

Wet-high intensity magnetic separation of magnesite ore waste

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Abstract

In this paper, investigation was carried out on a wet high intensity magnetic separation of the waste of magnesite ore where is stocked in open pit magnesite mine. The as received sample was subjected to physical, chemical, thermal and phase characterizations. The waste of magnesite ore sample containing 77.69% MgCO_3 and considerable amount of 3.14% Fe_2O_3 . The unwanted silica iron impurities were removed and the high grade magnesite was obtained through experiments. The results showed that the high grade of magnesite was obtained at 2T after the twice passing of the non magnetic portion of the processed sample. It is possible to upgrade magnesite to 91.03% and reduce iron content to 0.32% after the magnetic separation. After the calcination process at 1000 °C, the sample showed 52% of loss of ignition and yielded 85.39% MgO with a 0.32% Fe_2O_3 . The final product can be used in chemical and metallurgical applications where high magnesia content requires. The experiment results provide useful information on the wet magnetic separation of magnesite waste.

Key words: Magnesite waste, beneficiation, magnetic separation, calcination, magnesia,

1. Introduction

Magnesite is the major mineral of magnesium, the other minerals are talc, brucite and dolomite[1]. Other impurity minerals in magnesite ore are quartz, apatite and small amount of iron. It is formed through the carbonation of magnesian serpentine in the presence of water and carbon dioxide under moderate temperature and pressure. Chemical composition of magnesite ore is complex and varies country to country. Depending on magnesite ore, it contains MgO (42-47%), CaO (1.0-2.0%), SiO_2 (1%), Al_2O_3 (1%), Fe_2O_3 (1%) and 50% ignition loose. According to magnesia content of magnesite ore can be classified into three groups are high ($\text{MgO} > 46\%$), middle and low ($\text{MgO} < 43\%$) grades[2].

Due to physicochemical properties, magnesite is used in wide range of industrial applications including ceramic, metallurgy, cement, fertilizer, animal feed or refractory. More than 90% is used for sinter and caustic magnesia production as a fire resistance and slag materials in metallurgical application of steels, chemical, glass industries[3].

Magnesite ore occurs from ultrabasic and ultramafic peridotites in cryptocrystalline type ore. Turkey is one of the largest magnesite ore producer and refractory exporter country in the

world (7% of the total refractory production). The country has large magnesite ore deposits (approximately 2% of the world total) and are number of operators and exporter of magnesite ore. Approximately, 2 million tons of magnesite ore is mined in the country using for magnesia compounds such as dead burned, fused or chemical applications. Mining of magnesite ore in the country is carried out by open mining method in which the ore was beneficiated by hand/optical sorting, crushing screening, scrubbing and magnetic separation are used upgrading[4]. High calcium and silica content is restricted purification of magnesite. Therefore, it is very important to reduce unwanted oxides in it. After beneficiation processes, magnesite is subjected to calcination operation. According to calcination temperature, magnesia is named such as reactive magnesia (700-1000 °C), dead/hard burned magnesia (1000-2200 °C) and fused magnesia (above 2750 °C).

Magnetic separation is based on a direct physical separation method of minerals in magnetic field. Magnetic properties of minerals can be divided into three basic groups, ferromagnetic, para magnetic and diamagnetic. Magnetic susceptibility differences in particles sizes is accomplished wet or dry, at various intensities and in different basic machine configurations. Modern industrial application can be found different types. Choosing of magnetic separation method depends on many processing parameters including ore or mineral type and compositions and grades, particle size as well as their magnetic susceptibility. Addition of these, production and marketing factors are considered.

Magnetic separation has been used to upgrade and beneficiate variety of industrial minerals. First application on industrial minerals of magnetic separation process is to remove of iron oxide content such as hematite-limonite[5], taconite[6], pyrite (FeS_2), pyrohiotite (Fe_7S_8)[7]. It is dominant separation process in iron containing ores. There were well explained the principle of magnetic properties of materials[8,9] and applications in the literature [10]. Today, it is used from metallurgical, steels, scrap, food, medical, drug delivery or bio applications[11].

