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OPTIMIZING UPGRADING OF ABU-TARTUR PHOSPHATE ORE USING ATTRITION SCRUBBING AND MAGNETIC SEPARATION TECHNIQUES

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ABSTRACT 2.

Phosphate rock is one of key commodities from mining operations. The methods used to process phosphate ores primarily depend on the type of gangue minerals present in the extracted rock.The treatment of phosphate ore aims to increase the P_2O_5 percentage, adding economic value and contributing to the development of the national income. This study aimed at upgrading Abu Tartur phosphate ore and enriching the content of rare earth elements using attrition scrubbing and magnetic separation techniques. The attrition scrubbing was conducted to remove the clays. The impact of various parameters on attrition scrubbing, such as solid-liquid ratio, agitation speed, temperature, and time, was examined. The optimal variables for attrition scrubbing are a solid-liquid ratio of 40%, an agitation speed of 1200 rpm, a duration of 20 minutes, and a temperature of 40ºC. The results yielded phosphate rich fraction of 22.96 % P_2O_5, with a recovery of 80.25%, and total rare earth oxides (REOs) of 991.93 ppm. The dry high intensity magnetic separation was conducted using RER roll separator to remove iron oxides. The optimal obtained variables were an inclination angle of 86.75 degrees, a belt speed of 70.10 rpm, and a feed rate of 73.65 kg/hr. Under these conditions, the results showed a P_2O_5 concentration of 25.55% with recovery of 95.88%, Fe_2O_3 removal at 90.09% and total rare earth oxides (REOs) of 1141.40 ppm. A combination between attrition scrubbing and magnetic separation yielded a phosphate concentrate of 27.85% P_2O_5 and total rare earth oxides (REOs) of 1358.32 ppm.

KEYWORDS: Abu Tartur phosphate ore, Attrition scrubbing, Magnetic separation, and Total Rare Earth Elements

تحسين ترقية خام فوسفات أبو طرطور باستخدام تقنيات الغسيل الاحتكاكي والفصل المغناطيسي

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الملخص

تعد صخور الفوسفات من المنتجات الأساسية الناتجة عن عمليات التعدين. تعتمد تقنيات معالجة خامات الفوسفات بشكل أساسي على نوع المعادن العالقة والمصاحبة في الصخور المستخرجة. تهدف معالجة خام الفوسفات إلى زيادة نسبة خامس أكسيد الفوسفور، مما يضيف قيمة اقتصادية ويساهم في تنمية الدخل القومي. تهدف هذه الدراسة إلى ترقية خام فوسفات أبو طرطور

وإثراء محتوى العناصر الأرضية النادرة باستخدام تقنيات الغسيل الاحتكاكي والفصل المغناطيسي. أجريت عملية الغسيل الاحتكاكي لإزالة الطين. تم فحص تأثير العديد من المتغيرات على الغسيل الاحتكاكي، مثل نسبة المواد الصلبة إلى السائلة، وسرعة التقليب، ودرجة الحرارة، والوقت. المتغيرات المثلى للغسيل الاحتكاكي هي نسبة المواد الصلبة إلى السائلة ٤٠٪، وسرعة التقليب ١٢٠٠ دورة في الدقيقة، ومدة ٢٠ دقيقة، ودرجة حرارة ٤٠ درجة مئوية. أسفرت النتائج عن نسبة غنية بالفوسفات ٢٢,٩٦٪ من خامس أكسيد الفوسفور ، مع استرداد ٨٠,٢٥٪، وإجمالي أكاسيد الأرض النادرة ٩٩١,٩٣ جزء في المليون. كانت RERلإزالة أكاسيد الحديد. تم إجراء الفصل المغناطيسي الجاف عالي الكثافة باستخدام فاصل الأسطوانة المتغيرات المثلى التي تم الحصول عليها هي زاوية ميل ٨٦,٧٥ درجة، وسرعة حزام ٧٠,١٠ دورة في الدقيقة، ومعدل تغذية ٧٣,٦٥ كجم / ساعة. في ظل هذه الظروف، أظهرت النتائج تركيز خامس أكسيد الفوسفور بنسبة ٢٥,٥٥٪ مع استرداد ٩٥,٨٨٪، وإزالة الهيماتيت بنسبة ٩٠,٠٩٪ وإجمالي أكاسيد الأرض النادرة ١١٤١,٤٠ جزء في المليون. أدى الجمع بين الغسيل الاحتكاكي والفصل المغناطيسي إلى تركيز فوسفات بنسبة ٢٧,٨٥٪ من خامس أكسيد الفوسفور وإجمالي أكاسيد الأرض النادرة بمقدار ١٣٥٨,٣٢ جزء في المليون.

الكلمات المفتاحية : فوسفات ابوطرطور، الغسيل الاحتكاكي، الفصل المغناطيسي،العناصر الأرضية النادرة

1. INTRODUCTION

Phosphate ores are the main source of phosphorus, a crucial element with substantial economic significance, necessary for the production of phosphoric acid, fertilizers, and elemental phosphorus. The demand for phosphate has grown over the years, with global consumption reached up to 50 million tons by 2023 [1]. According to (IFASTAT), global phosphate fertilizer consumption fluctuated between 42 and 49 million tons of P_2O_5 from 2010 to 2020. The International Fertilizer Association (IFA) forecasts that demand for phosphate fertilizers will increase to 63-72 million tons of P_2O_5 by 2050, depending on agricultural practices and nutrient management strategies [2].

Phosphate ore is a significant economic resource in Egypt. In 2021, Egypt mined 7.5 million metric tons of phosphate rock. The country has three main mining areas: the Western Desert located between the El-Karga and El-Dakhla Oases (Abu-Tartur region), the Nile Valley close to Idfu, and the Red Sea coast between Safaga and Quesir. It is estimated that Egypt's total phosphate reserves surpass 3 billion tons [3]. The Abu Tartur phosphate deposit is among the largest in the Middle East, containing 1,000 million tons, of which 200 million tons are proven. It is situated in the Western Desert, approximately 60 km from El-Karga City and 10 km from the main road connecting El-Karga and El-Dakhla Oases. The phosphate ores in this area originate from sedimentary deposits and are part of the apatite group [4]. Various beneficiation techniques are used to upgrade phosphate ores, depending on the type of ore and associated gangue minerals [5]. Attrition scrubbing methods have been effectively used to enhance mid-grade, weathered sedimentary phosphate ores, like those found in Yunnan province, China [6]. Attrition scrubbing and classification are used when the primary gangue minerals are clays, characterized by their fine size and loosely bound grains with phosphorite pellets. Attrition in water liberates and disperses the clay particles, which are then removed by desliming or classification. This method is effectively utilized for enhancing phosphate rock from Egypt's Red Sea Coast [7]. Additionally, it can be applied when the accompanying gangue minerals primarily consist of coarse silica or chert fragments, as phosphorites are gathered from beneath the screens during a wet processing method [8]. Magnetic separation is utilized when one or more of the major gangue constituents is magnetic, to remove these constituents. This technique is mainly utilized for the beneficiation of igneous phosphate rocks, but it has also been employed to enhance certain sedimentary phosphate ores [9].

Phosphate rock, also known as phosphorite, is an important secondary resource for rare-earth elements, as it holds substantial quantities of phosphate minerals along with minor concentrations of rare earth elements (REEs) [10]. Phosphate rocks serve as an alternative source of REEs because they typically contain a significant amount of these elements. In recent years, phosphate deposits have gained attention as a potential source of rare earth elements (REEs) because (i) the mining and processing of phosphate rocks are already supported by the fertilizer industry, (ii) there is a high demand for these metals, and (iii) if not recovered, these elements would be lost and eventually accumulate in the soil [11]. As a result, these factors motivated researchers to create effective methods for extracting these metals from phosphate rocks [12].

However, phosphate rocks also possess numerous impurities, including Cd, Cu, Cr, As, Ti, Fe, and Mg [13]. Separation methods such as pyrometallurgy and hydrometallurgy have consequently been devised to refine phosphoric acid. Nowadays, hydrometallurgical processes are preferred because the cost of producing phosphoric acid through pyrometallurgy has increased. Heavy metals can be eliminated from wet phosphoric acid (WPA), which is phosphoric acid produced through hydrometallurgical processes, by using sulfide precipitation [14]. To further purify WPA by removing cationic impurities such as Fe, Al, Mg, and Ca, the pH can be raised to specific levels. For example, the elevated iron content in fertilizer-grade phosphoric acid (50% P_2O_5) is lowered to the industry-standard limit of 1.5% Fe_2O_3 through precipitation using K_2SO_4 [14]. Magnesium, along with a minor percentage of iron and aluminum, was eliminated by combining the raw phosphoric acid with hexafluorosilicic acid, resulting in the formation of $MgSiF6.6H_2O$, which can be easily separated [15].This study focuses on enriching Abu Tartur phosphate ore through attrition scrubbing and magnetic separation under various operating conditions to obtain a valuable phosphate concentrate rich in rare earth elements.

