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## Recovery and Valorisation by Flotation of Treatment Rejections to the Phosphates Case of Djebel-Onk Algeria

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**Abstract:** The main aim of this study is to develop a technological method for the valorization of the phosphate discharges for the case of Djebel-Onk located in Algeria. The method of enrichment, in use, does not give good results because losses in TPL rejections are important. The improvement of technology as well as the development of certain modern methods made it possible to reach very high contents of TPL. Thus, it is needed to think of improving the already existing technological chain in order to allow a higher recovery of useful mineral. According to the literature review and the world practice in the field of carbonated phosphates, it can be noticed that several factories treat phosphate with 11% in  $P_2O_5$ , level of which the enrichment remains rational and economical. The treatment of the rejections for the particular case of Djebel-Onk becomes necessary since it largely exceeds 20%. Moreover, it was proved that the most widely used method gives the best results is the reverse flotation. The advantage of this method is the mode of the reagents which remains unchanged in the two stages of flotation, except the medium (acid for the flotation of carbonates and basic for the flotation of phosphates). The geological investigation of the layer shows the phosphate is in the state of a mineral pseudolithic. The dimensions of phosphatic oolites vary from 600 to 100  $\mu m$ . In addition, they are regularly distributed between the various classes of particle-size. That encourages us to introduce the crushing factor (release of the phosphatic particles) and to study its interaction with the reagents of flotation via the qualitative characteristics of the concentrates finally obtained. The present research concerns the valorization of the discharges of phosphate with an industrial process, where the consumption of the reagents most used for the enrichment of phosphates is highlighted and to study the consumption-quality ratio (reagents concentrated) and their interaction with the degree of crushing.

**Key words:** Phosphates, collecting, depressing, crushing, flotation, TPL, factorial design

### INTRODUCTION

The rejections of Djebel Onk (+15 and +2 mm, +0.8 and -0.08 mm), of the dry line constitutes more than 20% in the weight of the crusher-run aggregate surroundings 2000 t/j which titrates approximatively 21% in  $P_2O_5$  (46% in TPL). That shows that the method currently applied does not give good results and that the technical-economic improvement of the indices requires the application of another more efficient method. According to the world practice the method which seems most efficient is the opposite flotation (Benamar *et al.*, 2000; Hernáinz *et al.*, 2005).

According to Miller *et al.* (2002) and the world practice in the field of carbonated phosphates, one notices that several factories treat phosphate with 11% in  $P_2O_5$ , threshold where enrichment remains rational and economic. Whereas the contents of the rejections of Djebel Onk largely exceeded the 20% (Table 5), therefore their treatment is a necessity.

The solution which is likely most suitable, is the recovery of these rejections and their treatment by flotation. The present study, is devoted to the study of this problem.

In addition moreover, the phosphate of Djebel Onk is an ore with carbonated gangue of sedimentary origin, the stage of calcinations in the current flow diagram for treatment generates the release of great quantities of carbon dioxide in the atmosphere. Flotation makes it possible to reduce the  $CO_2$  emissions from 80 to 85% of those produced by calcinations in case of replacing the proposed flotation by the current diagram.

**Mineral deposits and geology:** The Djebel Onk region is located within the eastern part of Nememcha mountains which belong to the saharian Atlas. Djebel Onk is a chalky calcareous mountain mass whose its south side lies rapidly under the Miocene and quaternary deposits (Léothsky, 1984).

This phosphate deposit which has a stratified sediment form constitutes an extension towards the west of the phosphates localized in the Tunisian territories. The mine belongs to the thanetian formation whose the basis is characterized by the appearance of the first important phosphate deposit. This latter is strongly clayey and it is succeeded above by the black marls and then the principal phosphate seat which makes approximately 30 m of thickness within a sediment of carbonated formation.

**Stratigraphie of the layer of kef essnoun:** The oldest sediments recognized in the area of Djebel Onk are cretaceous. In Djebel Onk where the exploitation is currently done, one has upwards:

- **Maestrichtian:** Massive, compact limestones (with sometimes of the fossil prints).
- **Danian:** It is characterized by argillaceous marls.
- **Montian:** Limestone, dolomitic limestone, lumachelles with dolomite and marl intercalations.
- **Thanetian:** Thanetien: is subdivided in two parts:
  - **Lower thanetian:** Is represented by marly schists with irregular limestone Intercalations with gastropods, phosphatic intercalations appear in the higher part of the marls.
  - **Higher thanetian:** Is formed of phosphate of dark gray color. One passes there from tender phosphates in bottom to phosphates coprolithic with coarse grains, one meets there also remains of shells, teeth and bones of fish.