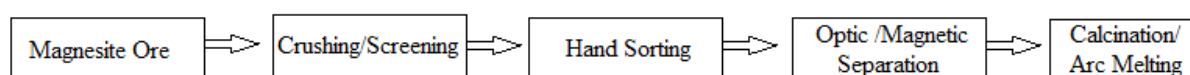


Figure 1. Flowsheet for magnesia processing.

A flowsheet for the processing of magnesite ore is given in Figure 1 that is generally accomplished by crushing/screening, hand sorting and dry magnetic separation. In addition those methods, froth flotation, leaching, agglomeration, coagulation or fluctuation methods can be used. Selection of beneficiation methods depends upon the mineralogical composition and the texture of the ore. To upgrade wanted content or to recovery of it, number of physical separation methods are applied such as frothflotation[12,13], electrostatic[14], gravity[15,16], or magnetic[17]. Each methods has advantages and disadvantages on applications, for example, flotation process treats very fine particles but needs some chemical agents and water which used media may creates environmental pollution and problems. Electrostatic route is environmentally friendly than flotation method from this point of view.

Extraction of magnesite from ore generates huge amount of soil waste. Due to availability of huge amount of magnesite ore tailing waste, it is important to utilize these waste as new magnesite potential source. Therefore the main objective of the present study is the characterization and the beneficiation of magnesite waste. High intensity wet magnetic separation was applied for the separation of magnetic and non magnetic fractions of the sample.

The present paper describes the characterization of magnesite tailing from Kütahya Magnesite Cooperation (KUMAS) Company and the application of the high intensity wet magnetic separation method was used to recovery of magnesite below -3.00+0.100 mm size fraction. Characterization was performed with thermal analysis, X-ray diffraction, scanning electron microscope, elemental analysis and infrared spectroscopy.

2. Experimental

2.1. Materials

The magnesite tailing was provided by the KUMAŞ Magnesite Inc. that was privatized in 2012, dominates in the magnesite mining industry[18]. ROM magnesite ore is beneficiated by hand sorting and dry magnetic separation at the mine sites for pre-concentration. Then it subjected to final beneficiation by high intensity dry magnetic separation. Depending on the impurity content of magnesite ore, the company has seven magnesite products, such as supper (Fe_2O_3 0.00-0.24%), extra (Fe_2O_3 0.25-0.29), special (Fe_2O_3 0.30-0.33), quality 1-4 (Fe_2O_3 0.34-0.72). It

produces 35 thousand tons of fused magnesite (FM), 275 thousand tons of dead burned magnesite (DBM) and 55 thousand tons of caustic calcinated magnesite (CCM). Magnesite tailing from the plant contains significant amount of magnesite as a low grade where is deposited in the plant to utilize the tailing in order to recovery of valuable oxide, to reduce storage and environmental problems. At the same time, tons of magnesite ore waste material has been stocked every year that damages the environment. A flowsheet was presented in Figure 2 for the experimental works.

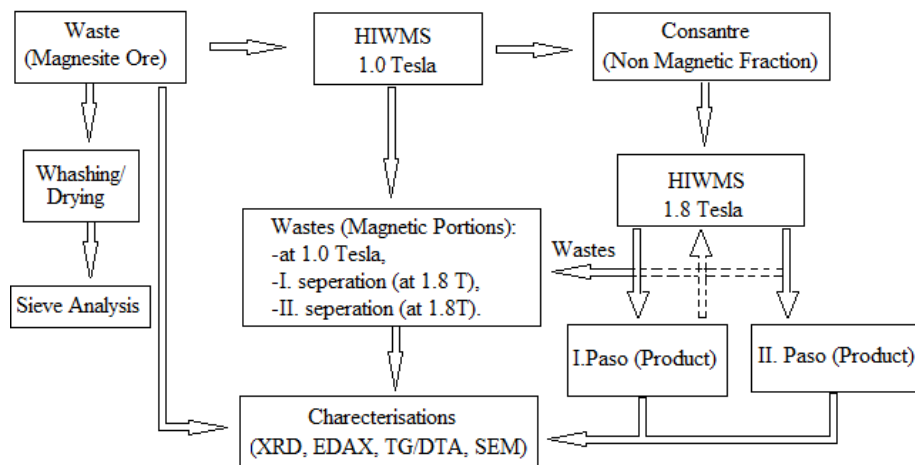


Figure 2. The flowsheet used for the characterization and the high intensity wet magnetic separation of the magnesite waste sample.

