

Article

Beneficiation of Low-Grade Phosphate Deposits by a Combination of Calcination and Shaking Tables: Southwest Iran

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Abstract: Three quarters of the world's phosphate deposits are of sedimentary origin and 75%–80% of those include carbonate gangue. In this study, carbonate sedimentary phosphate deposits of the Lar Mountains of southwest Iran are studied. These deposits consist mainly of calcite, fluorapatite, quartz, kaolinite and illite, with an average P₂O₅ grade of 9%–10% (low-grade). Various pre-processing and processing methods have been developed for concentrating low-grade phosphate up to marketable grade and this study aims to select the optimal method to produce an economically viable grade of phosphate concentrate from low-grade ore. Different concentration methods, including calcination and gravity separation, were applied on samples at both a laboratory and semi-industrial scale (pilot scale). Using an integrated method of calcination (performed in a rotary kiln) and shaking table for concentrating the low-grade phosphate ore, the results show promise at producing grades of 30.77% P₂O₅ with 60.7%–63.2% recovery.

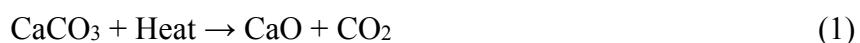
Keywords: calcination; Iran; low-grade phosphate ore; phosphate deposit; rotary kiln; shaking table

1. Introduction

1.1. Phosphate Production

Phosphate plays a significant economic role in developing countries because of the increasing demand on phosphate rock for fertilizer production and its importance in animal feed stocks, as well as food-grade phosphates and other industrial uses. The high demand for phosphate is typically fulfilled through mining and processing [1], an industry which, globally, produced 224 million tons in 2013 and is expected to reach 260 million tons in 2017 [2]. About 75% of the world's phosphate rocks are of sedimentary origin and 75%–80% of those contain carbonate gangue [3]. To meet the needs of the agricultural sector to produce phosphate and chemical fertilizers, several methods have been proposed for mining based on the characteristics and depth of phosphate ore [4]. Similarly, to concentrate low-grade phosphate ore to a marketable grade (~30% P₂O₅) [5] several pre-processing and processing methods are defined [1,6,7]. These are based on the ore type, associated gangue minerals and the amount of impurities, as well as factors such as the degree of liberation of apatite minerals, the cost of the beneficiation method [1,6]. The methods employed include gravity separation [6], magnetic separation [8–10], electrostatic separation [11–14], size reduction, screening [15], attrition, scrubbing, classification [7], heavy media separation [16], calcination [17], acidic leaching [18–20], direct flotation [21–24], reverse flotation [25,26] or the use of multiple methods. Sedimentary rocks have various chemical and mineralogical compositions in the gangue phase [16] and, therefore, based on the major associated minerals, the Lar phosphate deposit of southwest Iran is classified as a calcareous ore of sedimentary origin. In terms of processing, conventional techniques such as flotation and physical separation are difficult to remove the carbonate minerals from such ores (because of the similarity in physical properties of carbonates and phosphates) [6] and Calcination is another solution for upgrading these difficult-to-treat types of ores. Beneficiation by calcination is one of the better known processes which has been proposed in the past to treat carbonate-bearing sedimentary phosphate ores [27]. Calcination can be an effective mineral concentration method, and is used in the processing and production of more than 10% of global phosphate sales [28]. Calcination involves the thermal decomposition of carbonates and burning intended organic materials but has several disadvantages [11,29], such as high energy costs [5], low reactivity of final products and the high initial capital cost of calcination plants [6].

The thermal dissociation of carbonates is an endothermic reaction (Equation 1) with a significant energy requirement.



Furthermore, calcination produces low reactivity of the resulting phosphate [4] as well as a lower ratio of CaO/P₂O₅ compared with raw francolite [30]. It should be noted that despite the disadvantages, the calcined phosphate product is used as raw material in the production of phosphoric acid and chemical fertilizers [31].