2. Materials and Methods

2.1. Sample Preparation

The phosphate sample was processed through primary and secondary crushing stages, yielding a product entirely passing through a 6300 µm sieve. The crushed material was divided into roughly 20 kg portions using a ‘Denver’ Jones riffle sampler. One portion was further ground in rod mills to achieve a liberation particle size of 250 µm. This ground material was then deslimed via a 045 µm screen, with the retained fraction (-250 + 045 µm) serving as feed for magnetic separation. A separate portion was pulverized to -200 mesh (-75 µm) using a laboratory analytical mill for subsequent X-ray diffraction (XRD) and chemical characterization.

2.2. Chemical Analysis

A comprehensive chemical analysis of the sample was performed using X-ray fluorescence (XRF). For regular chemical analysis, the acid-insoluble residue (A.I.) was measured through standard procedures involving the opening and dissolution of samples with HCl and HNO_3. Silica content was determined using the standard gravimetric method, while the filtrate solution was utilized to measure P_2O_5 content via standard spectrometric methods. The concentration of rare earth elements was assessed using Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES).

2.3. X-ray Diffraction Analysis

The mineral composition of the sample was identified using X-ray diffraction (XRD). This analysis employed the powder pattern data from the ASTM, utilizing a cobalt radiation target with an iron filter at 30 kV and 20 mA. A 'Philips' X-ray diffractometer (PW 1730) was utilized for this purpose. The scan was conducted within the range of 2θ=1 to 2θ=80 degrees.

2.4. Sample preparation and attrition scrubing

The preparation process included screening with 250 µm and + 45 µm screens. Size fractions exceeding 250 µm and those below 45 µm were removed, while the remaining material was cleaned using a "Denver" attrition scrubbing machine. This machine features two stainless steel turbine-type propellers with opposite pitches, a stainless steel shaft, and a shaft collar. The attrition process aimed to remove clay minerals, focusing on factors like attrition time, pulp density, and attrition temperature. Subsequently, wet screening was conducted using a -250 µm sieve, discarding the size fraction smaller than 45 µm.

2.5. Magnetic Separation

Dry high-intensity magnetic separation was performed using a rare earth magnetic roll separator (RER) with a permanent field intensity of approximately 1.5 tesla. The feed characterized by -250+45 µm is fed onto a thin belt 250 µm. The feed material is passed through

the magnetic field, and the magnetic particles are attached to the roll and separated from the nonmagnetic stream. Three parameters are studied which are Splitter inclination, Belt speed, and feed rate. All the magnetic and nonmagnetic fractions were weighed and chemically analyzed.

3. RESULTS AND DISCUSSION

3.1. Characterization and Mineralogy of Phosphate Ore

Figure 1(a,b) represents the mineralogical study of Abu-Tartur phosphate ore which revealed that iron is found as pyrite, ankerite and iron oxide. Iron oxide is present as cryptocrystalline clusters of red to brown particles and is limited to the weathered outcrops. Composition of kaolin ore samples on their corresponding value of the Silicate Module. Silicate Module ($\nu SiO_2 / \nu Al_2O_3$) is the molar ratio of SiO_2 to the molar ratio of Al_2O_3. For example, the Silicate Module of the Troshkovsky kaolin ore sample is equal to [(52/60) / (31.9/102)] = 2.78.

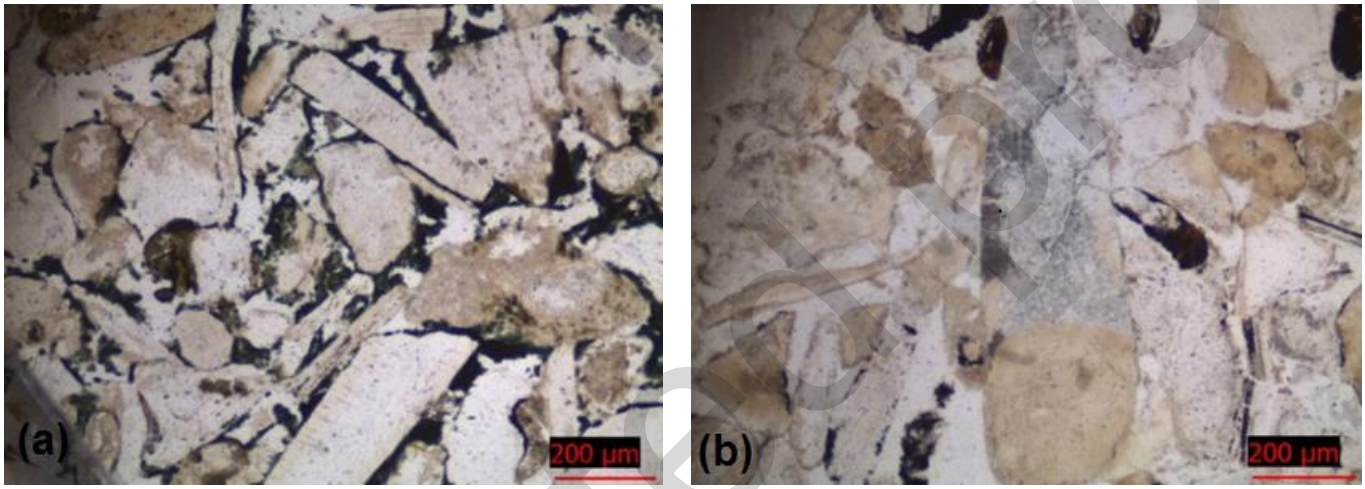


Fig. 1. The mineral constituents of the Abu-Tartur phosphate ore

3.1.1. Mineral Composition and Characterization

The mineralogical investigation of the representative sample using XRD revealed that the sample is consisting mainly of the following minerals shown in figure 2 and table 1.

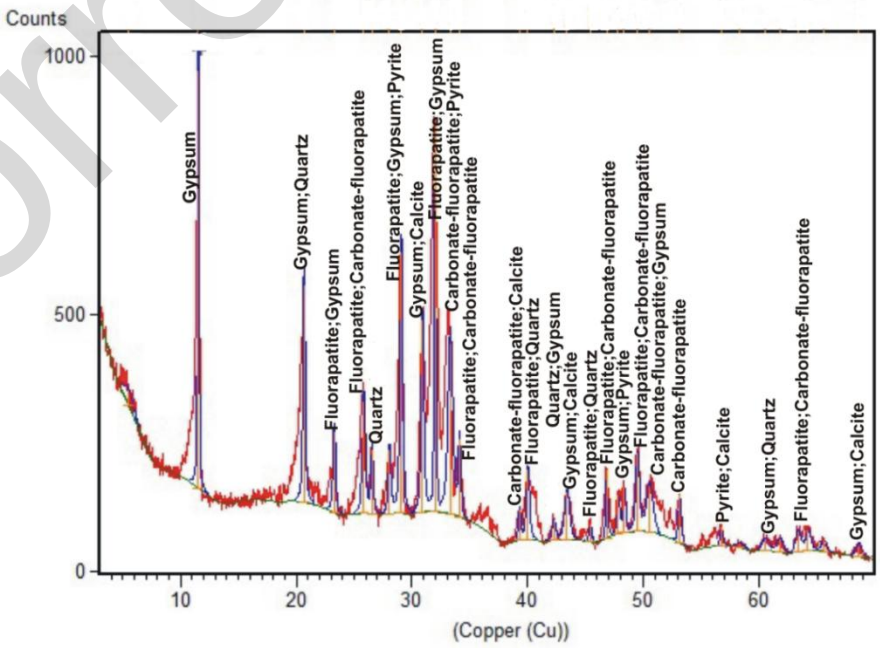


Fig. 2: XRD pattern of the phosphate sample.

As observed in Fig. 2, the Abu-Tartur phosphate sample primarily consists of the following key minerals: Fluorapatite, Gypsum, Quartz, Calcite, and Goethite. The phosphate-bearing minerals have been reported in all ore types as carbonate fluorapatite "francolite" and fluorapatite [11].

Table 1: Mineralogical composition of Abu Tartur phosphate ore.

Mineral Name	Chemical Formula
Gypsum	$CaSO_4.\ 2H_2O.$
Quartz	$SiO_2.$
Fluorapatite	$Ca_5\,(PO_4)_3F.$
Goethite	FeO_2H
Calcite	$CaCO_3$

3.1.2. Geochemistry

Table 2 presents the results of the chemical analysis of the representative sample conducted through XRF. The analysis indicates that the sample has a relatively low P_2O_5 content of 20.62% and a higher CaO content of 34.51%. The results also reveal the presence of additional calcium-bearing minerals besides apatite, such as calcite and gypsum, with SO_3 content at 4.79%. The sample has about 6.76% Fe_2O_3 and lower content of MgO (1.52 %) with silica content SiO_2 of (8.59 %). The loss on ignition (LOI) is about 11.43%. The total REOs are about 0.08% (800 ppm).

