.16	10.11	3.08	2.56	1.83
-0.036	9.18	55.76	25.52	2.60	11.02	8.59	20.51	6.38
Total	100.00	59.54	27.25	1.17	15.82	100.00	100.00	100.00

* Prior to crushing to –1 mm, the sample was scrubbed and deslimed to remove -0.10 mm slimes.
(scrubbing conditions: 1000 rpm impeller speed, 40% solids and 10 minutes)

Table 17. Size Distribution and Chemical Analysis of Sample FLA-5 After Being Crushed to –1 mm.*

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.50	38.51	62.53	28.62	1.67	10.40	40.21	31.22	34.48
0.50~0.315	19.57	61.16	27.99	1.87	11.23	19.99	18.05	18.92
0.315~0.16	17.69	56.92	26.05	1.70	16.73	16.82	14.63	25.45
0.16~0.10	5.96	57.03	26.10	1.75	17.44	5.69	4.88	8.94
0.10~0.071	3.89	60.87	27.86	1.82	11.57	3.94	3.41	3.87
0.071~0.036	3.54	61.22	28.02	2.02	9.20	3.61	3.42	2.84
-0.036	10.84	53.86	24.65	4.60	5.91	9.74	24.39	5.50
Total	100.00	59.89	27.41	2.05	11.63	100.00	100.00	100.00

* Prior to crushing to –1 mm, the sample was scrubbed and deslimed to remove -0.10 mm slimes. (scrubbing conditions: 1000 rpm impeller speed, 40% solid and 10 minutes)

through 17, the following conclusions can be made:

- No acceptable phosphate concentrate could be obtained simply by mechanical procedures like sizing, scrubbing, desliming, etc.
- Samples FLA-4 and FLA-5 contained a considerable amount of slimes. Part of the MgO content could be rejected at quite low P_2O_5 loss by scrubbing and desliming. Therefore it is necessary to scrub and deslime those two samples before grinding and flotation.
- After crushing to -1 mm, the MgO content was much higher in fine sizes than in coarse fraction, especially in samples FLA-2 and FLA-4. It was indicated that dolomite was more fragile than phosphate and silica.

MINERAL CHARACTERIZATION

Florida phosphate deposits were formed through marine chemical and biogenic deposition in the Tertiary Formation about 10 to 15 million years ago. Due to their quite young geological age, the lithification action was weak and the phosphate ore occurred as sandy aggregates. Most of the five samples contain about 25% P_2O_5, with the exception of 14.67% for FLA-3. All of them come from middle- to low-grade phosphate ore and must be upgraded for any utilization. The deleterious materials in the samples include MgO, Al_2O_3, Fe_2O_3, SiO_2, CaO, etc. in different amounts. The impurity minerals to be rejected consist of dolomite, calcite, silica, clay and others. Mineralogical studies should be made to gain a further understanding of micro-structures and interior textures of the samples in order to provide a basis for selecting a reasonable beneficiation process.

Mineralogical characterization studies were made on the samples aimed at the determination of their actual mineralogical composition, the interparticle relationship between different minerals, and the liberation degree of the minerals. All of the five samples were examined by X-ray diffraction (XRD) and polarized light microscopy (PLM) techniques.

X-ray Diffraction Analysis

X-ray diffraction analysis was made to identify the major mineral composition of the samples and to determine the unit cell parameters for quantifying the maximum theoretical P_2O_5 grade of the phosphate mineral.

Table 18 indicates the unit cell parameters, diffraction index and theoretical specific gravity of the phosphate mineral in each sample. The theoretical formula of the phosphate mineral in the samples is listed in Table 19. The chemical composition of the pure phosphate mineral calculated from the formula is given in Table 20, while the mineral composition of each sample is shown in Table 21. X-ray analysis results indicated that in the five samples tested there was no significant difference in the unit cell parameters, theoretical formula, chemical composition and mineral components of pure phosphate mineral.

Table 18. Unit Cell Parameters, Refractive Index and Specific Gravity of the Apatite Mineral in Each Sample.

Samples	Unit Cell Parameters		Refractive Index	Theoretical S.G (g/cm^3)
	a°	b°		
FLA-1	9.32816	6.89511	1.6022	3.11
FLA-2	9.32753	6.89476	1.6021	3.11
FLA-3	9.33156	6.89423	1.6030	3.11
FLA-4	9.33182	6.89994	1.6040	3.11
FLA-5	9.32980	6.89468	1.6030	3.11

Table 19. Theoretical Formula of the Apatite Mineral in Each Sample.

Samples	Theoretical Formula of Apatite
FLA-1	$Ca_{9.5934}Na_{0.2929}Mg_{0.1137}(PO_4)_{4.915}(CO_3)_{1.085}F_{2.434}$
FLA-2	$Ca_{9.5880}Na_{0.2970}Mg_{0.1150}(PO_4)_{4.902}(CO_3)_{1.098}F_{2.439}$
FLA-3	$Ca_{9.6270}Na_{0.2690}Mg_{0.1040}(PO_4)_{4.990}(CO_3)_{1.010}F_{2.400}$
FLA-4	$Ca_{9.6299}Na_{0.2666}Mg_{0.1035}(PO_4)_{5.000}(CO_3)_{1.000}F_{2.400}$
FLA-5	$Ca_{9.6100}Na_{0.2810}Mg_{0.1090}(PO_4)_{4.951}(CO_3)_{1.049}F_{2.4196}$

According to the method practiced in China, these phosphate minerals are usually named as carbonate-fluorapatite, corresponding to francolite.

Table 20. Chemical Composition of Pure Apatite Mineral in Each Sample.

Samples	Chemical Composition of Pure Apatite (%)						
	P_2O_5	CaO	CO_2	MgO	Na_2O	F^-	F^-=O
FLA-1	35.90	55.34	4.91	0.47	0.93	4.75	2.00
FLA-2	35.82	55.33	4.97	0.48	0.95	4.77	2.00
FLA-3	36.35	55.39	4.65	0.43	0.86	4.68	1.97
FLA-4	36.41	55.38	4.51	0.43	0.85	4.68	1.97
FLA-5	36.11	55.37	4.74	0.45	0.89	4.72	1.99

Table 21. Mineral Composition of Florida Dolomite Pebble Samples.

Samples	Mineral Composition (%)				
	Phosphate	Carbonates	Silicates	Clays	Others
FLA-1	64	15	16.5	3.5	1
FLA-2	68	11	16.5	3.5	1
FLA-3	39	49	9	2	1
FLA-4	69	11	14	4	2
FLA-5	70	15.5	9	3.5	2

In the five samples, there were two types of phosphate minerals. One is carbonate fluorapatite, which corresponds to what is usually called francolite in the USA, accounting for up to 98% of the total phosphate. This type of phosphate mineral has the characteristics of poor crystallinity, low diffraction index, specific gravity, unit cell parameters and theoretical P_2O_5 grade. Part of the PO_4^{-3} was substituted with CO_3^{-2} and some CO_2, MgO, Na_2O, etc. CLDRI's long experience in phosphate beneficiation research has shown that this type of phosphate mineral has poor floatability compared with well-crystallized fluorapatite. Another type of phosphate mineral is bioclaustics apatite, accounting for 2% of the total phosphate in the sample.

Polarized Light Microscopic Study

Polarized light microscopy (PLM) technique was used to examine and point-count polished sections of different size fractions of the samples for determining the free and locked mineral constituents or phase in each size fraction. The samples used for microscopic studies were first crushed to -1 mm, then sized to several fractions to make polished sections. The polished sections were then examined and point-counted under a microscope. The percentages of the liberated and interlocked phosphate and carbonate in each size fraction of the samples are listed in Tables 22 and 23. The existing sizes of phosphate and carbonate minerals in the as-received samples are presented in Table 24.

DESCRIPTION OF MINERAL TEXTURES

Phosphate Minerals

Polarized light microscopy (PLM) was used to determine the natural types of the phosphate minerals in terms of their textures in the samples. The results indicated that there were nine types of textures for the phosphate minerals observed, including sandy, massive, granular, dolomitic, granulated sandy, sandy dolomitic, granulated sandy dolomitic, sandy argillaceous, and bioclastics. They are briefly described as follows:

- **Sandy Phosphate.** In this texture, quartz clastics were cemented by francolite. The particle sizes of quartz clastics averaged 0.1 to 0.3 mm, with a maximum of 0.4 to 1.0 mm and a minimum of 0.02 to 0.05 mm. The content of phosphate minerals varied from 10% to 80%.

- **Massive Phosphate.** Francolite particles were in granular, platy and other irregular shapes of different sizes. Some francolite particles contained a small amount of fine-grained quartz or were thin-shelled by carbonate minerals. Francolite particles were usually in sizes of 1-4 mm.

- **Granular Phosphate.** Francolite granules were cemented by phosphate mineral. Such granules were usually in sizes of 0.2 to 1.5 mm. Some of them contained quartz clastics.

Table 22. Microscopic Determination of Liberated Phosphate in Different Size Fractions after Crushing the Samples to –1 mm.

	FLA-1			FLA-2			FLA-3			FLA-4			FLA-5		
Size (mm)	Free Phos. (%)[a]	Locked [b] Phosphate		Free Phos. (%)[a]	Locked Phosphate		Free Phos. (%)[a]	Locked Phosphate		Free Phos. (%)[a]	Locked Phosphate		Free Phos. (%)[a]	Locked Phosphate	
		>50%	<50%		>50%	<50%		>50%	<50%		>50%	<50%		>50%	<50%
+0.500	80	15	5	79	19	2	73	24	3	82	16.6	1.4	83	15	2
0.500~0.315	85	14	1	86	13	1	78	19	3	86	13	1	84	14.5	1.5
0.315~0.160	93	6	1	91	8	1	84	13	3	90	9	1	90	9	1
0.160~0.100	97	2	1	94	5	1	86	11	3	90	9.5	0.5	90	9	1
0.100~0.071	98	2	0	96	3	1	87	11	2	95	4.5	0.5	94	5	1
0.071~0.056	99	1	0	96	3	1	89	8	3	97	2	1	95	4	1
0.056~0.036	99	1	0	98	2	0	92	6	2						
-0.036	100	0	0	99	1	0	98	1	1	97	2	1	98	2	0

a: Liberated phosphate: The particles with over 90% phosphate were considered as liberated or free phosphate.

b: The particles with less than 90% phosphate were considered as locked. Those containing over 50% phosphate were classified as high-grade intergrowth, and those with less 50% phosphate as low-grade ones.

Table 23. Microscopic Determination of Liberated Dolomite in Different Size Fractions After Crushing the Samples to –1 mm.

Size (mm)	FLA-1			FLA-2			FLA-3			FLA-4			FLA-5		
	Free Dolo. (%)	Locked Dolomite		Free Dolo. (%)	Locked Dolomite		Free Dolo. (%)	Locked Dolomite		Free Dolo. (%)	Locked Dolomite		Free Dolo. (%)	Locked Dolomite	
		>50%	<50%		>50%	<50%		>50%	<50%		>50%	<50%		>50%	<50%
+0.500	86	9	5	78	7	15	95	3	2	93	6	1	83	8	9
0.500~0.315	90	6	4	85	10	5	96	3	1	93	6	1	83	11	6
0.315~0.160	92	5	3	87	8	5	96	2	2	94	5	1	87	7	6
0.160~0.100	92	5	3	88	5	7	96	2	2	96	3	1	87	7	6
0.100~0.071	93	5	2	94	3	3	96	2	2	97	1	2	87	6	7
0.071~0.056	93	5	2	94	2	4	96	3	1	98	1	1	93	4	3
0.056~0.036	94	2	4	95	2	3	98	1	1						
-0.036	97	2	1	97	2	1	99	1	0	99	1	1	95	3	2

Note:

1. Carbonate mineral intergrowth is usually that carbonate minerals are densely or sparsely disseminated in francolite. Further grinding could not improve the liberation significantly.
2. The percentage of liberated carbonates in -0.036 mm was estimated based on the trend of the results determined for coarser fractions.
3. Particles containing over 50% carbonates are considered as carbonate-rich intergrowth, and those with less 50% carbonates as low-carbonate intergrowth.

Table 24. Size Distribution of Phosphate and Carbonate Minerals in the As-received Samples.

Size (mm)	Phosphate Distribution (%)					Carbonate Distribution (%)				
	FLA-1	FLA-2	FLA-3	FLA-4	FLA-5	FLA-1	FLA-2	FLA-3	FLA-4	FLA-5
+1.65	19.9	11.3	26.4	24.0	20.6	21.5	32.1	37.6	22.5	18.4
1.65~1.00	21.8	32.5	24.5	20.3	33.6	6.5	21.1	24.3	3.7	9.8
1.00~0.50	33.1	32.2	27.9	29.5	22.3	23.8	17.0	22.4	19.0	16.2
0.50~0.31	14.6	12.3	11.5	12.1	12.5	6.0	10.4	7.9	22.8	15.5
0.31~0.10	8.1	9.2	6.5	11.3	8.4	14.8	12.1	5.3	21.9	21.9
-0.10	2.5	2.5	3.2	2.8	2.6	27.4	7.3	2.5	10.1	18.2
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0
Average Size(mm)	0.55	0.55	0.58	0.54	0.59	0.18	0.42	0.74	0.29	0.22

- **Dolomitic Phosphate.** Phosphate mineral and dolomite cemented by small amount of quartz clastics and francolite granules; dolomite also disseminated in francolite or thin-shelled francolite granules.

- **Granulated Sandy Phosphate.** It is similar to the granular texture with the exception that there were more quartz clastics. Both francolite granules and quartz clastics were in sizes of about 0.2 and 0.3 mm.

- **Sandy Dolomitic Phosphate.** Fine-grained dolomite and francolite cemented by quartz clastics in size of 0.1 to 0.3 mm. Dolomite disseminated in francolite in very fine particles, about 0.01 mm or finer. Some dolomite thin-shelled francolite in a thickness around 0.1 mm.

- **Granulated Sandy Dolomitic Phosphate.** This could only be observed in sample FLA-3. In such a texture, dolomite was cemented by quartz clastics and francolite granules, both of which were in size of 0.2 to 0.4mm.

- **Sandy Argillaceous Phosphate.** This was only seen in sample FLA-4. It was so named due to its being sandy francolite containing a significant amount of clayey minerals.

- **Bioclastics Phosphate.** Fossil shark teeth and fish bones could be observed. Some of them were substituted with francolite.

Gangue Minerals

The gangue minerals in the samples could be catalogued into five types in terms of the textures, including fine-grained dolomite, sandy dolomite, siliceous sand, sandy argillaceous dolomite and clays.

- **Fine-grained Dolomite.** This consisted of crypto- and micro-crystalline dolomite and small amount of calcite in size of 0.01mm. They were intergrown with each other. Such dolomite had the features of light specific gravity, homogeneous mass and smooth structure.

- **Sandy Dolomite.** There were some fine quartz clastics included in fine-grained dolomite.

- **Siliceous Sand.** There were two types, one was crystal silica in round and isometric granular shapes in size of 0.5 to 2.5 mm with maximum up to 5mm, and another was fine-grained allotriomorphic silicates intergrown with each other to form quartzite.

- **Sandy Argillaceous Dolomite.** Some quartz and other clastics were cemented by fine-grained and clay minerals.

Clays. Clay minerals like kaolin, montmorillonite cemented small amount of quartz, feldspar and other clastics, which occurred as soft-aggregates in yellow-white ovoid shape.

DESCRIPTION OF THE OCCURRENCE OF MAJOR MINERALS

Francolite

There were five types of occurrences of francolite mineral:

- Bulk agglomerates.
- Cement base cementing quartz clastics and francolite particles.
- Granular clastics cemented by francolite, carbonate and clay minerals.
- Cement base with carbonate minerals to cement quartz clastics.
- Mixed and disseminated with carbonate minerals.

In the last two cases, the phosphate mineral was disseminated with carbonates. It is very difficult or even impossible to separate dolomite from phosphate with flotation or any mechanical methods.

Carbonate Minerals

There were six types of occurrences of carbonate minerals, as observed under a microscope:

- Dolomite interlocked with small amount of calcite to form fine-grained dolomite.
- Micro-particles disseminated in francolite to form the cement base cementing quartz clastics.
- Coated on other minerals to form a thin shell.
- Granular, flat and villous particle aggregates distributed in the cement of francolite and dolomite.
- Interlocked with fine-grained quartz to form sandy dolomite.
- Very fine particles disseminated in francolite.

It could be predicted that in cases 1, 3 and 5, dolomite can be separated from phosphate through the flotation process, whereas in cases 2, 4 and 6, it will be difficult or impossible to remove because it is too hard to liberate dolomite from other minerals even when the ore is ground to very fine size.

Silicate Minerals

There were three types of occurrences of silicate impurities:

- Unit silica crystal in isometric, round-like particles
- Allothimorphic quartz in medium size particles and interlocked with each other.
- Clastics cemented by francolite and carbonate minerals.

From the mineralogical study results, the goal of the beneficiation research was determined to be the rejection of both MgO and silica impurities in order to achieve acceptable phosphate concentrate from Florida dolomitic pebble samples. There should be no problem removing the quartz impurity since it was well crystallized in quite coarse particle size. However, removing MgO is different. The MgO impurity is extremely difficult to reject in the following cases: (1) when the MgO exists in the lattices of the phosphate mineral, and (2) when the MgO-bearing minerals like dolomite are disseminated in phosphate ore in very fine particles.

TEST EQUIPMENT, FLOTATION REAGENTS, AND WATER

TEST EQUIPMENT

Grinding

An XMG-63 rod mill made in China was used in the testing. According to the liberation size of each sample as determined by mineralogical studies, the grinding fineness required was controlled by adjusting suitable grinding time. The dimensions of the mill are 105 x 135 mm (inside diameter x length of shell). The size, quantity, weight and percentage of the rods are as follows:

Diameter (mm)	Quantity	Weight (g)	%
17	7	1898.8	39.8
15	4	827.3	17.3
13	3	490.2	10.3
11	13	1556.7	32.6
Total	27	4773.0	100.0

The solid in grinding was 60% for all the samples except 50% for FLA-4.

Flotation

XFD-0.5L and XFD-0.75L flotation cells made in China and a Denver D-12 cell made in the USA were used for flotation tests. The volumes of the cells are:

XFD-0.5	0.5L
XFD-0.75	0.75L
Denver D-12	1.5L

Scrubbing

A Denver D-12 attrition box made in the USA was used for scrubbing the samples.

pH Meter

A meter by Orion Research was used to measure the pH of the flotation slurry during the flotation testing.

FLOTATION REAGENTS

The following flotation reagents were used in flotation testing:

- Phosphoric acid (H_3PO_4): As pH modifier and phosphate depressant, wet process acid with 28% P_2O_5, diluted to 5% P_2O_5 solution for addition.

- Sulfuric acid (H_2SO_4): As pH modifier, diluted to 5% solution for addition.

- Soda ash (Na_2CO_3): As pH modifier, diluted to 5% solution for addition.

- Caustic soda (NaOH): As pH modifier, diluted to 5% solution.

- Water glass (Na_2SiO_3): As silicate depressant, diluted to 5% solution.

- PA-31: As dolomite collector, manufactured by CLDRI, diluted to 2% solution.

- Fatty acid: Collector, provided by IMC-Agrico, saponified and diluted to 2% solution.

- Amine: As silica collector, provided by IMC-Agrico, diluted to 2% solution for addition.

- Fuel oil: As collector extender, provided by IMC-Agrico.

- Kerosene: As collector extender, provided by IMC-Agrico.

TEST WATER

Local tap water was used in the testwork. The typical water analysis was 12 mg/l $CO_3^{=}$, 247 mg/l HCO_3^{-}, 25-56 mg/l Ca^{++}, 18-27 mg/l Mg^{++}, 112 mg/l soluble salt and pH 6.88.

PROCESS DEVELOPMENT STUDIES

The beneficiation process was determined based on the characteristics of the test samples, particularly the composition of the minerals and their relationship, and on the final results to be expected. In order to obtain concentrate analyzing over 30% P_2O_5 and under 1% MgO, and to achieve an overall P_2O_5 recovery rate of more than 80% from the dolomitic pebble samples received, the sample must be ground to a given size for mineral liberation, then subjected to flotation for rejection of both dolomite and silica impurities. In order to maintain high P_2O_5 recovery in the flotation process, the phosphate in fine fraction must be recovered.

In light of the characteristics of Florida dolomitic phosphate pebble samples, a two-stage flotation process was decided upon. In this process, dolomite was floated first, followed by phosphate flotation or silica flotation. The two-stage flotation process has been investigated by many researchers and some achievements have been obtained. In those studies, the ground sample was usually deslimed to remove fine slimes prior to flotation. Although an acceptable flotation concentrate could be produced, the overall P_2O_5 recoveries of those processes were rather low, only 25 to 60%. With the CLDRI fine particle beneficiation technology, flotation was conducted without desliming the feed, and the high-quality concentrate could be achieved at very high overall P_2O_5 recovery, usually up to 80 to 90%.

In CLDRI's laboratory, an XMG-63 rod mill was used to grind the sample to the required fineness, and flotation tests were carried out on 200-gram batches using an XFD-0.5L flotation machine or on 400-gram batches using a Denver D-12 flotation cell.

CONDITIONAL TESTS

The key issue of the flotation process recommended was dolomite flotation. The factors affecting dolomite flotation included feed fineness, acid consumption, conditioning time, etc. The feed fineness determines the liberation degree of phosphate from other gangue minerals, particularly dolomite. The acid consumption affects the separation efficiency and the reagent costs of the flotation process. Therefore, the conditional tests were conducted on sample FLA-1 with the flotation flowsheet shown in Figure 2.