The thickness of the layer is evaluated of 30 m, the lithological variations of the phosphatic layers led to the establishment of the following lithological description:

- Fine phosphate pseudoolitic.
- Phosphate coprolithic.
- **Ypresian:** The ypresien is subdivided as a superior and inferior:
  - **Lower Ypresien:** Is represented by limestones with Phosphate intercalation, dolomitic limestones and flint levels.
  - **Higher Ypresien:** Is formed by limestones, dolomitic limestones. On meets intercalations of phosphated rocks, flint levels and geodes of quartz.
- **Lutetian:** The lutetian is subdivided in two parts:

- **Lower lutetian:** Is marked by a change of facies, the first sitted ones are tender white marls with geodes of quartz and flint and inserted limestones.
- **Higher Lutétien:** Is characterized by an evaporitic facies which is formed of white dolomite and gypsum.
- **Miocene:** The Miocene sediments rest on the various terms of the eroded tertiary sector. It is represented by conglomerates, white sands with medium and fine grains, no calcareous grains but affected by argillaceous intercalations, shale clay and sandstones with likings of gravels and rollers.
- **Quaternary:** The quaternary deposits cover the major party of the terrain; they are represented by sandy deposits, gravels and blocks. The stratigraphic cut built on the layer of Kef essnoun shows the following lithological succession:
  - The wall of the phosphatic beam is represented by laminated, dark marls of the lower thanetian in which settle, locally in the higher part, two slight dolomitic phosphate levels with an infra metric thickness.
  - The productive layer is represented by black gray phospharenites, of a thickness varying of 22 m in the North-East to 43 m in the south-west (average of 30.50 m). The phosphates of the productive layer of the Kef essnoun titrate 25.05 to 27.94% in  $P_2O_5$  and from 2 to 3% in MgO. The average ratio  $CaO/P_2O_5$  varies from 1 to 1.87%. The contents of  $SiO_2$  vary from 1 to 2.50%.
  - On the top of the productive layer, the summit phosphatic dolomite has an irregular thickness. It is generally expressed in the entire layer. This formation has a wick thickness in the South-West and thicker in the North-East.
  - On the top of the summit layer, the calcaro-dolomitic series with flint of the ypresien appears, on the top of which limestones of the lutetian settle locally, then sands of miocene and recent deposits (alluvia) of the quaternary. The total thickness of sterile covering varies from 40 m in North to 198 m in the South.

The Djebel Onk mine is divided into many layers namely: Djemijma, Kef Essnoun, Tarfaya, Margueb Ettir and Djebel el Onk Nord.

The total geological deposits are of 317 Mt minerals constituted of 26.53%  $P_2O_5$  and 2.16% MgO, where 168 Mt are proved (Kef Essnoun table), 50 Mt probable (slope breaking zone at the end of the table) and 99 Mt possible in the deep parts of the mine (Léothsky, 1984).

**Mineral properties:** The current sketch of Djebel Onk factory enrichment is characterized by the two following mineral properties:

- **Textural characteristics (1st property):** The phosphate is constituted of grains with dimensions of the order of 0.5 mm. In fact, the chemical analysis (Table 1) of various slices shows that the mineral is grains-rich with dimensions between 0.08 and 0.8 mm. According to the curve, a slice separation (-0.6+0.08) allows to obtain a rich concentrate. This separation becomes easier because of the sterilized gangue (crystallized calcium carbonate, calcite, dolomite and clay), which is softer than the tri-calcareous phosphate. A selective grinding and washing up allow the reduction of this matrix to a refined state without breaking the phosphate grains. The dry way and the workshop dismorass, use this property in the enrichment procedure.
- **Structural characteristics (2nd property):** The Djebel Onk phosphate is a carbonated matrix. A simple roasting at 900 to 950°C temperature allows the reduction of the dolomite, magnesite and calcite into periclase and lime. The latter, as being a toxic addition can be eliminated from the rich mineral by a hydrometallurgical treatment after suppression in water. The calcination and wash out workshops use this property as a basis in the enrichment method (Table 2).

**Mineralization:** The Djebel Onk mineral is constituted of a phosphate with a carbonated matrix. It belongs to the fluor carbonate apatite group and is composed essentially of (Table 3 and 4) (Léothsky, 1984).

- Phosphate as a fluorapatite contained inside phosphated nodules.
- Calcite (CaCO<sub>3</sub>) and dolomite Mg Ca(CO<sub>3</sub>)<sub>2</sub> that group the phosphated nodules (carbonated exogangue) and inside the nodules (carbonated endogangue).

