Jordan Journal of Civil Engineering, Volume **14**, No. **3**, **2020**

Beneficiation Study of Low-Grade Jordanian Kaolin to Increase the Brightness Index

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ABSTRACT

Kaolin deposits are found all over the world. The increasing demand for product quality in paper and fine ceramic industries and the increasing efficiency in the production processes make greater requirements in terms of the quality of raw materials and additives, such as kaolin. Raw kaolin contains various amounts of discoloring elements, such as anatase (TiO_2), mica and iron oxide (Fe_2O_3), which give low brightness and are detrimental in use. In order to improve the quality of kaolin for more demanding applications, it was upgraded by using wet high-intensity magnetic separation for removing the iron oxide and titanium oxide impurities from low-grade Jordanian kaolin. This is the first trial to upgrade low grade. Different variables affecting magnetic separation processes, such as solid percent, magnetic field and retention time, were studied. The results showed a dramatic reduction of Fe_2O_3 (from 1.71 wt. % to 0.54 wt. % with about 68 % removal) and TiO_2 (from 1.55 wt. % to 0.35 wt. %, with about 77% removal) contents. The results obtained in the present study are quite promising. It was possible to diminish Fe_2O_3 and TiO_2 contents improve the brightness index of kaolin concentrate from approximately 69% to 89%. As a result, it can be concluded that high-quality kaolin products can be used for many industries, such as white cement, ceramic and paper filling. The new laws of investment make such economic pre-feasibility more profitable in terms of return on investment (ROI) and pay back period (PBP).

KEYWORDS: Kaolin, Beneficiation, Magnetic separation, Attrition scrubbing, Brightness index, Flowsheet.

INTRODUCTION

Kaolin clay (or kaolin) is a commercial clay composed principally of an aluminum hydrous silicate clay mineral kaolinite [$Al_2Si_2O_5(OH)_4$]. Kaolin is used in many industrial applications due to its unique physical, physicochemical and chemical properties (Asmatulu, 2002; Kuşcu and Yıldız, 2016; Nouri et al., 2020; Wang et al., 2016). These include paper, paint, rubber, ceramic, glass, refractory, agriculture, waste treatment and cosmetic applications. It is also used in nanocomposites as coating, pigment, acid/base regulator, filler and geopolymer material (Gougazeh, 2013, 2018, 2019; Melhem and Hammoud, 2017); Vivek and Dhinakaran, 2017). The required quality and characteristics of kaolin depend on its use (Bundy, 1993; Gámiz et al., 2005; Jepson, 1984; Mukherjee, 2013; Murray, 2000, 2007). Typical impurities present in kaolin ore are: quartz, iron oxides, titaniferous minerals, mica, feldspar, organic matter,… etc. (Carty et al., 1998; Campos et al., 2017; Chandrasekhar and Ramaswamy, 2002; Murray and Keller, 1993; Pinheiro et al., 2005). The presence of impurities, particularly iron and titanium bearing minerals, imparts the color of kaolin (low brightness) and is detrimental in most kaolin applications (Gougazeh, 2018; Xu, et al., 2015; Milledgeville et al., 1997; Prasad et al., 1991). For most of the industrial applications, kaolin should be processed to obtain refined clay so as to match with standard specifications (Gougazeh, 2018; Raghavan et al., 2004).

Recently, research has been conducted on clay

Received on 26/8/2019.
Accepted for Publication on 13/5/2020.

samples to determine the surface properties and interfacial interactions of kaolin clay (Hu and Liu, 2003; Hu et al., 2003; Hu et al., 2005; Macht et al., 2011). Because of the kaolin particle size and its platelet structure, beneficiation and dewatering of the material are the main interests of many plants. In order to improve the quality of kaolin clay for industry, the discoloring impurities must be removed from the samples by suitable separation techniques (Raghavan et al., 2007; Asmatulu, 2002). These separations generally include magnetic separation, froth flotation, selective flocculation and size separation using hydrocyclone or centrifuge and leaching (Raghavan et al., 1997; Ma, 2011). Numerous publications are available on the removal of titaniferous impurities by froth flotation with different reagent combinations (Raghavan et al., 1997; Ma, 2011; Wang et al., 2016).

The studied area (Jabal Al-Harad kaolin deposit/ Batn El-Ghoul area) is located in southern Jordan, about 70 km southeast of Ma'an city. It is 280 km south of Amman. It covers 8 km2. The deposit is at a latitude of 29º 37' 52'' N and a longitude of 35º 57' 20'' E. The kaolin deposit reserve was estimated to be at least 180 million tons (Masri, 1998). The commercial kaolin deposit is concentrated in the north of Batn El-Goul area and is elongated from northeast to southwest. Presently, open pit mining activities are carried out below a 2-6 m thick soil cover (Gougazeh and Buhl, 2010).

Jabal Al-Harad kaolin deposit has been studied by Gougazeh and Buhl (2010), as well as by Khoury and El-Sakka (1986). As no systematic studies were carried out in the area under consideration concerning beneficiation and upgrading processes of kaolin deposits, this study was taken up to fill this lack of information and reports on methods for opening up kaolin deposits and specify the requirements to be met by kaolin products for varying industrial applications. The exploration and evaluation of raw materials for industrial uses represent a very important step for encouraging local industries. In addition, this step participates in forming a strong and healthy economy. There are many industrial raw materials that are imported for the local market, although some of these may be present, but not explored and evaluated.

