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Effect of blunging process on purification of halloysite ore from ferrous impurities by dry magnetic separation

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Abstract: The objective of this research is to study the effects of feed particle size, splitter angle, and washing process on Fe_2O_3 removal efficiency in the separation of ferrous impurities from halloysite ore by dry magnetic separation in order to increase the purity of halloysite sample after crushing and blunging processes separately. Firstly, after crushing ore in a jaw crusher and sizing to -2+1 mm, -1+0.5 mm, and -0.5+0.212 mm fractions, the sized materials were fed to REMS-type dry magnetic separator at a constant belt speed of 300 rpm with the splitter angles of 0, 15, 30° separately. Maximum Fe_2O_3 removal efficiency (*FRE*) (97.1%) was obtained in the nonmagnetic product at -0.5+0.212 mm size fraction and 0° splitter angle. The minimum Fe_2O_3 content (1.3%) was reached in the nonmagnetic product obtained in the experiment with the feed size of -2+1 mm and a splitter angle of 0°. Secondly, dry magnetic separation was applied to the washed -2+0.212 mm size fraction after drying at room temperature to evaluate the coarse particle-sized halloysite ore that was gained by mechanical dispersion in the aqueous medium towards sodium hexametaphosphate (SHMP), while a significant part of the clay minerals went into fine size after the dispersion process. In the experiment performed with a 0° splitter angle after washing, it was determined that halloysite concentrate of 0.4% Fe_2O_3 content could be obtained with 98.8% Fe_2O_3 removal efficiency. As a result of dry magnetic separation experiments, it was seen that Fe_2O_3 removal efficiency decreased as the splitter angle increased, while Fe_2O_3 content in magnetic and nonmagnetic products increased. It was determined that washing and cleaning of fine-sized minerals plastered on particle surfaces after mechanical dispersion and particle release of minerals with different magnetic properties increased the dry magnetic separation efficiency, and nonmagnetic products with very low Fe_2O_3 (0.4%) and high Al_2O_3 (31.9%) content was obtained. The blunging process in the presence of dispersant caused the dispersion of clay minerals and allowed to liberating of the ferrous minerals from the halloysite ore, hence the increase in the *FRE* for the magnetic separation.

Keywords: halloysite, dry magnetic separation, particle size, splitter angle, washing

1. Introduction

Halloysite deposits are located in different regions around the world (Wilson, 2004; Ece et al., 2008; Keeling, 2015). Halloysite reserves can often be seen as adjacent to kaolinite deposits, and halloysite can also be a mineral vein in the altered rocks. It is a kind of clay mineral with a tubular structure in the kaolin group, and the clay minerals of the kaolin group are mostly produced from quarries with open-pit mining methods. Selective mining and separation may be necessary as quality variability can be seen in the quarry. In this case, it is important to classify and stock the kaolin produced from the quarry to meet the criteria related to the subsequent enrichment or usage area.

Halloysite is more important compared to the kaolinite mineral in terms of industrial use and economics. The tubular structure of the halloysite is the main feature of this importance. In some places of the world, there are pure halloysite ores. However, there are also mixed ores, which consist of

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kaolinite and halloysite minerals together. This kind of ores is used in industrial areas such as ceramic body composition, and the advantages of halloysite mineral cannot be utilized in pure mineral form. Apart from kaolinite minerals, impurities such as quartz, feldspar, titanium, and ferrous minerals can also be found in halloysite deposits (Bordeepong et al., 2012; Joussein, 2016). Although halloysite reserves contain impurities in their current form, they need to be enriched to be used more economically. To ensure the liberation of fine-sized minerals, particle size reduction processes must first be applied. However, clay minerals are already present in fine-sized agglomerates due to their natural structure (Joo et al., 2012). On the other hand, halloysite experiences a decrease in the product value upon breakage of the tubes, especially during the size reduction process in the form of wet or dry grinding (Takahashi, 1957; Takahashi, 1959). To eliminate the negative effects of the milling process on the structure of the halloysite mineral, it should be prepared by wet mixing (Durgut et al., 2022a). Fast and powerful mechanical mixers are used to disperse agglomerated clay minerals into finer particles. The ore preparation process of clay minerals begins with the loading of water and chemical dispersants into a mechanical mixer. In this process, although the solids ratio in the pulp is usually between 30-60%, it is also studied at lower ratios depending on the structure of the clay.

In general, most clay mineral deposits contain iron minerals, which are harmful impurities that greatly reduce the whiteness of products sintered at high temperatures (Sumner, 1963; Park et al., 1974). Ferrous (+2 valence) iron minerals can create color problems in the ceramic industry because they turn into ferric (+3 valence) iron during the sintering stage, giving the products an orange color (Marabini et al., 1993). The iron content of industrial minerals can be reduced by physical, physico-chemical, and chemical processes. In most industrial applications, it is necessary to use pure clay with standard properties. For example, kaolin used in the paper and paint industries should be of high gloss and low yellowness in color. On the other hand, some low-quality clay minerals can be used in refractory and tile production as well as quarried (Abdel-Khalek et al., 2017). In order to improve the quality of clay for industrial use, discoloring impurities should be removed from the sample by proper techniques. However, difficulties arise in the separation of impurities that are finer in size than clay minerals. Separation at this size is generally done by magnetic separation, flotation, selective flocculation, hydrocyclone, and leaching methods (Asmatulu, 2002). Most kaolin group clay minerals used in paper production have versatile functions as a filler between paper fibers and as a white-gloss coating material. However, raw kaolin moves away from its white color due to impurities containing ferrous mica, tourmaline, pyrite, anatase, and rutile. To remove these impurities and produce high white material for paper or porcelain production, high field intensity gradient type magnetic separators are preferred. High-field intensity gradient-type magnetic separators are used in 75% of the world's white porcelain and paper production (Ohara et al., 2001; Yavuz et al., 2009).

Clay particles are agglomerated in an aqueous environment due to the electrical attraction of positive (+) and negative (-) charges on their surfaces (Li et al., 2022). In an aqueous environment, the surface charge of clay minerals is negative (-) in a wide pH range and the surface charge goes to a more negative (-) value as the pH value increases (Aksoy and Kaya, 2016). Thus, the clay particles are dispersed by moving away from each other. The surface charges of clay minerals in aqueous media are even more negative by using dispersants such as sodium silicate, sodium hexametaphosphate, tetrasodium pyrophosphate, and sodium polyacrylate, which ionize in water and give cation to the environment, and dispersion is achieved with lower mechanical energy (Durgut et al., 2022b).