Tests on Grinding Fineness

The test sample must be ground to a given size for liberating phosphate from gangue minerals, which can be considered as the basic precondition for achieving good separation performances. The reasonable grinding fineness depends on the occurrence of the minerals in the sample and the quality and the recovery of the final concentrate to be expected. It could be preliminarily predicted by measuring the interlocking sizes of major minerals and finally determined by flotation testing.

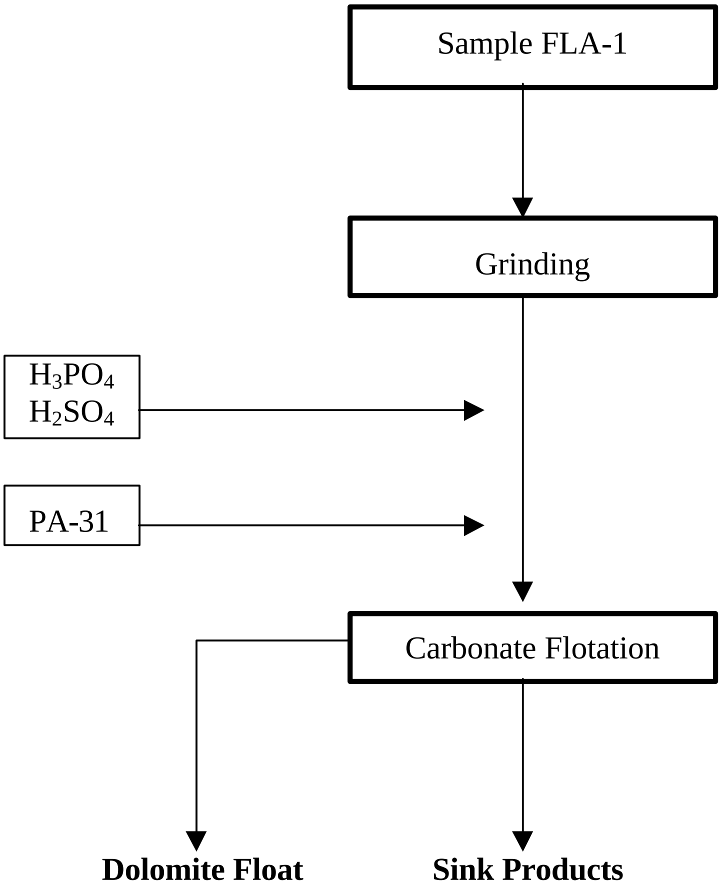

Figure 2. Flowchart for Tests on Grinding Fineness, Reagent Consumption and Conditioning Time for Sample FLA-1.

Figures 3 through 7 show the correlation curves of the grinding time as function of fineness (weight percentage of -200 mesh) for each sample. From the curves, it can be seen that the specified fineness can be obtained by control of the grinding time. The suitable fineness for a given sample was determined by liberation size investigation and flotation testing.

The results of dolomite flotation with differing degrees of feed fineness are summarized in Table 25. From Table 25, it can be seen that at the same reagent consumption, more MgO was rejected at higher P_2O_5 loss as the flotation feed got finer, while coarser flotation feed led to low MgO removal rate. If coarser feed was used, the reagent consumption had to be increased in order to reject more MgO into the float. It is quite important to determine the reasonable grinding fineness by taking both grinding and reagent costs into account. For sample FLA-1, the particle size of flotation feed recommended was 55.33% -200 mesh (equivalent to passing 0.2 mm), which was very close to the average distribution size of carbonate minerals in sample FLA-1 shown in Table 24.

Tests on Acid Consumption

Phosphoric acid consumption was one of the most sensitive parameters in dolomite flotation. It also contributed to a significant part of the total reagent cost of the process. It is very important to define the suitable phosphoric acid consumption, allowable fluctuation scope, and the possible amount to be substituted with other inexpensive acids. Table 26 presents the test results of phosphoric acid consumption for carbonate flotation. The test results indicated that phosphoric acid dosage was a very important factor in affecting the carbonate flotation process. Higher phosphoric acid consumption can improve the efficiency of separating dolomite from phosphate, but it will also cause some problems like collector usage increase, greater corrosion of process equipment due to low pH, high operation cost, etc.

From Table 26, it can be observed that the suitable phosphoric acid consumption for sample FLA-1 was 4.31 kg (P_2O_5) per ton of feed. Since phosphoric acid is rather expensive compared with other inorganic acids, further tests were conducted on substituting part of the phosphoric acid with sulfuric acid in an attempt to lower overall process reagent costs. Table 27 shows the test results of using mixed acids in carbonate flotation. As usage of sulfuric acid increases, the separation of dolomite from phosphate becomes less selective. Considering both separation performances and reagent costs, the appropriate ratio of phosphoric acid to sulfuric acid was determined to be 1.25:1.00 for dolomite flotation of sample FLA-1.

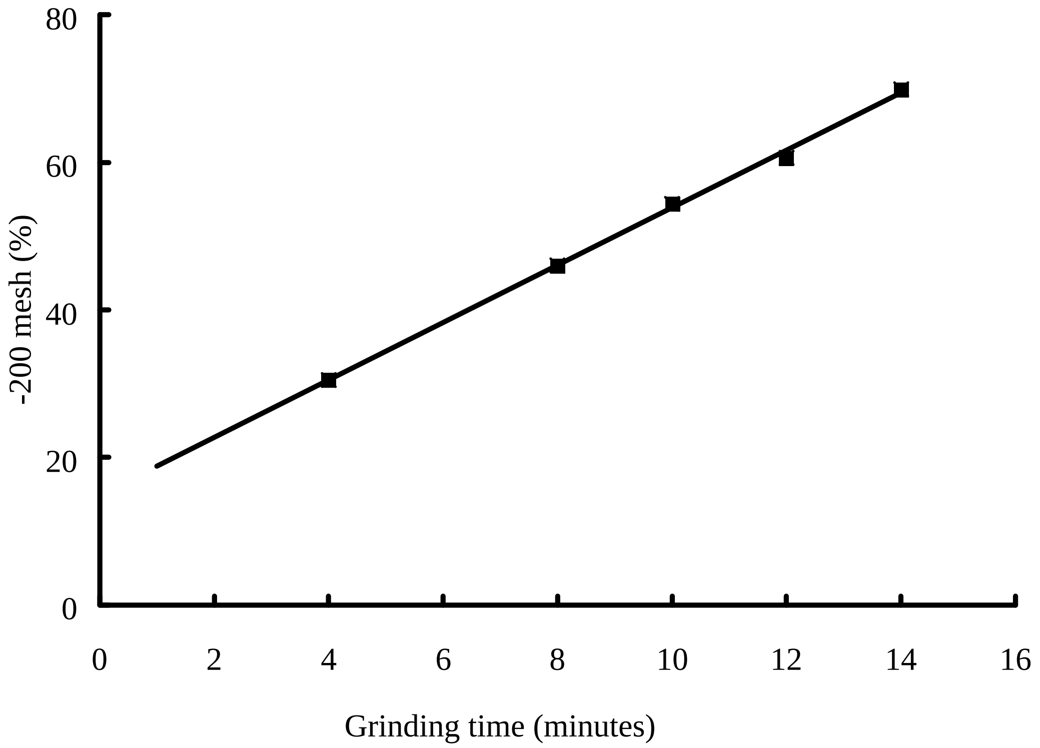

Figure 3. Percentage of -200 Mesh Fraction in Ground Product as Function of Grinding Time for Sample FLA-1.

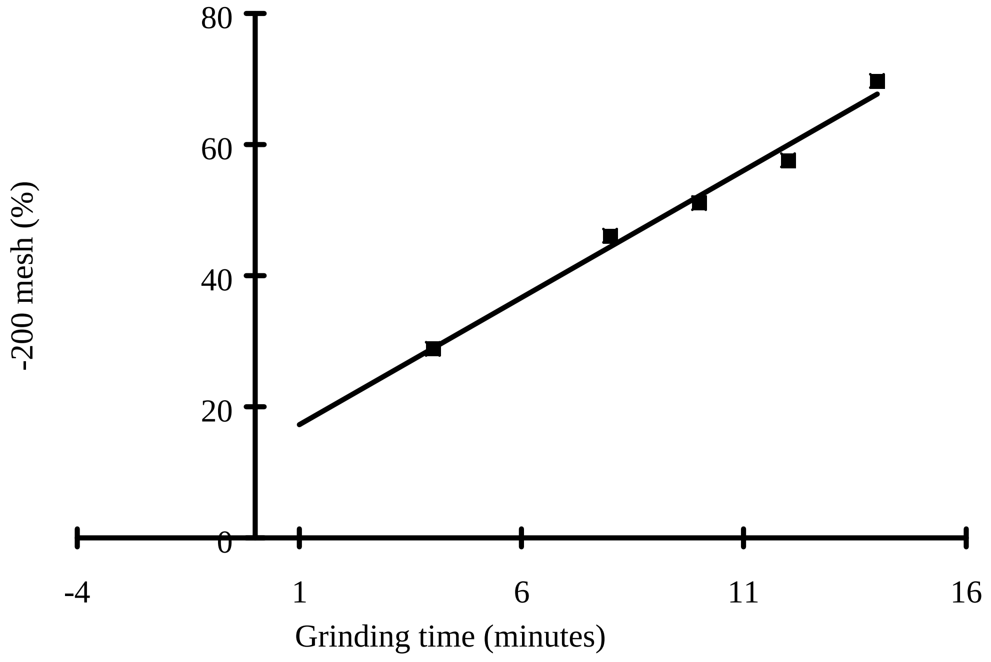

Figure 4. Percentage of -200 Mesh Fraction in Ground Product as Function of Grinding Time for Sample FLA-2.

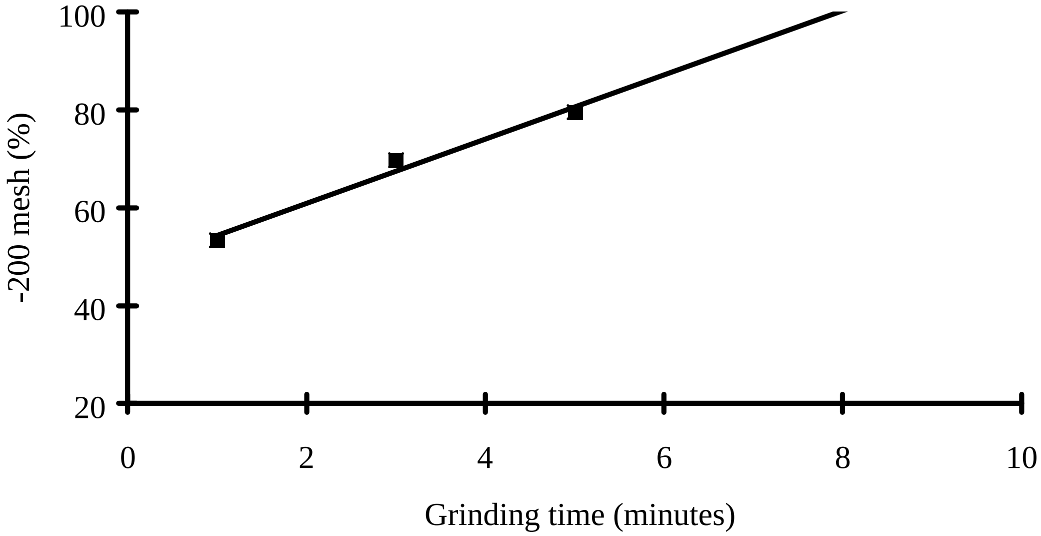

Figure 5. **Percentage of -200 Mesh Fraction in Ground Product as Function of Grinding Time for Sample FLA-3.**

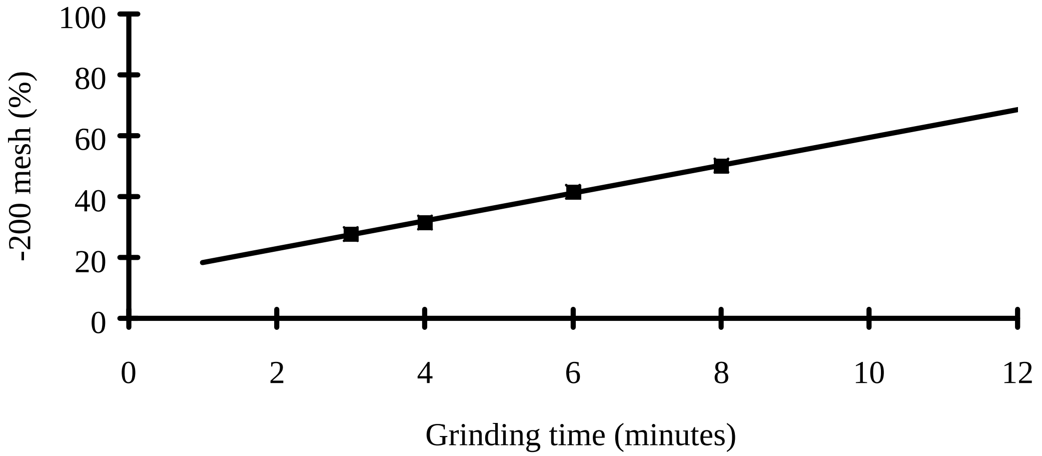

Figure 6. **Percentage of -200 Mesh Fraction in Ground Product**
Grinding Time for Sample FLA-4.

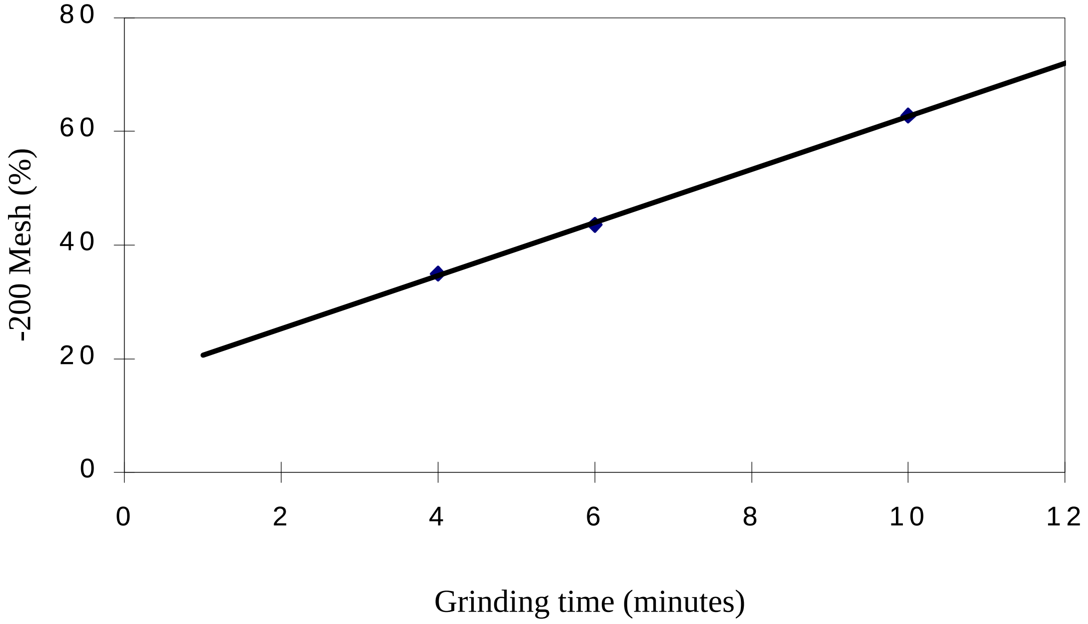

Figure 7. **Percentage of -200 Mesh Fraction in Ground Product as Function of Grinding Time for Sample FLA-5.**

Table 25. Flotation Results on Sample FLA-1 with Different Particle Sizes of Feed.

Reagent Dosage (lb./t)	Feed Size %-200 Mesh	Product	Weight (%)	Analysis (%)		Recovery (%)	
				P_2O_5	MgO	P_2O_5	MgO
H_3PO_4:8.62 (P_2O_5) H_2SO_4:0.00 PA-31:2.0	(8`) 45.99	Float	15.51	18.76	6.90	11.69	58.15
		Sink	84.49	26.03	0.91	88.31	41.85
		Feed	100.00	24.90	1.84	100.00	100.00
	(10`) 55.33	Float	18.80	19.31	6.54	14.64	66.13
		Sink	81.20	26.07	0.77	85.36	33.87
		Feed	100.00	24.80	1.86	100.00	100.00
	(12`) 60.61	Float	20.83	20.07	5.95	16.96	68.13
		Sink	79.17	25.86	0.73	83.04	31.87
		Feed	100.00	24.65	1.82	100.00	100.00
	(14`) 69.89	Float	25.06	20.71	5.19	20.91	71.82
		Sink	74.94	26.20	0.68	79.09	28.18
		Feed	100.00	24.82	1.81	100.00	100.00

Table 26. Effects of H_3PO_4 Consumption on Dolomite Flotation of Sample FLA-1.*

Reagent Dosage (lb./t Feed)		Product	Weight (%)	Analysis (%)		Recovery (%)	
H_3PO_4 (P_2O_5)	PA-31			P_2O_5	MgO	P_2O_5	MgO
3.08	1.0	Float	25.05	21.34	5.10	21.68	66.15
		Sink	74.95	25.89	0.87	78.32	33.85
		Feed	100.00	24.77	1.92	100.00	100.00
3.69	1.0	Float	21.04	20.43	5.43	17.31	61.29
		Sink	78.96	26.01	0.91	82.69	38.72
		Feed	100.00	24.84	1.86	100.00	100.00
4.31	1.0	Float	18.80	19.34	6.51	14.64	66.13
		Sink	81.20	26.07	0.77	85.36	33.87
		Feed	100.00	24.80	1.86	100.00	100.00
5.93	1.0	Float	15.31	18.88	6.91	11.58	60.92
		Sink	84.69	26.06	0.80	88.42	39.08
		Feed	100.00	24.96	1.74	100.00	100.00

* Particle size of flotation feed: 55.33% -200 mesh.

Table 27. Effects of H_3PO_4 to H_2SO_4 Ratio on Dolomite Flotation of Sample FLA-1.*

Reagent Dosage (lb./t feed)			Product	Weight (%)	Analysis (%)		Recovery (%)	
$H_3PO_4(P_2O_5)$	H_2SO_4	PA-31			P_2O_5	MgO	P_2O_5	MgO
8.6 (100%)	0 (0%)	2.0	Float	18.80	19.31	6.54	14.64	66.13
			Sink	81.20	26.07	0.77	85.36	33.87
			Feed	100.00	24.80	1.86	100.00	100.00
5.0 (56%)	4.0 (44%)	2.0	Float	18.45	20.20	5.85	15.35	61.34
			Sink	81.55	25.21	0.87	84.65	39.66
			Feed	100.00	24.29	1.79	100.00	100.00
4.0 (40%)	6.0 (60%)	2.0	Float	16.94	20.44	5.84	14.16	55.00
			Sink	83.06	25.26	0.98	85.84	45.00
			Feed	100.00	24.44	1.80	100.00	100.00
2.0 (20%)	4.0 (80%)	2.0	Float	16.58	21.35	4.95	14.33	46.59
			Sink	83.42	25.36	1.13	85.67	53.41
			Feed	100.00	24.70	1.76	100.00	100.00

* Particle size of flotation feed: 55.33% -200 mesh.

Tests on Conditioning Time

Based on CLDRI experience, the conditioning time of carbonate flotation has a direct effect on separation selectivity. Therefore, flotation tests were carried out to determine the suitable conditioning time to obtain optimum separation performances. The conditioning time represented the period between the addition of one reagent and another, or from the addition of the last reagent to the opening of the air valve to generate bubbles. The test results with different conditioning times are given in Table 28. The data indicate that longer conditioning time gives rise to high P_2O_5 and low MgO in dolomite flotation, which means poor separation selectivity. Therefore it is recommended that the conditioning time in dolomite flotation should be less than 1 minute, usually 30 seconds in the afterward testing.

FLOTATION TESTS ON SAMPLE FLA-1

It can be seen from the chemical composition of sample FLA-1 that about 65% MgO must be rejected to obtain a phosphate concentrate of less than 1% MgO at an overall P_2O_5 recovery exceeding 80%. To achieve this goal, different flotation processes were tried, including:

- Grinding-dolomite flotation-phosphate flotation;
- Grinding-dolomite flotation-sizing-phosphate flotation;
- Grinding-dolomite flotation-sizing-silica flotation.

According to the conditional tests, the optimal conditions for dolomite flotation of sample FLA-1 were: flotation feed size 55.33% -200 mesh (equivalent to 0.2 mm); phosphoric acid consumption 5.0 lb per ton of feed; sulfuric acid 4.0 lb per ton of feed, and PA-31 1.6 lb per ton of feed.

The size distribution of the flotation feed is shown in Table 29. It can be seen that the P_2O_5 grade is quite homogeneous in all size fractions while fine size contains much higher MgO content. Considerable MgO can be eliminated by desliming the flotation feed at a given cut-off size such as 400 mesh prior to flotation, but a significant amount of P_2O_5 will also be lost in the slimes. In order to ensure higher overall P_2O_5 recovery, all the ground slurry was subjected to flotation with no desliming.

For separation of silica from phosphate, both phosphate flotation using fatty acid as the collector and silica flotation using amines were tested. The appropriate reagent dosages were determined during the experiment.

The flowcharts of the flotation processes tested, including “grinding-dolomite flotation-phosphate flotation,” “grinding-dolomite flotation-sizing phosphate flotation,” and “grinding-dolomite flotation-sizing-silica flotation” are shown in Figures 8, 9 and 10, and the test results are listed in Tables 30, 31 and 32, respectively.