Table 1: TPL distribution as a function of the grains thickness

d (mm)	0.080	0.100	0.125	0.160	0.200	0.250	1.00
TPL (%)	57.00	63.50	64.60	65.00	62.60	61.40	54.0

Table 2: Properties of the principal constituents

Composition	Density (g cm <sup>-3</sup> )	Hardness	Tenor (%)
Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub>	3-3.5	5	26.00
CaCO <sub>3</sub>	2.72	3-2.5	48.05
MgCO <sub>3</sub>	3-3.2	3.5-4.5	0.50
Al <sub>2</sub> O <sub>3</sub>	2.5	Flaky	0.80
Fe <sub>2</sub> O <sub>3</sub>	5.26	5-6	0.70
CaSO <sub>4</sub>	2.32	2	--
Na <sub>2</sub> Ca (SO <sub>4</sub> ) <sub>2</sub>	2.80	2.5-3	--

- The sulfates and the chlorides are composed of sodium, potassium and limes.
- The silice is contained in clays and quartz.
- Iron and the aluminum in the form of oxides.
- Organic matters are very few.

**Actual technological scheme**

**Mechanical preparation:** In DO factory, the mechanical preparation is realized in two crushing stages (semi-hard mineral) ended up by a Sieving of 15 mm.

**Dry way:** This installation has for goal to obtain a dry concentrate less than 1% of humidity, having a TPL content greater than or equals 62% (about 28.5% of P<sub>2</sub>O<sub>5</sub>). The mineral reaching this installation, is delivered by the sieve 15 mm. Initial humidity being 3 to 7 %.

This unit uses during the enrichment, the first property of the mineral, it includes:

- A drying in a furnace of fluidization till a humidity of 1% and at the same time elimination of a fine part of silica.
- A sieving to 2 mm to eliminate the particles with a low concentration in TPL.
- A crushing in a hammer mill to release a part of the gangue stuck to the phosphatic grains.
- An elimination of fines of bottom titrate in TPL, by a separation to 0.08 mm in a draught by a set of selectors.

Table 3: Mineralogical composition

Chemical composition	Name
Ca <sub>3</sub> (PO <sub>4</sub> ) <sub>2</sub>	Tricalcareous phosphate
CaCO <sub>3</sub>	Calcium Carbonate (Calcit)
MgCO <sub>3</sub>	Magnésium carbonate
Ca Mg (CO <sub>3</sub> ) <sub>2</sub>	Dolomite
Al <sub>2</sub> O <sub>3</sub>	Alumine
Fe <sub>2</sub> O <sub>3</sub>	Hematite
CaSO <sub>4</sub>	Gypsum
NaCa(SO <sub>4</sub> ) <sub>2</sub>	Sulfate of soda
CaCl <sub>2</sub>	Chlorure of calcium
SiO <sub>2</sub>	Quartz
Sulfates, fluor and organic matter	

Table 4: Djebel Onk mineral chemical composition

Components	Composition (%)
P <sub>2</sub> O <sub>5</sub> Phosphate Anhydride	26.00
CO <sub>2</sub> Carbone Dioxide	4.00
MgO Periclase	0.50
CaO Lime	48.60
Na <sub>2</sub> O Sodium Oxide	0.08
K <sub>2</sub> O Potassium Oxide	0.02
Fe <sub>2</sub> O <sub>3</sub> Hematite	0.70
F Fluor	2.80
SiO <sub>2</sub> Quartz (Silice)	5.30
Cl Chlorine	0.12
Organic Matters, sulfate	-
Al <sub>2</sub> O <sub>3</sub> Alumine	0.80

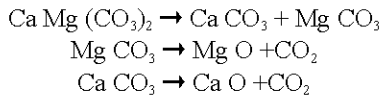
- A sifting to 0.8 mm, allowing the elimination of the coarse grains still rich in exogangue.

**Humid way:** This unit has as a goal the mineral enrichment by a humid way. The product made by the sieve 15 mm, is brought back to its commercial quality while undergoing three successive enrichment operations: dismorass, calcination and washing.

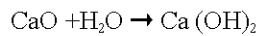
Finally, the product is dried in a rotary oven to have an output humidity less than 0.2% and a product titrated approximately by 75 to 77% in TPL.