After the kaolin group clay minerals are dispersed as a suspension, impurities can be removed from the ore. Industrially, impurities within kaolin ore are eliminated in the form of passing the suspension through equipment called a sandbox. Thus, the impurity group consisting of quartz and ferrous minerals with high specific gravity and coarser particle size is placed at the bottom of the pool, and fine-sized clay minerals are obtained by overflowing from the pool. Since mica minerals do not collapse quickly due to their flat shape, they can overflow and be recovered with the kaolin group clay minerals. Therefore, by feeding the kaolin into the vibrating sieve, the mica minerals are separated on the sieve due to the shape difference in this step (Murray, 2006). Another stage of the wet process is to classify clay minerals into coarse and fine particle sizes. This process is carried out with continuous reservoir-type centrifuges, hydro separators, and/or hydrocyclones (Oats et al., 2010; Boylu et al., 2012). The particle size-dependent separation process is carried out in such a way that the kaolin group clay minerals are fine and coarse in size group. After the coarse-sized fraction is filtered, clays are obtained by drying, while the fine fraction is fed into a high-gradient magnetic separator to remove iron and titanium-bearing impurities (Cieśla, 2003; Sakiewicz et al., 2016). Additionally, the separation of halloysite and kaolinite can be made in fine size after good dispersion. It has been suggested in the literature that the sedimentation behaviour of the particles, besides the effect of sucrose concentration is important in the separation of nano-sized clays in aqueous media and can be used in the purification of halloysite under appropriate conditions (Durgut et al., 2022b; Calvino et al., 2022).

In this study, the removal performance of ferrous minerals in halloysite ore was examined by applying dry magnetic separation tests to reduce the Fe_2O_3 content and increase Fe_2O_3 separation efficiency.

2. Materials and methods

2.1. Materials

In the experimental studies, halloysite ore was obtained from the mineral deposit in Kızıldam village of the Yenice district of Çanakkale province, Türkiye. The ore was initially crushed using a laboratory-type jaw crusher and reduced to -2 mm particle size. Then, the chemical analysis of the ore was carried out with an Axios Max model X-ray Fluorescence Spectrometer (XRF, MALVERN PANALYTICAL, The Netherlands), and the major element analysis is presented in Table 1.

Table 1. Chemical analysis of the halloysite ore

Compound	SiO_2	Al_2O_3	TiO_2	Fe_2O_3	CaO	MgO	Na_2O	K_2O	*LOI	TOTAL
(%)	50.9	29.7	0.7	3.9	0.2	0.4	0.2	1.1	12.0	99.1

*LOI: loss on ignition

Additionally, a MALVERN PANALYTICAL X'PERT PRO MPD X-ray diffractometer (XRD) was used in the mineralogical analysis of the obtained products. Mineralogical analyses were carried out with an angular range of 3-70° 2θ, step size 0.02°, divergence slit 1/4, anti-scatter slit 1/2, and qualitative and quantitative mineral analyses were performed from the obtained graphs. Hanawalt method (Jenkins and Snyder, 1998) was used in qualitative analyses and the Rietveld method (Young, 1995) was used in quantitative XRD analysis. The mineralogical analysis of the sample shown in Fig.1 indicated that the sample mainly contained kaolinite (43.4%) and halloysite (30.5%), and gangue minerals such as quartz (19.1%) and goethite (2.9%).

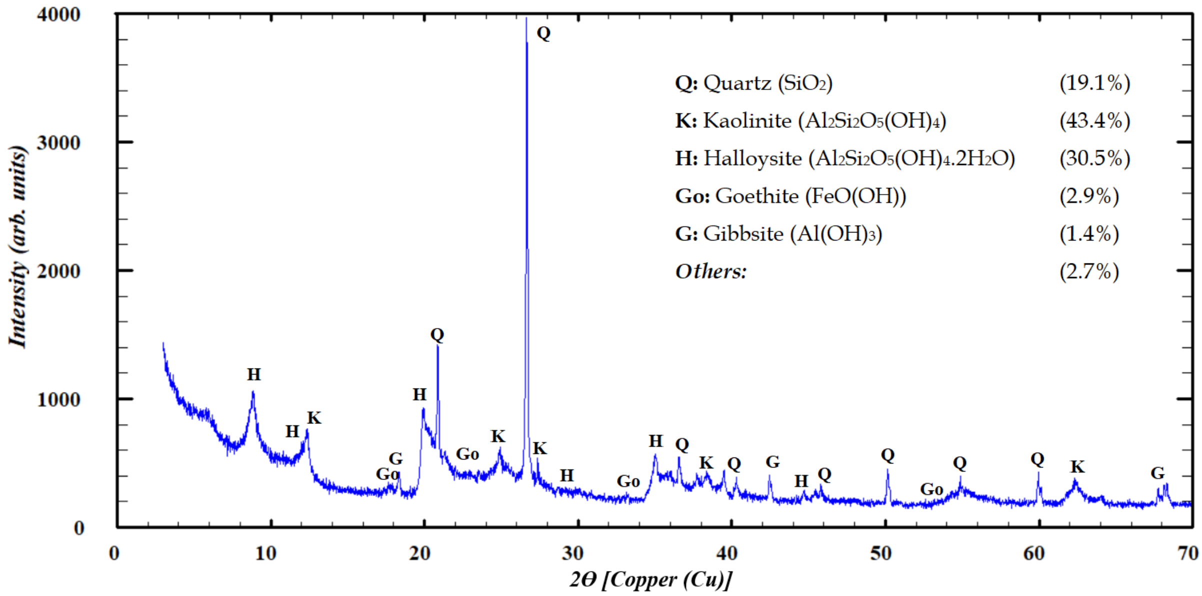

Fig. 1. The XRD result of the halloysite ore

Fourier-transform infrared spectroscopy (FTIR) spectrum of the halloysite ore was analysed with a BRUKER ALPHA ATR model spectrometer (Fig. 2). The Al-OH stretching vibration at 3600 cm^{-1} corresponded to halloysite, kaolinite, and gibbsite content in the sample. The low peaks of the hydroxyl stretching band with layer water at 3696 cm^{-1} and 1623 cm^{-1} wavelengths showed a 10 Å type of halloysite in the sample. Quartz mineral content was found with asymmetric and symmetrical Si-O stretching vibrations at 1083 cm^{-1} and 775 cm^{-1}, respectively. Also, the asymmetric Si-O bending vibrations at 450 cm^{-1} showed the quartz content in the formation (Durgut et al., 2022b). The OH stretching bands of goethite at 3416 cm^{-1} and 3133 cm^{-1} cannot be observed in the spectrum, however, OH bending bands at 892 cm^{-1} (d-OH) and 795 cm^{-1} would be the signs of goethite mineral.