Table 28. Effects of Conditioning Time on Dolomite Flotation of Sample FLA-1 .[a]

Reagent	Conditioning Time (Seconds)			Product	Weight	Analysis (%)		Recovery (%)	
(lb./t feed)	Acids [b]	PA-31[c]	Aeration[d]		(%)	P_2O_5	MgO	P_2O_5	MgO
H_3PO_4: 5.0 (P_2O_5) H_2SO_4: 4.0 PA-31: 2.4	15	15	15	Float	19.37	20.34	5.99	16.18	63.39
				Sink	80.63	25.31	0.83	83.82	36.61
				Feed	100.00	24.35	1.83	100.00	100.00
	30	30	15	Float	18.01	19.98	5.87	14.63	59.55
				Sink	81.93	25.70	0.89	85.37	40.45
				Feed	100.00	24.67	1.78	100.00	100.00
	60	60	15	Float	20.02	20.48	5.73	16.84	62.84
				Sink	79.98	25.32	0.85	83.16	37.16
				Feed	100.00	24.35	1.83	100.00	100.00
	120	120	15	Float	22.11	21.55	5.34	19.39	63.79
				Sink	77.89	25.46	0.86	80.61	36.21
				Feed	100.00	24.60	1.85	100.00	100.00

a: Particle size of flotation feed: 55.33% -200 mesh;
b: Agitation time after addition of acids;
c: Agitation time after addition of PA-31;
d: Time period between opening air-valve and skimming froth.

Table 29. Size Analysis of Flotation Feed for Sample FLA-1. (Grinding time: 10 Minutes, 55.33% -200 Mesh)

Size (mm)	Wt. (%)	Analysis (%)			Distribution (%)	
		BPL	P_2O_5	MgO	BPL	MgO
+0.160	1.63	47.9	21.92		1.47	
0.160~0.100	19.49	52.3	23.92		19.01	
0.100~0.071	23.55	52.5	24.05		23.08	
0.071~0.036	16.70	55.7	25.49		17.37	
-0.036	38.63	54.2	24.80		39.07	
Total	100.00	53.6	24.52		100.00	

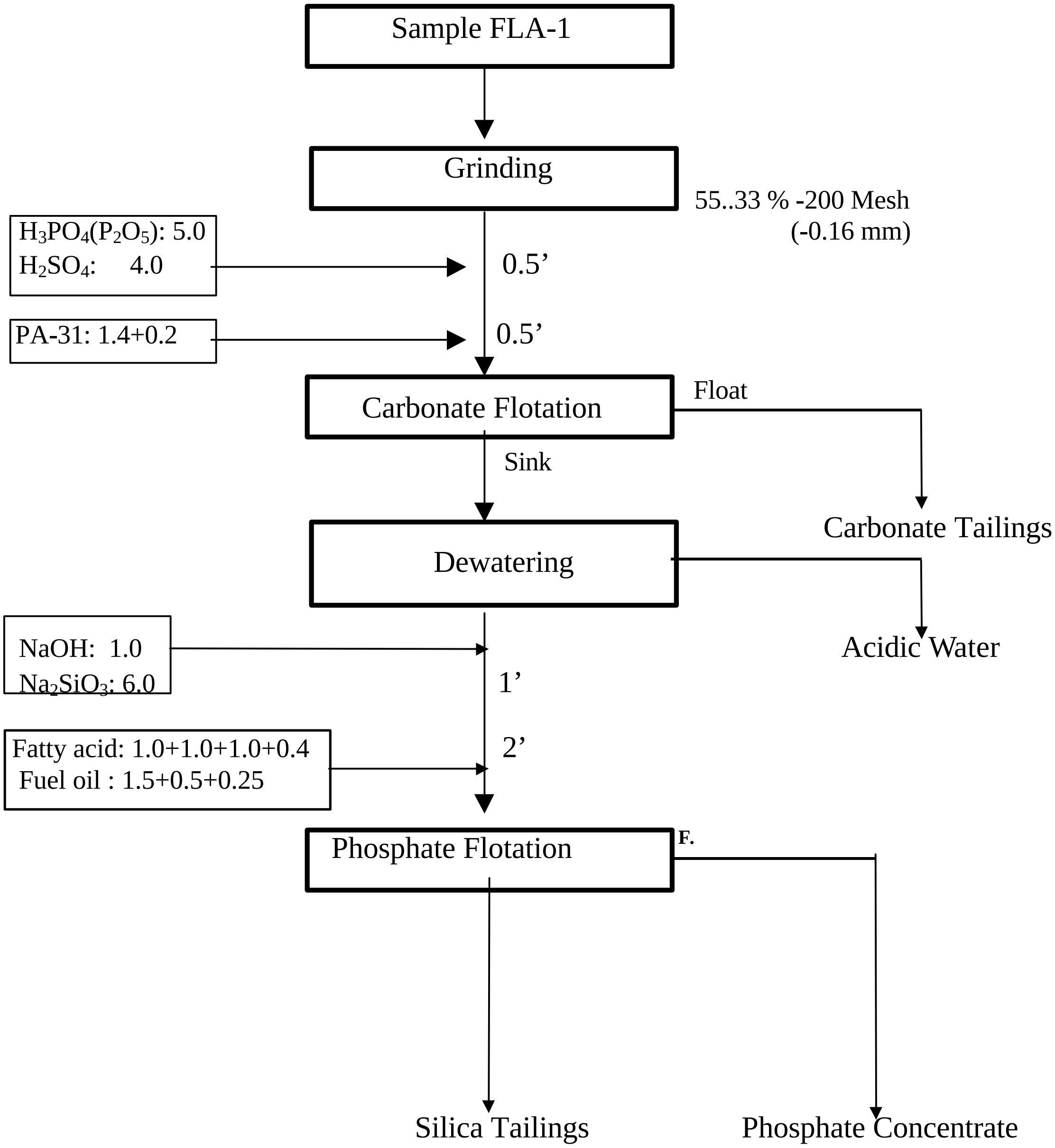

Figure 8. Carbonate and Phosphate Flotation Process for Sample FLA-1.

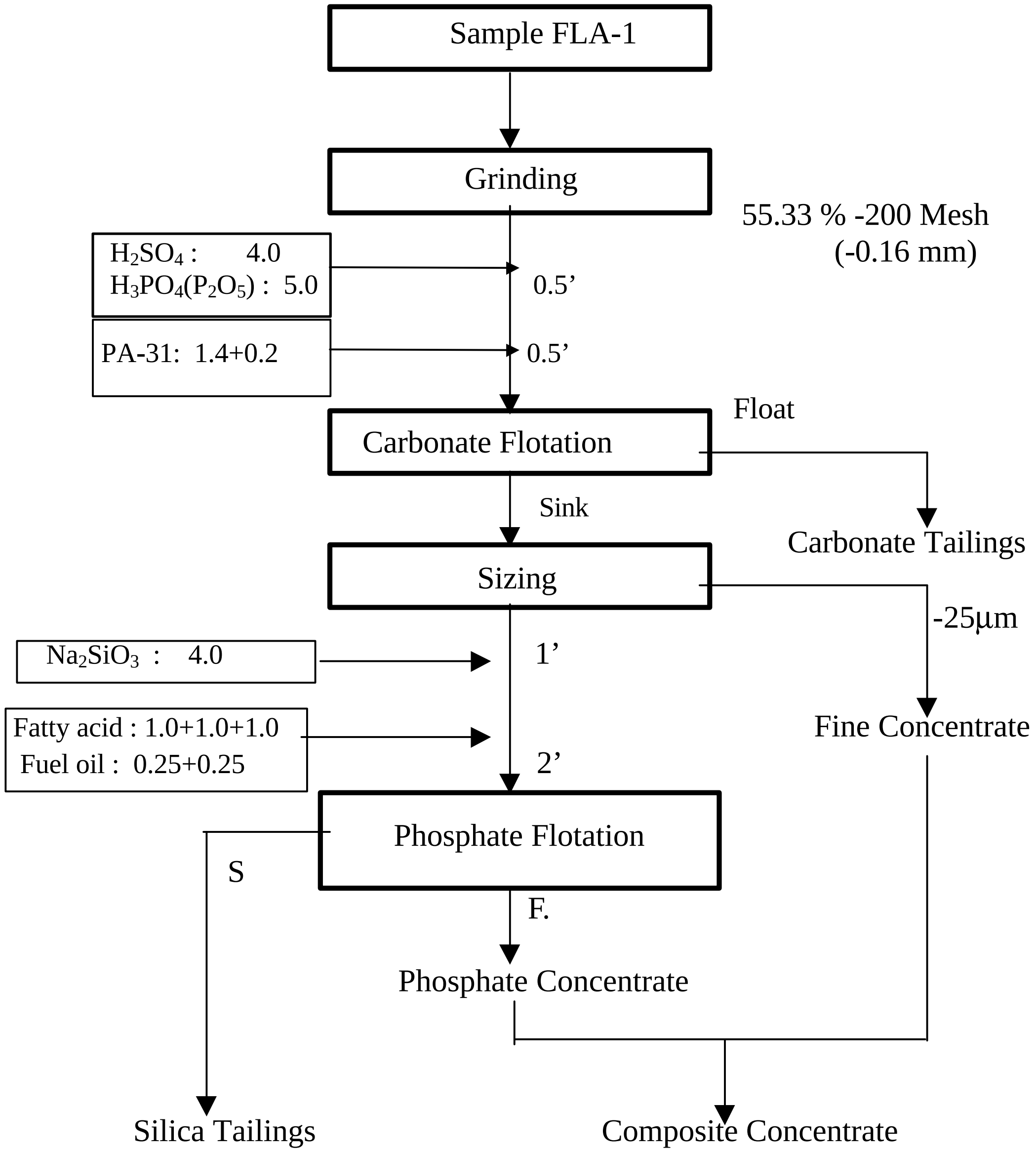

Figure 9. Carbonate Flotation-Sizing-Phosphate Flotation Process for Sample FLA-1.

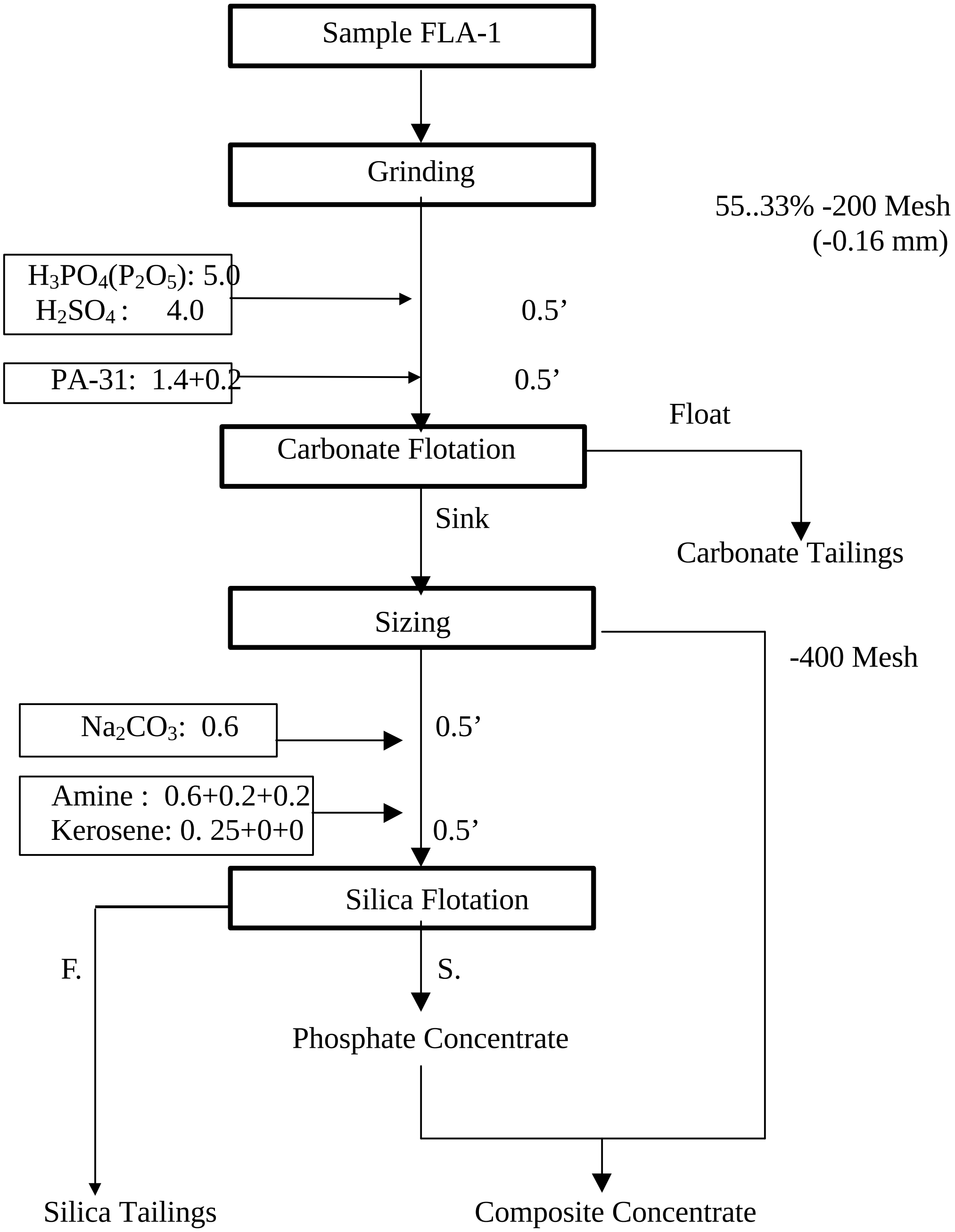

Figure 10. Carbonate Flotation-Sizing-Silica Flotation Process for Sample FLA-1.

Table 30. Flotation Results and Operating Conditions for Sample FLA-1 with Carbonate-Phosphate Flotation Process.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	19.01	45.35	5.93	8.66	16.00	65.70	8.94
Silica tailings	14.95	5.57	0.18	91.22	1.55	1.74	74.47
Phosphate concentrate	66.04	67.18	0.85	4.63	82.45	32.56	16.59
Head	100.00	53.37	1.72	18.45	100.00	100.00	100.00

II. Operating Conditions

Operation	Conditioning		Skimming Time (min.)	Reagent, lb./t Feed						
	Solids %	pH		H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	NaOH	Na_2SiO_3	Fatty Acid	Fuel Oil
Carbonate flotation I	35	4.5-5.5	7	5.0	4.0	1.4				
” ” II		4.8-5.8	5			0.2				
Phosphate flotation I	30	9.5	5				1.0	6.0	1.0	1.5
” ” II		9.0	2.5						1.0	0.5
” ” III		8.8	2						1.0	0.25
” ” IV		8.5	2						0.4	
Total reagent consumption, lb./t feed				5.0	4.0	1.6	1.0	6.0	3.4	2.25

Table 31. Flotation Results and Operating Conditions for Sample FLA-1 with Carbonate Flotation-Sizing-Phosphate Flotation Process.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	19.84	45.91	5.64	8.87	16.76	65.12	9.24
Silica tailings	13.38	4.46	0.16	94.13	1.08	1.16	66.09
-25μm fines	12.62	61.36	1.18	11.90	14.23	8.72	7.87
Flotation concentrate	54.16	68.18	0.80	5.90	67.93	25.00	16.80
Head	100.00	54.37	1.72	19.05	100.00	100.00	100.00
Composite concentrate	66.78	66.89	0.87	7.04	82.16	33.72	24.67

II. Operating Conditions

Operation	Conditioning		Skimming Time (min.)	Reagent, lb./t feed					
	Solids (%)	pH		H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2SiO_3	Fatty Acid	Fuel Oil
Carbonate flotation I	35	4.5-5.5	7	5.0	4.0	1.4			
” ” II		4.8-5.8	5			0.2			
Phosphate flotation I	25	8.5	5				4.0	1.0	0.25
” ” II		8.0	3					1.0	0.25
” ” III		7.5	2					1.0	
Total reagent consumption, lb./t feed				5.0	4.0	1.6	4.0	3.0	0.5

Table 32. Flotation Results and Operating Conditions for Sample FLA-1 with Carbonate Flotation-Sizing-Silica Flotation Process.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	19.50	45.63	5.62	9.66	16.32	64.33	9.68
Silica tailings	15.67	7.80	0.19	91.60	2.25	1.75	73.89
-400 mesh fines	15.77	61.17	1.16	13.09	17.69	10.53	10.61
Flotation concentrate	49.06	70.78	0.82	2.31	63.74	23.39	5.82
Head	100.00	54.48	1.71	19.42	100.00	100.00	100.00
Composite concentrate	64.83	68.42	0.91	4.92	81.73	33.92	16.43

II. Operating Conditions

Operation	Conditioning		Skimming Time (min.)	Reagent, lb./t Feed					
	Solids %	pH		H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation I	35	4.5-5.5	7	5.0	4.0	1.4			
” ” II		4.8-5.8	5			0.2			
Silica flotation I	25	8.5	3				0.6	0.6	0.25
” ” II		8.0	2					0.2	
” ” III		7.5	2					0.2	
Total reagent consumption, lb./t feed				5.0	4.0	1.6	0.6	1.0	0. 25

In order to further investigate the effect of particle size of flotation feed on separation performance, the “grinding-dolomite flotation-sizing-silica flotation” process was tried by increasing the grinding fineness to 41% -200 mesh (equivalent to 0.3 mm). The size distribution of the feed is presented in Table 33. The test flowchart is shown in Figure 11 and operating conditions and flotation results are listed in Table 34.

From the test results on sample FLA-1, it can be concluded that:

- The processes tested can produce the target results (phosphate concentrate with over 30% P_2O_5 and less than 1% MgO at an overall P_2O_5 recovery of more than 80%).
- The “grinding-dolomite flotation-sizing-silica flotation” process is recommended because of the advantages of low reagent cost, easy operation, etc. For amine flotation to remove silica, the sink product of dolomite flotation should be sized to remove fines such as the -400 mesh fraction. Otherwise the amine flotation does not work. The -400 mesh fines can be used as fine concentrate.
- After dolomite flotation, phosphate flotation can also be applied to reject silica by either desliming or undesliming the dolomite flotation underflow. The flotation results were quite similar, but total reagent cost can be lowered if the feed for phosphate flotation is deslimed.
- By increasing the grinding fineness from 0.2mm to 0.3mm, the target result can also be achieved; however, dolomite collector PA-31 usage will also be significantly increased.

FLOTATION TESTS ON SAMPLE FLA-2

Among the five samples received, sample FLA-2 contained the lowest MgO, only 1.12%. Phosphate concentrate of less than 1% MgO can be produced by eliminating about 35% of the total MgO in the feed.

For dolomite flotation of sample FLA-2, tests were carried out on grinding fineness, phosphoric acid consumption, proportion of phosphoric acid and sulfuric acid, etc. For separation of silica from phosphate, different processes like phosphate flotation or silica flotation were tried, and the reagents used included sodium hydroxide, sodium carbonate, sodium silicate, fatty acid, fuel oil, amine, kerosene, etc.

Size analysis indicated that after grinding, the MgO in the +150 mesh coarse fraction was very low, 0.63%. It was predicted that an acceptable concentrate could be obtained from this fraction only by rejecting silica and with no need to conduct dolomite flotation. The fine fraction was subject to dolomite flotation-sizing-silica flotation or dolomite flotation-sizing-phosphate flotation. In this way, the reagent consumption for dolomite flotation could be significantly reduced and the overall reagent costs could be lowered.

Table 33. Size Analysis for Sample FLA-1 by Increasing Particle Size of Flotation Feed. (Grinding time: 6 Minutes; Fineness: 41.01%-200 Mesh).