2.2. High intensity wet magnetic separator

When compare dry and wet magnetic separation techniques, conventionally, wet magnetic separation is used at finer size fractions, which is decreased impurity, high purity and better quality of products.

A high gradient electro magnet was used for the beneficiation of fine size $-3.00+0.10$ mm magnesite tailing. The magnetic separation experiments were carried out at the research laboratory of AKSA Magnet at Sakarya in Turkey. The separator (YMSM) consists of a separation canister packed with a strong ferromagnetic iron ball matrix. A magnetic field is trapped on the ball matrix which collect the magnetic particle and the non-magnetic portion is collected downstream. The intensity of the magnetic field developed between the coils was measured by means of Tesla.

Approximately, 50kg sample was made into slurry of desired initial pulp density with water (1:1 ratio). The slurry was stirred and passage through magnetic separator, the nonmagnetic slurry portion was collected downstream. The trapped magnetic portion was recovered from the ball matrix, after shutting down of the YMS (High Intensity Magnetic Separator) separator.

2.3. Analysis and characterizations

The particle size range of the magnesite tailing received was $-3.00+0.10$ mm. The sample sieving was carried out on six different size fractions between -3.00 to $+0.10$ mm, using a laboratory sieve vibrated for 5 min. and then the amount of material retained on each sieve was weighed.

For the determination of mineral phases in the received samples and the products, X-ray diffraction method (D/max Rigaku, Japan) was used at the condition of Cu K α radiation ($\lambda=0.15418$ nm) with a step size of 0.02° (2θ) and a scanning rate of 2° min^{-1} . Energy dispersive analytical X-ray (EDAX) was also used for basic chemical analysis.

Thermal analysis of the mixture was performed on a simultaneous thermal analyzer (Netzsch STA 400, Germany). TG/DTA was performed in an alumina crucible, under nitrogen atmosphere in the temperature range of $20-1450^\circ\text{C}$ and heating rate of 10°C/min .

2.4. Calcination and calcination behavior

Calcination is pyrometallurgical process which depends on temperature, time, pressure and particle size and heating rate. It is heating process of carbonates, hydroxides, sulfate, nitrate,, acetate are converted into their oxide forms in a furnace at high temperatures in air. Thermal treatment of magnesite is important process in preparation of pure magnesia that is used for any metallurgical or chemical applications. Magnesite decomposition drives off CO_2 and produces magnesia. According to the thermal treatment temperature can be classified into three products of magnesia are calcinated, dead burned and fused magnesia. Conventionally calcination of process for magnesite is carried out at temperature up to 1000°C . The main objective of behind thermal treatment of magnesite sample are removing of volatile content of water, organic content and dissociation of carbonate forms that goes to calcination.

3. Results and Discussions

3.1. Characterization of the magnesite waste

X-ray diffraction pattern of the received magnesite tailing sample was given in Figure 3. Magnesite exists in the shape of granularity and crystals in the ore. The main impurity minerals are dolomite, calcite, diabase, quartz and different forms of lizardite such as 1M ($\text{Mg}_3(\text{Si}_2\text{O}_5)(\text{OH})_4$), 1T (trigonal pyramids, $\text{Mg}_{2.82}\text{Fe}_{0.07}\text{Al}_{0.09})(\text{Si}_{1.94}\text{Al}_{0.06})\text{O}_5(\text{OH})_4$), and 2H (hexagonal plates, $\text{Mg}_{2.83}\text{Fe}_{0.05}\text{Al}_{0.10})(\text{Si}_{1.93}\text{Al}_{0.07})\text{O}_5(\text{OH})_4$). As expected, magnesite and lizardite were the second major phases in it. These impurities are important on successful mineral separation of any sample.