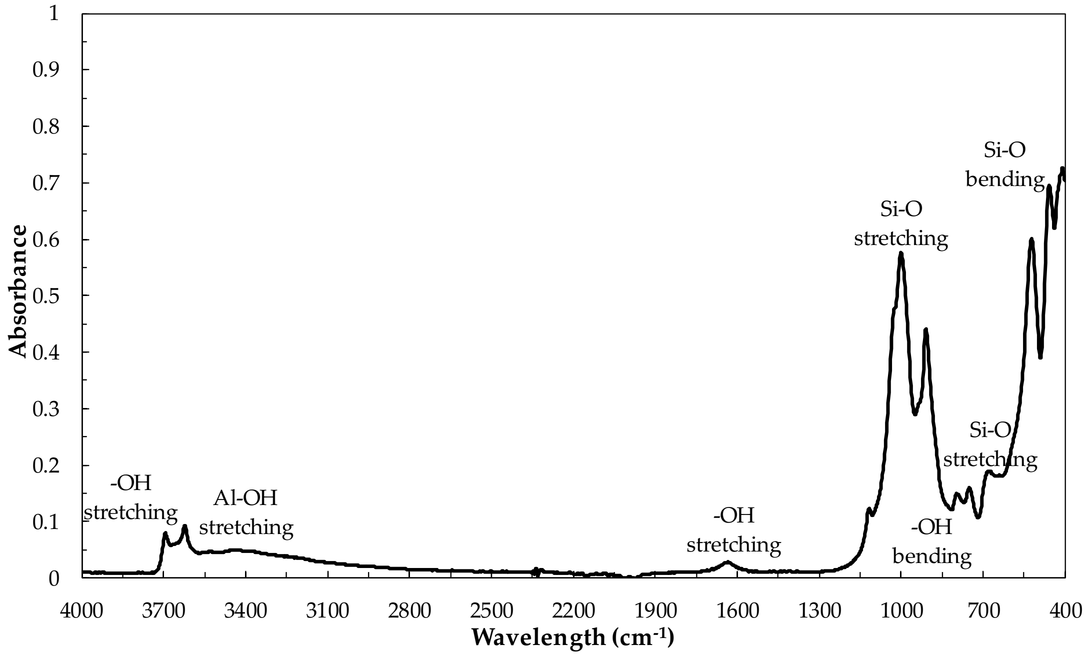

Fig. 2. The FTIR spectrum of the halloysite ore

Extra pure sodium hexametaphosphate (SHMP) with a total phosphate content of 65-70% obtained from Kimya, Türkiye was used as a dispersant to prepare halloysite ore in aqueous media by a mechanical mixing process before dry magnetic separation experiments.

2.2. Methods

2.2.1. Magnetic separation experiments with crushed sample

To remove magnetic minerals in halloysite ore, the dry magnetic separation tests were carried out with a roll-type rare-earth permanent magnet (REMS), belt type, high gradient magnetic separator, with a roll and belt of 13000 Gauss with a fixed magnetic field strength obtained from Manyet, Türkiye (Fig. 3a).

The feed size for the separation should be from 13 mm to 0.075 mm. A detailed description of separation using magnetic rolls can be found in Svoboda (1987). In this separation, as seen in Fig. 3b, the sample is fed into a vibratory feeder which distributes the particles uniformly (in a monolayer) on the belt. Then, the sample moves onto the magnetic roller. When a magnetic or nonmagnetic particle approaches a magnetic field, if the sum of gravitational force and magnetic forces is less than centrifugal force, the particle can be released from the magnetic roller. This indicates the diamagnetic material of low magnetic susceptibility. On the other hand, when the sum of magnetic force and gravitational force is greater than the centrifugal force, the particle will remain in contact with the magnetic roller and will be released upon leaving the magnetic field. This is the case for paramagnetic/ferromagnetic materials of high magnetic susceptibility. Each of these forces can be adjusted by varying the variables, magnetic roller speed, size fraction, feed rate, and angle, to optimize the efficiency.

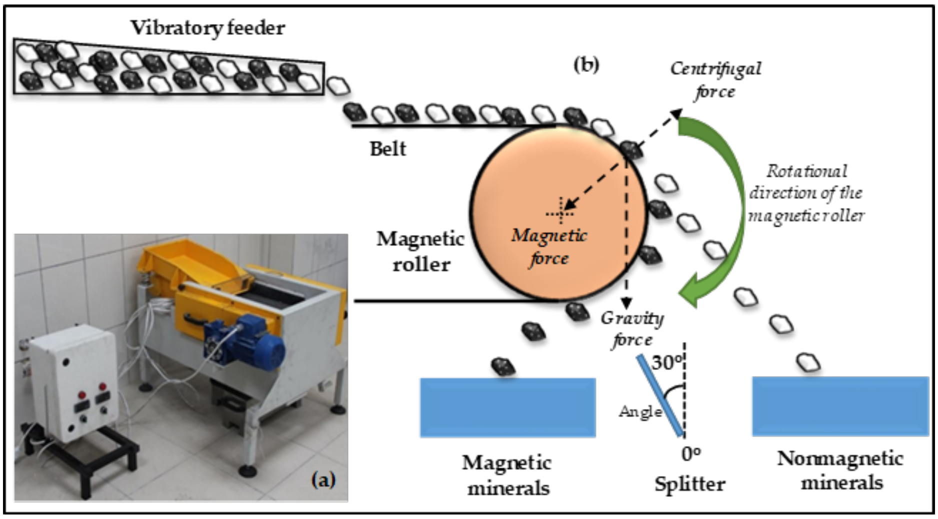

Fig. 3. (a) The magnetic separator used in this study (b) Principle of separation

Effective magnetic separation can be achieved by optimizing parameters such as magnetic roll speed, size fraction, feed rate, splitter angle, etc (Çınar and Durgut, 2019). Each variable can be adjusted to improve the separation efficiencies. For example, the efficiency of magnetic separation depends on particle size (Ozdemir et al., 2011). The gravitational and magnetic forces become dominant for particles larger than about 0.300 mm, hence paramagnetic and locked diamagnetic impurities report to magnetic products and thus are removed from the halloysite ore. As also known from the literature, especially grouping the ore into a narrow particle size fraction increases the Fe_2O_3 separation efficiency (Ibrahim et al., 2002). For this reason, the halloysite ore was crushed to -2 mm particle size total, and the crushed sample was sieved from 0.212 mm size to obtain the -2+0.212 mm size fraction for the magnetic separation experiments. The quantities of the material according to particle sizes after crushing were measured by weight: 28.9% of -2+1 mm, 22.6% of -1+0.5 mm, 15.2% of -0.5+0.212 mm, and 33.3% of -0.212 mm. The -2+0.212 mm size fraction was classified into -2+1, -1+0.5, and -0.5+0.212 mm size fractions due to the inability to treat relatively broad size ranges of particles.