The aim of the present paper is to investigate the possibility of using wet high-intensity magnetic separation for removal of colored contaminants, such as iron and titanium oxides, and consequently to improve the brightness of Jordanian kaolin from Jabal Al-Harad kaolin clay deposits in south Jordan which have not been earlier subjected to such beneficiation processes to be suitable for different industrial applications.

EXPERIMENTAL PROCEDURE

Materials

A representative bulk sample of about 200 kg of raw kaolin was collected from the new mine of Jabal Al-Harad, southern Jordan (Gougazeh and Buhl, 2010; Gougazeh, 2018; Gougazeh et al., 2019). The kaolin samples were broken with a rubber hammer and then subjected to disintegration or crushed carefully by a jaw crusher to a size smaller than 4 mm. The crushed products were attrition scrubbed at 4000 rpm and 40 % solid content using 0.1% Na-hexametaphosphate at pH 9.5 for 15 minutes. The products were washed on an industrial wet vibrating screen for 15 minutes through a 325 mesh (44 μm) screen. The percentage of the fraction less than 44 μm was about 97 wt. % of the original sample. The undersize fraction (44 μm) was characterized for its mineralogy, chemical constituents, particle size distribution and brightness.

Analytical Methods

The fraction less than 325 mesh (<44 μm – kaolin sample) was analyzed by X-ray diffraction (XRD), scanning electron microscope (SEM), X-ray fluorescence (XRF-chemical analysis) and the measurement of the brightness index. The analysis for mineral identification was performed with a Bruker AXS D4 ENDEAVOR X-ray diffractometer using Ni-filtered Cu-Kα radiation and with the generator operating at 40 kV and 40 mA. The goniometer velocity was 0.02º (2θ) per 1 s in the interval between 4 and 80º (2θ). The powder data was analyzed with the Stoe WinXPOW software package. Micrographs of the general texture and special features were taken with a JEOL JSM-6390A scanning electron microscope (SEM). Samples were prepared from the fraction less than 44 μm. A thin coating of Au was formed on each specimen by sputtering with an Edwards SCANCOAT SIX sputtercoator. SEM investigations were performed at 20 kV acceleration voltage. The chemical analysis was carried out with a Bruker S4 wavelength X-ray

dispersive fluorescence spectrometer (WDXRFS) with an Rh X-ray tube and the fraction (<44 μm) was prepared in a VANEOX automatic press in molding conditions of 20 mm of diameter, with a pressure of 20 tons and a time of 30 s, using boric acid (H_3BO_3) as caking in proportions of 1:0.1. The loss on ignition was determined by heating to 1000ºC for two hours. Size analysis of the feed sample (<44 μm) fraction was conducted using an Atterberg cylinder according to Stocke's law to separate the finer fractions of <2 μm, 2-6 μm, 6-20 μm and 20-44 μm. The degree of brightness of <44 μm (feed sample) and non-magnetic fractions was measured with a Carl-Zeiss Elrepho 96160 reflectance photometer ($MgCO_3$ as reference material) and expressed in ISO (International Standards Organization) units. Mineral identification analysis, chemical analysis and brightness were determined for the < 325 mesh (<44 μm – kaolin sample) and non-magnetic fractions. Autosorb – 3B, Company Quanta Chrome (Inorganic Chemistry Department, Hannover University) was used to determine the specific surface area of kaolin before and after treatment. Measurements were carried out according to the Brunauer–Emmet–Teller (BET) nitrogen adsorption method after outgassing at 200 °C for 2 h (Macht et al., 2011). A measurement time of 20 h was used to guarantee the achievement of equilibrium conditions.

Wet High-Intensity Magnetic Separation (WHIMS)

Impurities in kaolin which cause particular problems are iron-bearing minerals and fine-grained anatase. These tend to be more abundant in sedimentary kaolins as in Jabal Al-Harad kaolin deposit, south Jordan. The removal of coloring oxides is essential if high-brightness saleable grades are to be produced. Wet high-intensity magnetic separator is a standard processing technique used in kaolin industry.

Magnetic separation for the undersize fraction (<44 μm) was carried out in a Boxmag-Rapid separator in wet conditions and under a magnetic induction of approximately 14000 Gauss.

The kaolin samples were dispersed at 20% solid content using 0.1% Na-hexametaphosphate. The kaolin slurry was pumped vertically trough the magnetized canister at 180 sec retention time. The nonmagnetic product was collected from the canister, representing the kaolin concentrate. At the end of this cycle and after separation of the nonmagnetic fraction, the magnetic fraction was then de-energized and the bed flushed with high-velocity water to separate the magnetic fraction. The nonmagnetic and magnetic products were filtered, dried and weighed to calculate the weight percentage of both. Brightness index was measured for nonmagnetic products and complete chemical analysis for both nonmagnetic and magnetic products was conducted by X-ray fluorescence.

On the basis of chemical composition of nonmagnetic products, it was also determined that HIMS is efficient enough to capture submicron-sized magnetic particles and is therefore capable of magnetic separation to produce high-brightness kaolin.