Table 2: Chemical composition of Abu-Tartur phosphate ore sample

Comp.	P_2O_5	Al_2O_3	MgO	Fe_2O_3	CaO	K_2O	Na_2O	SiO_2	SO_3	F	CO_2	L.O.I.	Σ REOs
Wt, %	20.62	2.25	1.52	6.76	34.51	0.14	0.69	8.59	4.79	2.09	6.51	11.43	0.08

3.2. Attrition Scrubbing of Phosphate Ore Sample

Attrition scrubbing has been investigated to remove gangue minerals, such as clay minerals, from the surface of ore. These minerals were found both as aggregates and as coatings on phosphate grains in petrographic microscope images of thin section samples. Attrition scrubbing tests were conducted on the samples after they had been crushed, ground and sieved to a fraction of −250 +45 µm. The attrition scrubbing treatment aimed to enhance the grade and recovery of phosphate. Subsequently, the following main variable parameters were studied to obtain phosphate rich fraction with lower amounts of clays.

3.2.1. Effect of S/ L ratio on phosphate attrition scrubbing.

The effect of the solid/liquid ratio on the attrition scrubbing of phosphate ore sample was studied using ratios of 25%, 40%, 50%, and 60%. In these experiments, the liquid volume was kept constant while varying the amount of solid to determine the optimal solid/liquid ratio. The results are illustrated in Figure 3, under the conditions of a 20-minute attrition time, 40°C temperature, 1200 rpm attrition speed and ore particle size of -250 +45 µm. The results indicated that the solid/liquid ratio significantly affecting the concentration of ore and water, and thus influences the grade and recovery rate of P_2O_5.

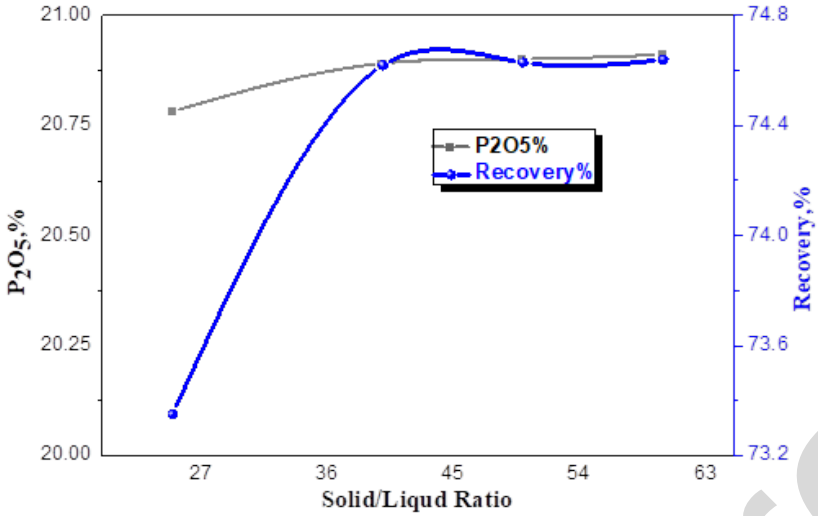


Fig. 3: Effect of solid/ liquid ratio on the phosphate attrition scrubbing

As shown in Figure 4, increasing the solid/liquid ratio from 25% to 50% enhances the grade of P_2O_5 and its recovery rate. At a 50% ratio, the P_2O_5 grade reaches its maximum value of 20.89%. Beyond this ratio, changes in the removal of clay minerals are negligible. Consequently, a solid/liquid ratio of 40% is considered optimal, achieving a P_2O_5 grade of 20.90% and a recovery rate of 74.63%. These findings are consistent with those reported by [17].

3.2.2. Effect of agitation speed.

The impact of agitation speed on the attrition scrubbing process of the phosphate ore sample was studied with speeds ranging from 900 to 1800 rpm. Although increasing the agitation speed improves the attrition scrubbing of phosphate sample, there is no significant effect beyond 1200 rpm. The results illustrated in Figure 4, show that as agitation speed increases, P_2O_5 grade rises from 20.92% to 21.28%, and recovery improves from 74.81% to 75.25%.

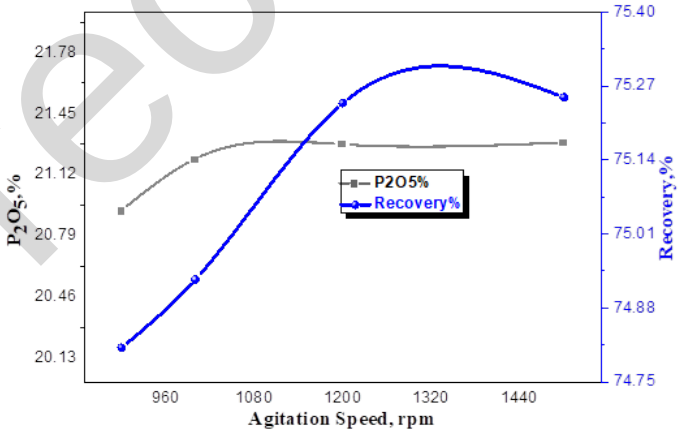


Fig. 4: Effect of agitation speed on the phosphate attrition scrubbing

3.2.3. Effect of attrition time

The impact of attrition time on phosphate attrition scrubbing was examined to identify the ideal duration for achieving the highest phosphate grade and recovery. Time intervals ranging from 5 to 30 minutes were tested. Experiments were conducted under a liquid/solid ratio of 40%, a reaction temperature of 40°C, 1200 rpm attrition speed and an ore sample particle size of -250+45 µm to study the impact of scrubbing time on phosphate beneficiation. These results are presented in Figure 5. As shown in Figure 6, at the first few minutes of scrubbing, the P_2O_5 content increased significantly.

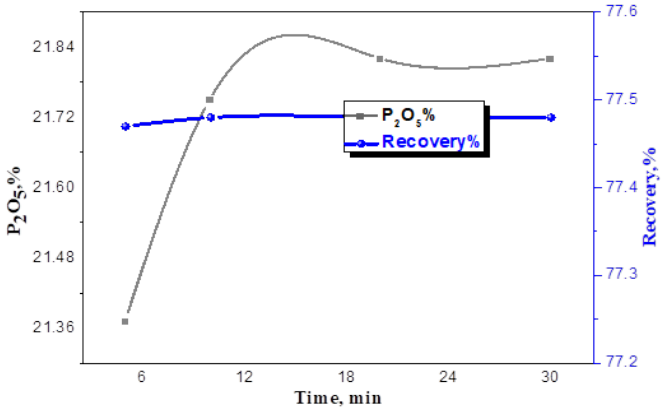


Fig. 5: Effect of attrition time on the phosphate attrition scrubbing

As the reaction time increased, the clay minerals content decreased, and the rate of increase in P_2O_5 became less pronounced. At 20 minutes, the clay minerals were nearly dissolved, and the P_2O_5 grade in the attrition scrubbing residues reached its maximum value, with a P_2O_5 grade and recovery of 21.83% and 77.96%, respectively. Thus, a 20-minute scrubbing time was found to be optimal. These results are consistent with the findings of [18].

3.2.4. Effect of attrition temperature

The impact of attrition temperature on leaching results was studied under these conditions: a liquid-to-solid ratio of 50:1, a reaction duration of 20 minutes, and an ore sample particle size ranging from -250 to +75 µm. The effect of temperatures of 20, 30, 40, 50, and 60°C was investigated. As shown in Figure 6, the grade of P_2O_5 increases with rising attraction temperatures. Specifically, at 40°C, the P_2O_5 grade reached 22.96%, with a recovery rate of 80.25%. Beyond 40°C, however, the increase in P_2O_5 grade became less pronounced, likely due to reduced scrubbing efficiency at higher temperatures. This observation is consistent with findings by [19].

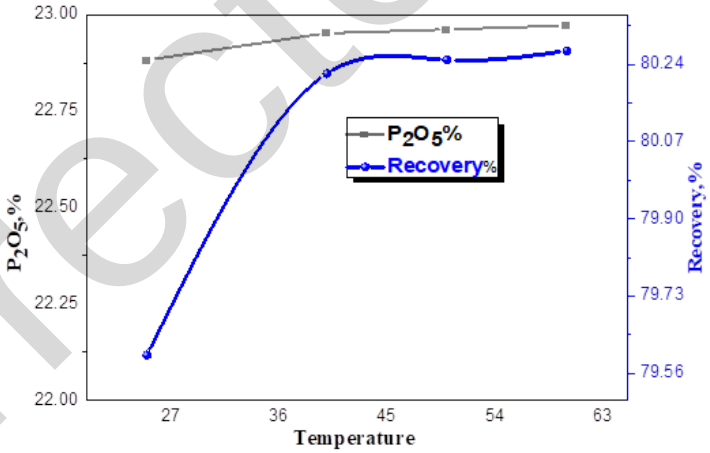


Fig. 6: Effect of attrition temperature on the phosphate attrition scrubbing

The optimum conditions for the attrition scrubbing of phosphate ore sample are: solid/liquid ratio 40%, Agitation speed 1200 rpm, attrition time 20 min and attrition temperature 40^0C. Applying these conditions, a phosphate rich fraction of 22.96% P_2O_5 with recovery of 80.25% was obtained (Tables 3 and 4). The XRF of the scrubbed phosphate shows that the clay minerals which are represented by Al_2O_3 and MgO were decreased from 2.25% and 1.52% in the original sample to 0.44% and 0.15% in the scrubbed sample respectively (Table 5). Consequently, the total content of rare earth elements (REE) was increased from 678 to 820 ppm (Table 5 and Table 6). The attrition scrubbing of Abu Tartur phosphate ore sample removed successfully the clay minerals. However, the scrubbed phosphate fraction requires further beneficiation processes in order to remove the iron oxides and silicates to obtain valuable phosphate concentrate enriched with rare earth elements.