This enrichment method uses the second property of the mineral. It includes:

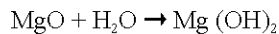
- A dismorass to make a pulp and giving a classification in order to eliminate the fine (-0.08 mm) and coarse (+ 2 mm) sterile with a low concentration in phosphates.
- A calcination which is a thermal treatment bound to deal with dismorassed mineral at a temperature of 900°C, allowing a concentration increase of 8% in TPL. One has a thermal destruction of the carbonates (dolomite, calcite and magnesite) with the formation of the unstable carbon acid decomposed itself to CO<sub>2</sub> and water, this undergoes according to the following reactions:



- A water washing of the calcinated mineral to remove steriles basically constituted of the lime set in the form of a lime milk according to the reaction:



- As well as the liberated magnesite during the calcination according to the reaction.



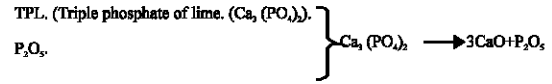
- **A drying:** The enriched mineral is wrung then dried to a temperature of 110°C in rotary ovens with an output humidity less than 0.2%.

### EXPERIMENTAL TECHNIQUE

**Rejections of the treatment chain:** Actually, the development and evaluation installation treat more than 10000 t/j of the mineral with 25% in P<sub>2</sub>O<sub>5</sub> (TPL = 54%). The set of fractions + 15 and + 2 mm, as well as the refusals

+0.8 mm and -0.08 mm of the dry line, constitute approximately 20% in weight of all coming, about 2000 t/j form the rejections with a titration of 21% in P<sub>2</sub>O<sub>5</sub>.

The rejections chemical analyses of the dry way of the 12 last months (Table 5) show the considerable losses in phosphates.



The percentage transformation indices from the various chemical forms

$$\begin{aligned} \text{P}_2\text{O}_5\% \times 2.1853 &= \text{TPL}\% \\ \text{TPL}\% \times 0.1997 &= \text{P}\% \\ \text{P}_2\text{O}_5\% \times 0.4364 &= \text{P}\% \\ \text{P}\% \times 5.0073 &= \text{TPL}\% \\ \text{TPL}\% \times 0.4576 &= \text{P}_2\text{O}_5\% \\ \text{P}\% \times 2.2914 &= \text{P}_2\text{O}_5\% \end{aligned}$$

(Martin Smith, 1986).

### Sampling

**Collect and preparation of the samples:** In this research, we are interested only in the dry way rejections since in this latter, the phosphates losses are the most important and do not undergo any chemical transformations, as in the humid way due to the roasting and dismorass.

The sampling is the choice procedure and samples analysis. The analysis has always as a goal the determination of the physical properties, the chemical composition and the component concentration useful in the matter check. A sample is a part of a substance, which contains the matter characteristics to be analyzed (density, concentration, components etc.). During the treatment, the mineralogical analysis of the sample allows to know structural and textural particularities, the useful minerals composition the associated character of the mineralogical components, their natural linkings, the crystallisation size or dessimination.

According to the data one makes the choice of the methods and technological schemes. Therefore it exists a quantity of matter that preserves the characteristics of the initial matter and is called minimal mass in order that the sample is the most representative.

The dry way rejection is divided in four categories according to their grading.

- 1st part: Rejection +15 mm (1st sieving 15 mm).
- 2sd part: Rejection+2 mm (sieving 2 mm).
- 3rd part: Rejection -0.08 mm
- 4th part: Rejection +0.8 mm (Sieving rejection 0.8 mm).

**Table 5: Analysis results of the 12 months rejections**

+	1	51.38	49.50	50.70	49.43	49.61	49.50	50.34	49.44	51.34	49.29	48.49	50.36
15 mm	2	23.61	22.77	23.33	22.74	22.83	22.77	23.16	22.75	23.62	22.67	22.51	23.17
+2 mm	1	51.64	52.10	52.60	54.11	52.00	55.18	53.68	54.71	53.87	52.36	54.79	53.10
-0.8 mm	2	23.75	23.96	24.19	24.89	23.92	25.38	24.69	25.16	24.78	24.09	25.20	24.42

**Table 6: Rejection grading analysis result (+15 mm)**

Classes (mm)	Weight (g)	$\gamma$ (%)	$\varepsilon\gamma_1$ (%)	$\varepsilon\gamma_1$ (%)	$\beta$ in $P_2O_5$ (%)
-60+50	240	1.00	1.0	100.00	23.61
-50+20	6280	25.00	26.0	99.00	22.74
-20+10	16000	64.00	90.0	74.00	22.77
-10+5	650	2.60	92.6	10.00	23.16
-5+0	1755	7.40	100.0	7.40	22.67
Total	24925	100.00	---	---	---