Size (mm)	Wt. (%)	Analysis (%)			Distribution (%)	
		BPL	P_2O_5	MgO	BPL	MgO
+0.200	7.94	51.7	23.67		7.67	
0.200~0.160	15.16	52.6	24.06		14.90	
0.160~0.100	22.94	53.3	24.41		22.86	
0.100~0.071	12.95	54.1	24.77		13.10	
0.071~0.036	13.31	56.3	25.77		14.00	
-0.036	27.70	53.1	24.31		27.47	
Total	100.00	53.5	24.50		100.00	

Table 34. Flotation Results and Operating Conditions for Sample FLA-1 in -0.3 mm Size with Carbonate Flotation-Sizing-Silica Flotation Process.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	19.86	45.80	5.90	6.20	16.81	66.10	6.49
Silica tailings	15.62	6.42	0.19	91.04	1.86	1.70	75.04
-400 mesh fines	9.75	61.56	1.18	13.33	11.11	6.78	6.86
Flotation concentrate	54.77	69.34	0.83	4.02	70.22	25.42	11.61
Head	100.00	54.09	1.77	5.42	100.00	100.00	100.00
Composite concentrate	64.52	68.16	0.88	5.42	81.33	32.20	18.47

II. Operating Conditions

Operation	Conditioning		Skimming Time (min.)	Reagent, lb./t Feed					
	Solids (%)	pH		H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation I	35	4.5-5.5	7	5.0	4.0	3.0			
” ” II		4.8-5.8	5	0.6		0.2			
Silica flotation I	25	8.5	3				0.6	0.4	0.2
” ” II		8.0	2					0.2	0.1
” ” III		7.5	2					0.2	
Total reagent consumption, lb./t feed				5.6	4.0	3.2	0.6	0.8	0.3

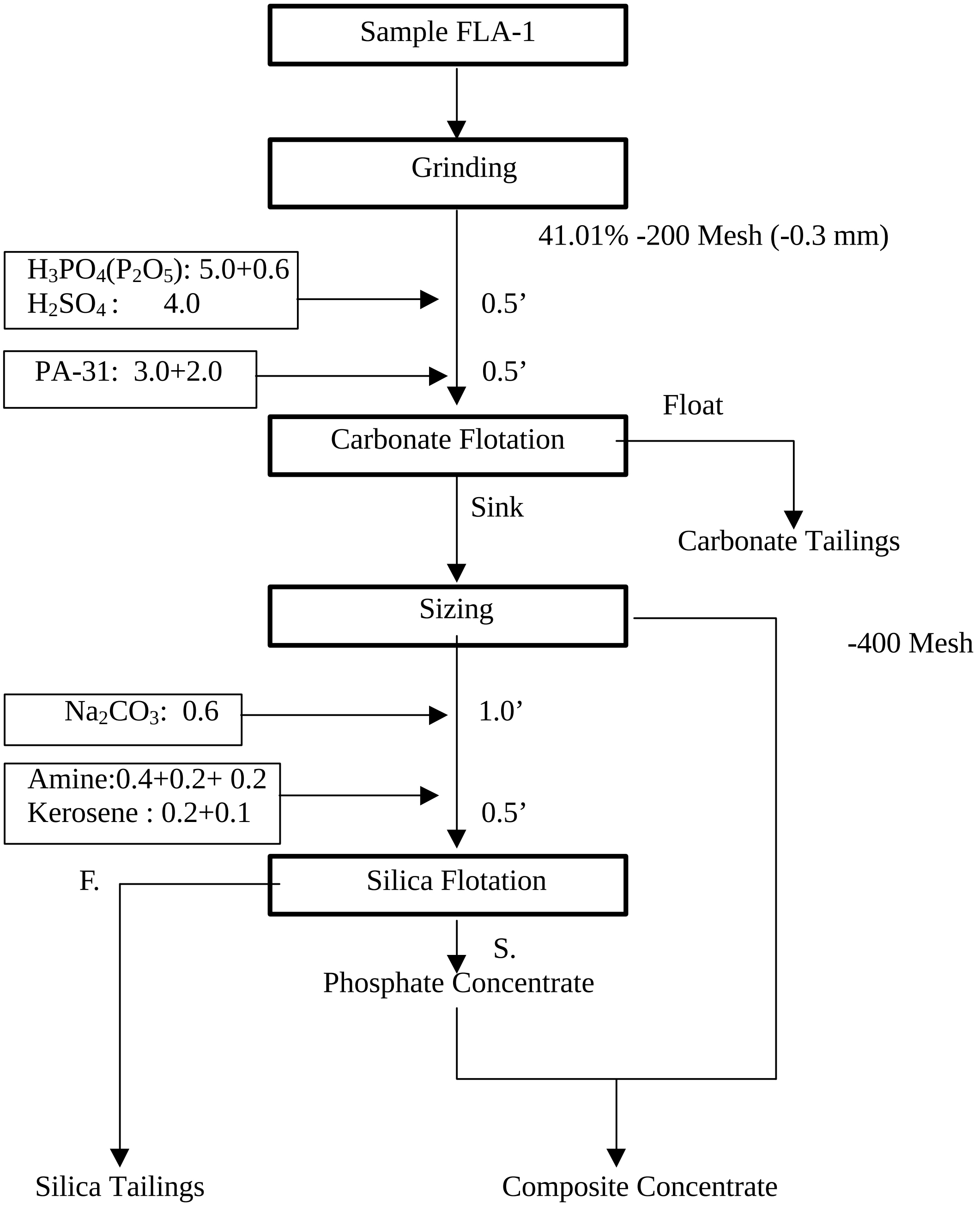

Figure 11. Carbonate Flotation-Sizing-Silica Flotation Process for Sample FLA-1 by Increasing Feed Size to -0.3 mm.

For sample FLA-2, the grinding time was four minutes and the fineness was about 29% -200 mesh, about 0.5 mm. The size distribution and chemical analysis of the flotation feed are shown in Table 35.

Three combination processes were tested for sample FLA-2, including “grinding-dolomite flotation-sizing-phosphate flotation”, “grinding, sizing, coarse size to silica flotation, fine size to dolomite flotation and silica flotation” and “grinding, sizing, coarse size to phosphate flotation and fines to dolomite flotation and phosphate flotation” as shown in Figures 12, 13, and 14 with the operating conditions and the flotation results listed in Tables 36, 37, and 38, respectively.

From the test results, the following could be concluded:

- With the flowsheets tested, high-quality phosphate concentrate analyzing 31% P_2O_5 and less than 1% MgO could be obtained at the overall P_2O_5 of over 90%, which was much better than the target results specified in the proposal.
- Among the flowsheets tested, the process “grinding, sizing, coarse size to silica flotation, fine size to dolomite flotation and silica flotation” consumed less reagents, but the flowsheet was quite complicated because two sizing operations were needed and different size fractions were subject to separate flotation steps. The flowsheet structure was rather simple for the process “grinding-dolomite flotation-sizing-phosphate flotation,” while the reagent consumption was higher compared with other processes.

FLOTATION TESTS ON SAMPLE FLA-3

Sample FLA-3 contained about 15% P_2O_5 and 9.5% MgO. In order to obtain a phosphate concentrate analyzing over 30% P_2O_5 and less than 1% MgO from this sample, it was calculated that at least 96% of the total MgO should be rejected from the feed. However, the elimination of so much MgO would also cause significant loss of P_2O_5. Therefore, it was predicted that the overall P_2O_5 recovery of the final phosphate concentrate would be limited; namely, it should be less than 80% with the existing flotation technology.

Preliminary tests were carried out on the scrubbing-desliming and calcination-digestion processes. The results indicated that only part of the materials could be discarded as tailings and no acceptable product could be produced. Therefore the grinding-flotation process was tested. Even though the average particle sizes of phosphate and carbonate minerals in the sample were 0.58 and 0.74 mm, respectively, as shown in Table 24, fine grinding was required for maximum liberation of phosphate from carbonate minerals because more than 96% of the total MgO must be rejected in order to achieve a concentrate with less than 1% MgO.

Based on the characteristics of sample FLA-3, grinding-dolomite flotation-silica flotation process was applied. The sample was ground to pass 100 mesh (0.15 mm). The

Table 35. Size Analysis of Flotation Feed for Sample FLA-2. (Grinding Time: 4 Minutes; Fineness: 28.9% -200 Mesh).

Size (mm)	Wt. (%)	Analysis (%)			Distribution (%)	
		BPL	P_2O_5	MgO	BPL	MgO
+0.315	9.46	52.6	24.06	0.51	8.85	4.63
0.315~0.200	27.83	55.0	25.16	0.65	27.16	16.67
0.200~0.160	10.12	55.1	25.20	0.74	9.90	6.48
0.160~0.100	11.10	56.0	25.61	0.60	11.02	6.48
0.100~0.071	12.59	58.2	26.62	0.89	13.00	10.19
-0.071	28.90	58.6	26.83	2.06	30.07	55.55
Total	100.00	56.3	25.77	1.08	100.00	100.00

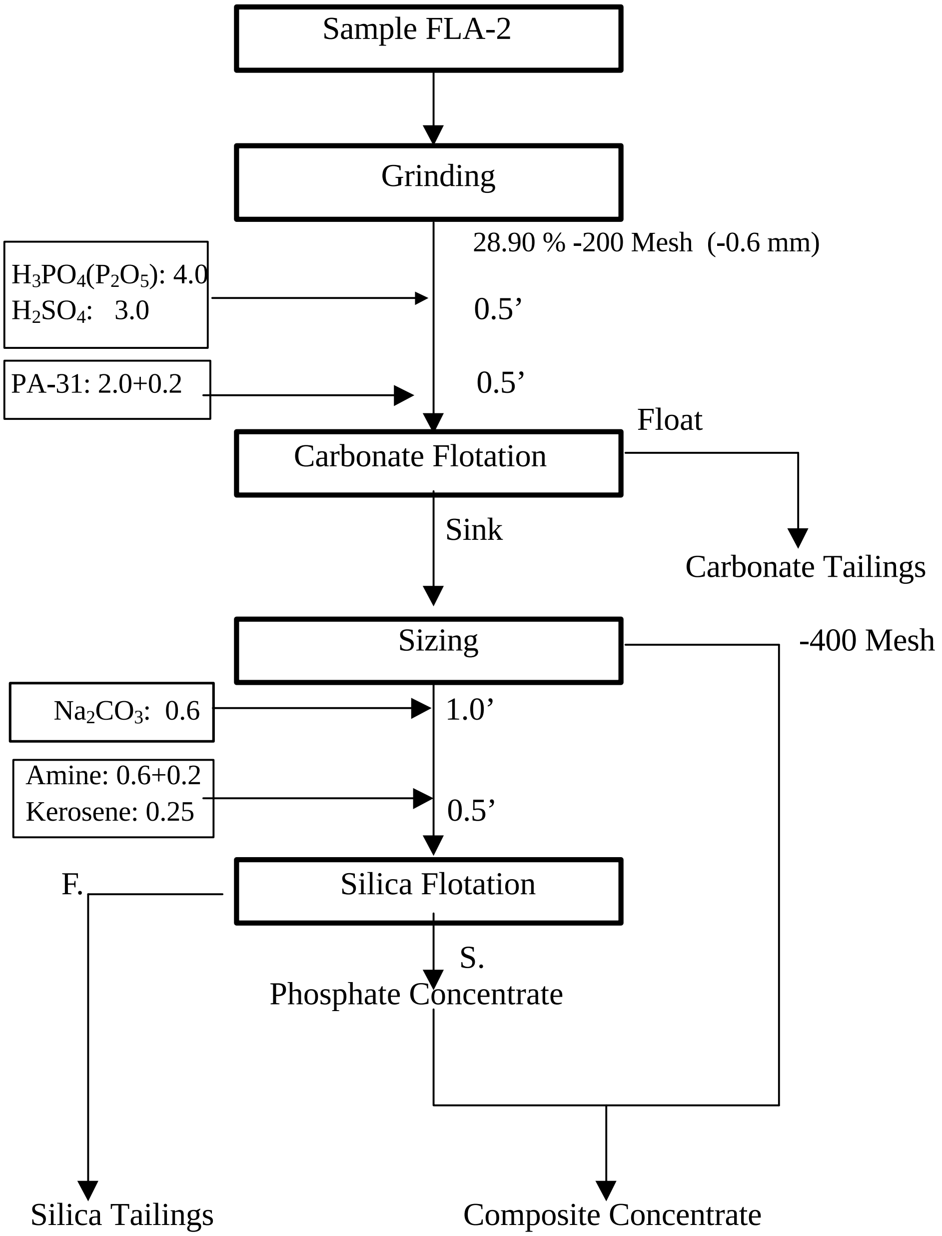

Figure 12. Carbonate Flotation-Sizing-Silica Flotation Process for Sample FLA-2.

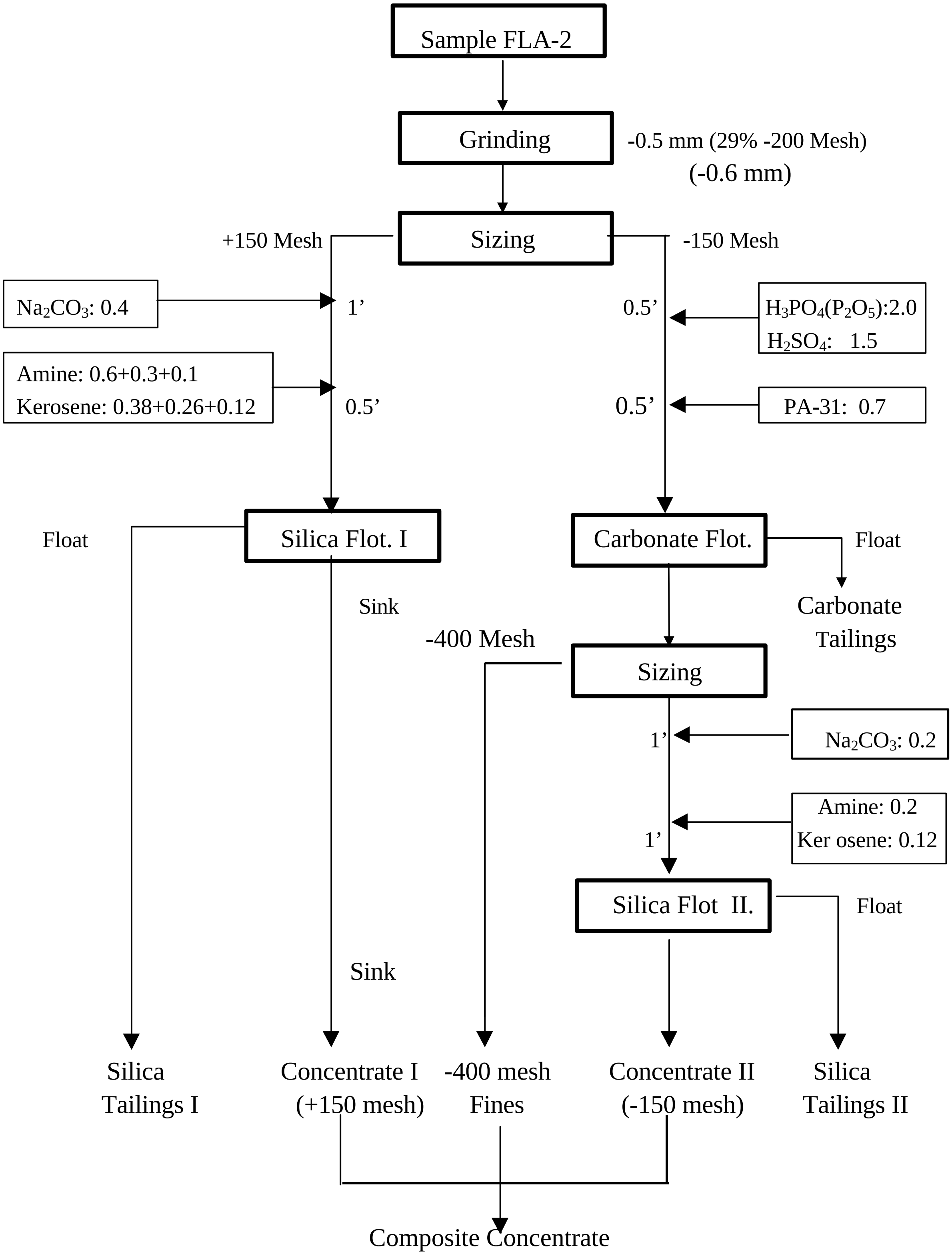

Figure 13. Sizing-Flotation Process for Sample FLA-2 Using Amine to Float Coarse Silica.

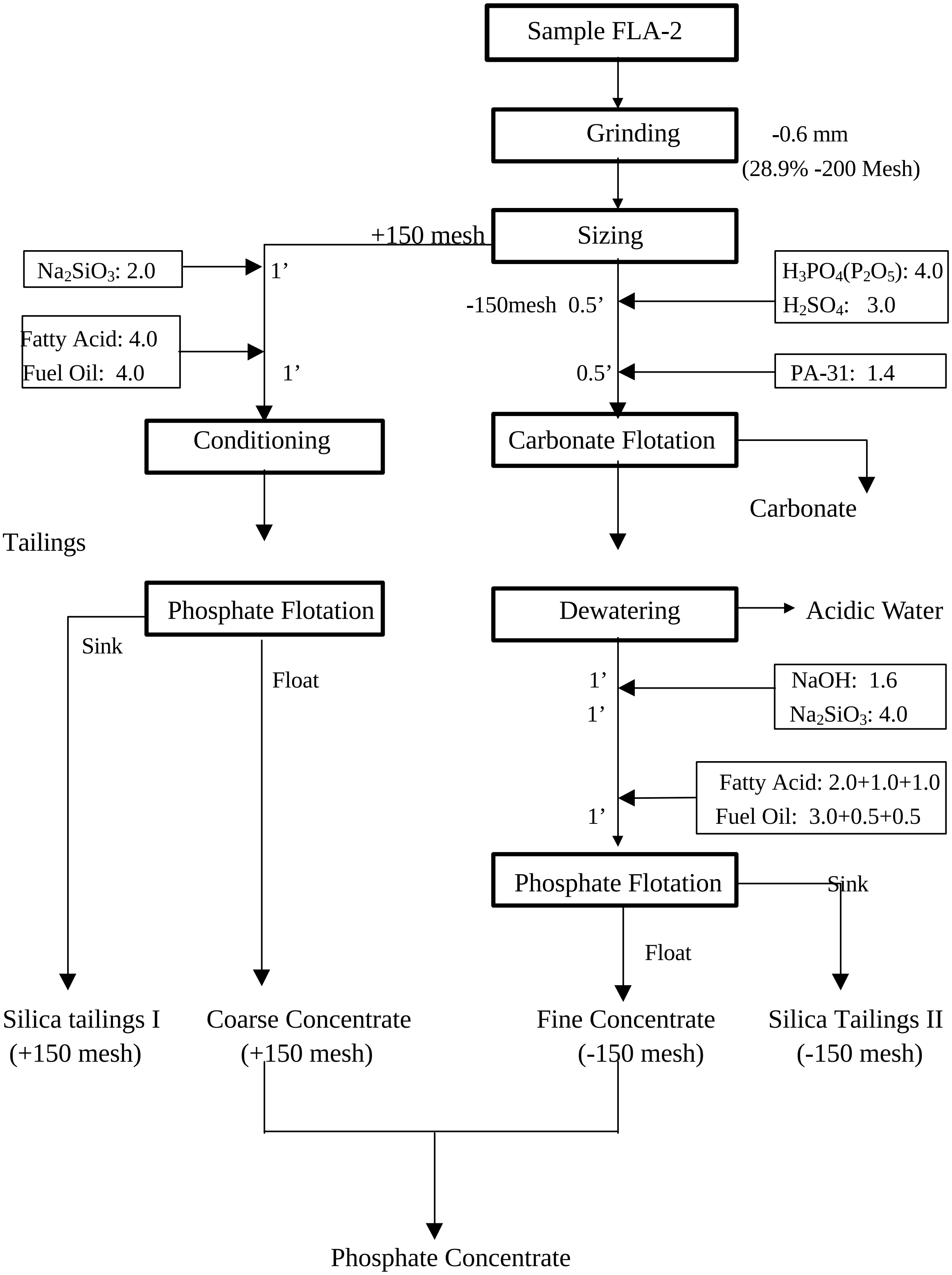

Figure 14. Sizing-Flotation Process for Sample FLA-2 Using Fatty Acid to Float both Coarse and Fine Phosphate.

Table 36. Flotation Results and Operating Conditions for Sample FLA-2 with Carbonate Flotation-Sizing-Silica Flotation Process.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	8.06	44.79	5.64	6.26	6.60	42.59	2.63
Silica tailings	16.11	4.79	0.10	92.89	1.38	1.85	77.02
-400 mesh fines	11.71	62.27	1.10	9.66	13.20	12.04	5.82
Flotation concentrate	64.02	68.04	0.74	4.40	78.82	43.52	14.53
Head	100.00	55.28	1.08	19.41	100.00	100.00	100.00
Composite concentrate	75.73	61.17	0.79	5.22	92.02	55.56	20.35

II. Operating Conditions (Starting Feed 200g)

Operation	Conditioning		Skimming time (min.)	Reagent, lb./t Feed					
	Solids %	pH		H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation I	35	4.5-5.5	9	4.0	3.0	2.0			
” ” II		4.8-5.8	4			0.2			
Silica flotation I	30	8.5	2				0.6	0.6	0.25
” ” II		8.0	1					0.2	
Total reagent consumption, lb./t feed				4.0	3.0	2.2	0.6	0.8	0.25

Table 37. Flotation Results and Operating Conditions for Sample FLA-2 with Sizing-Flotation Process Using Amine Flotation for Coarse Fraction.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Silica tailings I (+150mesh)	13.96	7.04	0.14	89.46	1.78	1.94	65.56
Concentrate I (+150mesh)	46.65	67.95	0.72	3.49	57.28	33.01	8.56
Carbonate tailings	9.01	46.28	5.02	7.33	7.54	43.69	3.46
Silica tailings II (-150mesh)	3.34	10.01	0.12	85.78	0.59	0.00	15.07
-400 mesh	8.52	62.47	1.13	9.35	9.63	9.71	4.20
Concentrate II (-150mesh)	18.52	69.31	0.66	3.24	23.18	11.65	3.15
Head	100.00	55.35	1.03	19.05	100.00	100.00	100.00
Composite concentrate	73.69	67.67	0.76	4.10	90.09	54.37	15.91

II. Operating Conditions (Starting Feed 400g)

Operation	Conditioning		Skimming Time (min.)	Reagent, lb./t Feed					
	Solids %	pH		H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Silica flotation I	35	9.0	3				0.4	0.6	0.38
” ” II		8.8	2					0.3	0.26
” ” III		8.5	1.5					0.1	0.12
Carbonate flotation	30	5.2-5.9	7	2.0	1.5	0.7			
Silica flotation (fine)	25	8.5	2				0.2	0.2	0.12
Total reagent consumption, lb./t feed				2.0	1.5	0.7	0.6	1.2	0.88

Table 38. Flotation Results and Operating Conditions for Sample FLA-2 with Sizing-Flotation Process Using Fatty Acid Flotation for Coarse Fraction.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	7.83	43.66	5.80	7.65	6.19	42.86	3.16
Silica tailings (+150mesh)	12.10	6.25	0.13	90.10	1.39	1.90	57.43
Silica tailings (-150mesh)	3.25	14.73	0.48	77.94	0.88	1.91	13.33
Concentrate (+150mesh)	48.32	65.55	0.73	6.62	57.56	33.33	16.86
Concentrate (-150mesh)	28.50	65.59	0.74	6.14	33.98	20.00	9.22
Head	100.00	55.04	1.05	18.98	100.00	100.00	100.00
Composite concentrate	76.82	65.59	0.73	6.44	91.54	53.33	26.08

II. Operating Conditions (Starting Feed 400g)

Operation	Conditioning		Skimming Time (min.)	Reagent, lb./t Feed						
	Solids (%)	pH		H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	NaOH	Na_2SiO_3	Fatty Acid	Fuel Oil
Phosphate flotation (coarse)	60	8.5	3					1.0	2.0	2.0
Carbonate flotation (fine)	30	4.8-5.8	7	2.0	1.5	0.7				
Phosphate flotation I	25	9.5	7				0.8	2.0	1.0	1.5
” ” II		8.8	4						0.5	0.25
” ” III		8.2	2						0.5	0.25
Total reagent consumption, lb./t feed				2.0	1.5	0.7	0.8	3.0	4.0	4.0

size distribution and chemical analysis of the flotation feed is presented in Table 39. Figure 15 shows the flotation flowchart, and the operating conditions and the flotation results are listed in Table 40.