Table 3: Optimum conditions of attrition scrubbing optimum variables

Attrition scrubbing optimum variables	
Solid/ liquid ratio	50%
Agitation speed	1200 rpm
Attrition time	20 min
Attrition temperature	40ºC

Table 4: P_2O_5 grade and recovery at attrition scrubbing at optimum variables

Phosphate Upgrading		
P_2O_5		Σ REEs
Assay, %	Recovery, %	820 ppm
22.96	80.25	

Table 5: The chemical analysis of Abu Tartur phosphate ore sample and the scrubbed phosphate sample

Comp.	P_2O_5	Al_2O_3	MgO	Fe_2O_3	CaO	K_2O	Na_2O	SiO_2	SO_3	F	CO_2	L.O.I	Σ REOs
Phosphate ore sample	20.62	2.25	1.52	6.76	34.51	0.14	0.69	8.59	4.79	2.09	6.51	11.43	0.08
Scrubbed Phosphate sample	22.96	0.44	0.15	5.62	37.45	0.18	0.75	8.66	4.55	2.38	5.88	10.86	0.10

Table 6: Total REEs of the phosphate ore and the scrubbed phosphate sample

Component	REEs ppm REOs	Phosphate ore sample	Scrubbed phosphate
Scandium	Sc	0.00	12.00
	Sc_2O_3	0.00	18.41
Yttrium	Y	149.00	169.50
	Y_2O_3	189.22	215.25
Lanthanum	La	99.80	119.25
	La_2O_3	117.04	139.85
Cerium	Ce	185.00	219.50
	CeO_2	227.26	269.64
Praseodymium	Pr	57.00	67.88
	Pr_6O_{11}	68.87	82.01
Neodymium	Nd	94.10	114.52
	Nd_2O_3	109.76	133.58
Promethium	Pm	0.00	0.00
	Pm_2O_3	0.00	0.00
Samarium	Sm	21.20	22.90
	Sm_2O_3	24.58	26.55
Europium	Eu	0.00	6.50
	Eu_2O_3	0.00	7.53
Gadolinium	Gd	30.00	35.28
	Gd_2O_3	33.05	38.87
Terbium	Tb	0.00	0.00
	Tb_4O_7	0.00	0.00
Dysprosium	Dy	16.20	18.80
	Dy_2O_3	18.59	21.58
Holmium	Ho	0.00	2.82
	Ho_2O_3	0.00	3.23
Erbium	Er	12.00	15.80
	Er_2O_3	13.72	18.07
Thulium	Tm	0.00	0.00
	Tm_2O_3	0.00	0.00
Ytterbium	Yb	14.10	15.25
	Yb_2O_3	16.06	17.37
Lutetium	Lu	0.00	0.00
	Lu_2O_3	0.00	0.00
Σ Total (ppm)	Σ REEs	687.40	820.00
	Σ REOs	818.15	991.93
Thorium	Th	30.83	36.58
	ThO_2	35.09	41.63
Uranium	U	2.31	2.75
	U_3O_7	2.73	3.25

3.3. Magnetic Separation of Abu Tartur Phosphate Ore Sample

A rare earth magnetic roll separator (RER) was utilized to perform dry high-intensity magnetic separation on the phosphate ore sample, operating with a permanent field intensity of approximately 1.5 tesla. The phosphate feed sample of size (-250 + 45µm) is fed onto a thin belt

250 µm. The feed material is passed through the magnetic field, and the magnetic (or weakly magnetic) particles are attached to the roll and separated from the non-magnetic stream. RER magnetic separation was achieved in order to separate iron from phosphate. Three parameters are studied which are Splitter inclination, Belt speed, and feed rate.

3.3.1. Applying Box-Behnken Design

The Box-Behnken Design, an experimental design method, was employed to optimize the recovery of phosphate ore with the RER magnetic separator and to assess the interactions between various parameters (refer to Tables 7-9). In this design, three factors are considered. The optimal conditions, according to the experimental setup, are determined using a second-order polynomial function, which establishes a correlation between the response and the factors studied. The equation is typically expressed as [14]:

$$Y = \beta_o + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \beta_{11} X_{12} + \beta_{22} X_{22} + \beta_{33} X_{32} \quad \textbf{(1)}$$

Where Y is the predicted response; phosphate recovery%, X_1, X_2 and X_3 are studied variables inclination angle, belt speed and feed rate;respectively β_{ij} are equation constants and coefficients.

Table 7: Box-Behnken Design with 3 levels for phosphate upgrading

Run	Coded factor levels		
	A (x_1)	B (x_2)	C (x_3)
1	-1	0	-1
2	0	0	0
3	0	0	0
4	0	0	0
5	0	1	-1
6	0	0	0
7	1	0	-1
8	-1	0	1
9	0	-1	1
10	-1	1	0
11	1	0	1
12	0	-1	-1
13	1	1	0
14	0	0	0
15	-1	-1	0
16	1	-1	0
17	0	1	1

Table 8: RER separator factor levels

Symbol	Parameter	Unit	(-1)	(0)	(+1)
A (x_1)	Splitter inclination	degree	80.00	85.00	90.00
B (x_2)	Belt speed	rpm	60.00	75.00	90.00
C (x_3)	Feed rate	kg/hr	60.00	80.00	100.00

Table 9: Box-Behnken Design results for RER separator

Run	Splitter inclination, degree	Belt speed, rpm	Feed rate, kg/hr	Wt, %	P_2O_5, %	P_2O_5 recovery, %	Fe_2O_3, %	Fe_2O_3 removal, %
1	80.00	75.00	60.00	76.75	24.64	91.71	1.65	75.59
2	85.00	75.00	80.00	75.00	25.48	92.68	0.57	91.57
3	85.00	75.00	80.00	94.90	25.49	92.59	0.56	91.72
4	85.00	75.00	80.00	75.00	25.48	92.68	0.57	91.57
5	85.00	90.00	60.00	68.80	23.72	79.14	1.86	72.49
6	85.00	75.00	80.00	75.10	25.47	92.76	0.58	91.42
7	90.00	75.00	60.00	74.25	24.95	89.84	1.25	81.51
8	80.00	75.00	100.00	51.50	24.52	61.24	0.91	86.54
9	85.00	60.00	100.00	62.22	23.57	71.12	1.20	82.25
10	80.00	90.00	80.00	50.50	24.78	60.69	1.61	76.18
11	90.00	75.00	100.00	76.30	24.39	90.25	1.13	83.28
12	85.00	60.00	60.00	79.15	25.40	97.50	0.72	89.35
13	90.00	90.00	80.00	59.65	24.12	69.77	0.82	87.87
14	85.00	75.00	80.00	75.10	25.47	92.76	0.58	91.42
15	80.00	60.00	80.00	54.45	24.24	64.01	0.74	89.05
16	90.00	60.00	80.00	66.40	25.06	80.70	1.40	79.29
17	85.00	90.00	100.00	63.20	24.93	76.41	0.49	92.75

The ANOVA data for the magnetic phosphate upgrading system suggests that the experimental results align well with the polynomial model equation, indicating the model's accuracy, as shown in Table 10. The elevated R^2 values suggest that the quadratic equation effectively represents the system within the specified experimental domain. It is evident that there is a strong correlation between the predicted and actual values, as the Predicted R^2 is closely aligned with the Adjusted R^2, with a difference of less than 0.2. The high Adequate Precision ratios demonstrate a sufficient signal [20]. The model F-values suggest that the model is significant, indicating it can be utilized to explore the design space [21].