Using gravity methods after calcination is an effective process to increase the concentrate grade and leads to a more economical design. There are numerous advantages to gravity separation: low startup cost, low energy consumption during the crushing process, high efficiency, lack of environmental impacts and high selectivity compared with other methods, such as flotation [32]. The gravity separation method is not applicable to all mineral compounds and requires the determination of a concentration criterion (CC) based on the relationship between the specific gravity (SG) of heavy and light minerals and the fluid (water or air) in which they are found [33], as follows:

$$\text{Concentration Criterion} = \frac{\text{SG of heavy mineral} - \text{SG of fluid}}{\text{SG of light mineral} - \text{SG of fluid}}$$

Mineralogical studies have shown that calcite, quartz, apatite (collophane) and glauconite with specific gravities of 2.7, 2.65, 3.2 and 2.4 g/cm³, respectively, are the primary minerals of the phosphate ore. Consequently, the CC, based on the equation of Taggart (1951) [33] is 1.29, a value which indicates that gravity separation will not be commercially viable and cannot be used as the only pre-concentration method. Because carbonate is very finely dispersed in phosphate, gravity separation is also ineffective [16] and the similarity in specific gravities of dolomite, calcite and apatite also render the technique ineffective as the only mineral concentration process. Dependence of the foregoing phenomena it can be stated that this method should be applied together with calcination as an effective process to concentrate the low-grade phosphate ore. In this study the degree of liberation was considered the fundamental selection criterion for screening using the shaking table and, hence, parameters such as deck slope, feed water flow rate and dressing water flow rate were optimized to increase the efficiency of process.

Of the gravitational methods, the use of shaking tables (such as the Wilfley table) is considered an efficient method for ore concentration. In this method, a solid-liquid separation process is based on cross flow of light and heavy particles on an inclined, riffled table as the particles simultaneously spread out. As the table shakes the differential motion, riffled deck and cross flowing water causes particle separation (Figure 1), with the riffles helping to transmit the shaking motion to the particles as well as preventing direct washing of particles off the table. The vibration is asymmetrical, being slow in the forward direction and quick in the reverse direction. The particle feed enters at the corner of the table at a concentration of 25% solids, along with dressing water introduced from the upper edge to aid separation and displacement across the table. Finally, the particles move diagonally across the deck in accordance with their specific gravity where they can be variously collected.

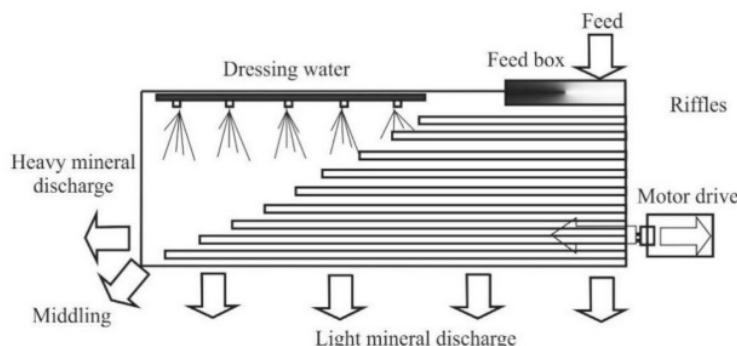


Figure 1. Schematic illustration of the arrangement of a riffled shaking table; note the feed input at the top right corner of the table.

1.2. Background of the Study

As 75% of the world's phosphate carbonate reserves are of sedimentary origin [3], much research has been done into the use of calcination to reduce carbonate gangue. In each of the cases described in this section, new methods involving calcination were used to concentrate phosphate carbonate ore.

Kaljuvee *et al.* (1995) [34] examined phosphate samples from Kazakhstan with an initial P₂O₅ grade of 21%–23% and CO₂ content of 8%–10% CO₂. This is a carbonate ore containing primarily fluorapatite, quartz, dolomite and minor amounts of pyrite and calcite. The similarity in mineralogical composition in the region meant that common concentration methods (flotation, wet separation) were not effective. An integrated method of fluidized-bed calcination at a temperature of 900 °C and air separation for the concentration of the intended sample was used. The final P₂O₅ grade of the concentrate reached 28% with recovery at more than 85%.