RESULTS AND DISCUSSION

Characterization of Kaolin before Beneficiation

Chemical analysis, particle size distribution analysis and mineralogical analysis of the fraction less than <44 μm (kaolin sample) were performed using XRF and the results are indicated in Table 1. The results showed that the kaolin sample has high contents of Al_2O_3 (32. 07 wt. %) and SiO_2 (51.05 wt. %). The sample has relatively high amounts of Fe_2O_3 (1.71 wt. %) and TiO_2 (1.55 wt. %). It has a lower percentage of loss of ignition (11.25 wt. %). These results are in agreement with XRD analysis which confirms that kaolin is the dominant mineral followed by quartz and traces of anatase (Fig. 1). The XRD pattern (Fig. 1) indicates the presence of kaolinite, identified by the presence of the basal peaks 7.23 Å (001) and 3.86 Å (002) and the triplets 4.48, 4.35 and 2.56 Å and 2.53, 2.49 and 2.33 Å. Also identified, as impurities, were muscovite at 9.95 and 4.98 Å and quartz at 3.3 Å. TiO_2 and Fe_2O_3 occur in small amounts (Table 1), mainly in the form of anatase and hematite. The occurrence of muscovite is consistent with K_2O.

Table 1. Characterization of kaolin sample (<44 µm)

Method	Constituents/ Particulate	
Chemical analysis	SiO_2	51.07
	TiO_2	1.55
	Al_2O_3	32.07
	Fe_2O_3	1.71
	MnO	0.01
	MgO	0.09
	CaO	0.11
	Na_2O	0.06
	K_2O	1.18
	P_2O_5	0.12
	LoI	11.25
Particle size distribution analysis	< 44 µm	97.35
	< 44 – 20 µm	1.72
	< 20 – 6 µm	5.12
	< 6 – 2 µm	21.24
	< 2 µm	71.92
Mineralogical analysis	Kaolin	Major
	Quartz	Minor
Brightness [%]		72.24

The morphologies of the raw kaolin are shown in Fig. 2. As can be seen, Jabal Al-Harard kaolin exhibits a pseudo-hexagonal plate-like shape which is similar to other kaolinitic minerals reported elsewhere (Ekosse, 2000, 2001, Murray, 2000, 2007).

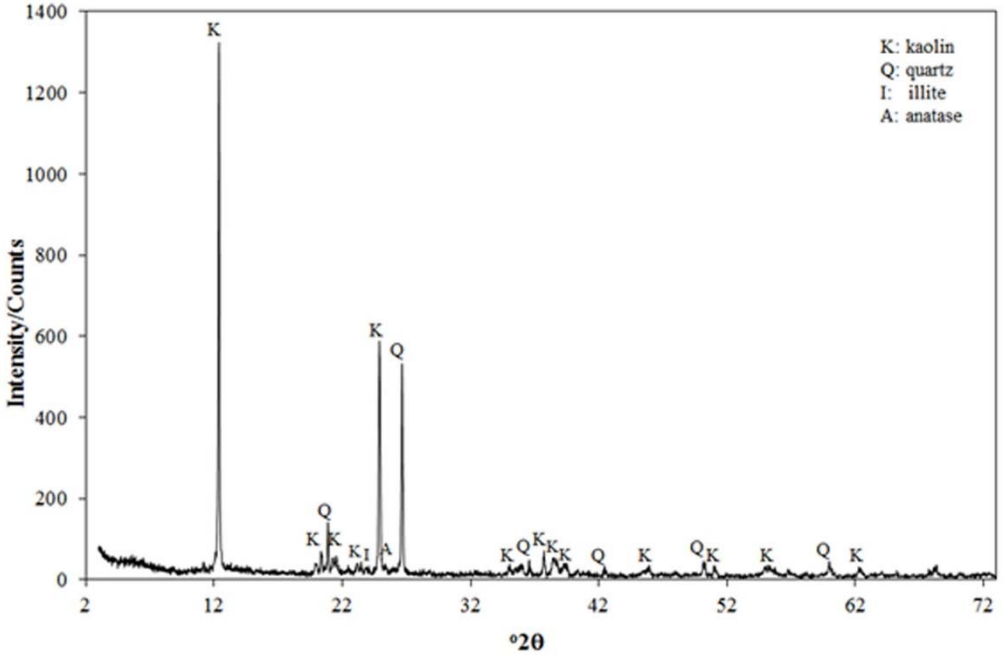

Figure (1): X-ray diffraction pattern of raw kaolin (fraction <44 µm)