Table 10: ANOVA for response surface quadratic model of RER separator

The statistical parameters	P_2O_5		Fe_2O_3	
	Assay, %	Recovery, %	Assay, %	Removal, %
Standard deviation	0.015	0.28	0.021	0.31
R-Squared	0.9998	0.9998	0.9990	0.9990
Adj. R-Squared	0.9994	0.9995	0.9977	0.9977
Pred. R-Squared	0.9967	0.9968	0.9853	0.9853
Adequate precision	165.37	174.04	83.68	83.68

F-value	3195.39	3672.08	780.40	780.40

The equations, expressed in terms of actual factors for the phosphate upgrading system, enable predictions regarding the responses at specific factor levels. These responses include P_2O_5 assay %, P_2O_5 recovery %, Fe_2O_3 assay %, and Fe_2O_3 removal % in the non-magnetic phosphate concentrate, as outlined below:

P_2O_5 assay % = -122.5145 +2.8737 A +0.5933 B +0.0949 C -0.01416 A^2 -0.00255 B^2 -0.00125 C^2 -0.00493 A * B -0.0011 A * C +0.00253 B * C **(2)**

P_2O_5 recovery % = -2928.7247 +70.7724 A +9.0567 B -8.9732 C -0.4337 A^2 -0.05805 B^2 +0.00352 C^2 -0.02534 A * B +0.**0772** A * C +0.0197 B * C **(3)**

Fe_2O_3 assay % = +88.5158 -2.2785 A +0.4058 B -0.1447 C +0.01476 A^2 +0.00089 B^2 +0.00074 C^2 -0.00483 A * B +0.00155 A * C -0.00154 B * C **(4)**

Fe_2O_3 Removal % = -1209.4046 +33.7049 A -6.0035 B +2.1399 C -0.21834 A^2 -0.01325 B^2 -0.01087 C^2 +0.07149 A * B -0.02292 A * C +0.0228 B * C **(5)**

The response surfaces of the P_2O_5 assay and recovery % as well as the Fe_2O_3 assay and removal % as a function of RER studied parameters are presented in Figures 7 - 10. It is displayed that the splitter inclination angle is an effective variable for magnetic phosphate upgrading using RER dry high intensity magnetic separator. There is an interaction between the splitter inclination angle and the other studied variables. At lower inclination angle, increasing the belt speed resulted in an increase in phosphate assay (P_2O_5%) and recovery (Figures 8a, 9a). While, at higher inclination angle increasing belt speed up to 70 rpm increased both phosphate assay and recovery up to 25% and 94% respectively. Further increase in the belt speed resulted in a slight decrease in the phosphate assay and recovery.

Also, at lower splitter inclination angle increasing feed rate values resulted in an increase in phosphate assay but with minimum recovery of approximately 60% (Figures 7b, 8b). While, at higher inclination angle increasing the feed rate up to 75 kg/hr increased both phosphate assay and recovery up to 25% and 90% respectively. Further increase in the belt speed more than 75 kg/hr resulted in lower phosphate assay but with higher phosphate recovery up to 93%. Successful phosphate upgrading was accomplished with high assay and recovery at reduced roll speed and feed rate values (Figures 7c and 8c). A reduced feed rate combined with a slower belt speed allows for the efficient separation of iron particles from phosphate particles at a splitter inclination of 85 degrees (Figures 9c and 10c). Also, increasing both splitter inclination and belt speed leads to higher removal efficiency of iron from phosphate ore (Figures 9a and 10a).

The higher recovery and lower assay phosphate values obtained at splitter inclination angle more than 87 degree, are due to the fact that; if the goal is to obtain the non-magnetic fraction (phosphate) as the desired product, positioning the splitter close to the roll (with a high splitter inclination angle) will lead to a high recovery rate. However, this may result in some residual iron oxide being present in the phosphate concentrate [16, 17]. So, the most efficient splitter inclination degree for obtaining both high phosphate grade and recovery with maximum iron oxide removal is approximately 87 degrees. Additionally, the increased recovery and decreased phosphate assay values observed at belt speeds exceeding 70 rpm can be attributed to the rise in centrifugal force, which extends the trajectory arc of the non-magnetic phosphate fraction. This occurs because the higher belt speed allows the centrifugal force to surpass the magnetic attraction on the roll's surface, causing some iron oxide to be lost into the non-magnetic phosphate stream [20, 21]. So, the most efficient belt speed for obtaining both high phosphate grade and recovery with maximum iron oxide removal is approximately 70 rpm.

The objective is to have a one-particle thick layer on the belt as the feed passes over the roll [18, 19]. The higher recovery and lower grade obtained at feed rates more than 75 kg/hr, are due to the

feed has more than one-particle thick layer. So, the most efficient feed rate for obtaining both high phosphate grade and recovery with maximum iron oxide removal is approximately 75 kg/hr.

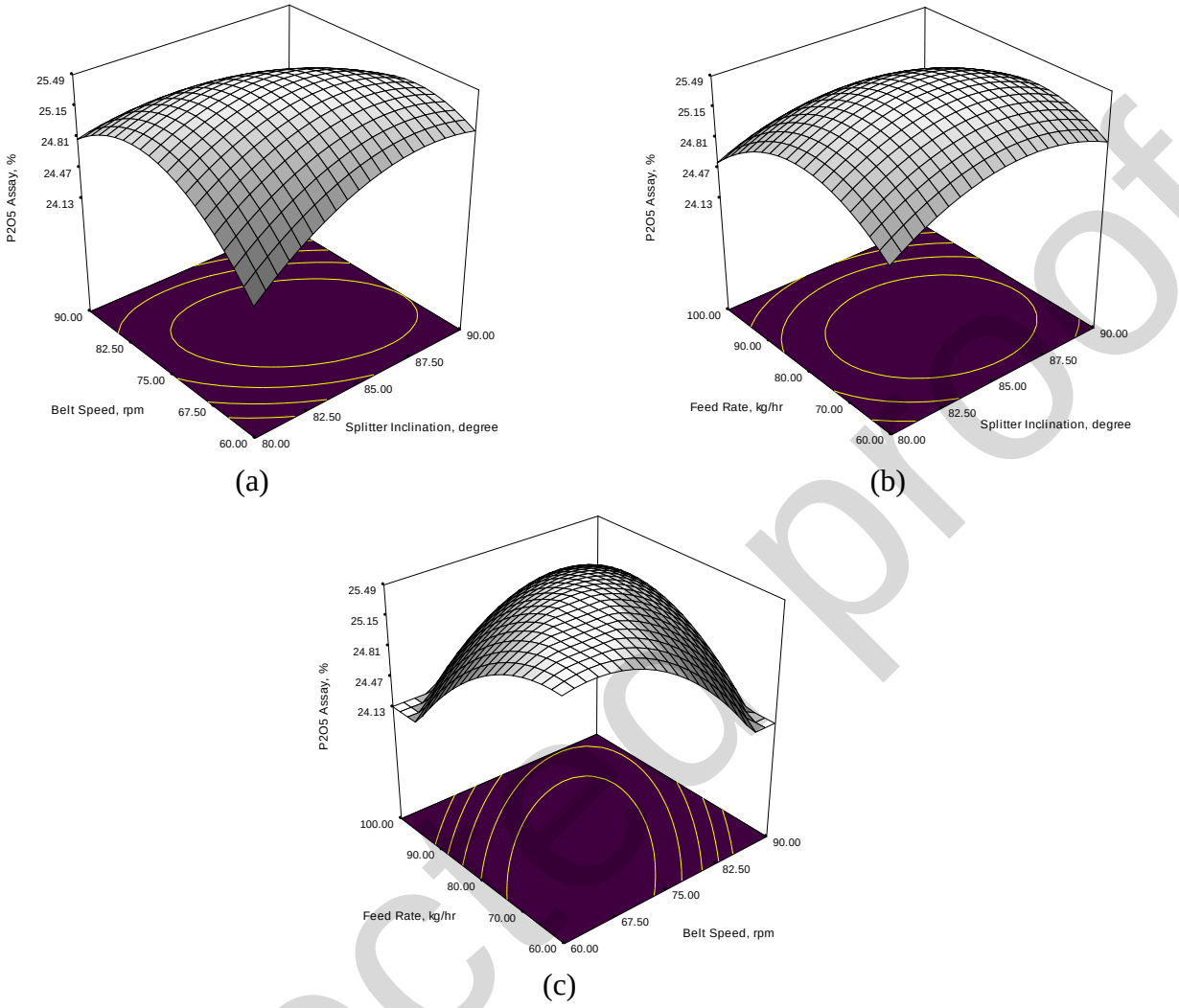
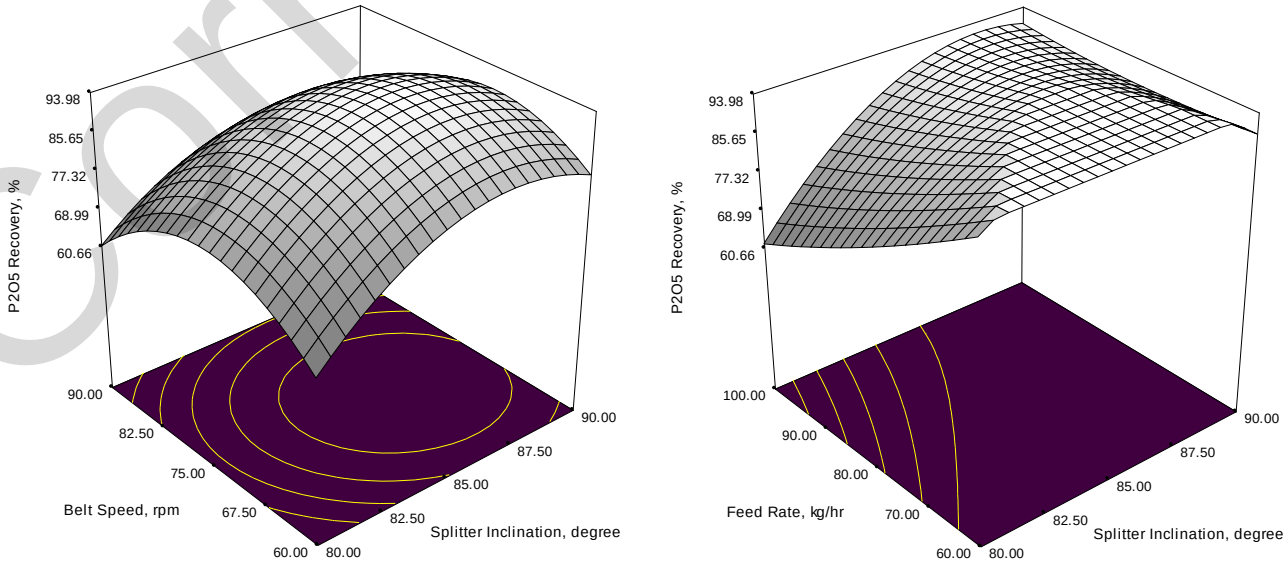


Fig. 7. The response surface plots a, b, and c illustrate the P_2O_5 assay percentage resulting from the primary effects of RER magnetic separation parameters: inclination angle, belt speed, and feed rate.