**Table 7: Rejection sieving result (+2, +0.8 and -0.08 mm)**

Classes (mm)	Weight (g)	$\gamma$ (%)	$\varepsilon\gamma_1$ (%)	$\varepsilon\gamma_1$ (%)	$\beta$ in $P_2O_5$ (%)
-4+2	10505	21.01	21.01	100.00	20.02
-2+1	25000	50.00	71.01	78.99	20.97
-1+0.5	2885	5.77	76.78	28.99	23.35
-0.5+0	11610	23.22	100.00	23.22	22.15
Total	50000	100.00	--	--	--

Rejection (+2 mm, -0.08 and +0.8 mm) In the same batch. The sample minimal mass was calculated according to the following formula:

$$Q = K \times D^a$$

- $Q_{\min}$  = Sample minimal Mass in kg.
- D = Particles dimension in mm.
- a = Degree index (1.1 a 1.3), we take a = 1.2
- K = Proportionality coefficient (0.1) from the experiment.

- Product 15 mm (D = 15 mm).

$$Q_{\min} = 0.1 \times 15^{1.2} = 2.6 \text{ kg} = 2600 \text{ g.}$$

- Product 2 mm (D = 2 mm).

$$Q_{\min} = 0.1 \times 2^{1.2} = 0.23 \text{ kg} = 230 \text{ g.}$$

- Product 0.8 to 0.08 (D = 0.8 mm)

$$Q_{\min} = 0.1 \times 0.8^{1.2} = 0.080 \text{ kg} = 80 \text{ g.}$$

The rejections are evacuated to the workshops exterior in forms of two piles (+15 mm in a pile and the products +2, +0.8 and -0.08 mm in another pile). The sampling method consists of taking a test quantity of about 0.5 kg each 4 h during 5 days. (2 test quantities per post during 3 posts).

$$Q_{\text{totale}} = 0.500 \times 2 \times 3 \times 5 = 15 \text{ kg.}$$

**Table 8: Rejections chemical analysis results**

Concentration (%)								
$P_2O_5$	CaO	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	S	F	CO <sub>2</sub>
20.70	42.70	5.30	1.95	0.46	0.41	0.88	2.22	20.90

For the transportation every sample type is packed in a plastic bag, then in a second bag to avoid the losses during the transportation. The sample (+2, +0.8 and -0.08 mm) is evacuated in the same pile, therefore it is necessary to take one sample and to treble the mass 45 to 50 kg.

**Grading analysis:** It is very interesting to do a grading selection in order to see if there is a good distribution of  $P_2O_5$  among the various rejections grading slices.

Product sieving (+15 mm) (Table 6) and Product sieving (+2, +0.8 and -0.08 mm) (Table 7).

The phosphate nodules have a grading that varies from 600 to 100 surrounded by a strongly carbonated gangue (Calcite and dolomite). According to the grading classification, one notices that the  $P_2O_5$  concentration is divided uniformly between the various slices. Therefore, it is necessary to treat the entire rejections if one wants to recover the most possible mineral. The rejections (-0.08 and 0.8 mm), representing about 30% of the rejections total, can be introduced after the classification cycle since they contain a great concentration in fine product.

The sampling procedure is good, since each sample mass is more than sufficient in comparison with the minimal calculated mass.

**Collect of a sample for the chemical analysis:** The rejections of various grading which will be considered as all coming for the experiments were mixed after a crushing to 6 mm by the cone method and crown and then divided in four parts. We take one of these parts and mix it well by the same method and so on after a crushing until getting a small mass (about 20 g) which will be used for the chemical analysis (Table 8).

**Influential factors on the flotation procedure:** One can summarize the factors which influence on the flotation procedure of the as follows:

- The size of the floating mineral (crushing times).
- The flotation pulp density (fraction liquid- solid).
- The flotation reactive regime (specific consumption  $\text{kg t}^{-1}$  and introduction order).

Table 9: Values of the studied factors variations

Studied factors	Symbols	Values of the highest level (+)	Values of the lowest level (-)	Basis value (Theoretical) (0)
Grinding time (min)	A	14.0	6	10.0
Dejector (kg)	B	0.6	0.4	0.5
Collector (kg)	C	1.8	1.0	1.4

- The ventilation and pulp agitation intensity.
- The flotation period.
- The flotation scheme.

**Factors choices to be studied:** According to the bibliographical research (Ghiani and Saba, 1982; Viniti, 1984) and the world-wide practice in the field of the flotation application procedure (as well as the reactive consumption), the most influential factors on the flotation are:

**1st factor:** The floating mineral size or crushing times. The crushed mineral was grinded in a crushing with balls laboratory during 8 or 14 mn.