Sinirkaya *et al.* (2011) [35] examined the changes of P₂O₅ in samples of phosphate rock from Turkey during simultaneous sulfidation-calcination. The ore had an initial P₂O₅ grade of 23%–27% and consisted primarily of calcite, fluorapatite and quartz. In this case, a fluidized-bed reactor was used for calcination, and the results showed a temperature decrease during calcination, a reduction in particle size as well as rapid quenching of sulfated samples, which resulted in an increase of P₂O₅ to more than 36%.

Khoshjavan and Rezai (2011) [28] examined phosphate rock samples of northern Iran that have an average P₂O₅ grade of 11.9% and consist of apatite (collophane), quartz, calcite and dolomite. Using calcination and subsequent two-stage flotation and desliming, a concentrate with a P₂O₅ grade of more than 31% P₂O₅ and recovery of more than 62% was obtained. However, in contrast with flotation methods, gravity separation has several advantages such as [33]: (a) a lower installed cost per ton of throughput than flotation, (b) a lower installed power requirement per ton of throughput, (c) gravity separation does not use expensive reagents, (d) the environmental impact of gravity plant effluent is considerably less than for flotation.

The present work investigates sedimentary phosphate carbonate reserves of southwest Iran that consist mainly of calcite, fluorapatite, quartz, kaolinite and illite. The phosphate ore has an average P₂O₅ grade of 9%–10%, classified as low-grade. As a result, various methods are used to increase concentration, such as calcination and gravity separation, and hence the research presented here aims at selecting the optimum method(s) for producing salable phosphate concentrates from low-grade deposits.

2. Material and Methods

2.1. Ore Samples

The studied samples were selected from the phosphate rock reserves of the Lar Mountains in southwest Iran. The initial size of representative sample was 4 cm diameters. The degree of crushing to generate a representative sample for mineral liberation and concentration purposes had to be considered and, accordingly, optical microscopic studies were performed using a Zeiss Axioplan 2 Polarized Light Microscope (Kharazmi University, Tehran, Iran) on 45 thin sections of nine sample size fractions. The results showed that, in terms of mineral liberation, 80% of apatite ore of 100 µm (150 mesh) in size was liberated. To perform X-ray diffraction (XRD) and X-ray fluorescence (XRF)

analysis, the prepared samples were sent to the Kansaran-e-Binaloud Laboratory. The results of XRD analysis are presented in Table 1 and the results of major-element XRF analysis are presented in Table 2. The results of XRD analysis show that the minerals calcite, fluorapatite and quartz are the primary minerals of the ore and kaolinite, illite and other feldspathic minerals occur at 5% or less.

Based on the tabulated results, it is evident that the Lar Mountains samples, with an initial P_2O_5 grade of 9%–10%, are of a very low grade, and given the high CaO concentration, can be considered as sedimentary phosphate ore with carbonate gangue.

Table 1. X-ray diffraction (XRD) analysis of a representative phosphate ore sample from the Lar Mountains.

Mineral Type	Calcite	Quartz	Kaolinit	Illite	Fluorapatite	Other Minerals	Total
%	65	8	5	2	18	2	100

Table 2. Major element geochemical analysis of a representative phosphate ore sample from the Lar Mountains. L.O.I: Loss on Ignition.

Content	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	Na ₂ O	K ₂ O	MgO	TiO ₂	MnO	P ₂ O ₅	S	L.O.I	Other	Total
%	9.24	2.45	1.67	46.01	0.28	0.38	0.72	0.102	0.012	9.164	0.003	28.98	0.989	100

2.2. Guidelines for Concentrating Ore

2.2.1. Primary Crushing

At the primary crushing stage, to avoid production of fine particles and reduce production of phosphate waste and recovery loss, phosphate representative sample with initial dimensions of 4 cm were subjected to two-stage jaw crushing in the open circuit, cone crushing in the closed circuit and finally screened to 2.4 mm (8-mesh). At the end of the crushing process, 100% of the product smaller than 2.4 mm was considered feedstock for the next stage of concentration.