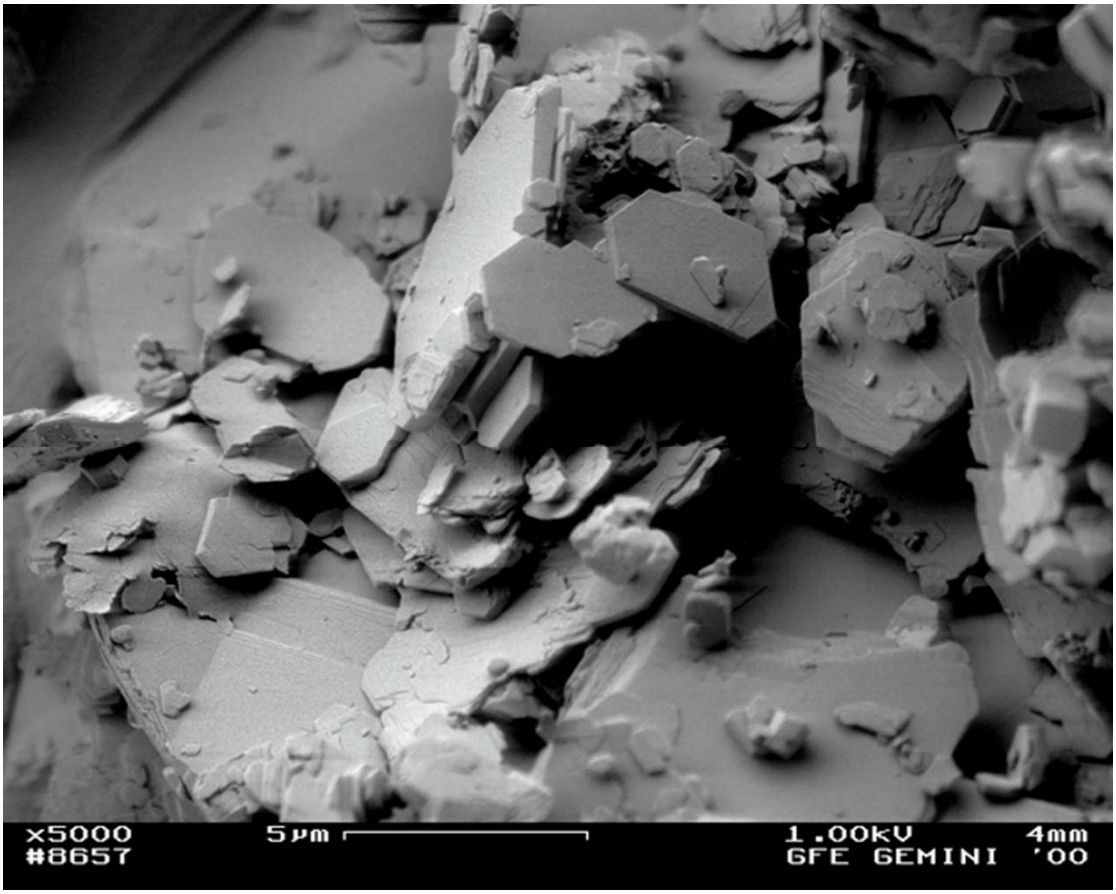

Figure (2): SEM photomicrographs of kaolin showing kaolinite plates with a hexagonal shape

The results of beneficiation of Jabal Al-Harad kaolin by magnetic separation will be discussed later. The main chemical constituents of the studied kaolin samples are Al_2O_3 and SiO_2, in addition to small amounts of impurity oxides Fe_2O_3, TiO_2, MgO and CaO (Table 1). The level of coloring oxides (Fe_2O_3 + TiO_2) in the studied kaolin <44 µm kaolin fractions (average 2.53 %) is relatively high as compared with that of the common high-grade kaolin which should in total contain less than 1.2% Fe_2O_3 + TiO_2 (Konta, 1979).

Grain size analysis results of the studied representative kaolin samples (Table 1) show that the average weight percentages of <44 µm, <20 µm, <6 µm and <2 µm size fractions are about 97%, 95%, 79% and 72%, respectively. These investigations suggest that the grain size less than 44 µm is a key for kaolin concentrate.

Beneficiation of Jabal Al-Harad Kaolin

Different beneficiation techniques were suggested, such as attrition scrubbing, degritting and multi-classification using hydrocyclone. Using these techniques helped in removing coarse grit of silica and carbonates. Successful separation of iron oxides to enhance the brightness of the kaolin product could be achieved by a combination of magnetic separation and reductive bleaching. Meanwhile, appreciable amounts of associated carbonates could be removed using the froth flotation process. Applying a combined beneficiation flowsheet succeeded in producing a high-grade kaolin concentrate (~80 wt. % below 2 µm) low in both iron oxides (0.85 %) and CaO (0.1%) with a significant improvement in its brightness to 89.60 %. Such a concentrate can be used in ceramics and paper industries.

The beneficiation of kaolin from crude ore to finished product relies on a wide variety of processing techniques (Flowsheet in Fig. 3). Based on chemical analysis, the concentration of kaolin from Jabal Al-Harad as well as the successful separation of iron and titanium oxides (the coloring materials) to enhance the brightness of the kaolin product could be achieved using wet high-intensity magnetic separation. Applying such a combined beneficiation flowsheet (Fig. 3) succeeded in producing a high-grade kaolin concentrate (low in both iron oxides (0.55%) and TiO_2 (0.43%)) with a

significant improvement in its brightness to 89.60 %. The kaolin concentrate produced showed suitable characteristics for use in paper industries, as filler and coating, and in ceramic industries (Campos et al., 2017).