Also, separation efficiency can be affected significantly by changing the angle of the splitter. Depending on the position of the splitter, the separation of magnetic and nonmagnetic minerals from each other is possible. Therefore, the effect of the splitter angle on the separation efficiency was investigated at different angles such as 0, 15, and 30°.

With this purpose, the magnetic separation experiments were carried out at a constant belt speed of 300 rpm, a feed mass of 800 g, 53.3, 44.4, and 33.3 g/sec feed rates, and 0, 15, and 30° splitter angles for particle size fractions of -2+1, -1+0.5, and -0.5+0.212 mm. The adjustment of the feed rate is very important because the optimum feed rate depends on roll speed. When the feed rate increases, the separation efficiency decreases because of the magnetic force decrement on the particles (İbrahim et al., 2002; Zong et al., 2018). Therefore, in this study, the feed rate for each size fraction was kept very low during the experiments to produce a high-purity sample. Roll speed can result in the loss of magnetic particles from the belt. Finally, after the dry magnetic separation process, two different products were collected, a magnetic and a nonmagnetic product. The experimental parameters for the dry magnetic separation are also presented in Table 2.

Fig. 4 shows the flowsheet of the magnetic separation process for the -2+0.212 mm size of the crushed halloysite ore.

Table 2. The experimental parameters for the dry magnetic separation

Feed Size (mm)	-2+1, -1+0.5, -0.5+0.212
Blade Angle (°)	0, 15, 30
Belt Speed (rpm)	300
Feed Mass (g)	800
Feed Rate (g/sec)	53.3, 44.4, and 33.3

Finally, the iron removal efficiencies in the magnetic separation experiments were calculated according to Eq. 1 based on the decrease in the Fe_2O_3 content in the nonmagnetic product according to the material feed.

$$\%\mathrm{FRE} = 100 - \left(\frac{\mathrm{NPQ} * \mathrm{NPQ}_{\mathrm{Fe2O3}}}{\mathrm{FCF}_{\mathrm{Fe2O3}}}\right) \tag{1}$$

where FRE: %Fe_2O_3 removal efficiency, NPQ: Nonmagnetic product quantity (%), NPQ_{Fe2O3}: %Fe_2O_3 content in the nonmagnetic product after magnetic separation, FCF_{Fe2O3}: %Fe_2O_3 content of feed.

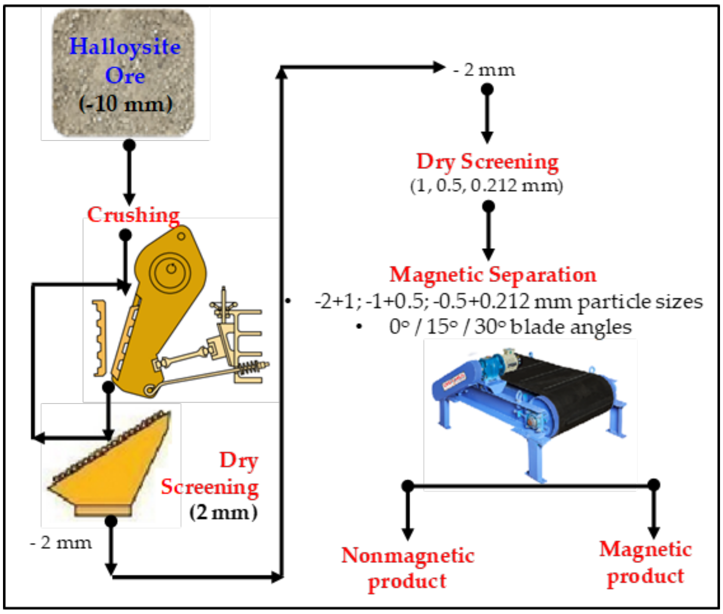

Fig. 4. Flowsheet for magnetic separation experiments of the crushed halloysite ore

2.2.2. Magnetic separation experiments with blunged sample

Even though crushing is the best method of reducing the size of the particles, especially for hard and non-plastic ores, it is not recommended for clay minerals because crushed gangue minerals deteriorate the effective separation. Therefore, clay minerals can be dispersed using mechanical mixers in an aqueous environment, and it will be easy to separate from associated gangue minerals through size classification methods due to their plastic behavior and natural fine sizes (Murray, 2006). Also, the tubular form of halloysite mineral is affected negatively during dry and wet grinding, causing a devaluation of the product (Takahashi, 1957; Takahashi, 1959).

Our previous study indicated that the use of dispersants enhanced the mechanical dispersion effect for plastic clay mineral separation from hard minerals in an aqueous medium (Durgut et al., 2022b). Moreover, the dispersant type showed the greatest effect on the mechanical dispersion conditions, and SHMP was determined as the optimum dispersant along with 7.5 kg/Mg dosage in terms of the contents of Al_2O_3, and SiO_2 values of the samples.

For this reason, the sample was blunged in the presence of 7.5 kg/Mg of SHMP using a laboratory scale mixer (blunger) at the obtained optimum conditions (35% of solids ratio, 500 rpm blunging speed, 4 h of blunging time, pH of 7.4, and 25.0°C) from the previous study (Durgut et al., 2022b). In this manner, the plastic clay minerals were liberated from non-plastic hard minerals without changing their sizes to reduce the particle size of the halloysite rather than the crushing process. Then, the blunged suspension was wet screened using 2 mm and 0.212 mm sieves to obtain the -2+0.212 mm fraction size as before. Then, the sieved sample was dried at 60°C for 24 h in an oven, and the dried sample was subjected to a magnetic separation process at a 300 rpm belt speed and splitter angles of 0, 15, and 30°. Finally, the iron removal efficiencies (FRE) in the magnetic separation experiments were calculated using Eq. 1. The quantities of the material according to particle sizes after blunging were measured as by weight 17.5% of +0.212 mm and 82.5% of -0.212 mm. The experimental parameters for the dry magnetic separation with the blunged sample are also presented in Table 3. Fig. 5 shows the flowsheet of the magnetic separation process for the -2+0.212 mm size of halloysite ore.