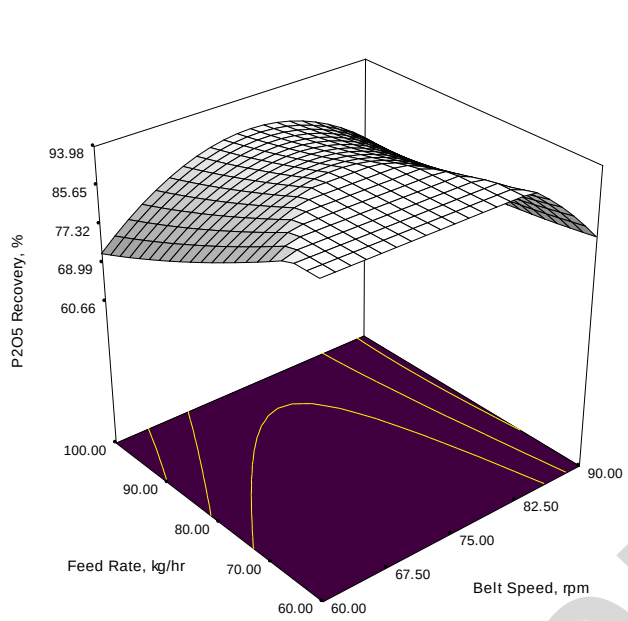
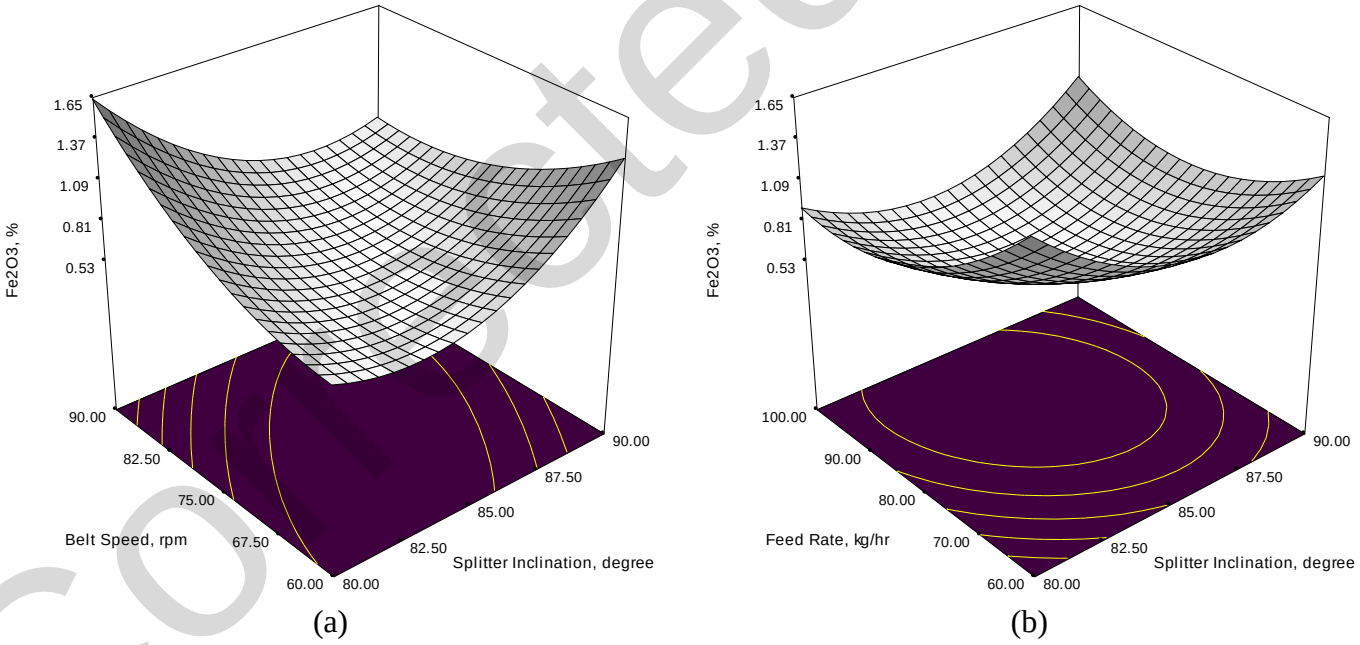
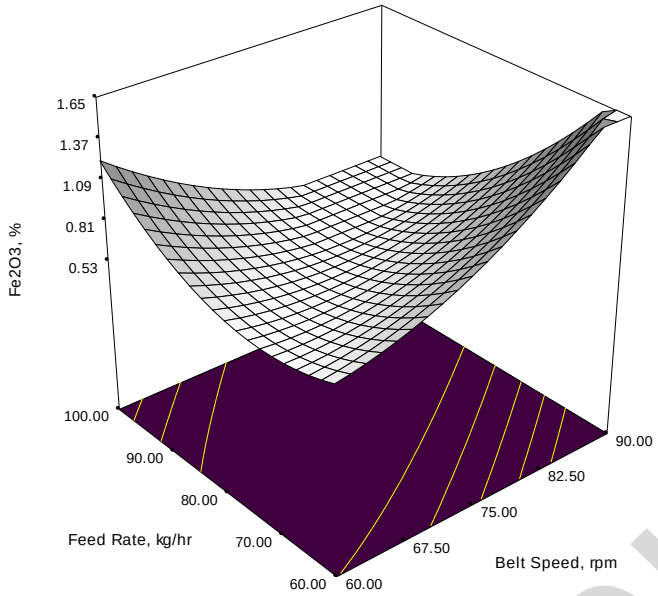


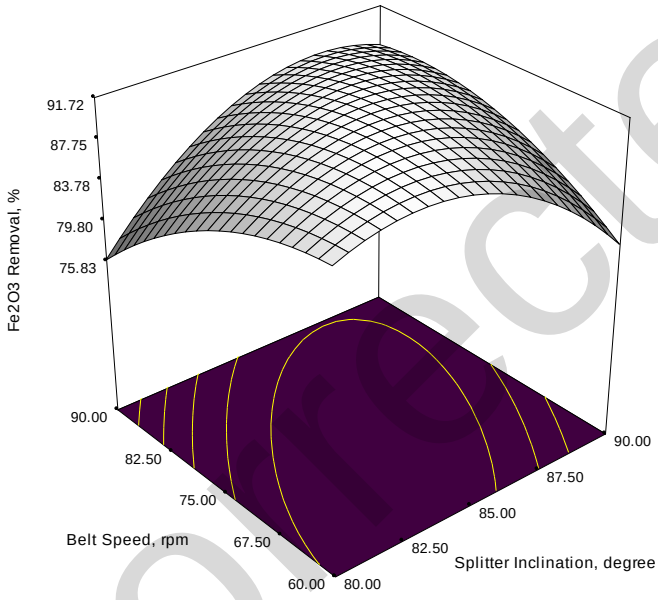
Fig. 8. The response surface plots a, b, and c illustrate the P_2O_5 recovery percentage influenced by the primary factors of RER magnetic separation: inclination angle, belt speed, and feed rate.