2.2.2. Calcination

According to the results of XRD and XRF analysis, in terms of mineralogical content, it is evident that the major impurities in the phosphate ore are calcite and silica. In this study, potential issues with calcination, such as appropriate crushing to minimize the generation of slimes and determining the optimum time and temperature, were overcome by proper design of the rotary kiln, modifying the energy recovery system and finally, optimizing the overall concentration circuit. Rotary kiln applied in order to modify energy recovery system in calcination process. Fundamentally, rotary kilns are heat exchangers in which energy from a hot gas phase is extracted by the bed material. During its passage along the kiln, the bed material will undergo various heat exchange processes, a typical sequence for kilns being drying, heating, and chemical reactions that cover a broad range of temperatures. The most common configuration in rotary kiln systems is counter current flow whereby the bed and gas flows are in opposite directions. This phenomenon will be the modified energy recovery system of kilns in comparison with other mineral processing equipment. To minimize the production of slimes, a two-stage crushing circuit involving jaw crushing and cone crushing was implemented. For this

purpose, a representative sample was fed into the jaw crusher and the product, after passing through a 2.4-mm screen, was fed into the cone crusher. Subsequent calcination took into account three main parameters: particle size range, temperature and duration of calcination, based on previous experiences, and laboratory testing. Optimal conditions were obtained via design tests using DX7 Software (Stat-Ease Incorporation, Minneapolis, MN, USA). Accordingly, tests using varying parameters were carried out, as follows: fractions > 2.4 mm, fractions 150 μm to 2.4 mm, fractions < 75 μm , temperatures of 850, 950 and 1050 °C, and three different durations of 60, 120 and 180 min. The resulting phosphate grade and degree of recovery of each of the tests was then evaluated.

Calcination was carried out at a semi-industrial, pilot-scale using a rotary kiln. Thermal processes in the rotary kiln provide the impetus for the physic-chemical reactions, in conjunction with using the correct combination of solid grains to ensure proper heat transfer [15]. A short dry kiln was used, which required the feed particles to first be pre-heated to 130–200 °C prior to entering the main reactor [36]. This pre-heating prevents an extreme temperature drop in the rotary kiln, which would lead to poor heat transfer between the solid and gas phases. The rotary drum used consisted of a steel cylinder 1.5 m in diameter and 10 m in length with countercurrent flow. The bed motion in the transverse plane of the drum is rolling, conducted at a rotation rate of 3 rpm. Studies of rotary kiln bed behavior have allowed for the development of predictively estimating bed behavior under a given operational condition. One such tool is a bed behavior diagram of Henein (1980) as shown in Figure 2 [37] presents a typical behavior for a sand bed for a 41 cm (1.35 ft) diameter pilot kiln. Given the angle of repose, kiln geometry, and speed, users of such diagrams can predict what bed behavior to expect within the kiln cross section. The Froude number (Fr) assigned to the bed level in this study was considered to be between 0.5×10^{-3} and 0.2×10^{-1} . Also, according to the bed behavior diagram, the bed depth of particles based on a kiln rotation speed of 3 rpm and mentioned Froude number, measured 0.2 meters.

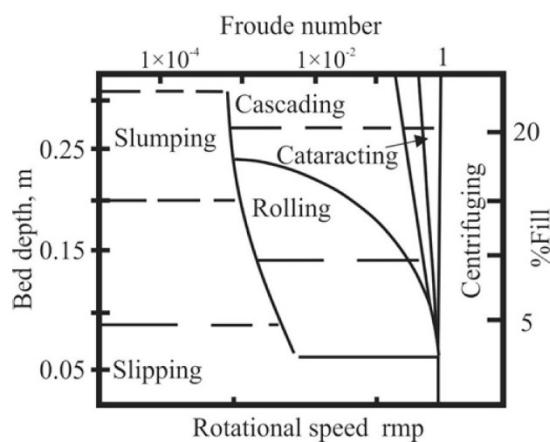


Figure 2. Bed behavior diagram for a rotary kiln indicating particle behavior in relation to rotational speed, bed depth and % fill (adapted from [22]).

As shown in Figure 3, various zones found within a rotary kiln can be discerned in the transverse plane: the active layer, including the upper part of the bed where surface renewal occurs [36] and the passive layer, which has a shear rate of zero. In the active layer, particles slide over each other via

granular flow, and are returned to the top of the passive layer where particles move as a solid mass concentrically around the axis of the kiln.