Table 3. The experimental parameters for the dry magnetic separation with the blunged sample

Feed Size (mm)	-2+0.212
Blade Angle (°)	0, 15, 30
Belt Speed (rpm)	300
Feed Mass (g)	800
Feed Rate (g/sec)	44.4

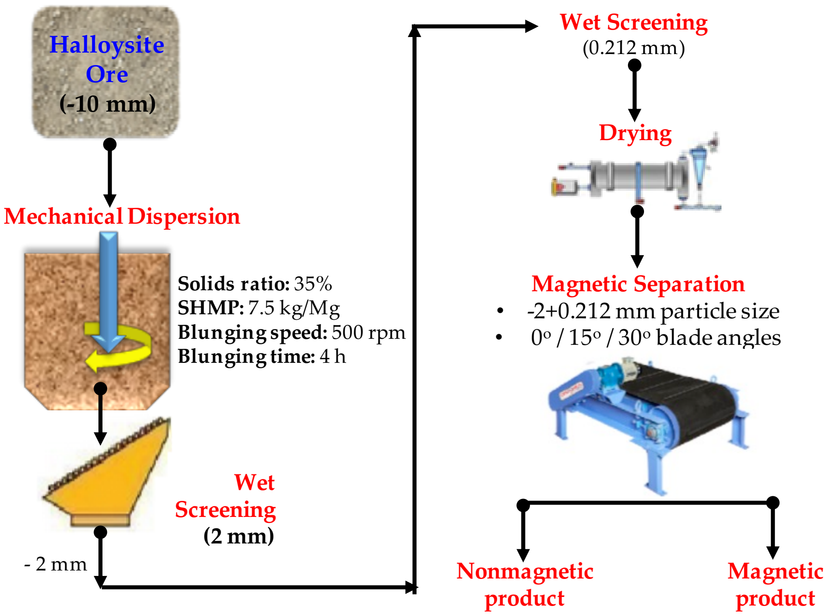

Fig. 5. Flowsheet for magnetic separation experiments of the blunged halloysite ore

3. Results and discussion

3.1. Magnetic separation experiments with crushed sample

The magnetic separation of the crushed halloysite ore was conducted for three different size fractions, -2+1, -1+0.5, and -0.5+0.212 mm, while the splitter angle ranged from 0° to 30°. The results of the magnetic separation including the contents of Fe_2O_3 and Al_2O_3 are presented in Table 4. Fe_2O_3 removal efficiencies (FRE) were also calculated by applying chemical analysis to magnetic and nonmagnetic products after magnetic separation experiments by comparing Fe_2O_3 contents in the feed material. Fig. 6 shows FRE values for magnetic and nonmagnetic products obtained as a result of the magnetic separation experiments. As shown in Fig. 6, the magnetic separation significantly improved the grade of halloysite ore for each size fraction. Al_2O_3 content is an indicator of kaolin-type clay mineral concentration. The increase of Al_2O_3 content in the nonmagnetic products at each size fraction except -0.5+0.212 mm at 0°C splitter angle indicated clay mineral increment. The specific gravity of iron-bearing minerals is higher than that of clay minerals (Roy et al., 2008; Ramakgala and Danha, 2019). The specific gravity of the particles and the magnetic force are determinant factors of the particle direction out of the magnetic separator. In the finer size, these factors also played a role in the Al_2O_3 and Fe_2O_3 contents of products.

FRE value was achieved as maximum (97.1%) in the -0.5+0.212 mm size group at 0° splitter angle, while the minimum (48.0%) value was reached in the -0.5+0.212 mm size group at 30° splitter angle. The nonmagnetic product with the lowest Fe_2O_3 content (1.3%) was obtained in the test performed in the -2+1 mm size group at 0° splitter angle, while the magnetic product with the highest Fe_2O_3 content (28.8%) was obtained in the dry magnetic separation test performed in the -2+1 mm size group at 30° splitter angle.

Table 4. Results of the magnetic separation experiments for three different size fractions of the crushed halloysite ore as a function of splitter angle

		-2+1 mm			-1+0.5 mm			-0.5+0.212 mm		
Splitter Angle	% Value	F	M	NM	F	M	NM	F	M	NM
0°	Weight	100	64.5	35.5	100	78.4	21.6	100	94.2	5.8
	Fe_2O_3	5.56	7.88	1.34	3.95	4.58	1.67	3.47	3.58	1.75
	FRE	100	91.4	8.6	100	90.9	9.1	100	97.2	2.9
	Al_2O_3	28.9	26.9	32.5	29.8	29.5	31.0	30.2	30.2	29.9
15°	Weight	100	26.7	73.3	100	35.5	64.5	100	54.9	45.1
	Fe_2O_3	5.56	15.59	1.87	3.95	7.32	2.10	3.47	4.74	1.93
	FRE	100	74.9	24.7	100	65.8	34.3	100	75.0	25.1
	Al_2O_3	28.9	22.0	31.4	29.8	28.5	30.5	30.2	29.9	30.5
30°	Weight	100	12.1	87.9	100	14.4	85.6	100	18.4	81.6
	Fe_2O_3	5.56	28.75	2.35	3.95	14.3	2.19	3.47	9.08	2.21
	FRE	100	62.6	37.2	100	52.1	47.5	100	48.1	52.0
	Al_2O_3	28.9	11.8	31.3	29.8	26.4	30.3	30.2	30.0	30.2