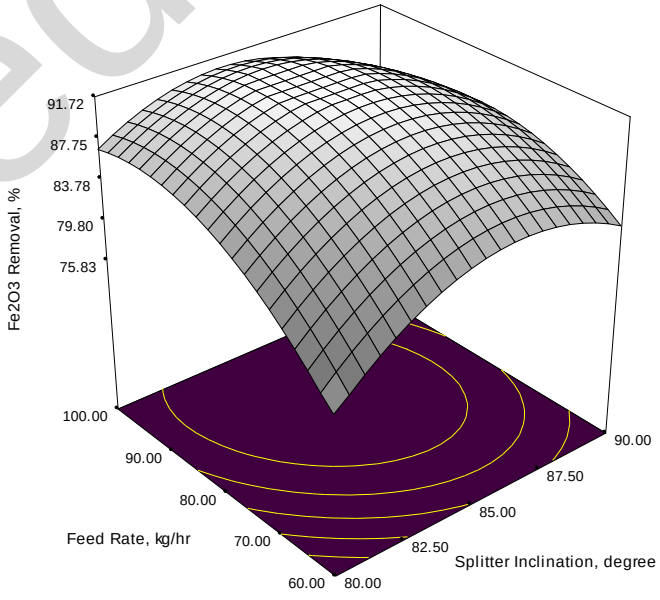


(c)

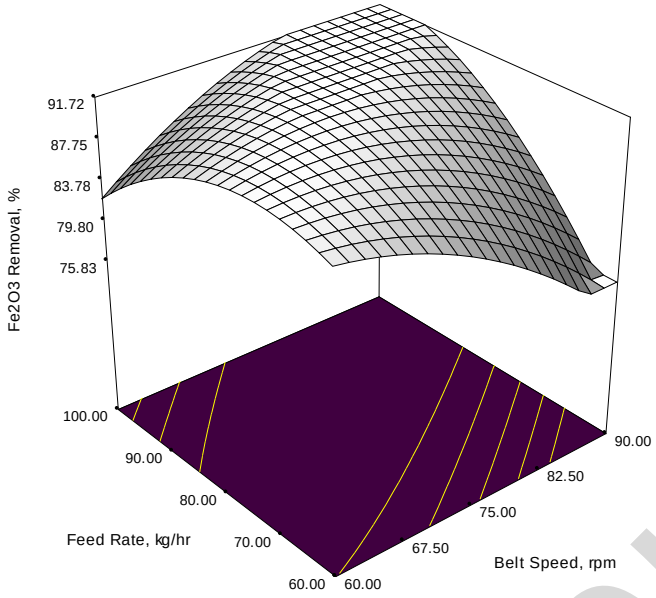
Fig. 9: The response surface plots a, b, and c illustrate the Fe_2O_3 assay percentage as influenced by the primary factors of RER magnetic separation: inclination angle, belt speed, and feed rate.



(a)

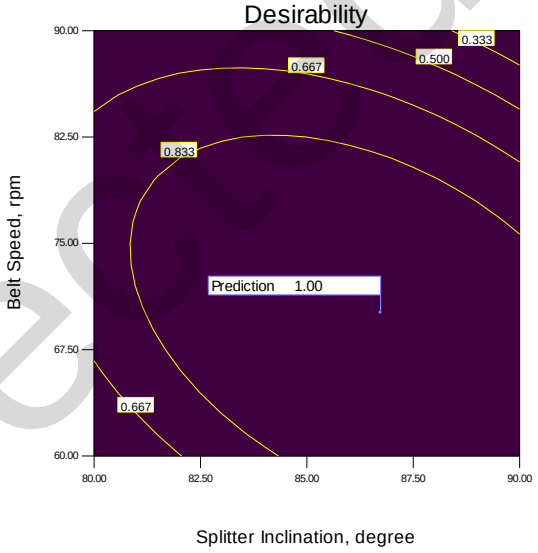


(b)



(c)

Fig. 10. The response surface plots a, b and c of Fe_2O_3 removal % resulting from the main effects of RER magnetic separation; inclination angle, belt speed and feed rate



(a)

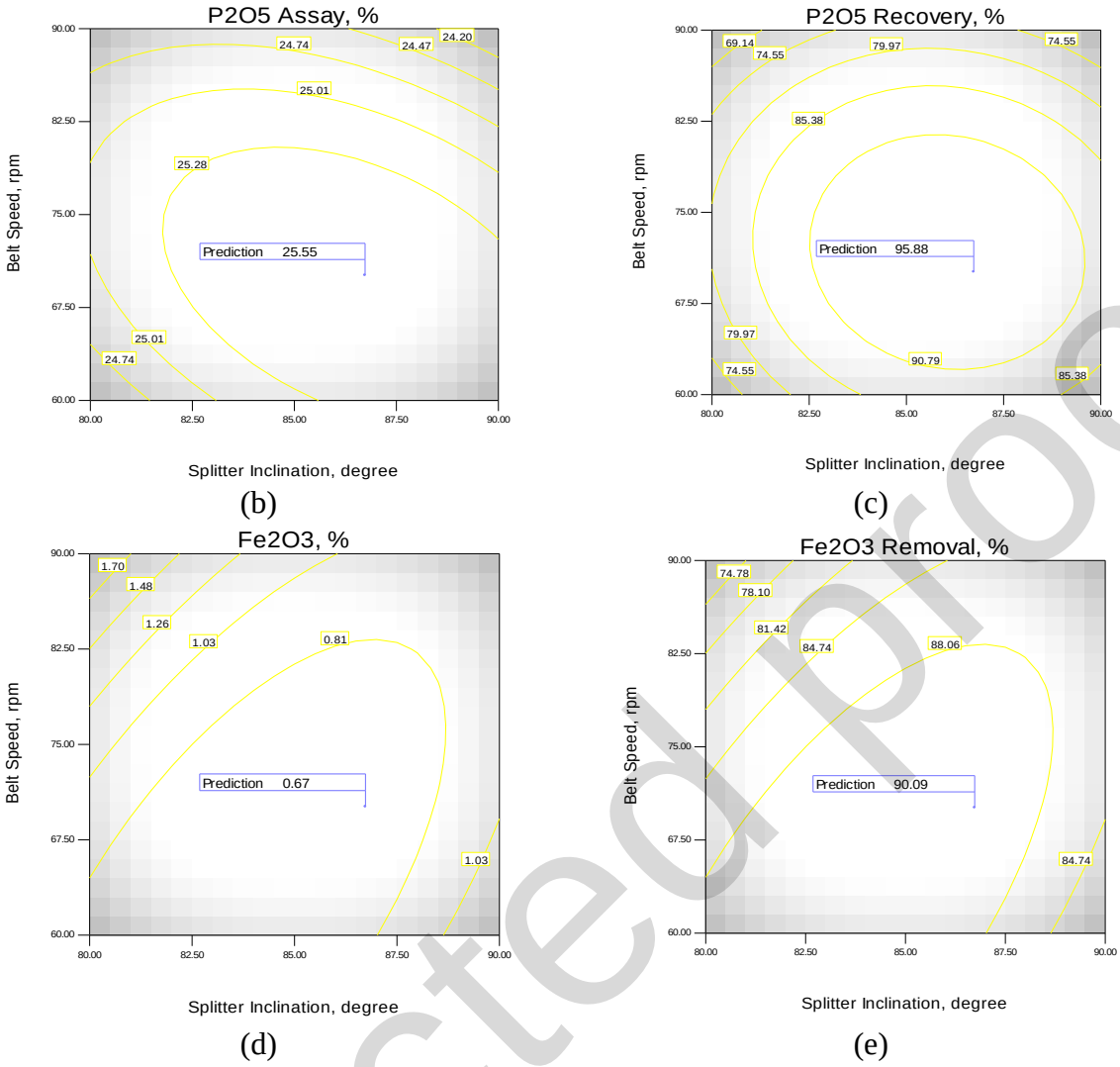


Fig. 11: RER Optimization of phosphate; a- Desirability, b- P_2O_5 assay %, c- P_2O_5 recovery %, d- Fe_2O_3 % and e- Fe_2O_3 removal %.

The optimal parameters determined by the Box-Behnken design for phosphate beneficiation using a magnetic separator are: a splitter inclination of 86.75 degrees, a belt speed of 70.10 rpm, and a feed rate of 73.65 kg/hr, as illustrated in Table 9. Applying these optimum parameters, a phosphate concentrate is obtained with 25.55 P_2O_5% with recovery of 95.88%. This concentrate has only 0.67 Fe_2O_3 % with 90.09 removal% (Figure 11a-e and Table 10).

Table 11: RER Optimum variables

RER Optimum variables		
Inclination angle, degree	Belt speed, rpm	Feed rate, kg/hr
86.75	70.10	73.65

Table 12: Phosphate upgrading at RER Optimum variables

Phosphate Upgrading			
P_2O_5		Fe_2O_3	
Assay, %	Recovery, %	Assay, %	Removal, %
25.55	95.88	0.67	90.09

The XRF of the non-magnetic phosphate fraction shows that the iron oxides were decreased from 6.76 % in the original sample to 0.67% in the non-magnetic sample. The total content of rare earth elements is increased from 678 to 942.24 ppm (Table 12). The dry high intensity magnetic separation of Abu Tartur phosphate ore sample removed successfully the iron content with 90% removal efficiency. However, the non-magnetic phosphate fraction requires further beneficiation processes in order to remove the silicates and clays to obtain phosphate concentrate enriched with rare earth elements.