To achieve the experimental objectives, particles from the crushing process were calcined at 950 °C for 120 min, determined from other experimental results and charts. To increase the residence time, some dams within the kiln were designed. After performing calcination and dissociation of carbonates, the remaining compounds enter a washing phase in which hot calcined particles at 800–950 °C are rapidly quenched with cold water, resulting in rapid heat loss release of CaO. The CaO reacts with water and forms Ca(OH)₂ according to Equation (2).



After removing the Ca(OH)₂ slurry, the particles were filtered and dried before entering another crushing phase in preparation for separation using a shaking table.

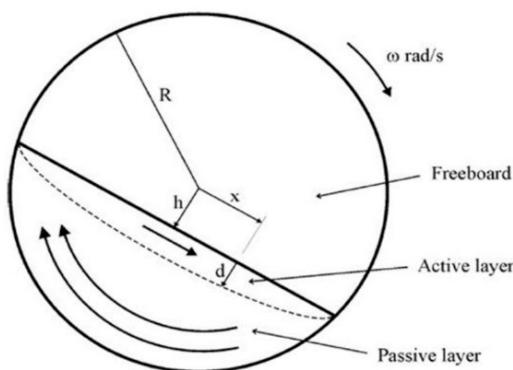


Figure 3. Schematic cross section of a rotary kiln drum, indicating the distribution of active and passive regions in relation to the bed and freeboard (adapted from [36]).

2.2.3. Shaking Table

The factors influencing effective mineral separation on a shaking table include particle size, density and shape, riffle design, deck shape and slope, water flow, feed rate and shaking motion and speed [38]. In terms of particle size, it must be noted that the quenched and crushed calcined phase is screened to 150 mesh, and coarser particles that remain are then crushed to 150 mesh (based on mineralogical results and in terms of mineral liberation, 80% of apatite ore of 100 µm (150 mesh) in size was liberated), filtered and dried along with already screened particles, prior to concentration using the shaking table.

With respect to mineralogical densities, and as mentioned in Section 1.1, representative samples of the phosphate ore contain calcite, quartz, fluorapatite (collophane) and glauconite with specific gravities of 2.7, 2.65, 3.2 and 2.4 g/cm³, respectively.

The degree of liberation was considered the fundamental selection criterion for screening using the shaking table and, hence, parameters such as deck slope, feed water flow rate and dressing water flow rate were optimized. To achieve this, several experiments were performed on a laboratory scale and final waste, middling and concentrate products were scrutinized for the highest concentrations and recovery rates. It was found that the optimal deck slope was 10°, the optimal feed water flow rate was 8 liters per minute and the dressing water flow rate was 10 liters per minute.

3. Results and Discussion

The research presented here has been concerned with a pilot study on improving efficiency and recovery of higher phosphate grades from low-grade phosphate ores of southwest Iran. In the first step, samples were calcined in a rotary kiln, after crushing. The effects of three parameters on the process were assessed, namely temperature, particle size and residence time. Three size fractions were studied, namely <75 µm, 75–150 µm and >2.4 mm (results provided in Tables 3–5, respectively).

Table 3. Results of calcination of phosphate ore <75 µm in size.

Particle Size (Micron)	Calcination Temperature (°C)	Time (min)	Initial Weight of the Sample (gr)	Weight Loss (%)	P ₂ O ₅ (%)	Recovery (%)
<75	850	120	100	7.43	10.1	95.4
<75	950	120	100	9.44	10.4	96.1
<75	1050	120	100	16.83	11.3	95.9

Table 4. Results of calcination test for the fractional class of 75–150 µm.

Particle Size (Micron)	Calcination Temperature (°C)	Time (min)	Initial Weight of the Sample (gr)	Weight Loss (%)	P ₂ O ₅ (%)	Recovery (%)
75–150	850	120	100	4.91	10.1	98
75–150	950	120	100	8.47	10.6	99
75–150	1050	120	100	18.89	11.9	98.5

Table 5. Results of calcination test fractional class of >2.4 mm.