FRE: Fe_2O_3 removal efficiencies, *M:* Magnetic, *NM*: Nonmagnetic, *F*: Feed

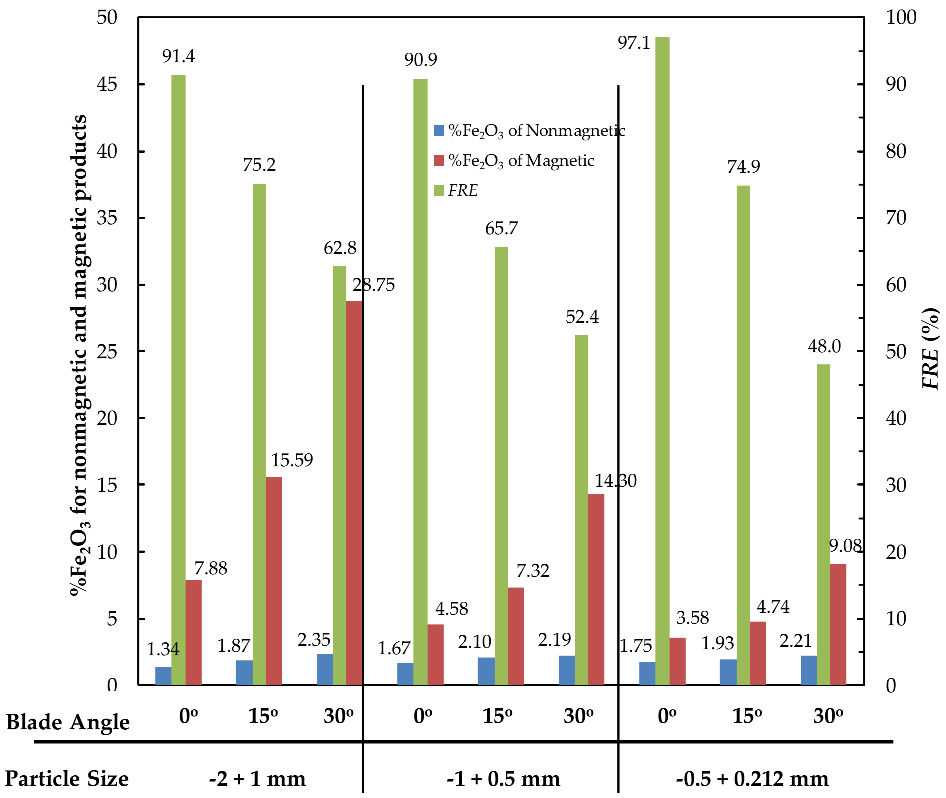

Fig. 6. Results of dry magnetic separation applied to the crushed halloysite ore

In REMS-type roll separators, minerals with low magnetic sensitivity are less affected by magnetic field strength. Thus, coarser minerals with low magnetic sensitivity and finer nonmagnetic minerals are thrown in the same direction and left on the magnetic roller (Gehauf, 2004). In dry magnetic separation experiments, it was observed that goethite minerals with low magnetic sensitivity were mixed into the nonmagnetic product as the splitter angle increased. Thus, the Fe_2O_3 content in the nonmagnetic product increased and the Fe_2O_3 removal efficiency decreased. In dry magnetic separation, when the splitter angle is adjusted to separate coarser minerals with low magnetic sensitivity, they will move together with nonmagnetic finer minerals. If the splitter angle is adjusted to separate finer nonmagnetic minerals, coarser minerals with low magnetic sensitivity will also be mixed into the product (Zong et al., 2018). As a result of the dry magnetic separation tests carried out after the dry screening, the nonmagnetic product with minimum Fe_2O_3 content (1.3%) was reached at -2+1 mm feed size at 0° splitter angle, while the magnetic product with maximum Fe_2O_3 content (28.8%) was reached at -2+1 mm feed size at 30° splitter angle. From here, the effect of the particle size is obvious.

Additionally, X-ray diffraction analysis was performed for the magnetic product obtained from the magnetic separation experiments performed at a splitter angle of 30° with a particle size of -2+1 mm to determine the mineral types reporting into the magnetic product. As seen in Fig. 7, the mineralogical analysis of the magnetic product shows that it consists of 26.0% goethite (FeO(OH)), 5.0% hematite (Fe_2O_3), 35.8% quartz, 16.8% kaolinite, 13.1% halloysite, and 3.3% other minerals. When evaluated together with the chemical analysis of the sample, it is understood that the 28.8% Fe_2O_3 content comes from the goethite and hematite minerals.

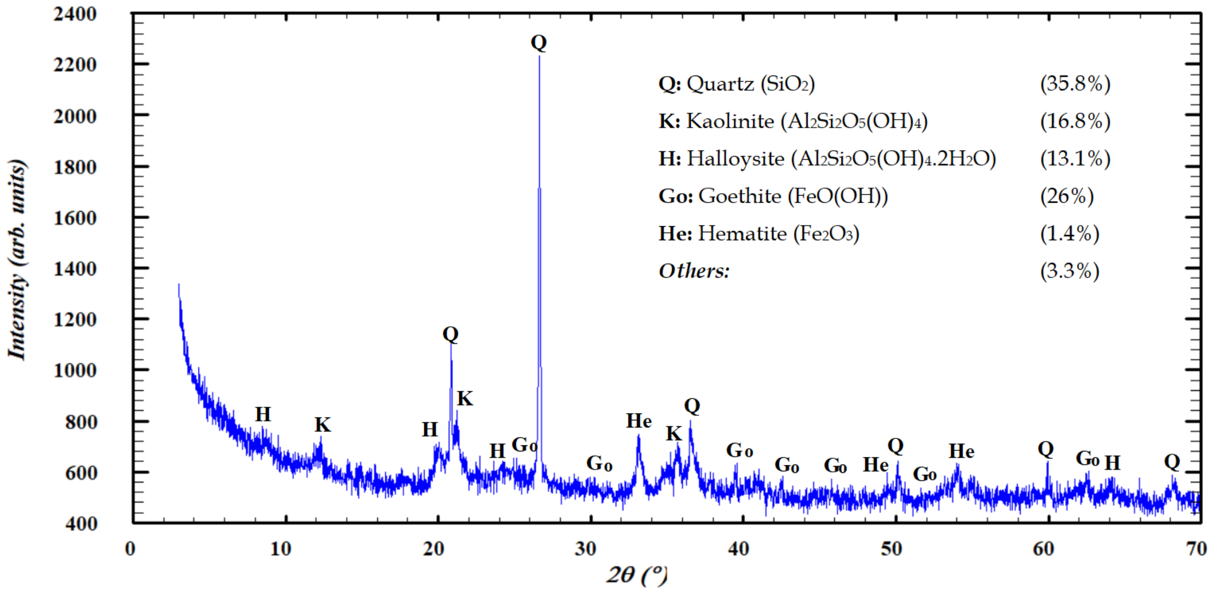

Fig. 7. Quantitative XRD analysis of the magnetic product from the magnetic separation experiments performed at a splitter angle of 30° with a particle size of -2+1 mm

3.2. Magnetic separation experiments with the blunged sample

In this part of the study, the halloysite ore was first blunged in the presence of SHMP, then the blunged sample, which is a material that is mechanically dispersed and washed in the aquatic environment, was dried in an oven overnight, and finally, the dried sample was subjected to a dry magnetic separation at 300 rpm of roller speed and splitter angles of 0, 15, 30°. The results for the magnetic separation for the blunged halloysite ore including the contents of Fe_2O_3 and Al_2O_3 are presented in Table 5. Fig. 8 shows FRE values for magnetic and nonmagnetic products obtained as a result of the magnetic separation experiments.