**2nd factor:** Dejector consumption. Influence of the liquid glass quantity introduced after crushing nothing before the collector in the agitation. Quantity 400 or 600 g t<sup>-1</sup>.

**3rd factor:** Collector consumption. Influence of the oleic acid quantity introduced after the dejector in the agitation operation. Quantity 1000 g t<sup>-1</sup> or 1800 g t<sup>-1</sup>.

During all experiments, one must keep constant the other factors (the pulp dilution, flotation environment pH, air flow machines flotation, excitation...) and changes only one factor at once among the three most influential factors according to the factorial plan of the experiment (Table 9 and 12).

**Calculation**

**Sample weight for each experiment:** From the practice, we have to take the solid concentration between 25 and 35% (in our case, we take BS = 30%).

**Starting data**

- Solid = 30%.
- Liquid = 70%.
- Volume of the used cell = 1300 mL
- (Flotation machine of Russian benchmark Mekhanover)
- Filling up coefficient = 0.75
- Real used volume (Vr) = 975 mL.
- Mineral density (phosphate) = 2.8 g cm<sup>3</sup>

Table 10: Reactive flotation consumption

Reactives	Consumption	
	kg t <sup>-1</sup>	g/362 g
Collector: oleic acid C <sub>17</sub> H <sub>33</sub> COOH	1.4	0.50
dejector: liquid glass Na <sub>2</sub> SiO <sub>3</sub>	0.5	0.18
Regulator: phosphoric acid	1.6	0.52
Foam: pine oil	0.055	0.02

Table 11: Reactive and consumption for the inverse flotation

Reactives	Carbonates flotation	Consumption (kg t <sup>-1</sup> )	Phosphate flotation	Consumption (kg t <sup>-1</sup> )
Regulator	85% acid Phosphoric	pH 4.8 in 5.2	Sodium carbonate	pH 7.5 in 8.5
Dejector	Liquid glass	0.5	Liquid glass	0.5
Foam	Pine oil	0.055	Pine oil	0.055
Collector	Oleic acid	1.5	Oleic acid	1.5

Thus:

$$\left. \begin{array}{l} 2.8 \text{ g of solid} \text{ ----- } 1\text{mL} \\ 30 \text{ g of solid} \text{ ----- } X\text{mL} \end{array} \right\} X = 10.70\text{mL}$$

And:

$$\left. \begin{array}{l} 30\% \text{ of solid} \text{ --} 10.7 \text{ mL} \\ 70\% \text{ of liquid} \text{ ---} 70 \text{ mL} \end{array} \right\} V_{\text{total}} = 80.7 \text{ mL}$$

In 80.70 mL we have 30 g of solid  
In 975 mL we have X g of solid

$$X = \frac{30 \times 975}{80.70} = 362 \text{ g}$$

Thus:

$$\text{Solid occupied volume} = \frac{362}{2.8} = 129.29 \text{ mL}$$

$$\text{Water occupied volume} = 975 - 129.29 = 845.75 \text{ mL}$$

**Consumption of flotation reactive**

- **Environment regulator:** after crushing of 362 g of TV in water, we put the obtained pulp in the flotation cell. We add water and put in motion the rotor, then one adds drop by drop phosphoric acid and controlling the pulp acidity with a pH paper until we get pH = 4 to 5. (About 15 drops, drop weight is approximately 0.03841 g. Therefore for a tone of the mineral, we will have 1.6 kg of environment regulator).
- **Dejector:** For a tone 0.5 kg therefore for 362 g of mineral, we will have 0.18 g.

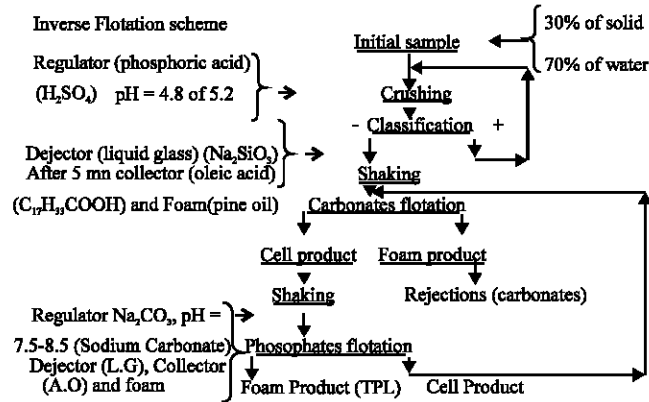


Fig. 1: Inverse flotation scheme

Table 12: Factorial experiments table and results

Experiments	Factors			Results		
	(A)	(B)	(C)	$\beta$ (%)	$\gamma$ (%)	$\varepsilon$ (%)
1	+	+	+	30.65	57.42	85.02
2	-	+	+	26.10	75.20	94.82
3	+	-	+	28.60	66.61	91.70
4	-	-	+	25.38	70.61	86.57
5	+	+	-	26.60	70.33	90.37
6	-	+	-	24.62	78.70	92.85
7	+	-	-	25.68	68.96	85.55
8	-	-	-	24.10	64.26	74.81