Particle Size (mm)	Calcination Temperature (°C)	Time (min)	Initial Weight of the Sample (gr)	Weight Loss (%)	P ₂ O ₅ (%)	Recovery (%)
>2.4	850	120	500	7.12	10.2	96.7
>2.4	950	120	500	11.18	10.6	96
>2.4	1050	120	500	14.3	11.14	97.4

For the fractional class of 75–150 µm, when considering temperature and residence time as the two key variables, it was found that the best recovery is related to a temperature of 1050 °C and residence time of 120 min. Results lead to the production of concentration with average grade of 11.9% P₂O₅ and recovery as 98.5% (Table 4).

For the fractional class >2.4 mm, maximum P₂O₅ grade is related to a temperature of 1050 °C in residence time of 120 min which led to the production of concentrate with an average P₂O₅ grade of 11.14% and recovery of 97.4% (Table 5).

The laboratory studies showed that these three size fractions were not ideal for calcination in the rotary kiln at a pilot-project scale.

According to the results obtained from the chemical analyses which is shown in Table 6, the ideal fraction for calcination are particles in the 0.15–2.4-mm range.

Additionally, for the 0.15–2.4-mm-size fraction, when considering temperature and residence time as the two key variables, it was found that the best recovery is related to a temperature of 950 °C and

residence time of 120 min. Rapid quenching of the calcined samples also led to the production of concentrate with an average P_2O_5 grade of 17.15% and recovery of 96.04% (Figure 4).

Table 6. Results of calcination test fractional class of 0.15–2.4-mm.

Calcination Temperature (°C)	Time (min)	Initial Weight of the Sample (gr)	Weight Loss (%)	P_2O_5 (%)	Recovery (%)
850	120	1250	29.28	13.65	98.5
950	120	1250	45.12	17.15	96.04
1050	120	1250	46.48	16.2	88.47

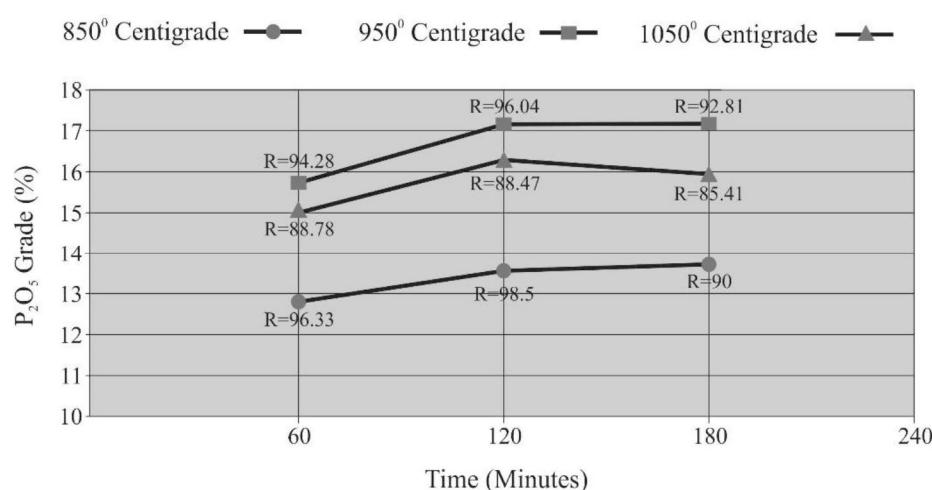


Figure 4. The effect of time and temperature on the grade and recovery (R) of P_2O_5 for the 0.15–2.4-mm fraction

Shaking table studies performed by assigning values to variables such as particle size, desk slope, feed water flow rate, dressing water flow rate and finally base on four mentioned parameters, the grade and recovery variation *versus* effective parameters were investigated. Accordingly, the samples carried out from calcination tests was divided in various fractions (<100, 100–270, 270–500, 500–1000 μm) before testing. The following comparative experiments were established. The first experiment, for the fraction of 500–1000 μm , the deck slope 6°, the feed water flow rate of 8 liters per minute and table water flow rate 6 liters per minute lead to the high grade and low recovery of 40.75% and 14.71%, respectively (Table 7).