The data demonstrated that CLDRI's fine particle flotation technology could be applied to beneficiate a high-dolomite phosphate sample like sample FLA-3 to obtain a phosphate concentrate with over 30% P_2O_5 and less than 1% MgO and an overall P_2O_5 recovery of 60%. The main reason for the low overall P_2O_5 recovery could be the extremely high MgO content. In the process, more than 97% of the total MgO was removed to dolomite tailings. The P_2O_5 recovery of the process could be higher if the final concentrate could tolerate more than 1% MgO.

Comparison tests were also conducted in the attempt to increase the grinding fineness. The results indicated that increasing the particle size of flotation feed could reduce the quality of the concentrate and increase the reagent consumption significantly.

FLOTATION TESTS ON SAMPLE FLA-4

The sample FLA-4 contained a lot of primary slimes and was very sticky. The data listed in Table 11 indicate that about 65% of the total MgO could be eliminated at about 10% P_2O_5 loss by desliming -150 mesh fines after scrubbing the sample. This could be considered an effective method for MgO removal. For such a high-slime sample, not only could scrubbing and desliming raise the quality of the feed to the flotation operation, but also could improve the flotation behavior. The scrubbing was conducted in a 3000 ml Denver flotation cell and desliming was done using a 150 mesh sieve. The operating conditions for scrubbing were: 40% solid, 1500 rpm impeller speed and five minutes. The size distribution of the scrubbed sample is given in Table 41, and that of the deslimed sample in Table 42. The deslimed sample was air-dried and crushed to -1.0 mm. The size analysis is presented in Table 16.

From Table 42, it can be seen that the P_2O_5 grade was increased from 24% in the as-received sample to 26.5% in the +150 mesh fraction while MgO was reduced from 2.0% to 1.2%, which would be beneficial to the flotation operation.

During the grinding test prior to flotation, it was found that the MgO was enriched in fine fraction after sample FLA-4 was ground. The remaining MgO could be less than 1% if the fines were removed. In this way, the carbonate flotation could be omitted and reagent consumption and cost could be lowered. In accordance with this feature of the sample, size and chemical analyses were carried out to investigate the MgO distribution when the sample was ground to different degrees of fineness. The data are shown in Table 43.

Table 43 indicated that P_2O_5 could be upgraded to 27% and MgO could be reduced to 0.8% after the ground sample was deslimed at 20μ to 38μ. The plus fraction could only be subject to a silica rejection step to obtain the target result. For separating

Table 39. Size Analysis of Flotation Feed for Sample FLA-3. (Grinding Time: 6 Minutes; Fineness: 69.71% -200 Mesh).

Size (mm)	Wt. (%)	Analysis (%)			Distribution (%)	
		BPL	P_2O_5	MgO	BPL	MgO
+0.160	1.11	51.6	23.62	1.45	1.80	0.22
0.160~0.100	16.72	49.1	22.49	2.88	26.06	5.20
0.100~0.071	12.46	44.3	20.27	4.84	17.53	6.51
-0.071	69.71	24.7	11.30	11.65	54.61	88.07
Total	100.00	31.5	14.43	9.22	100.00	100.00

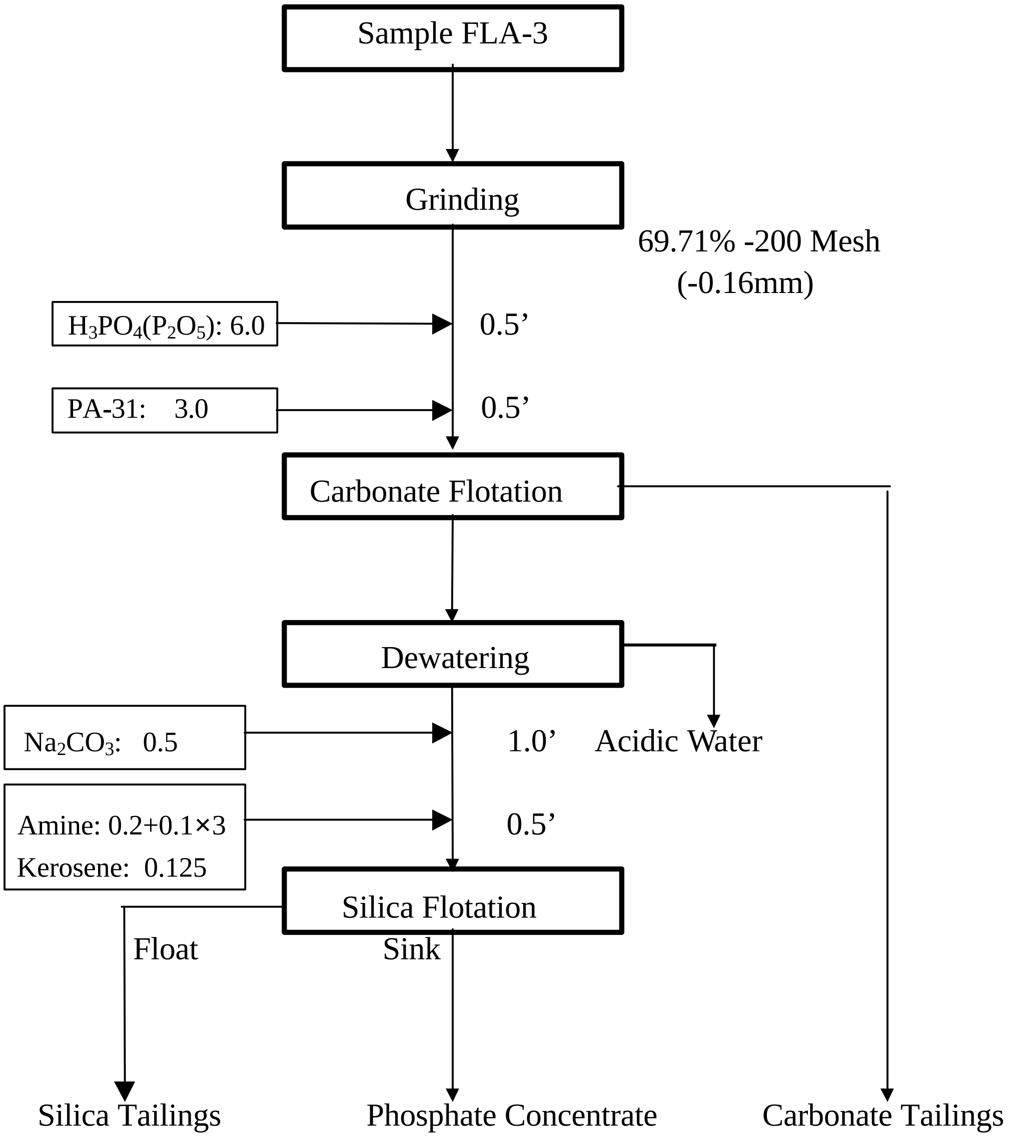

Figure 15. Carbonate Flotation-Phosphate Flotation Process for Sample FLA-3.

Table 40. Flotation Results and Operating Conditions for Sample FLA-3 with Carbonate Flotation-Silica Flotation Process.

I. Flotation Results							
Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	64.38	17.74	14.57		37.22	97.10	
Silica tailings	8.47	7.69	0.22		2.14	0.21	
Flotation concentrate	27.15	68.59	0.96		60.64	2.69	
Head	100.00	30.70	9.66		100.00	100.00	100.00

II. Operating Conditions (Starting Feed 400g)								
Operation	Conditioning		Skimming time (min.)	Reagent, lb./t Feed				
	Solid (%)	pH		H_3PO_4 (P_2O_5)	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation I	35	4.5-5.5	7	6.0	3.0			
” ” II		4.8-5.8	5	2.0	1.0			
Silica flotation I	30	8.5	0.5			0. 5	0.2	0.125
” ” II		8.2	1.5				0.1	
” ” III		8.0	1.5				0.1	
” ” VI		8.0	1				0.1	
Total reagent consumption, lb./t feed				8.0	4.0	0. 5	0.5	0.125

Table 41. Size Analysis after Sample FLA-4 Was Scrubbed at 40% Solids and 1000 rpm for 5 Minutes.

Size (mm)	Weight (%)	Grade (%)		P_2O_5 Recovery (%)
		BPL	P_2O_5	
+0.100	83.83	58.6	26.84	93.71
0.100-0.038	2.54	27.7	12.68	1.33
0.038-0.020	1.24	22.1	10.11	0.54
-0.020	12.39	18.6	8.53	4.42
Total	100.00	52.5	24.01	100.00

Table 42. Chemical Analysis of +150 Mesh Fraction for Sample FLA-4 after Scrubbing and Desliming.

Chemical Analysis (%)										
BPL	P_2O_5	MgO	CaO	CO_2	SiO_2	Fe_2O_3	Al_2O_3	F	A.I.	I.L
57.6	26.36	1.21	39.26	4.78	15.26	2.16	1.46	2.36	15.96	7.75

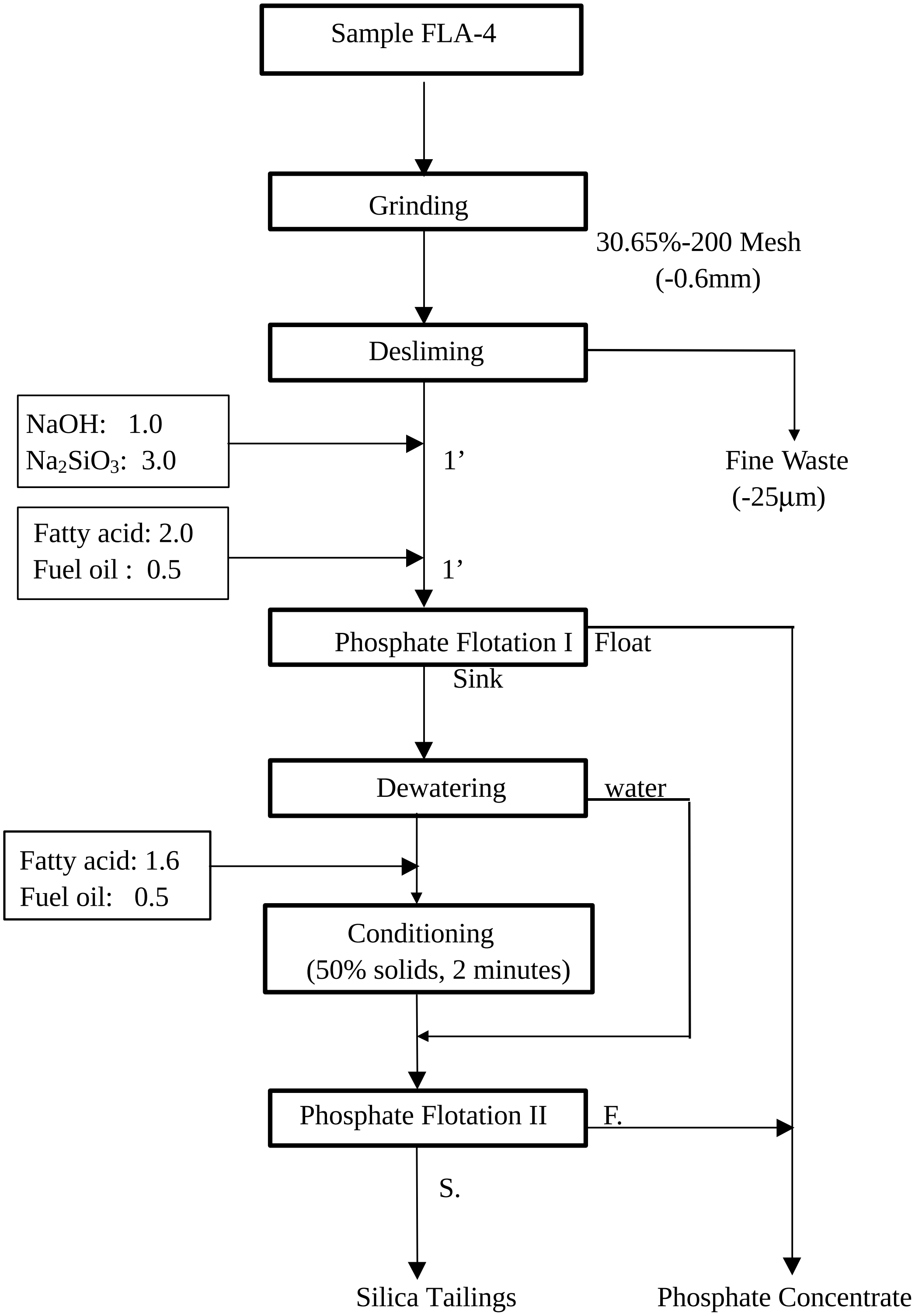

Figure 16. Sizing-Phosphate Flotation Process for Sample FLA-4.

Table 43. Size and Chemical Analysis of Sample FLA-4 at Different Grinding Times.

Time (min.)	Size (mm)	Weight (%)	Analysis (%)			Distribution (%)		Passing Size (mm)
			BPL	P_2O_5	MgO	BPL	MgO	
4	+0.500	5.81	63.6	29.11	0.61	25.82	12.50	0.60
	0.500-0.315	18.04						
	0.315-0.071	45.50	57.5	26.32	0.81	44.57	30.83	
	0.071-0.038	9.78	62.9	28.81	1.04	10.49	8.33	
	-0.038	20.87	53.8	24.63	2.80	19.12	48.34	
	Total	100.00	58.7	26.88	1.20	100.00	100.00	
6	+0.100	47.11	58.1	26.58	0.76	46.93	31.30	0.30
	0.100-0.071	11.36	58.8	26.93	0.78	11.47	7.83	
	0.071-0.020	23.19	62.0	28.37	0.99	24.66	20.00	
	-0.020	18.34	53.9	24.65	2.54	16.94	40.87	
	Total	100.00	58.3	26.68	1.15	100.00	100.00	
8	+0.100	35.85	58.1	26.58	0.73	35.88	21.85	0.20
	0.100-0.071	13.98	57.7	26.41	0.75	13.90	8.40	
	0.071-0.020	29.99	60.7	27.77	0.99	31.36	25.21	
	-0.020	20.17	54.3	24.84	2.62	18.86	44.54	
	Total	100.00	58.0	26.56	1.19	100.00	100.00	

silica from phosphate, it was discovered that amine flotation was not as effective as expected for this sample. There was little silica float even when amine consumption was increased to 4.0 lb per ton of feed. No improvement was achieved in amine flotation after considerable efforts were made, such as adjusting flotation pH, scrubbing and desliming the flotation feed before addition of amine collector, etc. Therefore, phosphate flotation was employed for separation of phosphate from silica.

Prior to flotation, the sample was ground to 31% -200 mesh (about 0.6 mm). The -25μ fraction was deslimed to remove part of the MgO impurity. In flotation testing, the reagent dosages like sodium carbonate, sodium hydroxide, sodium silicate, fatty acid and fuel oil were optimized.

For sample FLA-4, two flotation processes were tested. One was grinding-desliming-phosphate flotation. In this process, the sample was ground to 31% -200 mesh and deslimed at 25μ. The deslimed sample was subject to two-stage phosphate flotation. The flowchart is given in Figure 16, with the operating conditions and flotation performance in Table 44.

Another process was grinding-dolomite flotation-sizing-phosphate flotation. The ground sample was sent to dolomite flotation first. The underflow of dolomite flotation was sized to separate -25μ (or -38μ) fraction as fine concentrate. The plus portion was subject to phosphate flotation. The flowchart is shown in Figure 17 and the operating conditions and flotation results are given in Table 45.

Both of the processes could produce the acceptable concentrate up to the required specifications. The overall P_2O_5 recovery of the grinding-dolomite flotation-sizing-phosphate flotation process was higher than that of the grinding-desliming-phosphate flotation process, but the reagent consumption was also higher. From an economical viewpoint, the grinding-desliming-phosphate flotation process could be recommended for such samples.

FLOTATION TESTS ON SAMPLE FLA-5

Because of high slime content, sample FLA-5 was scrubbed and deslimed to remove -150 mesh fine fraction before preparing the flotation feed. The scrubbing was carried out in a 3000 ml Denver cell at the conditions of 65-70% solids and 1000 rpm impeller speed and for a duration of 10 minutes. The scrubbed sample was deslimed using a 150 mesh sieve. The size distributions and chemical analyses of scrubbed, deslimed and crushed (to -1 mm) samples are presented in Tables 46, 47 and 48 separately.

From Tables 46 and 47, it can be seen that it was necessary to pre-handle the sample by scrubbing and desliming to eliminate some of the MgO impurities before

Table 44. Flotation Results and Operating Conditions for Sample FLA-4 with Sizing-Phosphate Flotation Process.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Slimes	18.72	54.36	2.59	12.57	17.18	48.00	14.62
Silica tailings	9.92	7.69	0.74	81.51	1.29	7.00	50.31
Phosphate concentrate	71.1.36	67.69	0.63	7.90	81.53**	45.00	35.07
Head	100.00	59.26	1.00	16.08	100.00	100.00	100.00

II. Operating Conditions (Starting Feed 400g)

Operation	Conditioning		Skimming Time (min.)	Reagent, lb./t Feed			
	Solids %	pH		NaOH	Na_2SiO_3	Fatty Acid	Fuel oil
Phosphate flotation I	28	9.0-9.5	3	1.0	3.0	2.0	1.5
” ” II	40*	8.5-9.0	1			0.4	0.7
Total reagent consumption, lb./t feed				1.0	3.0	3.6	2.2

* After dewatering, conditioned in a 500ml Denver conditioner.

** BPL recovery is 76.40% after the deduction of BPL loss in primary slimes.

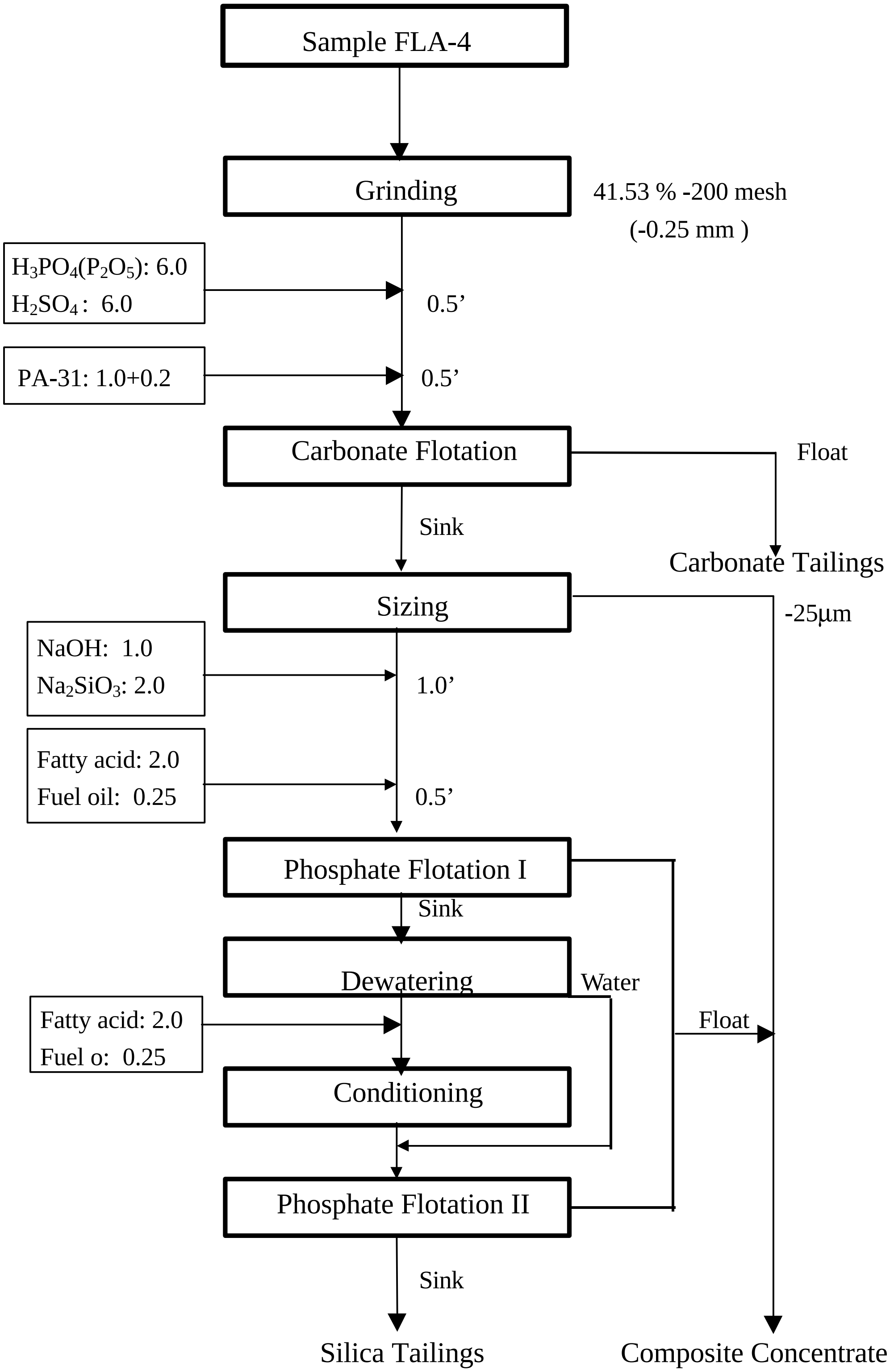

Figure 17. Carbonate Flotation-Sizing-Phosphate Flotation Process for Sample FLA-4.