In this study, the experimental procedure was started with dry magnetic separation. This study aimed to remove iron minerals or decrease the iron content of the halloysite ore using a magnetic separator. For this purpose, the dry sieving was performed and obtained 3 different size fractions, namely, -2+1, -1+0.5, and -0.5+0.212 mm, and used for the dry magnetic separation experiments. The results indicated that the dry magnetic separation was not successful in terms of the iron content of the nonmagnetic samples. The reason for this can be attributed to the particles' surfaces coated with clay minerals which cause a limitation in the magnetic properties of the minerals hence the separation recovery was low. As also indicated in the literature (Tripathy et al., 2015), the distribution and abundance of the gangue- bearing slime coated on coarse-size particles as well as the complex mineralogical association of the gangue minerals caused differences in findings from bulk separation

Table 5. Magnetic separation experiment results for the blunged halloysite ore as a function of splitter angle

Splitter Angle		-2+0.212 mm		
		Feed	**Magnetic**	**Nonmagnetic**
0°	Weight (%)	100	71.5	28.5
	Fe_2O_3 (%)	9.60	13.25	0.44
	FRE (%)	100.0	98.7	1.3
	Al_2O_3 (%)	26.4	24.20	31.90
15°	Weight (%)	100	35.4	65.6
	Fe_2O_3 (%)	9.60	20.16	3.75
	FRE (%)	100	74.3	25.6
	Al_2O_3 (%)	26.4	21.20	29.60
30°	Weight (%)	100	19.9	80.1
	Fe_2O_3 (%)	9.60	28.36	4.94
	FRE (%)	100	58.8	41.2
	Al_2O_3 (%)	26.4	17.90	28.50

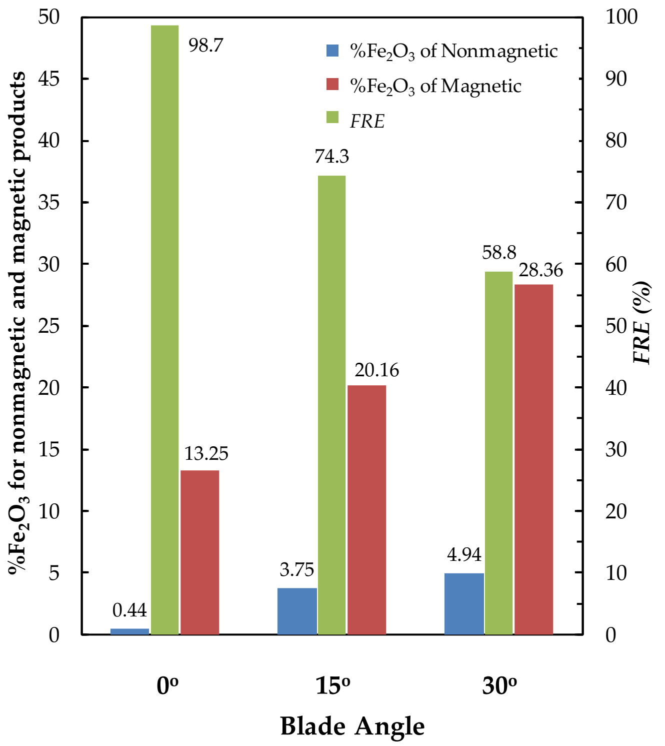

Fig. 8. Results of dry magnetic separation applied to the blunged halloysite ore

and split beneficiation. Therefore, the blunging process was used to disperse minerals so that the particle surfaces would be clean. Then, the sample was dried and sieved to obtain the -2+0.212 mm size fraction. It was clear that the wet magnetic separation could be used after the wet preparation process especially to manufacture a qualified final product, however, the industrial materials were also sold in dry form in the industry and it was an alternative way to enrich the halloysite mineral and opportunity to compare the blunged material after wet and dry preparation methods. It was seen that the wet-prepared material was gained with maximum iron removal efficiency (98.8%) after the drying process.

In the dry magnetic separation experiments performed at 0, 15, and 30° splitter angles, it was seen that as the splitter angle decreased, the Fe_2O_3 content in the nonmagnetic product decreased and a product richer in kaolin group clay minerals was obtained. With the increase in the splitter angle, a magnetic product with low Al_2O_3 content and high Fe_2O_3 content was obtained. In dry magnetic separation experiments, the lowest Fe_2O_3 content (0.4%) was reached in the nonmagnetic product taken at 0° splitter angle. In addition, according to the product fed, the content of Al_2O_3 increased from 26.4% to 31.9%. The highest Fe_2O_3 content (28.3%) was reached in the magnetic product at a splitter angle of 30°. The washing process before the dry magnetic separation cleaned the mineral surfaces, especially the ferrous goethite and hematite minerals, which were more affected by the magnetic field (Ruan et al., 2019). Thus, it was determined that the efficiency of separation increased. Moreover, as the splitter angle decreased, it was seen that a cleaner nonmagnetic product would be obtained in terms of Fe_2O_3 (Fig. 9).

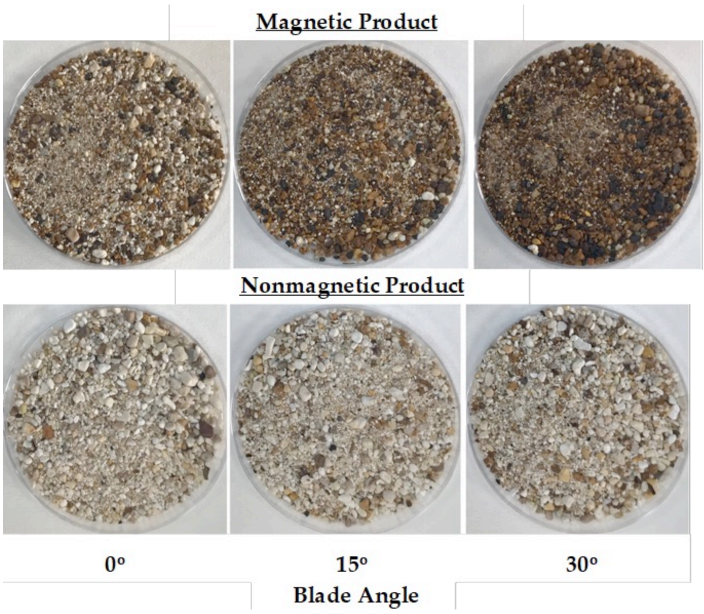

Fig. 9. Images of the products obtained after dry magnetic separation applied to the -2+0.212 mm size group after mechanical dispersion