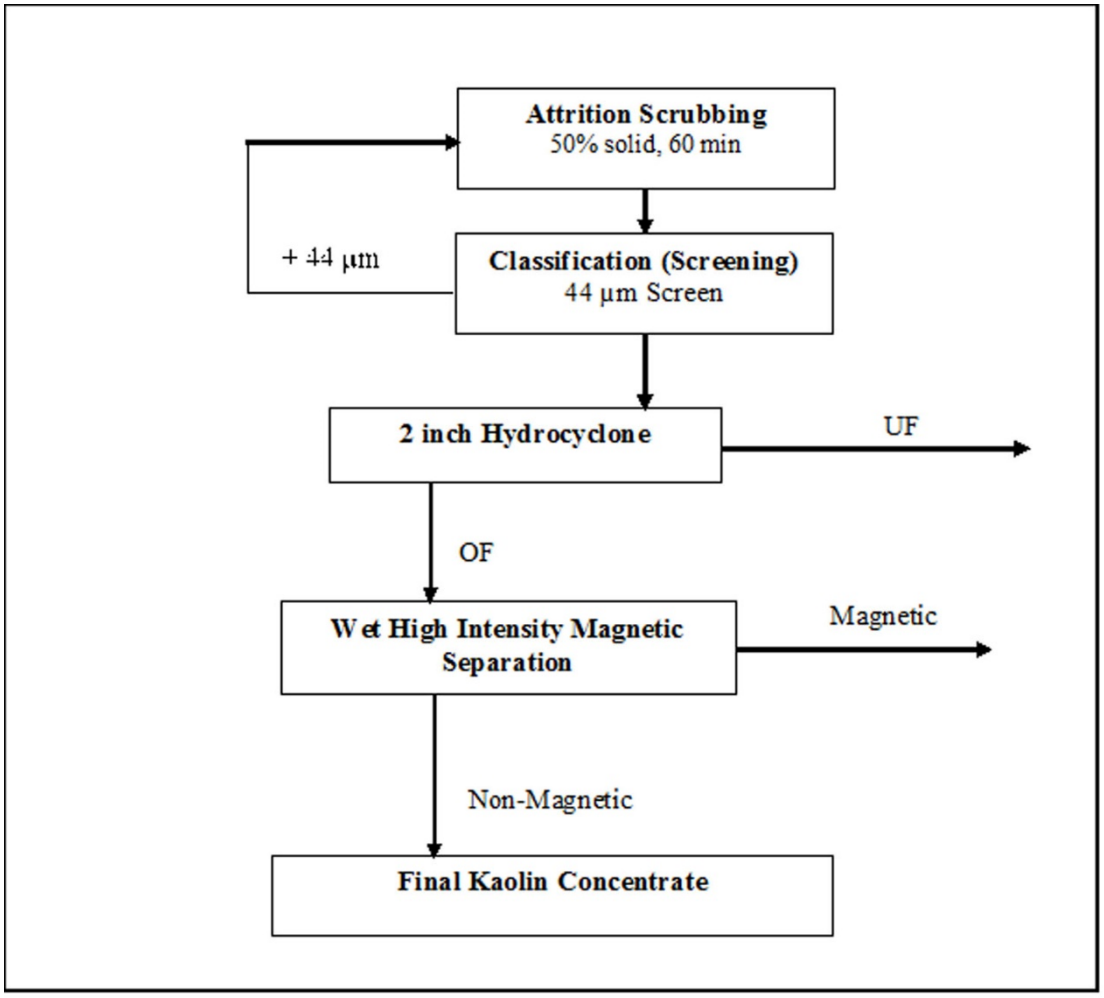

Figure (3): General flowsheet for beneficiation of Jabal Al-Harad kaolin, south Jordan

Hydrocycloning

Hydrocyclone separation was carried out for the <44 µm screened materials. The test results of separation using hydrocyclone are given in Table 2. It is shown that the overflow fraction represents about 74% by weight of kaolin samples. The hydrocyclone overflow was taken as a pre-concentrate for the magnetic separation test to produce high-quality kaolin. The particle size analysis of such pre-concentrate (overflow product) showed that it has a very fine grain size distribution, where about 80% by weight of the grains are below ~ 2 µm for the kaolin sample.

Table 2. Classification of wet screening products (<44 µm faction) using a 2-inch hydrocyclone

Fraction	Operational wt. %	Overall wt. %
Overflow	76.23	74.21
Underflow	23.77	23.14
Total	100	97.35

Wet High-Intensity Magnetic Separation Test

The wet high-intensity magnetic separator was used to increase product brightness by removing iron and titanium minerals, such as hematite and anatase.

The hydroyclone overflow product for both samples L and U was subjected to magnetic separation using "Boxmag Rapid" wet high-intensity magnetic separator by the procedure given earlier and the results under optimized conditions are given in Table 3. The results show that the maximum removal of iron oxide reached 68%, while TiO_2 removal reached 72%, hence increasing the degree of brightness from 72.24% to 89.60% in the nonmagnetic fraction (kaolin product) at a magnetic field intensity of 14000 Gauss (Table 3).

Table 3. Wet high-intensity magnetic separation (WHIMS) tests conducted on the overflow of II-inch hydrocyclone using "Boxmag Rapid" magnetic separator

Fraction	Operational wt. %	Overall wt. %	Fe_2O_3 wt. %	Fe_2O_3 % removal	TiO_2 wt. %	TiO_2 % removal
Non-Magnetic	94.17	69.88	0.55	68	0.43	72
magnetic	5.83	4.33	6.46		7.41	
Total	100	74.21	1.74		1.65	

The results of Table 3 and Figures from 4 to 6 indicate that a high-quality product of kaolin was achieved by using wet high-intensity magnetic separation process. In the final kaolin product, the maximum removal of iron oxide reached 68%, while TiO_2 removal reached 72% with a better degree of brightness (89.6%) at a magnetic field intensity of 14000 Gauss. The final results indicate that the optimum separation efficiency was observed at a retention time of 2 min at a material feeding rate of about 280 ml/min (Fig. 5). The concept of retention time involves the control of flow rate in a canister in order to balance the viscous drag of the medium on the suspended particles in the slurry against the force of magnetic attraction induced in the matrix by the background field. Control of retention time is still the crux of the entire process, where it controls the product quality and the production rate (Campos et al., 2017). The results also showed that at such conditions, it is possible to remove 68% of associated iron oxide and about 72% of TiO_2 with a concentrate assaying 0.55% and 0.43% of Fe_2O_3 and TiO_2, respectively and the maximum brightness achieved was 89.6%.