Table 45. Flotation Results and Operating Conditions for Sample FLA-4 with Carbonate Flotation-Sizing-Phosphate Flotation Process.

I. Flotation Results

Product	Wt. %	Analysis, %			Distribution, %		
		BPL	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	10.38	48.70	4.67		8.60	42.60	
Silica tailings	11.36	6.82	0.50		1.30	5.22	
Fine concentrate	14.27	58.06	1.60		14.12	20.00	
Flotation concentrate	63.99	69.66	0.58		75.98	32.18	
Head	100.00	58.67	1.15		100.00	100.00	
Composite concentrate	78.26	67.54	0.77		90.10**	52.18	

II. Operating Conditions

Operation	Conditioning		Skimming time (min.)	Reagent, lb./t Feed						
	Solid %	pH		H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	NaOH	Na_2SiO_3	Fatty Acid	Fuel oil
Carbonate flotation I	35	4.2-5.0	10	6.0	6.0	2.0				
” ” II		4.5-5.5	8			0.4				
Phosphate flotation I	40*	9.2	4				1.0	2.0	2.0	0.25
” ” II		9.5	1						0.6	0.25
Total reagent consumption, lb./t feed				6.0	6.0	2.4	1.0	2.0	2.6	0.5

* After dewatering, conditioned in a 500 ml Denver conditioner.

** The BPL recovery is 84.43% after the deduction of BPL loss in primary slimes.

Table 46. Size Distribution and Chemical Analysis after Sample FLA-5 Was Scrubbed and Deslimed at 150 Mesh.

Products	Weight (%)	Analysis (%)			Distribution (%)	
		BPL	P_2O_5	MgO	BPL	MgO
Sands (+150 mesh)	93.20	59.0	27.00	1.95	96.69	72.22
Slimes (-150 mesh)	6.80	27.6	12.65	10.28	3.31	27.78
Total	100.00	56.9	26.02	2.52	100.00	100.00

Table 47. Chemical Analysis of Flotation Feed for Sample FLA-5.

Chemical Analysis (%)										
BPL	P_2O_5	MgO	CaO	CO_2	SiO_2	Fe_2O_3	Al_2O_3	F	A.I.	I.L
57.8	26.43	1.92	42.56	7.06	10.79	1.74	1.40	2.29	11.36	9.30

Table 48. Flotation Results of Sample FLA-5 at Different Feed Sizes.

Grinding Fineness		Product	Wt. (%)	Analysis(%)		Distribution(%)	
-200 Mesh (%)	Passing (mm)			BPL	MgO	BPL	MgO
35	-0.4	Dolomite tailings	16.03	41.4	8.62	11.18	64.79
		Silica tailings	7.34	6.3	0.12	0.77	0.47
		-400 mesh	10.91	64.8	1.12	11.87	5.63
		Flotation concentrate	65.72	68.9	0.94	76.18	29.11
		Head	100.00	59.4	2.13	100.00	100.00
		Composite concentrate	76.63	68.3	0.97	88.05*	34.74
44	-0.25	Dolomite tailings	19.03	43.2	7.66	13.87	70.87
		Silica tailings	9.21	9.5	0.10	1.47	0.49
		-400 mesh	12.47	65.9	1.08	13.87	6.31
		Flotation concentrate	59.29	70.7	0.78	70.79	22.33
		Head	100.00	59.2	2.06	100.00	100.00
		Composite concentrate	71.76	69.9	0.82	84.66**	28.64
63	-0.15	Dolomite tailings	19.61	44.8	7.04	14.82	65.71
		Silica tailings	8.62	9.9	0.10	1.44	0.48
		-400 mesh	18.55	64.8	1.16	20.27	10.48
		Flotation concentrate	53.22	70.7	0.93	63.47	23.33
		Head	100.00	59.3	2.10	100.00	100.00
		Composite concentrate	71.77	69.2	0.99	83.74***	33.81

*The BPL recovery is 85.14% after the deduction of BPL loss in primary slimes.

** It should be 81.86% after the same deduction.

*** It should be 80.97% after the deduction is made.

flotation. After the pre-treatment, the MgO content was reduced from 2.5% to 2.0%. Of the total MgO in the as-received sample, 27.78% was removed, while P_2O_5 loss was only 3.31%.

According to the characteristics of sample FLA-5, the process “grinding-dolomite flotation-sizing-silica flotation” was used, which was consistent with the processes for other samples. With the flowchart shown in Figure 18, the flotation tests on different feed size are listed in Table 48 with the corresponding reagent consumption in Table 49.

The data in Tables 48 and 49 indicated that the target results could be obtained when the percentage of -200 mesh size in the flotation feed was increased from 35 to 63. But the consumption of the flotation reagents, particularly dolomite collector, was significantly increased, which increased the total reagent cost. This phenomenon was also demonstrated in the test results from other samples. Therefore, the reasonable grinding fineness of a given sample should be decided by considering comprehensive factors, such as energy consumption for grinding, reagent consumption for flotation, separation performances, dewatering of the products, grinding in wet phosphoric acid production, etc. For sample FLA-5, the feed size to flotation was chosen to be 44% -200 mesh (passing 0.3 mm), with the size distribution in Table 50.

In the “grinding-dolomite flotation-sizing-phosphate flotation” process, phosphoric acid consumption was the major portion of the total flotation reagent cost, accounting for 40-60 %. Substituting other inexpensive acids for phosphoric acid was attempted to reduce the total reagent cost. The tests indicated that sulfuric acid could fully substitute for the phosphoric acid to obtain the target results from sample FLA-5, and the total reagent cost was thus considerably reduced. Figure 19 shows the test flowchart with the operating conditions and the flotation results in Table 51. Amine flotation was applied to remove silica, with reagent dosage and operating conditions similar to those for other samples.

From the test results of the sample FLA-5, it can be concluded that:

- With the “grinding-dolomite flotation-sizing-phosphate flotation” process, the beneficiation results obtained from the sample FLA-5 exceeded the target specified in the proposal;
- Increasing the particle size of flotation feed can lead to significant increase in reagent consumption, particularly dolomite collector. Therefore, the reasonable grinding fineness should be determined through evaluation on comprehensive factors.
- Sulfuric acid could be used to substitute for phosphoric acid to produce the target results.

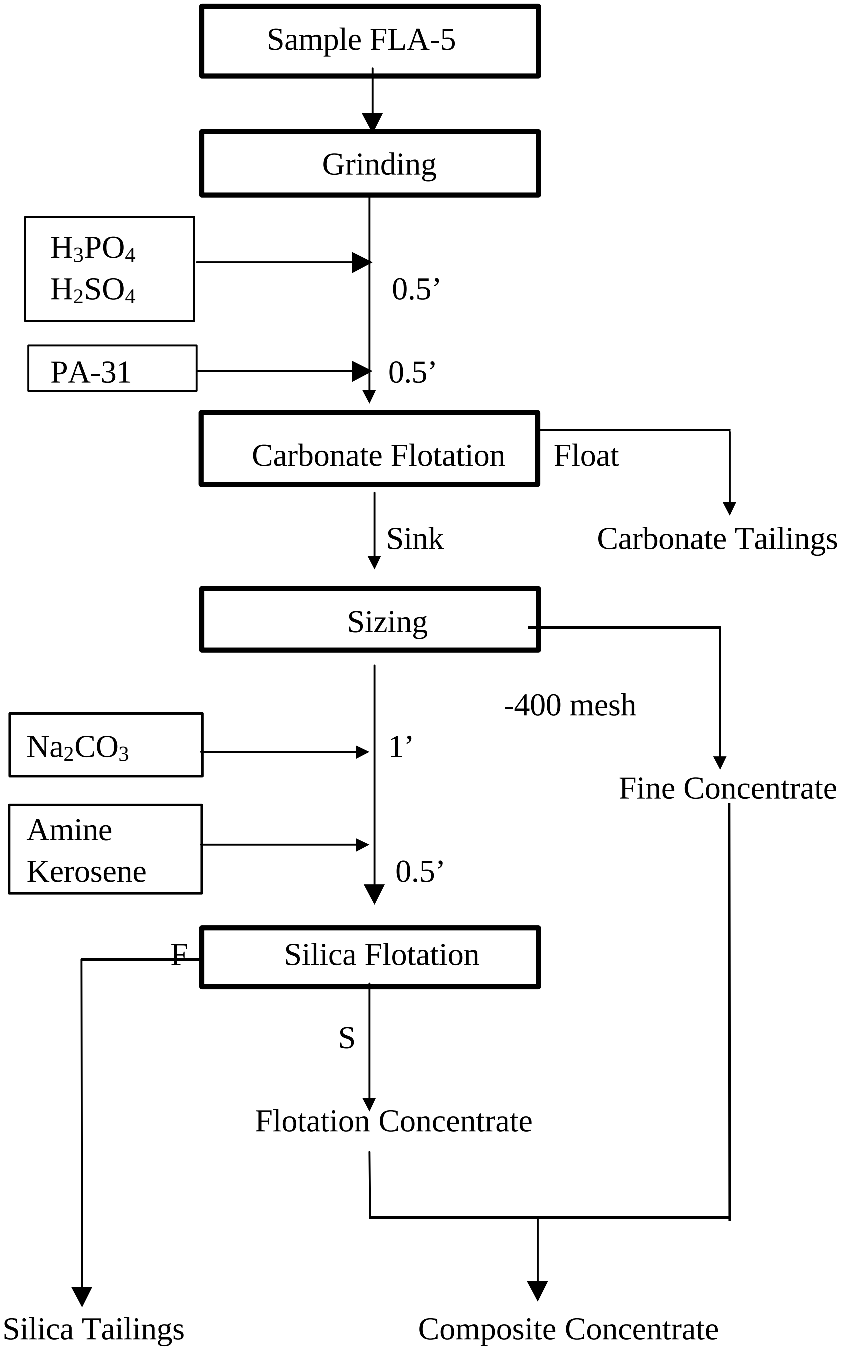

Figure 18. Principal Flotation Process for Sample FLA-5.

Table 49. Reagent Dosages for Flotation of Sample FLA-5 at Different Feed Sizes.

Grinding Fineness		Reagent Dosages (lb./t Feed)					
-200 Mesh (%)	Passing (mm)	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
35	0.5	5.6	4.0	5.6	0.6	0.8	0.25
44	0.3	5.0	4.0	3.6	0.6	0.8	0.25
63	0.15	5.0	4.0	1.6	0.6	0.8	0.25

Table 50. Size Distribution of Flotation Feed at 44%-200 Mesh for Sample FLA-5. (Grinding Time: 6 minutes;Fineness: 43.57%-200 Mesh).

Size (mm)						
+0.25	+0.20	+0.16	+0.071	+0.036	-0.036	Total
1.37	5.96	12.68	36.42	12.26	31.31	100.00

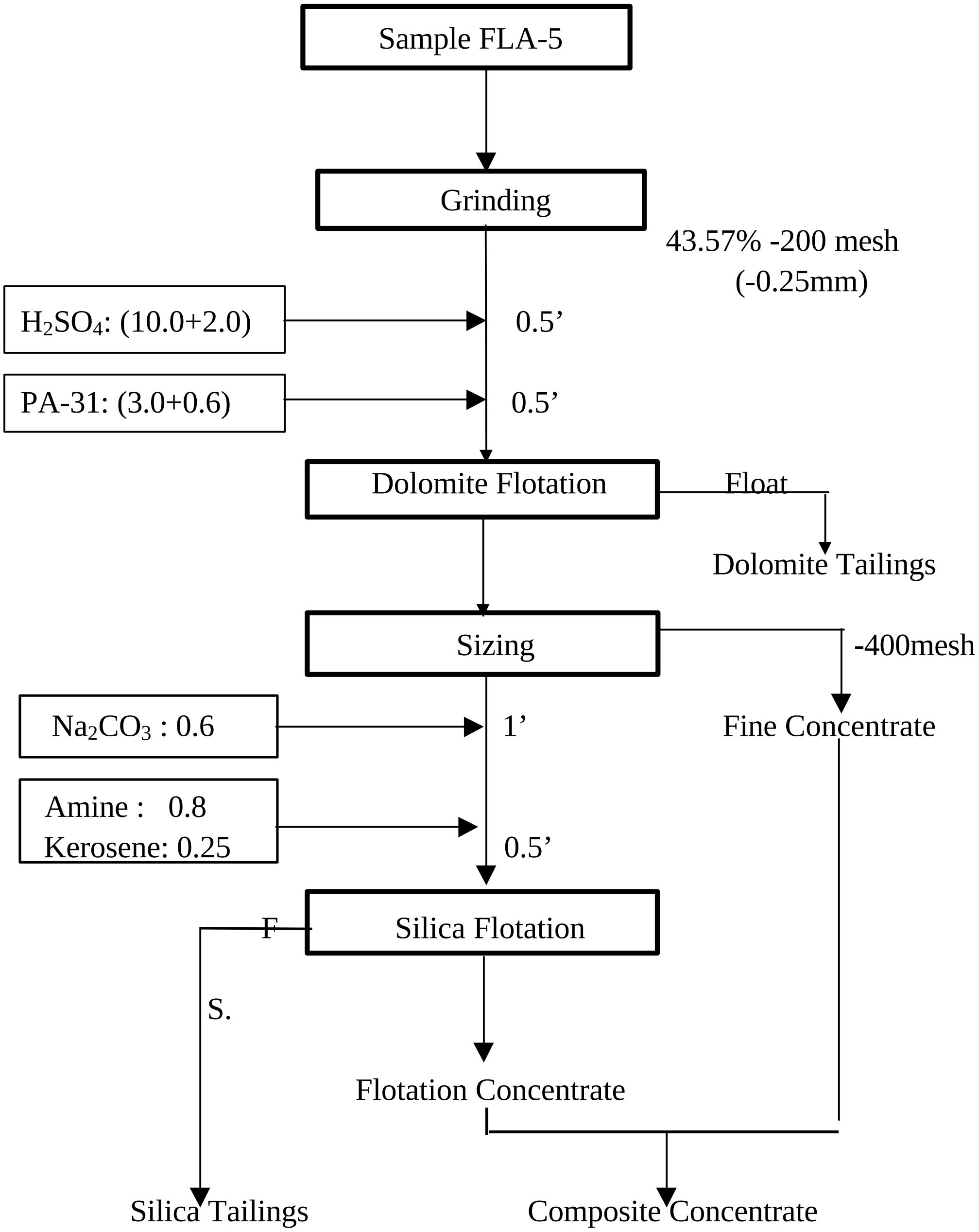

Figure 19. Flotation Process for Sample FLA-5 Using H_2SO_4 as pH Modifier.

Table 51. Flotation Results and Operating Conditions for Sample FLA-5 with Carbonate Flotation-Sizing-Silica Flotation. (Feed Size 44% -200 Mesh).

I. Flotation Results

Product	Weight (%)	Analysis (%) BPL	Analysis (%) MgO	Distribution (%) BPL	Distribution (%) MgO
Carbonate tailings	19.62	44.4	7.02	14.85	66.35
Silica tailings	8.14	7.2	0.31	1.01	1.44
-400mesh (fine concentrate)	12.72	64.5	0.63	13.99	3.85
Flotation concentrate	59.52	69.0	0.99	70.15	28.36
Head	100.00	58.6	2.08	100.00	100.00
Composite concentrate	72.24	68.2	0.93	84.14*	32.21

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent dosage, lb./t Feed					
	Solids (%)	Time (min.)	Solid (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation	34~35	0.5+0.5	34~35	7+4	4.9~6.0		10.0+2.0	3.0+0.6			
Silica flotation	24~25	1+1	24~25	1.5+1+1	8.5				0.6	0.8	0.25
Total reagent consumption, lb./t feed							12.0	3.6	0.6	0.8	0.25

* The BPL recovery is 81.35% after the deduction of BPL loss in primary slimes.

TESTS WITH DENVER CELL

All the above tests were carried out with the XFD-0.5L cell made in China. The proposal specified that comparison tests should be conducted with different types of flotation machines in order to make the CLDRI fine particle flotation technology more applicable to Florida usage. Therefore, flotation tests were carried out with a Denver D-12 cell on samples FLA-1, FLA-2, FLA-4 and FLA-5. For samples FLA-1, FLA-2 and FLA-5, the "grinding-dolomite flotation-sizing-silica flotation" process was used with fatty acid as the dolomite collector, and the "grinding-desliming-phosphate flotation" process was used for sample FLA-4. The operating conditions and the flotation results for samples FLA-1, FLA-2, FAL-4 and FLA-5 are listed in Tables 52, 53, 54 and 55, respectively.

The test results with the Denver cell indicated that similar flotation performances could be achieved to those obtained with the XFD-0.5L cell. The Denver cell could be used to meet the requirements of the fine particle flotation process if relevant adjustments and partial modification were made. To scale up to industrial operation, further pilot testing should be conducted. After suitable modification, the fatty acid collector currently used in the Florida area could be used to substitute for the PA-31, however the flotation results showed that the selectivity of the modified fatty acid collector seemed little bit poorer than that of PA-31. It is expected that this can be improved by further research work.

REUSE OF PROCESS WATER

Wet process phosphoric acid was used as a depressant in dolomite flotation. Its consumption is one key factor in the flowsheet. In order to reduce phosphoric acid consumption, the process water was recycled to grinding and dolomite flotation to investigate its effects on reagent dosages.

In laboratory testing, the process water of dolomite flotation from the first test run, including that from the underflow and float, was decanted and used for grinding and dolomite flotation in the second test run. The pH value of the recycled water was about 5.3-5.8. The process water of the second run was used for the third run and that of the third run was used for the fourth. The test was conducted on sample FLA-5 with the process "grinding-dolomite flotation-sizing-silica flotation." The operating conditions and the flotation results of the five runs are listed in Table 56.

It can be seen that recycling the process water in the dolomite flotation stage caused no problem in achieving the target results. The total acid consumption could be decreased by 10 to 20% due to the acidity of the recycled water. There was no considerable difference in PA-31 collector dosages.

Table 52. Flotation Results and Operating Conditions for Sample FLA-1 with Carbonate Flotation-Sizing-Silica Flotation Process Using Denver Flotation Cell and Fatty Acid Collector. (Particle Size of Flotation Feed: 55.3%-200 Mesh).

I. Flotation Results

Product	Weight (%)	Analysis (%) BPL	Analysis (%) P_2O_5	Analysis (%) MgO	Distribution (%) BPL	Distribution (%) MgO	
Carbonate tailings	18.38	45.5	20.83	5.42	15.62	57.80	
Silica tailings	14.46	5.0	2.28	0.10	1.35	0.58	
-400mesh (fine concentrate)	16.55	59.4	27.20	1.30	18.35	12.72	
Flotation concentrate	50.61	68.5	31.34	0.99	64.68	28.90	
Head	100.00	53.6	24.52	1.73	100.00	100.00	100.00
Composite concentrate	67.16	66.2	30.32	1.07	83.03	41.62	

I Operating Conditions

Operation	Conditioning Solids (%)	Conditioning Time (min.)	Flotation Solids (%)	Flotation Time (min.)	pH	Reagent dosage, lb./t Feed H_3PO_4 (P_2O_5)	H_2SO_4	Fatty acid	Na_2CO_3	Amine	Kerosene
Carbonate flotation	24~26	0.5+0.5	24~26	8.0+4.0	5.3~6.3	5.0	4.0	1.4+0.2	0.6	0.6	0.25
Silica flotation	16~18	1.0+0.5	16~18	1.5	8.5						
Total reagent consumption, lb./t feed						5.0	4.0	1.6	0.6	0.6	0.25

Table 53. Flotation Results and Operating Conditions for Sample FLA-2 with Carbonate Flotation-Sizing-Silica Flotation Process Using Denver Flotation Cell and Fatty Acid Collector. (Particle Size of Flotation Feed: 28.9%-200 Mesh).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	7.57	47.6	21.78	5.24		6.36	40.00	
Silica tailings	17.17	9.3	4.26	0.08		2.82	1.00	
-400mesh (fine concentrate)	10.59	62.9	28.77	1.35		11.76	14.00	
Flotation concentrate	64.67	69.3	31.70	0.70		79.06	45.00	
Head	100.00	56.7	25.93	1.00		100.00	100.00	100.00
Composite concentrate	75.26	68.4	31.29	0.78		90.82	59.00	

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed					
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	Fatty Acid	Na_2CO_3	Amine	Kerosene
Carbonate flotation	24~26	0.5+0.5	24~26	8.0+2.0	5.3~6.0	4.0	3.0	1.4+0.2			
Silica flotation	20~22	1.0+0.5	20~22	2.0	8.5				0.6	0.6	0.25
Total reagent consumption, lb./t feed						4.0	3.0	1.6	0.6	0.6	0.25

Table 54. Flotation Results and Operating Conditions for Sample FLA-4 with Grinding-Desliming-Phosphate Flotation Process Using Denver Flotation Cell and Fatty Acid Collector. (Particle Size of Flotation Feed: 31.5% -200 Mesh).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Slimes reject (-25 micron)	18.20	54.4	24.88	2.69	12.85	16.93	45.37	14.02
Silica tailings	12.12	10.1	4.64	0.66	80.35	2.09	7.41	58.36
Flotation concentrate	69.68	68.0	31.10	0.73	6.62	80.98	47.22	27.62
Head	100.00	58.5	26.76	1.08	16.69	100.00	100.00	100.00
Composite concentrate	69.68	68.0	31.10	0.73	6.62	80.98*	47.22	27.62

II.Operating Conditions

Operation	Condition		Flotation		Reagent Dosage, lb./t Feed						
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	NaOH	Na_2SiO_3	Fatty acid	Fuel Oil	Amine	Kerosene
Phosphate flot. I	24~26	1.0+1.0	24~26	3.5		1.0	3.0	2.0	1.5		
Phosphate flot. II	58	2.0		1.0				0.4	0.75		
Total reagent consumption, lb./t feed						1.0	3.0	2.4	2.25		

* The BPL recovery is 75.89% after the deduction of BPL loss in primary slimes.