- **Collector:** For a tone 1.4 kg, therefore for 362 g of mineral, we will have 0.5 g.
- **Foam:** For a tone 0.055 kg, therefore for 362 g of mineral, we will have 0.02 g. The summarizes the reactive flotation consumption:

The experiment factorial plan is a complete one, the experimental number in relation with factors number is determined by the following formula:

$$n = q^k$$

- n = Experiment number.
- q = Level number of each factor (2).
- k = Factors number (3).
- => n = 2<sup>3</sup> = 8 experiments.

**The reactive loading points:** The most used flotation scheme in the case of the carbonated phosphates is the inverse flotation (Fig. 1 and Table 11). After the mineral crushing, one makes a flotation of the carbonates in an acid environment (pH = 4.8 to 5.2) with 85% phosphoric acid. The foams product containing the carbonate is considered as a rejection. The cell product rich in phosphates will be considered as concentrated.

One can predict phosphates principal flotation. The phosphates flotation will occur in a basic environment created by the carbonate of sodium (pH = 7.5 to 8.5) the foams product rich in phosphates is more enriched with relevant operations.

As a general rule, one adds the flotation reactive in the following order.

- **Dejector:** It is loaded in the crushing, the classifier or shaking vessel.
- **Environment regulator:** One adds the regulator in the crushing cycle or in shaking vessel.
- **Collector:** One adds the collector in the shaking vessel to about 5 mn after the dejector.
- **Foam:** One adds the foam it in the flotation cell 5 mn after the collector.

All the essays were carried out in an identical manner, on a laboratory cell of Russian Benchmark. The results from the point of view concentration and content, yield and extraction coefficient of the phosphates are given in the Table 12, (the cell products results of the various experiments for the carbonates flotation).

## RESULTS

According to the grading classification, one notices that the P<sub>2</sub>O<sub>5</sub> concentration is divided uniformly between the various slices. Therefore, it is necessary to treat the entire rejections if one wants to recover the most possible mineral.

The carbonates are concentrated in the foam product; the phosphate ore is concentrated in the product of cell. IT is directed towards the cycle of flotation of phosphates in the alkaline medium (pH = 7.6-8) creates by the sodium carbonate.



It can be noticed that according to (Table 12) that certain consumptions of reagents and degrees of release (crushing) determine high recoveries of phosphate but with concentrates of insufficient quality. While other values do not allow a good depression of phosphatic minerals, this gives us low contents of phosphates but the outputs of weights more higher.

**DISCUSSION**

**Regression equations:** The multiple linear regressions are an analysis method of quantitative data. It has for a goal to make obvious the relationship between a variable called explained, which is denoted by Y and many other variables called explanatory denoted by  $X_1, X_2, \dots, X_k$ .

The k variables  $X_i, i = 1, \dots, k$  can be either random, or controllable which means they are known without error. In what follows, we will assume that the variables  $X_i, i = 1, \dots, k$  are controllable. We are interested in the linear model, which means at the models of the type:  $Y = a_0 + a_1 X_1 + a_2 X_2 + \dots + a_k X_k$ . Where  $a_0, a_1, \dots, a_k$ . Where real numbers are called model coefficients.

Average calculation for each index:

$$\sigma\gamma = \frac{\sum \gamma_i}{8} = 69.01\% = a_0$$

$$\sigma\beta = \frac{\sum \beta_i}{8} = 26.46\% = b_0$$

$$\sigma\varepsilon = \frac{\sum \varepsilon_i}{8} = 87.71\% = c_0$$

**Correction coefficient calculation (fitting) for each factor:**

- For the crushing :  $k\gamma = -3.10$   
 $k\beta = +1.40$   
 $k\varepsilon = +0.44$
- For the dejector:  $k\gamma = +1.32$   
 $k\beta = -0.54$   
 $k\varepsilon = -2.05$
- For the collector:  $k\gamma = -1.60$   
 $k\beta = +1.20$   
 $k\varepsilon = +1.81$