Figure 6 shows the relationship between the solid percent and magnetic separation efficiency. As shown in the Figure, the removal efficiency increases with increasing the solid percent starting from 2 till reaching the most efficient separation at a solid percent (8%), where iron oxide removal reached 68% (0.70% Fe_2O_3) and TiO_2 removal reached 72% (0.80% TiO_2). After that, the removal efficiency started to decrease gradually till a solid percent of 16%.

Accordingly, the data obtained by WHIMS magnetic separation correlates with the mineralogical composition (XRD) of the kaolin deposit (Fig. 7). The XRD results of the beneficiated kaolin (kaolin product) indicated that kaolin is the dominant mineral and quartz was found as traces. The absence of iron and titanium oxides is obvious in the XRD pattern obtained after beneficiation (Fig. 7), demonstrating that the most iron occurs as its oxides and does not substitute in the kaolin structure and indicating that the mineralogical characteristics of the kaolin have not been significantly affected. It is seen that all test results are in excellent agreement with results in the literature (Raghavan et al., 1997, 2007). It could be concluded that by increasing the applied magnetic field, there is a remarkable increase in the upgrading degree of the product.

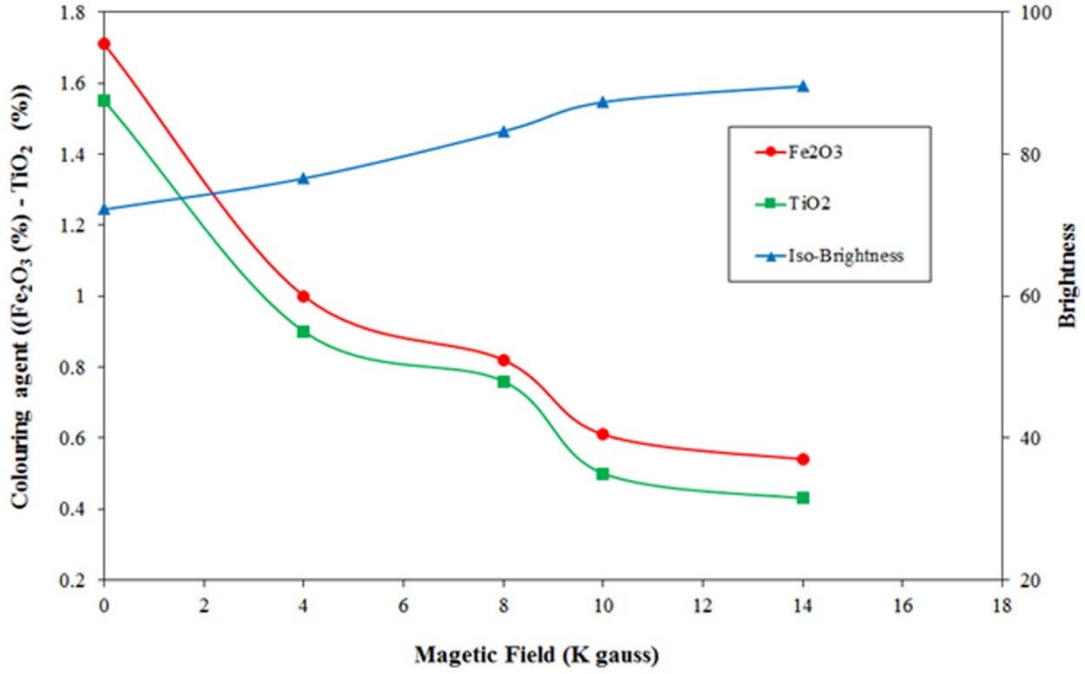

Figure (4): Kaolin product grade as a function of magnetic field strength

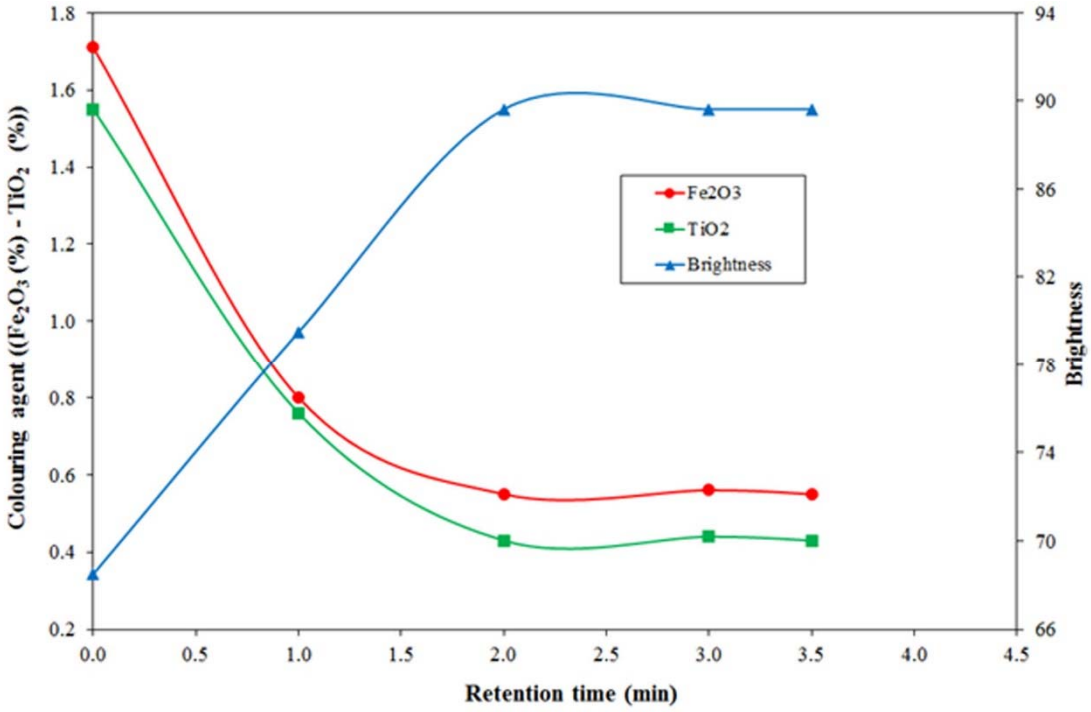

Figure (5): Kaolin product grade as a function of retention time

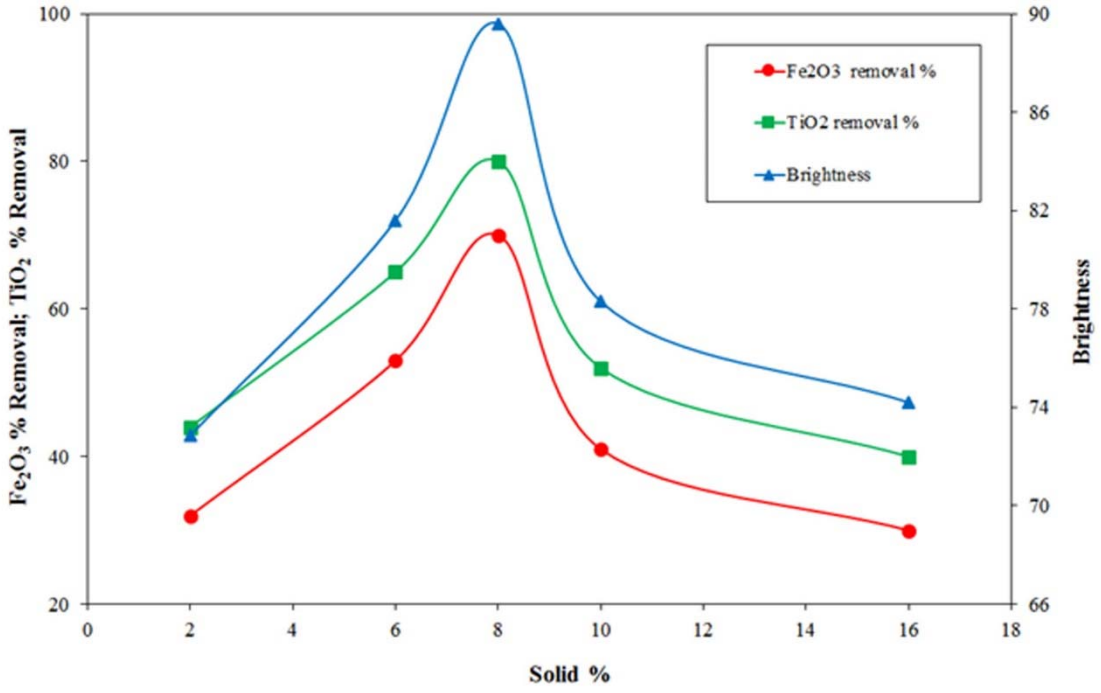

Figure (6): Kaolin product grade as a function of solid percent

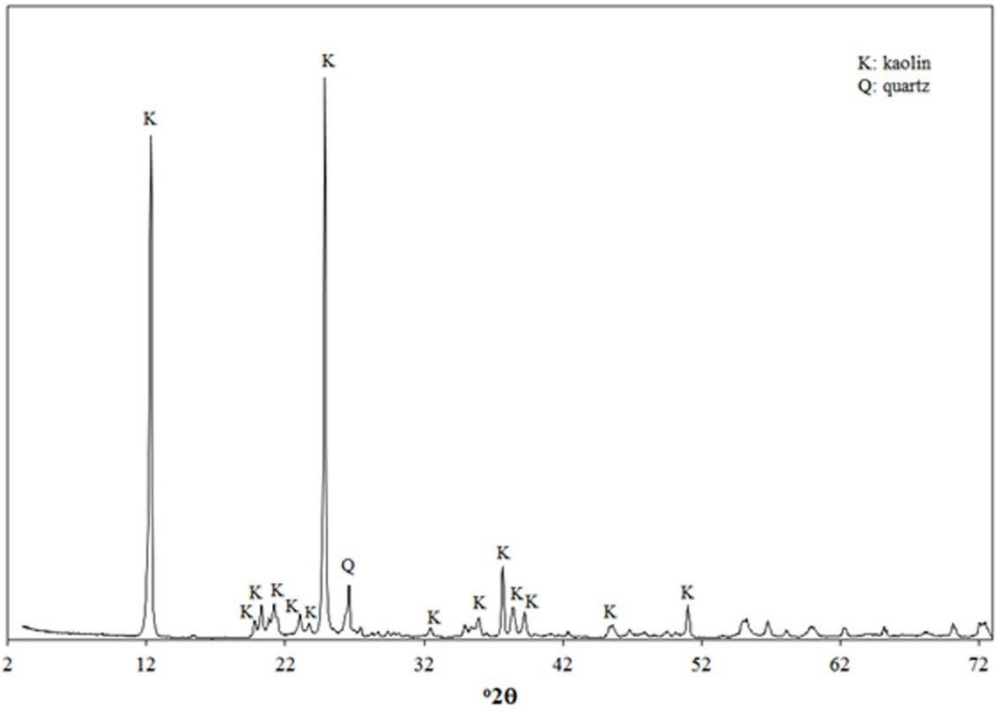

Figure (7): X-ray diffraction pattern of the beneficiated kaolin (final kaolin product)