Table 55. Flotation Results and Operating Conditions for Sample FLA-5 with Carbonate Flotation-Sizing-Silica Flotation Process Using Denver Flotation Cell and Fatty Acid Collector. (Particle Size of Flotation Feed: 43.6%-200 Mesh).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	18.59	45.1	20.66	6.78	5.11	14.30	63.64	7.97
Silica tailings	7.79	6.4	2.95	0.12	90.60	0.86	0.50	59.23
-400mesh (fine concentrate)	12.62	63.9	29.24	1.20	9.15	13.74	7.58	9.65
Flotation concentrate	61.00	68.4	31.30	0.91	4.53	71.10	28.28	23.15
Head	100.00	58.7	26.85	1.98	11.92	100.00	100.00	100.00
Composite concentrate	73.62	67.6	30.94	0.96	5.31	84.84*	35.86	32.80

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed					
	Solid (%)	Time (min.)	Solid (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	Fatty Acid	Na_2CO_3	Amine	Kerosene
Carbonate flotation	24~26	0.5+0.5	24~26	7.0+4.0	5.0~5.5		12+2	2.0+0.6			
Silica flotation	17~19	1.0+0.5	17~19	1.0	8.0				0.6	0.6	0.5
Total reagent consumption, lb./t feed							14	2.6	0.6	0.6	0.5

* The BPL recovery will be 82.03% after the deduction of BPL loss in primary slimes.

Table 56. Flotation Results and Operating Conditions for Sample FLA-5 with Carbonate Flotation-Sizing-Silica Flotation Process Using Recycled Water. (Particle Size of Flotation Feed: 63% -200 Mesh).

Recycled Water (times)	1. Flotation Results (Composite Concentrates) (%)					
	Weight	P_2O_5	BPL	MgO	Recovery (P_2O_5)	
					Flotation Feed	Scrubbing Feed
Fresh water	72.71	31.51	68.8	0.98	83.34	80.58
1st.	70.63	31.58	69.0	0.96	81.87	79.16
2nd.	72.98	31.54	68.9	0.96	84.42	81.63
3rd.	72.13	31.67	69.2	0.97	82.87	80.13
4th.	72.10	31.50	68.8	0.98	82.73	79.99
5th.	71.44	31.44	68.7	0.99	82.94	80.19

2. Reagent Dosage lb./t (Flotation Feed)

Recycled Water (times)	Dolomite Flotation			Silica Flotation			pH	
	H_3PO_4	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene	Dolo.Flot.	Cyc.Water
Fresh water	3.0	8.0	1.2	0.6	0.6	0.25	5.1-5.6	6.9
1st.	2.0	6.0	1.2	0.6	0.6	0.25	5.1-5.6	5.3
2nd.	2.0	6.0	1.2	0.6	0.6	0.25	5.1-5.5	5.6
3rd.	2.0	6.0	1.4	0.6	0.6	0.25	5.1-5.5	5.4
4th.	2.0	6.0	1.4	0.6	0.6	0.25	5.1-5.8	5.8
5th.	2.0	6.0	1.4	0.6	0.6	0.25	5.1-5.5	5.6

PRODUCT CHARACTERIZATION

CHEMICAL ANALYSIS OF CONCENTRATE

Phosphate concentrate from the five high-dolomite pebble samples was chemically analyzed, and the data are shown in Table 57. It can be seen that in terms of P_2O_5 and MgO, all the phosphate concentrate could meet the requirements specified in the proposal. The ratio of CaO to P_2O_5 is around 1.5, and the MER value is between 0.11 to 0.15. The MER is higher than 0.1 because of the high content of Fe_2O_3 and Al_2O_3 in the product. Microscopic studies indicated that there was some iron oxide contamination on the surface of the phosphate mineral, and this is difficult to remove by the flotation method. There were two types of aluminum-bearing minerals in the phosphate pebble samples; one was a clay mineral and another was aluminum-bearing phosphate, such as wavellite. The aluminum oxide in clay minerals could be removed by scrubbing, desliming or flotation, while it was impossible to separate aluminum-bearing phosphate by beneficiation procedures.

SIZE ANALYSIS OF CONCENTRATE

All the phosphate concentrates were analyzed for size distribution and chemical composition. The analysis data are presented in Tables 58 through 64.

From size analysis, it could be observed that the MgO content became lower in finer sizes, which indicated that the CLDRI flotation technology was very effective in beneficiating fine-grained phosphate. There are two possible reasons why there was more MgO in coarse sizes than in fines. The first is that dolomite was not well liberated from phosphate in coarse sizes; or possibly the flotation process, operating conditions and reagent regimen were more suitable for floating fine dolomite, while not effective enough for coarse sizes.

In China, most of the phosphate ores must be ground very fine for liberation, such as 70% -200 mesh for the Wengfu Phosphate Processing Plant and 93% -200 mesh for the Dayukou Plant. It is understood that the concentrate produced from Florida dolomitic pebbles is quite coarse compared to the particle sizes of the flotation concentrates from the above-mentioned plants. In China, there is no technical problem in dewatering and transporting such fine phosphate concentrate.

Table 57. Chemical Analysis of Flotation Concentrates from Different Samples.

Sample	Product	Analysis, %										
		P_2O_5	BPL	MgO	A.I.	CaO	CO_2	SiO_2	Fe_2O_3	Al_2O_3	F	I.L
FLA-1	Flotation conc.	32.39	70.77	0.82	2.79	47.67	6.04	2.00	1.85	1.69	3.36	8.53
	-400 mesh fines	27.99	61.16	1.16	14.39	41.63	5.02	12.96	2.23	1.31	2.81	8.32
	Composite conc.	31.31	68.41	0.91	5.61	46.20	5.79	4.67	1.94	1.60	3.23	8.48
FLA-2	+150 mesh conc.	31.44	68.70	0.72	4.08	46.71	5.44	2.16	1.82	2.24	3.34	7.68
	-150 mesh conc.	32.06	70.05	0.66	3.59	47.53	5.70	3.07	1.79	1.40	3.40	7.71
	-400 mesh fines	28.94	63.23	1.13	9.69	43.09	5.84	8.65	2.03	2.42	2.99	8.94
	Composite conc.	31.32	68.43	0.76	4.61	46.50	5.55	3.14	1.84	2.05	3.31	7.83
FLA-3	Concentrate	31.77	69.42	0.96	3.40	47.99	5.48	1.93	1.43	1.29	3.34	8.46
FLA-4	Concentrate	30.98	67.69	0.63	8.50	46.20	4.80	6.67	1.79	1.01	3.18	7.22
FLA-5	Flotation conc.	31.30	68.39	0.91	4.83	47.24	5.00	2.12	1.71	2.04	3.44	7.89
	-400 mesh fines	29.24	63.89	1.20	8.93	44.13	5.24	6.80	2.05	1.50	2.97	8.72
	Composite conc.	30.94	67.60	0.96	5.53	46.71	5.04	2.92	1.77	1.95	3.36	8.03

Table 58. Size Distribution and Chemical Analysis of Composite Concentrate of Sample FLA-1. (Particle Size of Flotation Feed: 41.01% -200 mesh; Flotation Process: Grinding-Carbonate-Silica Flotation).

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.200	10.01	68.0	31.10	0.99	4.58	9.99	12.50	8.08
0.200-0.160	17.02	68.2	31.20	0.98	4.00	17.05	21.25	11.95
0.160-0.100	25.82	69.2	31.65	0.81	4.03	26.24	26.25	18.28
0.100-0.071	15.22	70.2	32.14	0.57	4.51	15.70	11.25	12.13
0.071-0.036	16.82	70.2	32.12	0.30	4.83	17.34	6.25	14.23
-0.036	15.11	61.6	28.17	1.18	13.33	13.68	22.50	35.33
Total	100.00	68.0	31.14	0.80	5.69	100.00	100.00	100.00

Table 59. Size Distribution and Chemical Analysis of Composite Concentrate of Sample FLA-1. (Particle Size of Flotation Feed: 55.33% -200 Mesh; Flotation Process: Grinding-Carbonate-Silica Flotation).

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.160	0.89	69.5	31.81	0.98	2.60	23.87	23.96	12.08
0.160-0.100	22.43							
0.100-0.071	26.89	70.1	32.10	0.98	2.50	27.77	27.08	13.27
0.071-0.036	25.46	70.5	32.27	0.75	2.32	26.45	19.79	11.68
-0.036	24.33	61.2	27.99	1.16	13.09	21.91	29.17	62.97
Total	100.00	67.9	31.08	0.96	5.05	100.00	100.00	100.00

Table 60. Size Distribution and Chemical Analysis of Composite Concentrate of Sample FLA-2. (Particle Size of Flotation Feed: 28.9% -200 Mesh; Flotation Process: Grinding-Sizing-Flotation).

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.315	5.98	67.2	30.76	0.63	7.63	5.91	5.06	10.22
0.315-0.200	30.00	68.2	30.23	0.77	4.22	30.09	29.11	28.22
0.200-0.160	11.42	68.2	31.20	0.86	3.40	11.43	12.66	8.67
0.160-0.100	15.91	69.0	31.59	0.85	2.80	16.15	17.72	10.00
0.100-0.071	12.13	69.2	31.69	0.67	3.74	12.33	10.13	10.00
0.071-0.036	13.00	69.7	31.91	0.57	3.10	13.33	8.86	8.89
-0.036	11.56	63.2	28.94	1.13	9.35	10.76	16.46	24.00
Total	100.00	68.0	31.14	0.79	4.50	100.00	100.00	100.00

Table 61. Size Distribution and Chemical Analysis of Composite Concentrate of Sample FLA-2. (Particle Size of Flotation Feed: 28.9% -200 Mesh; Flotation Process: Grinding-Carbonate Flotation-Sizing-Silica Flotation).

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.315	5.61	68.4	31.32	0.61	13.05	5.66	3.75	15.30
0.315-0.200	28.53	68.1	31.16	0.69	4.44	28.58	25.00	26.63
0.200-0.160	12.77	68.6	31.40	0.88	3.33	12.89	13.75	9.01
0.160-0.100	16.00	69.2	31.66	0.84	2.30	16.39	17.50	7.76
0.100-0.071	9.98	69.9	31.99	0.69	2.42	10.25	8.75	5.03
0.071-0.036	11.55	70.3	31.18	0.65	2.09	11.96	10.00	5.03
-0.036	15.46	62.7	29.71	1.10	9.66	14.27	21.25	31.24
Total	100.00	68.0	31.11	0.80	4.77	100.00	100.00	100.00

Table 62. Size Distribution and Chemical Analysis of Composite Concentrate of Sample FLA-3. (Particle Size of Flotation Feed: 69.71% -200 Mesh; Flotation Process: Grinding-Carbonate Flotation-Silica Flotation).

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.160	2.95	66.0	30.21	1.82	4.78	2.81	5.05	4.46
0.160-0.100	30.03	67.0	30.67	1.46	3.20	29.04	44.45	30.57
0.100-0.071	32.97	70.1	31.07	0.82	3.00	33.33	27.27	31.53
0.071-0.036	24.13	71.1	32.53	0.69	3.14	24.76	17.17	24.20
-0.036	9.92	70.3	32.16	0.61	2.94	10.06	6.06	9.24
Total	100.00	69.3	31.71	0.99	3.14	100.00	100.00	100.00

Table 63. Size Distribution and Chemical Analysis of Composite Concentrate of Sample FLA-4. (Particle Size of Flotation Feed: 30.65% -200 Mesh; Flotation Process: Grinding-Desliming-Phosphate Flotation).

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.315	2.49	70.8	32.38	0.46	3.74	2.66	1.52	1.09
0.315-0.200	30.79	68.5	31.36	0.56	6.56	31.76	25.76	24.40
0.200-0.160	17.16	66.5	30.44	0.63	9.16	17.16	16.67	18.96
0.160-0.100	21.41	64.6	29.58	0.66	10.46	20.81	21.21	27.05
0.100-0.071	12.02	65.5	29.97	0.70	9.54	11.83	12.12	13.89
0.071-0.036	10.56	65.7	30.08	0.77	8.00	10.45	12.12	10.14
-0.036	5.57	63.7	29.16	1.25	6.69	5.33	10.60	4.47
Total	100.00	66.5	30.42	0.66	8.28	100.00	100.00	100.00

Table 64. Size Distribution and Chemical Analysis of Composite Concentrate of Sample FLA-5. (Particle Size of Flotation Feed: 43.57% -200 Mesh; Flotation Process: Grinding-Carbonate Flotation-Sizing-Silica Flotation).

Size (mm)	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
+0.200	5.80	69.8	31.95	0.63	4.00	5.97	4.00	4.50
0.200-0.160	13.98	69.2	31.67	1.02	3.96	14.28	14.00	10.76
0.160-0.100	27.45	67.3	30.81	1.16	4.23	27.27	32.00	11.70
0.100-0.071	16.70	68.4	31.30	1.03	4.62	16.86	17.00	15.07
0.071-0.036	18.93	69.7	31.91	0.64	4.37	19.47	12.00	16.24
-0.036	17.14	63.9	29.24	1.20	9.15	16.15	21.00	30.73
Total	100.00	67.8	31.02	1.00	5.11	100.00	100.00	100.00

SUPPLEMENTAL TESTS

In late August 1998, FIPR dispatched a five-member delegation to CLDRI to conduct an on-site assessment of CLDRI's test work. It was conducted in a workshop format, involving a laboratory demonstration, a tour of CLDRI facilities, presentations, and discussions. After an evaluation of the research work done by CLDRI, the delegation recommended a series of tests as the extension of the laboratory research project. These tests would consist of :

a. Increasing particle size of flotation feed;
b. Conditioning the flotation feed at high solid percent;
c. Using sulfonated fatty acid as a dolomite collector to substitute for PA-31;
d. Determining the effectiveness of limestone removal with CLDRI process.

INCREASING PARTICLE SIZE OF FLOTATION FEED

The tests aimed at increasing the particle size of the flotation feed were conducted on samples FLA-1, FLA-2, FLA-4 and FLA-5.

In the tests on sample FLA-1, flotation tests were carried out at a feed particle size of 55.33% -200 mesh, about 0.2 mm, with the operating conditions and test results listed in Tables 30, 31 and 32. In order to investigate the possibility of increasing the particle size of the flotation feed, the sample was ground to 41% -200 mesh for flotation. The size distribution and chemical analysis of the ground sample are given in Table 33. Figure 11 shows the flotation flowsheet, with the operating conditions and the test results listed in Table 34. Comparing the flowsheet and test results with those in Figure 10 and Table 32, it can be seen that, using the same process, the same flotation results could be obtained, but the reagent consumption was significantly increased, particularly the dolomite collector, from 1.6 lb/t of feed for -0.2 mm feed to 5.0 lb/t of feed for -0.3 mm feed.

Similar tests were carried out on sample FLA-5. From Tables 48 and 49, it can be seen that the target result could be obtained when the particle size of the flotation feed was increased from 63% -200 mesh (about 0.16 mm) to 35% -200 mesh (about 0.4 mm), but dolomite collector PA-31 dosage was increased from 1.6 to 5.6 lb per ton of feed.

For sample FLA-2, the particle size of the flotation feed was increased from 28.9% -200 mesh (about 0.4 mm) to 21.88% -200 mesh (about 0.5 mm). The size distribution and chemical analysis of 21.88% -200 mesh feed are given in Table 65. Table 66 presents the operating conditions and the flotation results with the "grinding-dolomite flotation-sizing-silica flotation" process as shown in Figure 12. Table 67 gives the beneficiation performances with the "grinding, sizing, coarse size for phosphate flotation, fine size for dolomite flotation-sizing-silica flotation" flowsheet. From Tables 66 and 67, it can be seen that same flotation results could be obtained when the particle

Table 65. Size Distribution and Chemical Analysis as Sample FLA-2 Was Ground to 21.88%-200 Mesh.

Size (mm)	Weight (%)	Analysis (%)			Distribution (%)	
		BPL	P_2O_5	MgO	BPL	MgO
+0.500	2.76	48.6	22.24	0.36	2.38	0.92
0.500~0.400	9.64	55.0	25.17	0.44	9.49	3.67
0.400~0.315	13.37	55.5	25.39	0.52	13.24	6.42
0.315~0.200	23.22	54.1	24.78	0.78	22.46	20.18
0.200~0.160	9.39	55.2	25.26	0.87	9.26	7.34
0.160~0.100	11.68	56.9	26.06	0.74	11.88	8.26
0.100~0.071	8.06	60.0	27.48	0.88	8.63	6.42
0.071~0.036	6.68	61.1	27.97	1.14	7.31	7.34
-0.036	15.20	56.5	25.88	2.81	15.35	39.45
Total	100.00	55.9	25.60	1.09	100.00	100.00
+0.10	70.06	54.9	25.11	0.73	68.71	46.79
-0.10	29.94	58.4	26.71	1.94	31.29	53.21

Table 66. Flotation Results and Operating Conditions for Sample FLA-2 by Increasing Feed Size to 21.88% -200 Mesh. (Flotation Process: Grinding-Carbonate Flotation-Sizing-Silica Flotation).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	8.98	48.2	22.05	5.23	4.90	7.73	45.19	2.25
Silica tailings	15.16	6.6	3.02	0.15	90.65	1.80	1.92	70.25
-400mesh (fine concentrate)	6.74	63.5	29.06	0.86	9.66	7.66	4.77	3.32
Flotation concentrate	69.12	67.0	30.67	0.71	6.84	82.81	47.12	24.18
Head	100.00	55.9	25.60	1.04	19.56	100.00	100.00	100.00
Composite concentrate	75.86	66.7	30.53	0.73	7.09	90.47	52.89	27.50

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed					
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation	32~34	0.5+0.5	32~34	7+3	5.1~5.9	4.0	3.0	2.0+0.2			
Silica flotation	27~29	1.0+0.5	27~29	4	9.0~8.5				0.6	1.4	0.75
Total reagent consumption, lb./t feed						4.0	3.0	2.2	0.6	1.4	0.75

Table 67. Flotation Results and Operating Conditions for Sample FLA-2 by Increasing Feed Size to 21.88% -200 Mesh. (Flotation Process: Grinding-Sizing-Flotation).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Silica tailings (+150mesh)	14.27	6.4	2.94	0.14	90.71	1.65	1.89	66.91
Flotation concentrate (+150mesh)	55.64	67.2	30.76	0.84	5.48	66.99	44.34	15.77
Carbonate tailings	6.35	44.2	20.03	6.08	6.62	4.97	36.79	2.17
Silica tailings (-150mesh)	2.47	11.3	5.16	0.15	83.86	0.51	0.94	10.70
-400 mesh (fine concentrate)	6.13	63.6	29.09	1.08	7.90	6.97	6.60	2.48
Flotation concentrate (-150mesh)	15.14	69.7	31.92	0.66	2.54	18.91	9.44	1.97
Head	100.00	55.8	25.54	1.06	19.34	100.00	100.00	100.00
Composite concentrate	76.91	67.4	30.84	0.83	5.08	92.87	60.38	20.22

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed					
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosen
Silica flot. (+150mesh)	29	1.0+0.5	29	1+1	9.0~8.5				0. 5	1.3	0.64
Carbonate flotation	36	0.5+0.5	36	7+3	5.2~6.0	1.5	1.0	0. 5			
Silica flot. (-150mesh)	23	1.0+0.5	23	1+1+1	9.0~8.5				0.15	0.3	0.188
Total reagent consumption, lb./t feed						1.5	1.0	0. 5	0.65	1.6	0.828

size of the flotation feed was increased from 0.4 mm to 0.5 mm, and the reagent consumption was also increased, but not too much.

For sample FLA-4, the flotation test was carried out by increasing the particle size of the flotation feed from 31.52% -200 mesh (about 0.35 mm) to 22.45% -200 mesh (about 0.5 mm). The size distribution of -0.5 mm feed is provided in Table 68. The "grinding-desliming-phosphate flotation" process, as shown in Figure 16, was used. The operating conditions and flotation results are listed in Table 69.

The test results indicated that when the particle size of the flotation feed was increased, the target results could be obtained by increasing the reagent consumption.

CONDITIONING FLOTATION FEED AT HIGH SOLIDS

Tests on conditioning the flotation feed at high solids percent were conducted for dolomite flotation of sample FLA-1 and phosphate flotation of sample FLA-4.

Dolomite Flotation

In the test, the ground slurry was conditioned with mixed acids and PA-31 collector at 60% solids, then subjected to dolomite flotation. The underflow of the dolomite flotation was sized to produce a fine concentrate and the plus fraction was sent to amine flotation to remove silica. The operating conditions and flotation results are given in Table 70.

It can be seen that there were no remarkable changes in the efficiency of dolomite flotation and in reagent consumption when the solids were increased from 30% to 60%. Therefore, the ground slurry could be directly sent to conditioning for dolomite flotation with no thickening or dilution.

Phosphate Flotation

The test was conducted on sample FLA-4 with the "grinding-desliming-phosphate flotation" process. The operating conditions and the flotation results are listed in Table 71.

From Table 71 it can be observed that increasing the conditioning solids of phosphate flotation was favorable to floating coarse particles, which could improve the overall phosphate recovery, but there was no evident decrease in the consumption of the fatty acid collector.

Table 68. Size Distribution and Chemical Analysis as Sample FLA-4 Was Ground to 22.45% -200 Mesh.