3.3.2. A combination between the attrition scrubbing and the magnetic separation processes

In a trial to further upgrade Abu Tartur phosphate ore sample and enriching the total content of rare earth elements, a combination between attrition scrubbing and magnetic separation is applied. In this regard the RER Magnetic separation could be used as a valuable technique to further upgrade the scrubbed phosphate fraction. During this process, the phosphate fraction, which has already been scrubbed to remove surface clays contaminants, is subjected to RER magnetic separation. The magnetic minerals, including iron oxides and other magnetic gangue materials, are separated from the non-magnetic phosphate particles. This not only enhances the quality of the phosphate concentrate but also aids in enriching the total content of rare earth elements (REEs). As the REEs are associated with nonmagnetic minerals that exhibit weak magnetic properties, allowing for their concentration alongside the nonmagnetic phosphate product. This process is conducted as following:

1- The attrition scrubbing of Abu Tartur Phosphate ore sample of size fraction -250 +45µm was conducted using water at the obtained optimum parameters: solid/liquid ratio 40%, Agitation speed 1200 rpm, attrition time 20 min and attrition temperature 40^{o}C. Applying these conditions, a phosphate rich fraction of 22.96% P_2O_5 with recovery of 80.25% was obtained as previously displayed in Table 4. It was observed that the attrition scrubbing removed successfully the fundamental clay minerals.

2- The scrubbed phosphate fraction is subjected to further RER dry high intensity magnetic separation in order to remove the iron oxides and obtain phosphate concentrate enriched with rare earth elements. The most optimal parameters derived from the Box-Behnken design for phosphate beneficiation using a magnetic separator are as follows: splitter inclination at 86.75 degrees, belt speed at 70.10 rpm, and feed rate at 73.65 kg/hr, as detailed in Table 11. Applying these optimum parameters on the scrubbed phosphate fraction, a phosphate concentrate (nonmagnetic product) is obtained with 27.85 P_2O_5% (Table 13). The XRF of the non-magnetic scrubbed phosphate concentrate ensures successful removal of both clay minerals and iron oxides. Consequently, the total content of rare earth elements is enriched up to 1120.65 ppm (Table 14).

Figure 12 shows the X-ray diffraction of the non-magnetic scrubbed phosphate rich fraction. It is clear that the main mineral present in the nonmagnetic concentrate is phosphate, along with some silica content. The SEM-EDX images, Figure 13, evidences the effective separation of clays and iron flakes from phosphate as well as enrichment of the total rare earth elements.

Table 13: The chemical analysis of phosphate ore sample, non-magnetic phosphate fraction and the non-magnetic scrubbed phosphate concentrate

Component	Phosphate ore sample	Non-magnetic Phosphate fraction	Non-magnetic scrubbed phosphate concentrate
P_2O_5	20.62	25.55	27.85
Al_2O_3	2.25	2.40	0.52
MgO	1.52	1.65	0.18
Fe_2O_3	6.76	0.67	0.63
CaO	34.51	38.65	40.15
K_2O	0.14	0.19	0.18
Na_2O	0.69	0.74	0.76
SiO_2	8.59	8.72	8.79
SO_3	4.79	3.22	3.21
F	2.09	2.39	2.56
CO_2	6.51	5.78	5.51
L.O.I.	11.43	9.92	9.50
Tot. REOs	0.08	0.11	0.14

Table 14: Total REEs of phosphate ore sample, non-magnetic phosphate fraction and the non-magnetic scrubbed phosphate concentrate, analysed with (ICP-OES)

Component	REEs ppm / REOs	Phosphate ore sample	Non-magnetic phosphate fraction	Non-mag scrubbed phosphate
Scandium	Sc	0.00	18.00	24.50
	Sc_2O_3	0.00	27.61	37.58
Yttrium	Y	149.00	195.70	230.50
	Y_2O_3	189.22	248.53	292.72
Lanthanum	La	99.80	134.73	150.57
	La_2O_3	117.04	158.01	176.59
Cerium	Ce	185.00	252.90	299.85
	CeO_2	227.26	310.66	368.34
Praseodymium	Pr	57.00	73.96	91.45
	Pr_6O_{11}	68.87	89.36	110.49
Neodymium	Nd	94.10	125.27	150.77
	Nd_2O_3	109.76	146.12	175.86
Promethium	Pm	0.00	0.00	0.00
	Pm_2O_3	0.00	0.00	0.00
Samarium	Sm	21.20	31.22	28.50
	Sm_2O_3	24.58	28.50	33.05
Europium	Eu	0.00	7.20	7.85
	Eu_2O_3	0.00	8.34	9.09
Gadolinium	Gd	30.00	38.70	41.95
	Gd_2O_3	33.05	42.64	46.22
Terbium	Tb	0.00	5.40	4.55
	Tb_4O_7	0.00	6.35	5.35
Dysprosium	Dy	16.20	20.74	22.24
	Dy_2O_3	18.59	23.80	25.52
Holmium	Ho	0.00	8.50	12.48
	Ho_2O_3	0.00	9.74	14.30
Erbium	Er	12.00	15.60	18.55
	Er_2O_3	13.72	17.84	21.21
Thulium	Tm	0.00	0.00	2.85
	Tm_2O_3	0.00	0.00	3.25
Ytterbium	Yb	14.10	18.61	28.50
	Yb_2O_3	16.06	21.19	32.45
Lutetium	Lu	0.00	0.00	5.54
	Lu_2O_3	0.00	0.00	6.30
Σ Total (ppm)	Σ REEs	678.40	942.23	1120.65
	Σ REOs	818.15	1141.40	1358.32
Thorium	Th	30.83	42.15	49.89
	ThO_2	35.09	47.96	56.87
Uranium	U	2.31	3.16	3.75
	U_3O_7	2.73	3.73	4.42

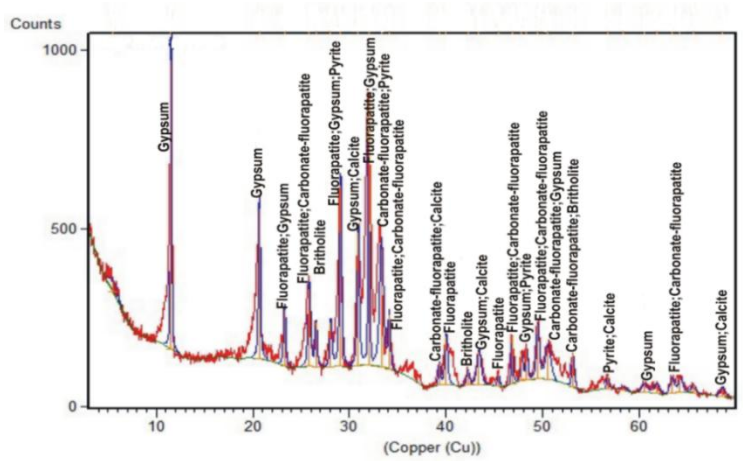


Fig. 12: XRD of the non-magnetic scrubbed phosphate concentrate

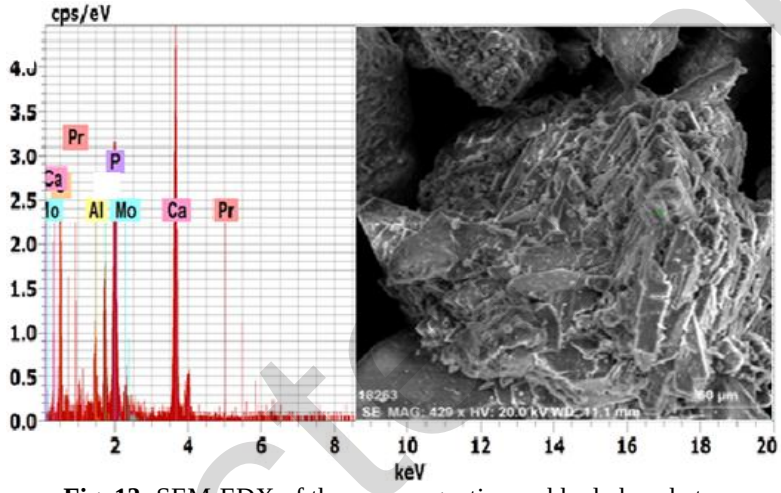


Fig. 13: SEM-EDX of the non-magnetic scrubbed phosphate concentrate.

Conclusions

In this study, a range of processing methods were utilized, including classification, attrition scrubbing, magnetic separation, and a combination of these techniques. The attrition scrubbing of Abu Tartur phosphate ore sample successfully removed clay minerals. However, the scrubbed phosphate fraction requires further beneficiation to remove iron oxides and silicates to obtain a valuable phosphate concentrate enriched with rare earth elements. Under attrition scrubbing optimal conditions (40% solid-liquid ratio, 1200 rpm agitation speed, 20 minutes of scrubbing, and 40ºC attrition temperature), the grade improved to 22.96% P_2O_5, with an 80.25% recovery and 820 ppm total rare earth elements (REEs) was obtained. The Box-Behnken design was employed to assess the RER magnetic separation of Abu Tartur phosphate ore. The optimal parameters identified were a splitter inclination of 86.75 degrees, a belt speed of 70.10 rpm, and a feed rate of 73.65 kg/hr. This resulted in a nonmagnetic concentrate containing 25.55% $P_2O_5$and 0.67% Fe_2O_3, with P_2O_5 recovery of 95.88% and 90.09% iron oxide removal.

A combination between attrition scrubbing followed by magnetic separation at the recorded optimal parameters yielded a phosphate concentrate with 27.85% P_2O_5. The results of the nonmagnetic scrubbed phosphate concentrate confirmed the successful removal of both clay minerals and iron oxides as well as, enriching the total content of rare earth elements to 1120.65 ppm.

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