Specific Surface Area

The specific surface area was measured by BET, taking a multipoint surface of 13.03 m^2g^{-1} for the beneficiated sample, as compared to 14.78 m^2g^{-1} for the raw kaolin sample. The decrease is difficult to explain, since the morphology on the fine scale is quite similar.

Pre-fesibility Study of Beneficiation of Al-Harad Kaolin Deposit

Commercially available kaolin deposits are

extensive. Careful and extensive geological, mineralogical and chemical studies (Gougazeh and Buhl, 2010) were conducted to achieve effective separation, beneficiation, upgrading and concentration of kaolin from Jabal Al-Harad kaolin clay deposit in south Jordan (Gougazeh, 2013; Gougazeh, 2018; Gougazeh et al., 2019), which can represent a value added to the Jordanian economy by preventing the importation of high-quality kaolin. The desired properties, large quantities of good-quality raw material (exceeding 180 million tons), and simple mining process, strongly recommend that deposit to be utilized in the ceramic and paper industries. In addition, the existence of a developed infrastructure at nearby Aqaba city and port of Aqaba will be important factors in the economic potential of the studied deposits. In the meantime, the Jordanian government has created a new healthy climate to encourage investment in Jordan.

The mass balance of the proposed beneficiation plant was calculated based on the suggested flowchart in the current study. It was found that 15.000 tons of raw material of kaolin (RMK) should be processed to yield 10.482 tons of kaolin concentrate that covers the local forecast of the annual demand on kaolin in ceramic industry in 2020 (Personal Communication), applying the feed rates of RMK in the mass balance of the proposed beneficiation flowsheet.

Financial Evaluation of the Project

Both "Return on Investment" (ROI) and "Pay Back Period" (PBP) were calculated at different alternatives and different selling prices. In each alternative, net annual cash flow, net profit, depreciation, taxes and interest have been estimated at different levels of selling prices (Vogely, 1985; Behrens and Hawranek, 1991; Pascoe, 1992).

The new healthy climate of investment will have a positive impact on the economic viability of projects in Jordan. For this reason, the pre-feasibility study of beneficiation of Jordanian kaolin is recalculated in light of this new situation.

Although the total capital cost required for installation of a plant beside the mines is calculated as about 2,518,500 US$ and the operating cost is nearly about 600,000 US$, yet the economic viability of the project is significantly improved. This clearly indicates the impact of the new facilities presented to investors in the new law of investment on making the projects more profitable, especially in the new industrial communities and desert areas.

CONCLUSIONS

From the results of this investigation, the following conclusions can be drawn:

- The results showed that the fractions <44 µm of kaolin samples from Jabal Al-Harad kaolin clay deposit in south Jordan were composed mainly by well-crystallized pseudohexagonal kaolinite in booklet morphology, followed by quartz as accessory and traces of illite/muscovite, hematite and anatase dispersed through the kaolin. The chemical composition of kaolin is essentially SiO_2 and Al_2O_3, with minor amounts of TiO_2, Fe_2O_3, MnO, MgO, Cao, Na_2O, K_2O and P_2O_5.
- Simple hydrocycloning and laboratory wet high-intensity magnetic separation processes used in this study were found to be very successful in producing kaolin with finer particle-size distribution, increasing the brightness index and removing of impurities such as iron and titanium oxides. Those processes were also able to increase the Al_2O_3 and reduce the SiO_2 contents in kaolin from Jabal Al-Harad, south Jordan.
- Separation with a magnetic induction of 14000 Gauss was able to remove iron and titanium oxides, which resulted in an increase in the brightness index of the kaolin product. This led to increase the brightness of kaolin from 72% to 89.6% in nonmagnetic kaolin (kaolin product).
- The beneficiated Jabal Al-Harad kaolin is sufficiently low in iron and titanium along with high brightness (89.6%). Therefore, it is supposed to be a source of white burning industrial kaolins as fine ceramics and as filler in paper, paint and plastics industries.
- The results indicated that kaolin deposits from Jabal Al Harad, south Jordan have the potential to be processed to kaolin products of premium grades with simple beneficiation. It is recommended that kaolin and clay suppliers in Jordan look into the possibility of upgrading their products instead of just selling their kaolin or clay in raw form.

Acknowledgments

The author would like to express his gratitude to the Institute of Mineral Resources and Raw Materials Engineering, Aachen University of Technology (RWTH-Aachen), Germany for technical support of this research work. Special thanks are due to the Institute of Mineralogy, Leibniz University, Hannover, Germany for allowing the use of research facilities. Special thanks go to Prof. Dr. J.-Ch. Buhl and Dipl. Geow. Valeriy Petrov for assistance with the acquisition of XRD and SEM data.

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