Size (mm)	Weight (%)	Analysis (%)			Distribution (%)	
		BPL	P_2O_5	MgO	BPL	MgO
+0.500	2.72	64.0	29.27	0.83	2.98	1.82
0.500~0.400	8.82	64.4	29.47	0.85	9.70	6.36
0.400~0.315	13.16	62.2	28.47	0.78	13.99	9.09
0.315~0.200	24.39	57.1	26.12	0.82	23.76	18.18
0.200~0.160	10.39	55.1	25.23	0.98	9.77	9.09
0.160~0.100	11.28	57.2	26.18	0.80	11.00	8.18
0.100~0.071	6.79	61.1	27.97	0.66	7.09	3.64
0.071~0.025	8.72	62.9	28.80	1.22	9.36	10.00
-0.025	13.73	52.6	24.11	2.70	12.35	33.64
Total	100.00	58.6	26.81	1.10	100.00	100.00
+0.025	86.27	59.5	27.24	0.85	88.65	66.36

Table 69. Flotation Results and Operating Conditions for Sample FLA-4 with Grinding-Desliming-Phosphate Flotation Process by Increasing Feed Size to 22.45% -200 Mesh.

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Slimes (-25micron)	12.98	52.2	23.91	3.08	12.60	11.52	40.82	10.31
Silica tailings	12.61	17.6	8.04	0.69	69.81	3.75	19.39	55.35
Flotation concentrate I	27.88	66.6	30.46	0.61	6.98	31.55	20.41	12.26
Flotation concentrate II	32.87	68.5	31.34	0.63	6.21	38.28	9.18	12.83
Flotation concentrate III	13.66	64.2	29.37	0.83	10.75	14.90	10.20	9.25
Head	100.00	58.8	26.91	0.98	15.90	100.00	100.00	100.00
Composite concentrate	74.41	66.9	30.64	0.65	7.34	84.73*	48.94	34.34

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed			
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	NaOH	Na_2SiO_3	Fatty Acid	Fuel Oil
Phosphate flotation I	28~30	1+2	28~30	3.5	10~9.5	1.0	3.0	2.0	1.5
Phosphate flotation II	62	2	20~22	1	9.5~9.0			1.0	0. 75
Phosphate flotation III	40	2	9~10	1	9.0~8.5			0.4	0. 5
Total reagent consumption, lb./t feed						1.0	3.0	3.4	2.75

* The BPL recovery will be 79.40% after the deduction of BPL loss in slimes.

Table 70. Flotation Results and Operating Conditions for Sample FLA-1 by Conditioning at High Solids for Dolomite Flotation (Particle Size of Flotation Feed: 41.01% -200 Mesh; Flotation Process: Grinding-Carbonate Flotation-Sizing-Silica Flotation).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	21.38		21.20			18.69		
Silica tailings	14.58		2.52			1.53		
-400 mesh (fine concentrate)	8.59		27.38			9.69		
Flotation concentrate	55.45		30.64			70.09		
Head	100.00		24.24			100.00		
Composite concentrate	64.04		30.20			79.78		

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed					
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation	60	0.5+0.5	32~34	7+3	5.5~6.1	5.0+0.6	4.0	3.0+0.6	0.6	0.4+0.2+0.2	0.25+0.25
Silica flotation	23~25	1.0+0.5	23~25	2+1+1	8.5						
Total reagent consumption, lb./t feed						3.6	4.0	3.6	0.6	0.4	0.5

Table 71. Flotation Results and Operating Conditions for Sample FLA-4 by Conditioning at High Solids for Phosphate Flotation (Particle Size of Flotation Feed: 22% -200 Mesh; Flotation Process: Grinding-Desliming-Phosphate Flotation).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Slimes (-25micron)	13.64	51.9	23.76			12.16		
Silica tailings	14.53	21.0	9.60			5.22		
Flotation concentrate	60.18	67.6	30.93			69.86		
Flotation concentrate	11.65	63.8	29.18			12.76		
Head	100.00	58.2	26.64			100.00		
Composite concentrate	71.83	66.9	30.64			82.62*		

I. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed			
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	Na_2CO_3	Na_2SiO_3	Fatty Acid	Fuel Oil
Flotation conc.	70	2	28~30	2	10~9.5	2.0	3.0	3.6	3.0
Flotation conc.	65	2	9~10	1	9.0~8.5			0.4	0. 5
Total reagent consumption, lb./t feed						2.0	3.0	4.0	3.5

*The BPL recovery will be 77.42% after the deduction of BPL loss in primary slimes.

USING SULFONATE FATTY ACID AS A DOLOMITE COLLECTOR

In order to investigate the possibility of using sulfonate fatty acid to substitute for PA-31 as a dolomite collector, sodium dodecyl benzene sulfonate and sodium dodecyl sulfonate were used in dolomite flotation. During the verification testing done by CLDRI investigators in the IMC-Agrico laboratory in December 1998, sulfonate fatty acid was also used.

Sodium Dodecyl Benzene Sulfonate

This test was done on sample FLA-1 with the “grinding-dolomite flotation-sizing-silica flotation” process. The operating conditions and the flotation results are given in Table 72.

Sodium Dodecyl Sulfonate

The same sample and the same process were tested with sodium dodecyl sulfonate as a dolomite collector. Table 73 shows the operating conditions and the flotation results.

Sulfonated Fatty Acid

This test was conducted in the IMC-Agrico laboratory on sample FLA-5 with the “grinding-dolomite flotation-sizing-phosphate flotation” process. The operating conditions and flotation results are presented in Table 74.

The data indicated that using sulfonate-type collectors failed to obtain the target result due to the poor selectivity of separating the dolomite from the phosphate. All the sulfonate collectors produced abundant and stable bubbles, which led to poor fluidity of the froth product. It can be concluded that a sulfonate-type collector cannot be satisfactorily used to substitute for PA-31 in dolomite flotation.

DETERMINING THE EFFECTIVENESS OF LIMESTONE REMOVAL WITH THE CLDRI PROCESS

The test sample, which contained high limestone, was taken from Pine Level. The sample was analyzed at 48.0% BPL, 1.93% MgO, 36.69% CaO, 16.43% insoluble, 1.71% Fe_2O_3, 1.95% Al_2O_3, etc. During grinding for mineral liberation, it was found that the sample was easily over-ground; part of the ground material stuck firmly on the surfaces of the rods and mill and was difficult to wash off. The flotation test was run in the IMC-Agrico laboratory while conducting verification tests in December 1998. The “grinding-dolomite flotation-sizing-silica flotation” process was tried. The operating conditions and the flotation results are shown in Table 75. It can be seen that the results

Table 72. Flotation Results and Operating Conditions for Sample FLA-1 with Sodium Dodecyl Benzene Sulfonate (SDBS) as Collector. (Particle Size of Flotation Feed: 55.33% - 200 Mesh; Flotation Process: Grinding-Carbonate Flotation-Sizing-Silica Flotation).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	27.17		24.00			26.72		
Silica tailings	13.33		1.49			0.82		
-400mesh (fine concentrate)	9.42		25.13			9.71		
Flotation concentrate	50.08		30.57			62.75		
Head	100.00		24.40			100.00		
Composite concentrate	59.50		29.71			72.46		

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed					
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	SDBS	Na_2CO_3	Amine	Kerosene
Carbonate flotation	32~35	0.5+0.5	32~35	7+3	5.2~6.3	5.0	4.0	1.6+0.4			
Silica flotation	22~24	1+0.5	22~24	1.5+1+1	8.5				0.6	1.0	0.5
Total reagent consumption, lb./t feed						5.0	4.0	2.0	0.6	1.0	0.5

Table 73. Flotation Results and Operating Conditions for Sample FLA-1 with Sodium Dodecyl Sulfonate (SDS) as Collector. (Particle Size of Flotation Feed: 55.33%-200 Mesh; Flotation Process: Grinding-Carbonate Flotation-Sizing-Silica Flotation).

I. Flotation Results

Product	Weight (%)	Analysis (%)				Distribution (%)		
		BPL	P_2O_5	MgO	A.I.	BPL	MgO	A.I.
Carbonate tailings	17.70	52.44	24.00			17.35		
Silica tailings	13.41	4.41	2.02			1.10		
-400mesh (fine concentrate)	16.83	54.25	24.83			17.06		
Flotation concentrate	52.06	66.29	30.34			64.49		
Head	100.00	53.53	24.50			100.00		
Composite concentrate	68.89	63.37	29.00			81.55		

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage, lb./t Feed					
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	SDS	Na_2CO_3	Amine	Kerosene
Carbonate flotation	32~35	0.5+0.5	32~35	5+3	5.2~5.9	5.0	4.0	1.6+0.2			
Silica flotation	22~24	1+0.5	22~24	1.5+1+1	8.5				0.6	1.0	0.5
Total reagent consumption, lb./t feed						5.0	4.0	1.8	0.6	1.0	0.5

Table 74. Flotation Results and Operating Conditions for Sample FLA-5 Using Sulfonated Fatty Acid and Florida Deep Well Water. (Particle Size of Flotation Feed: -0.2 mm; Process: Grinding-Carbonate Flotation-Sizing-Silica Flotation).

I. Flotation Results

Products	Wt. (%)	Analysis (%)								Distribution (%)	
		BPL	P_2O_5	MgO	A.I.	Fe_2O_3	Al_2O_3	CaO	CO_2	BPL	MgO
Carbonate tailings	25.95	52.52	24.03	3.20	6.16	1.65	1.06	42.23	11.63	23.37	50.30
Silica tailings	6.88	6.53	2.99	0.10	83.62	1.21	0.13	4.57	0.94	0.79	0.61
-400mesh(fine conc.)	10.65	62.91	28.79	1.15	6.28	1.79	1.11	44.77	5.61	11.50	7.27
Flotation conc.	56.52	66.40	30.39	1.22	1.02	1.54	1.02	47.29	6.03	64.34	41.82
Calc.head	100.00	58.34	26.70	1.65	8.60	1.57	0.98	42.77	7.09	100.00	100.00
Composite concentrate	67.17	65.85	30.14	1.21	1.83	1.58	1.03	46.89	5.96	75.84*	49.09

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage (lb./t Feed)					
	Solids (%)	Time (min.)	Solids (%)	Time (min.)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	Sulfonate Fatty Acid	Na_2CO_3	Amine	Kerosene
Carbonate flotation	30-32	0.5+0.5	28-30	5.0+3.0	4.5-5.5	5.0	4.0	1.6+0.4	---	---	---
Silica flotation	20-22	1.0+0.5	18-20	1.5+1.0	9-8	---	---	---	0.6	0.8	0.2
Total reagent consumption (lb./t feed)						5.0	4.0	2.0	0.6	0.8	0.2

* The BPL recovery will be 73.33% after the deduction of BPL loss in primary slimes.

Table 75. Flotation Results and Operating Conditions for the Pine Level Sample with Grinding-Carbonate Flotation-Sizing-Phosphate Flotation Process Using Four Corners Plant Water.

I. Flotation Results

Products	Wt. (%)	Analysis (%)							Distribution (%)	
		BPL	P_2O_5	MgO	A.I.	Fe_2O_3	Al_2O_3	CaO	BPL	MgO
Carbonate tailings	35.73	38.96	17.83	3.22	20.62	1.97	2.90	32.52	28.57	58.49
Silica tailings	5.76	10.15	4.65	0.25	78.55	0.81	0.31	7.50	1.20	0.73
-400mesh(fine conc.)	16.30	42.79	19.58	1.91	25.22	2.36	3.31	31.27	14.32	15.85
Flotation concentrate	42.21	64.52	29.53	1.16	2.40	1.42	0.89	46.90	55.91	24.93
Calculated head	100.00	48.70	22.29	1.96	17.01	1.74	1.97	36.94	100.00	100.00
Composite concentrate	58.51	58.47	26.76	1.37	8.76	1.68	1.56	42.55	70.23	40.78

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage (lb/t Feed)						
	Solids (%)	Time (min)	Solids (%)	Time (min)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Na_2SiO_3	Fatty Acid	Fuel Oil
Carbonate flotation	30-32	0.5+0.5	28-30	5.0+4.0	4.5-5.5	7.0	4.0	1.6+0.4	---	---	---	---
Phosphate flotation	20-22	1.0+0.5	16-18	1.5+1.0	9-8	---	---	---	1.0	2.0	2.0	0.24
Total reagent consumption (lb/t feed)						7.0	4.0	2.0	1.0	2.0	2.0	0.24

were not satisfactory. The removal rate of MgO in dolomite flotation was not as good as that for other samples. After dolomite flotation, -400 mesh fines in the underflow contained only about 43% BPL with quite high MgO, which could not be used as fine concentrate. Even though only a few runs were made on this sample, it can be possibly concluded that the process developed for the other samples might not work well on it and that further studies should be done to develop a suitable flowsheet for the effective recovery of phosphate from such a high-limestone phosphate resource.

VERIFICATION TESTING IN IMC-AGRICO'S LABORATORY

In the proposal, it was specified that as a final step in the process development, a series of lab tests should be made at IMC-Agrico Company's facilities with local deep-well water and plant water to verify there are no problems in the application of Chinese technology in Florida. Therefore, CLDRI investigators carried out such verification tests in IMC-Agrico Company's lab in order to investigate the effects of the water sources on the CLDRI flotation process.

The sample for the verification test was taken from a pebble pile in IMC-Agrico's laboratory yard, which was chemically similar to sample FLA-5. The sample contained 54.44% BPL, 3.32% MgO, 9.33% insoluble, 0.99% Al_2O_3 and 1.50% Fe_2O_3. The sample was scrubbed in a modified hand-drill scrubber at the conditions of 60% solids and 380 rpm impeller speed for 6 minutes and sized to reject -150 mesh fine particles since it had been contaminated with some primary slimes during long-time storage. The deslimed sample contained 58.41% BPL, 2.10% MgO, 9.38% insoluble, 1.00% Al_2O_3 and 1.54% Fe_2O_3. The +150 mesh fraction was air-dried and roll-crushed to -1 mm. The crushed sample (400 grams per batch) was ground to 55% -200 mesh in a rod mill and then subjected to flotation. The flotation was carried out in a Denver D-12 cell. Dolomite was first floated with wet phosphoric acid and sulfuric acid as a pH modifier and depressant, and with PA-31 as collector. The underflow was sized to remove -400 mesh as fine concentrate. The +400 mesh fraction was subjected to either amine flotation or fatty acid flotation for the separation of silica from phosphate. This flotation concentrate was combined with -400 mesh fines to form a final product. The principal flotation flowsheet is given in Figure 19. During flotation testing, both deep-well water and plant water were used.

Tables 76 and 77 present the operating conditions and the flotation results of the "grinding-dolomite flotation-sizing-silica flotation" and "grinding-dolomite flotation-sizing-phosphate flotation" processes. Both of those tests were conducted using Florida deep-well water. Table 78 shows the operating conditions and the flotation results of the "grinding-dolomite flotation-sizing-silica flotation" process using Four Corners plant water.

Verification test results indicate that CLDRI fine particle flotation technology had very good adaptability to the fluctuation of water quality. The tests further verified there should no problems in the application of the CLDRI process in Florida.

Table 76. Verification Test Results for Sample FLA-5 with Grinding-Carbonate Flotation-Sizing-Silica Flotation Process Using Florida Deep Well Water.

I. Flotation Results

Products	Wt. (%)	Analysis (%)							Distribution (%)	
		BPL	P_2O_5	MgO	A.I.	Fe_2O_3	Al_2O_3	CaO	BPL	MgO
Carbonate tailings	18.52	41.06	18.79	8.10	4.12	1.80	0.92	42.09	13.03	72.81
Silica tailings	6.31	6.29	2.88	0.03	94.09	0.26	0.05	2.16	0.68	0.00
-400mesh(fine conc.)	16.07	64.69	29.61	0.74	7.05	1.92	1.09	46.23	17.85	5.83
Flotation conc.	59.10	67.43	30.86	0.74	3.91	1.55	0.90	47.79	68.44	21.36
Calc.head	100.00	58.25	26.66	2.06	10.14	1.57	0.88	43.60	100.00	100.00
Composite concentrate	75.17	66.86	30.60	0.74	4.58	1.63	0.94	47.46	86.29*	27.19

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage (lb./t Feed)					
	Solids (%)	Time (min)	Solids (%)	Time (min)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation	30-32	0.5+0.5	28-30	5.0+4.0	4.5-5.5	5.0	4.0	2.4+0.6	---	---	---
Silica flotation	20-22	1.0+0.5	18-20	1.0	8-9	---	---	---	0.6	0.6+0.2	0.20
Total reagent consumption (lb./t feed)						5.0	4.0	3.0	0.6	0.8	0.20

* The BPL recovery will be 83.43% after the deduction of BPL loss in primary slimes.

Table 77. Verification Test Results for Sample FLA-5 with Grinding-Carbonate Flotation-Sizing-Phosphate Flotation Process Using Florida Deep Well Water.

I. Flotation Results

Products	Wt. (%)	Analysis (%)								Distribution (%)	
		BPL	P_2O_5	MgO	A.I.	Fe_2O_3	Al_2O_3	CaO	CO_2	BPL	MgO
Carbonate tailings	18.95	42.44	19.42	5.30	3.87	1.59	1.01	41.72	17.94	13.96	62.50
Silica tailings	7.81	5.33	2.44	0.11	81.76	0.69	0.19	3.87	0.83	0.72	0.63
-400mesh (fine conc.)	16.78	63.89	29.24	0.82	5.15	1.82	1.17	46.48	4.67	18.63	8.75
Flotation concentrate	56.46	68.01	31.13	0.79	0.95	1.50	0.97	49.61	5.19	66.69	28.12
Calculated head	100.00	57.60	26.36	1.60	8.52	1.51	0.95	44.02	7.18	100.00	100.00
Composite concentrate	73.24	67.10	30.71	0.81	1.91	1.57	1.02	48.89	5.07	85.32*	36.87

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage (lb./t Feed)						
	Solids (%)	Time (min)	Solids (%)	Time (min)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	NaOH	Na_2SiO_3	Fatty Acid	Fuel Oil
Carbonate flotation	30-32	0.5+0.5	28-30	5.0+3.0	4.5-5.5	5.0	4.0	2.0	---	---	---	---
Phosphate flotation	20-22	2.0+1.0	18-20	2+2+1	9-8	---	---	---	0.4	4.0	3.0	0.28
Total reagent consumption (lb./t feed)						5.0	4.0	2.0	0.4	4.0	3.0	0.28

* The BPL recovery will be 82.50% after the deduction of BPL loss in primary slimes.

Table 78. Verification Test Results for Sample FLA-5 with Grinding-Carbonate Flotation-Sizing-Silica Flotation Process Using Four Corners Plant Water.

I. Flotation Results

Products	Wt. (%)	Analysis (%)								Distribution (%)	
		BPL	P_2O_5	MgO	A.I.	Fe_2O_3	Al_2O_3	CaO	CO_2	BPL	MgO
Carbonate tailings	18.20	41.40	18.95	5.58	4.21	1.53	0.97	38.76	18.61#	12.85	62.96
Silica tailings	6.67	3.32	1.52	0.07	90.70	1.11	0.07	2.10	0.63	0.37	0.00
-400mesh(fine conc.)	18.58	64.88	29.69	0.81	6.47	1.80	1.14	45.88	4.71	20.57	9.26
Flotation concentrate	56.55	68.68	31.43	0.79	1.35	1.54	0.99	48.78	5.15	66.21	27.78
Calculated head	100.00	58.65	26.84	1.62	8.78	1.56	0.95	43.30	7.22	100.00	100.00
Composite concentrate	75.13	67.74	31.00	0.80	2.62	1.60	1.03	48.06	5.04	86.78*	37.04

II. Operating Conditions

Operation	Conditioning		Flotation			Reagent Dosage (lb./t Feed)					
	Solids (%)	Time (min)	Solids (%)	Time (min)	pH	H_3PO_4 (P_2O_5)	H_2SO_4	PA-31	Na_2CO_3	Amine	Kerosene
Carbonate flotation	30-32	0.5+0.5	28-30	5.0+3.0	4.5-5.5	5.0	4.0	1.6+0.4	---	---	---
Silica flotation	20-22	1.0+0.5	18-20	1.5+0.5	9-8	---	---	---	0.6	0.6	0.2
Total reagent consumption (lb./t feed)						5.0	4.0	2.0	0.6	0.6	0.2

* The BPL recovery will be 83.91% after the deduction of BPL loss in primary slimes.

CONCLUSIONS

From the laboratory tests conducted on Florida high-dolomite pebble samples, the following conclusions could be made:

- With CLDRI fine particle flotation technology, the target of achieving a phosphate concentrate from Florida dolomitic phosphate pebble analyzing over 65% BPL and less than 1% MgO at an overall BPL recovery of more than 80% can be reached, with the exception of low-BPL recovery for sample FLA-3 due to its extremely high MgO content, up to 9.6%.
- It is inevitable that the dolomitic pebble sample must be ground in order to liberate dolomite from the phosphate prior to flotation. The determination of reasonable grinding fineness should be dependent on comprehensive economic factors such as energy for grinding in both beneficiation and acid production, reagent consumption, product dewatering and transport, etc.
- Phosphoric acid consumption is the critical factor in determining the overall reagent cost of the process. Inexpensive sulfuric acid can partially substitute for phosphoric acid in order to achieve the target results at reasonably low reagent cost. The appropriate proportion of phosphoric acid to sulfuric acid depends on the characteristics of the test sample and the desired target results.
- The verification tests conducted at IMC-Agrico Company’s facilities demonstrated that the CLDRI fine particle flotation technology had good adaptability to the fluctuation of water quality and could be well practiced in Florida.
- Pilot testing should be carried out prior to any industrial application of CLDRI fine particle flotation process for beneficiating Florida dolomitic phosphate resources. The laboratory test results could be used as the criteria in determining the process flowsheet for pilot testing