Also, results of shaking table tests in second experiment denote that separation considering the parameters such as the slope of deck as 8°, feed water flow rate of 7 liters per minute and dressing water flow rate of 7 liters per minute, for the fraction of 270–500 microns produces a concentrate with the grade (concentration) of 39.22% P_2O_5 and recovery of 16.38% (Table 7).

In the third experiment for the fraction of 100–270 μm , the table slope was decreased to 4° and the feed water flow rate and dressing water flow considered as 6 and 5 liters per minute respectively. These changes lead to an increase in concentrate grade but the recovery remarkably decreased to 10.74% (Table 7).

In the fourth experiment, to attain a higher grade, slope of deck was increased to 10°, the feed water flow rate was set up to 8 liters per minute and dressing water flow rate was raised to 10 liters per

minute. As a result, for the fraction of <100 µm, as described in Section 2.2.3, the ideal shaking table parameters (10° deck slope, feed water flow rate of 8 liters per minute and dressing water flow rate of 10 liters per minute) result in production of a concentrate with a P₂O₅ grade of 30.77% and recovery of 63.2%. However, with regard to recovery loss caused by calcination and screening, the overall recovery reduced to 60.7%. The results of shaking table separation are provided in Table 7.

Table 7. The results of shaking table recovery of calcined phosphate ore for the fraction of <100, 100–270, 270–500, 500–1000 µm.

Sample	Particles Size (µm)	Weight (gr)	Weight (%)	P ₂ O ₅ (%)	Recovery (%)	CaO (%)
Concentrate	500–1000	77.5	6.19	40.75	14.71	-
Middling	500–1000	934.47	74.63	15.7	68.32	-
Waste	500–1000	240.03	19.17	12.88	14.39	-
Feed	500–1000	1252	100	17.15	100	-
Concentrate	270–500	89.67	7.16	39.22	16.38	-
Middling	270–500	775.31	61.92	18.18	67.99	-
Waste	270–500	387.02	30.91	12.66	22.81	-
Feed	270–500	1252	100	17.15	100	-
Concentrate	100–270	57.69	4.6	39.98	10.74	-
Middling	100–270	388.4	31.02	21.98	39.75	-
Waste	100–270	805.91	64.36	12.58	47.21	-
Feed	100–270	1252	100	17.15	100	-
Concentrate	<100	441	35.22	30.77	63.2	45.11
Middling	<100	564	45.05	12.72	33.41	56.74
Waste	<100	247	19.73	2.95	3.39	47.56
Feed	<100	1252	100	17.15	100	51.65

4. Conclusions

Integrated methods of calcination and the use of a shaking table to concentrate low-grade phosphate ore (P₂O₅ 9%–10%) from southwest Iran show that it is possible to produce concentrate with a P₂O₅ grade of 30.77% and recovery of 60.7%.

Short, dry rotary kilns were used to calcine the ore after preheating to temperatures of 130–200 °C. In this method, the optimum time and temperature were determined to be 120 min and 950 °C, respectively. The ideal size fraction on which to perform calcination was found to be 150 µm to 2.4 mm. With respect to shaking table mineral separation, it was found that the optimal slope of the deck was 10°, a feed water flow rate of 8 liters per minute and a dressing water flow rate of 10 liters per minute.

In conclusion, it has been shown that it is possible to effectively employ an integrated and technical method of phosphate concentration that overcomes the inherent problems of the ore, including low-grade, slimes generation and physical property similarities between constituents. Additionally, this method does not incur a high operational cost compared with other methods, and hence it is possible to acquire a marketable product of appropriate grade for further processing into fertilizer, or to produce technical- or food-grade phosphoric acid.

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Author Contributions

Aisan Ramadi conceived and designed the experiments, performed the experiments, contributed reagents and wrote the paper; Shahram Shariati analyzed the data, contributed analysis tools, Provide the representative sample and Armin Salsani contributed materials, provide the representative sample and wrote the paper.

Conflicts of Interest

The authors declare no conflict of interest in publishing this article.

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