**Regression equation:**

$$\begin{aligned} \gamma &= a_0 - 3.10 X_1 + 1.32 X_2 - 1.60 X_3 \\ \beta &= b_0 + 1.40 X_1 - 0.54 X_2 + 1.20 X_3 \\ \varepsilon &= c_0 + 0.44 X_1 - 2.05 X_2 + 1.81 X_3 \end{aligned}$$

This means

$$\begin{aligned} \gamma &= 69.01 - 3.10 X_1 + 1.32 X_2 - 1.60 X_3 \\ \beta &= 26.46 + 1.40 X_1 - 0.54 X_2 + 1.20 X_3 \\ \varepsilon &= 87.71 + 0.44 X_1 - 2.05 X_2 + 1.81 X_3 \end{aligned}$$

**Explanation of the regression curves:** The regression curves allow us to optimize the flotation procedure; therefore, one has to concentrate on the qualitative indices which are the concentration  $\beta$  in % and the extraction degree of  $\varepsilon$  in %.

$$\beta = 26.46 + 1.40 X_1 - 0.54 X_2 + 1.20 X_3$$

$$\varepsilon = 87.71 + 0.44 X_1 - 2.05 X_2 + 1.81 X_3$$

One notices that:

**X1:** If one increases the value of  $X_1$  (crushing), by a step (4 mn) the concentration will be improved by 1.40% and the extraction coefficient by 0.44%.

**X2:** If one decreases  $X_2$  (dejector) by a step (0.1 kg t<sup>-1</sup>) the concentration will increase by 0.54% and the extraction coefficient by 2.05%.

**X3:** If one increases  $X_3$  (collector) by a step (0.4 kg t<sup>-1</sup>) the concentration will increase by 1.2% and the extraction coefficient by 1.81%.

In this manner, one succeeds to optimize the flotation procedure by acting on the studied factors.

Concerning the second floor of the flotation (phosphates direct flotation), we will act in the same manner on the regression equations (this step is not included in this study).

**CONCLUSIONS**

When one uses only the gravimetric laundry (cases of Djebel Onk), one tends to eliminate big mineral particles which can contain phosphate nodules. The resulted rejections represent an important loss which can be recovered by flotation, since in this type of procedure we carry out the total mass of the mineral in the flotation cells and therefore we considerably increase the recuperation probability of the phosphate particles.

Thus, it is necessary to liberate the most possible phosphate nodules by trying various degrees of crushing (crushing times). Moreover, there is a hardness difference between the phosphates and the sterile gangue constituted principally by carbonates which are more softer than phosphates.

The economic effects of the flotation of phosphates are of an increasing importance. The benefit of flotation can be estimated by the increase of the recoveries on those of the gravimetric laundries and the reduction in the cost price of the treatment. Flotation allows the recovery of an additional tonnage of the rejections of the gravimetric section which is equal or higher to this latest.

Flotation makes it possible to enrich a rough ore by low content, which is becoming necessary in consequence of the exhaustion of the reserves with high content. For example, phosphate exploitation by gravimetric had a revise life of only few years. By adding flotation and the extraction of the ore with low content, the life of this exploitation can be extended by several tens of years.

The aim of the flotation is first of all is to increase the content of the products of the laundry, for example the content can be 68 to 70% of TPL, whereas much higher contents is required. The flotation increase the content until an average of 77% of TPL, or of advantage still.

It is very useful to choose the reactive which act directly on the particles surfaces (dejector and collector), since they are the determining factors of the flotation procedure. The best choice of these two reactive as well as their consumption, will allow the considerable improving of the phosphates recuperation While based on the real consumption values of some laundry reactive being varied in well defined proportions and planning series of experiments related to various possible combinations of the chosen factors, one arrives to establish a mathematical model (regression equations) that will allow us to optimize the proposed enrichment procedure.

#### RECOMMENDATIONS

- Before realizing an installation of a known capacity, it is essential to go through the pilot phase which will have as a goal to check the validity of the proposed procedure.

- To install a tractable laundry of current installation followed by a thickener for the treatment of the huge quantity of the stocked rejections near to the Djebel Onk mining complex.
- To predict a reconstitution installation of the used waters in the flotation procedure to a reuse in the beginning of the procedure since the deposit is in a semi dry region.
- The check of the pH during the flotation will be regulated and verified automatically during the flotation since this is a very important parameter in the inverse flotation.
- Finally, to propose this procedure for the treatment of the all coming regarding its simplicity, economy and yield in comparison with the current procedure.

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