The Fe_2O_3 contents of dry magnetic feed materials according to the particle sizes were given as 5.56% for -2+1 mm, 3.95% for -1+0.5 mm, and 3.47% for -0.5+0.212 mm after crushing. On the other hand, -2+0.212 mm sized material after the blunging process involved 9.6% Fe_2O_3 as feed material for dry magnetic separation. Firstly, the dispersion process resulted in washing clayey material on the surface of the coarse-sized material into fine size. Secondly, after the blunging process the washed surfaces of coarse-sized material which combined hard, iron-bearing minerals mostly, could be separated efficiently with dry magnetic separation. After the -2+0.212 mm size fraction obtained by the dry sieving of halloysite ore and the wet screening after mechanical dispersing under optimum conditions, the dried material was subjected to dry magnetic separation to see the change in the Fe_2O_3 removal efficiency of the washing process. The experiments were carried out at a drum speed of 300 rpm and splitter angles of 0, 15, and 30°. Fe_2O_3 removal efficiencies were determined as 92.2%, 72.2%, and 57.1% for the dry processing and 98.8%, 74.4%, and 58.8% for the wet processing at splitter angles of 0, 15, and 30°, respectively (Fig. 10). After mechanical dispersion and washing processes iron-bearing minerals covered by the clayey particles was removed in the aqueous environment, therefore Fe_2O_3 removal efficiency in the nonmagnetic product increased by dry magnetic separation.

In these experimental studies, halloysite ore was mineralogically mostly composed of halloysite, kaolinite, quartz, and a small amount of other impurities. Our previous studies showed that the zeta potential values of kaolinite and quartz were negative in all measurements and were increasingly negative from pH 3 to 11 (Durgut et al., 2022b). The results indicated that the dispersant use enhanced the mechanical dispersion effect for plastic clay mineral separation from hard minerals in an aqueous medium. The reason for this can be attributed to the mineral surface charges becoming more negative as the concentration increased in the zeta potential measurements.

In addition, the increase in zeta potential caused a decrease in viscosity or shear forces in colloidal suspensions composed of homogeneously charged silica and clay particles (Kobayashi et al., 2005; Singh et al., 2005). For example, while the viscosity of halloysite ore prepared with distilled water was measured as 217.5 mPa.s, the viscosity value of the halloysite ore in the presence of SHMP was measured as 3.3 mPa.s for 10 kg/Mg of dispersant dosage (Durgut et al., 2022b).

Overall, it can be concluded that the success of mechanical energy in the dispersion of clay minerals mechanically dispersed in the presence of dispersant was due to the increase in the electrostatic repulsion forces caused by the dispersants between the particles.

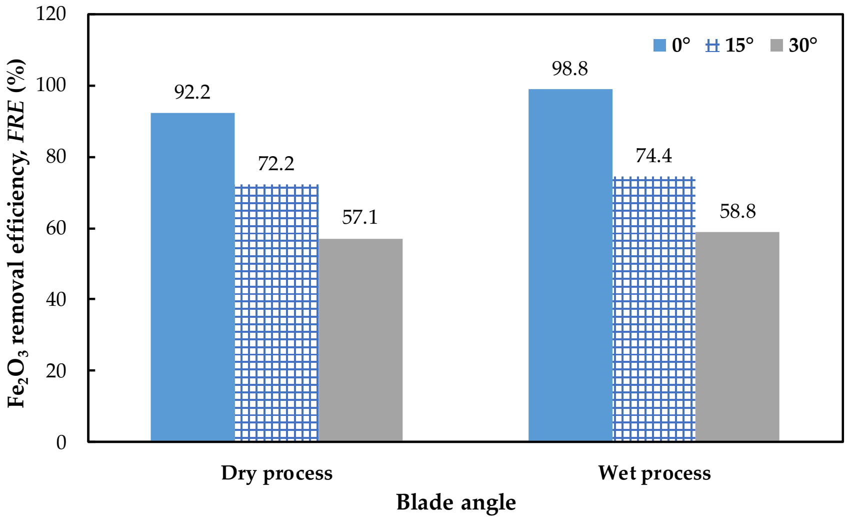

Fig. 10. Fe_2O_3 removal efficiencies in nonmagnetic products as a result of the application of dry magnetic separation to materials brought to -2+0.212 mm size by dry and wet screening

4. Conclusions

The importance of clay ores increases by removing their impurities. Nowadays, clay minerals have no alternative in ceramic body compositions due to their properties such as providing dry strength before firing and crystal phase formation after firing. In addition, as the iron content of a clay ore to be used in the ceramic industry decreases, the whiteness color value after firing will increase and the colors of the decoration applied on the tile will be more vivid. This study aimed to investigate the separation of ferrous impurities from halloysite ore by dry magnetic separation with the crushed and blunged halloysite ore in order to increase the purity of halloysite mineral. With this purpose, the effects of several parameters, namely feed particle size, splitter angle, and washing process on the Fe_2O_3 removal efficiency (FRE) were studied in detail. The results indicated that the Fe_2O_3 removal efficiency in the nonmagnetic product increased in the dry magnetic separation process by mechanical dispersion and washing the clayey particles covering the minerals with Fe_2O_3 content in the aqueous environment. As a result of dry magnetic separation experiments, it was seen that Fe_2O_3 removal efficiency decreased as the splitter angle increased, while Fe_2O_3 content in magnetic and nonmagnetic products increased. It was determined that washing and cleaning of fine-sized minerals plastered on particle surfaces after mechanical dispersion and particle release of minerals with different magnetic properties increased the dry magnetic separation efficiency, and nonmagnetic products with very low Fe_2O_3 (0.4%) and high Al_2O_3 (31.9%) content was obtained.

Also, the results for the magnetic separation for the -2+0.212 mm size group whose surfaces were cleaned after the blunging process showed that the magnetic separation was more successful than the dry-sized material although the size range was very large because the coating of fine-sized clay or iron minerals on the coarse material surface of the material that is crushed and classified as dry causes a large amount of inefficient separation.

As a result of the blunging process with the help of dispersant SHMP, the net surface charge of the minerals in the suspensions became more negative, the viscosity of the suspension decreased and the amount of material liberated more. Hence, the removal efficiency of ferrous minerals from the halloysite ore increased. It is thought that it would be appropriate to use high-speed dispersion followed by wet magnetic separation and leaching processes in order to recover the remaining Al_2O_3 content in the magnetic product. When kaolinite and halloysite minerals are compared in industrial terms, it is seen that halloysite is much more important in technological and economic terms. Since clay minerals represent very fine-sized materials, it is planned to carry out studies on the separation of halloysite and kaolinite minerals in fine-sized medium in the coming period.

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