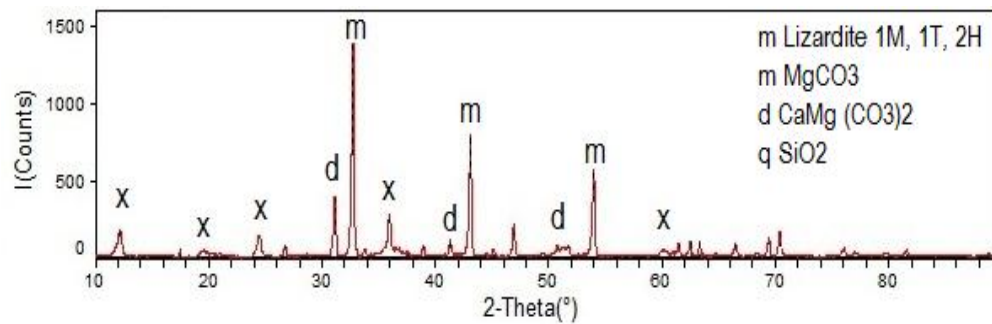


Figure 3. XRD pattern of the as received magnesite waste sample.

The relative proportions of the particle size was determined by sieve analysis test. The collected data was presented in Table 1. The particle size range of the sample was between - 3.00 to 0.1 mm. As seen from it, the dominant particle size range was -2.00+1.00 mm that corresponds to 40.21% of the total retained quantity. The particle size of the second large portion corresponds to -1.00+0.5 mm that equals to approximately ¼ of the retained quantity of the total.

Table 1. Sieve analysis of the magnesite tailing sample.

Sieve size (mm)	-3.00+2.00	-2.00+1.00	-1.00+0,500	-1.00+0,250	-0.5+0,100	-0,100
Weight %	12.21	40.21	26.73	13.82	4.95	2.08

In order to evaluate magnesia content of the tailing relating to the chemical analysis of the processed samples data collected from the X-ray fluoresce (XRF) method. Each data was in good agreement with others that were a linear relationships between them. The chemical composition of the magnesite tailing (T) sample was given in Table 2 at the first line. The main

oxide was MgO in the form of magnesite with a percentage of 77.69%, and the second oxide is silica with 12.10% in the various minerals in form of serpentine, lizardite and quarts. The iron content of the received sample was 3.14%. There were a numbers of trace impurities such as Al, Mn, K, Zn, Ni and Cr oxides less than 0.5% in total.

Table 2. XRF analysis of the received and the processed samples.

Samples	Mg*	SiO ₂	Ca*	Fe ₂ O ₃	Al ₂ O ₃	NiO	Cr ₂ O ₃	MnO	K ₂ O	ZnO	Mst	Loi
T	77.69	12.10	6.68	3.14	0.19	0.10	0.10	0.06	0.01	0.003	4.67	37.01
W	62.46	26.02	3.52	6.96	0.49	0.20	0.19	0.11	0.08	0.005	5.05	17.02
P	91.03	3.92	4.58	0.32	0.05	n.a.	0.07	0.01	n.a.	0.002	3.31	48.00
T : Magnesite tailing as received, W: Magnetic portion, P: Magnesite portion, Mst: Moisture content, Loi: Lost of ignition, *forms in CO ₃ structure												

3.2. High intensity wet magnetic separation

As seen from the sieve analysis of the received magnesite ore tailing, the size range was - 3.00+0.1 mm. The sample was mixed with water in 1:1 ratio, a slurry obtained and then subjected to magnetic separation process at 1T (Tesla) and 1.8T (Tesla). After the process, the two samples, P was the non-magnetic portions and W is the magnetic portions were analyzed by the XRF and XRD methods.

For the further purification of nonmagnetic portion of the first processed portion was subjected to the second separation process. The chemical analysis of the second nonmagnetic portion was determined by XRF method. The result was given in Table 2 the second and the third lines. These results show that, magnesite content reached up to 91.03% after the two times application of the wet magnetic separation process to the magnesite tailing sample. The process was very effective at 1.8 T for concentrating of high quality magnesite.

Table 3. The effect of magnetic field on the sieve analysis.

Mag. intensity	Sample	-3.0+2.0 mm	-2.0+1.0 mm	-1.0+0,50 mm	-1.0+0,25 mm	-0.5+0,10 mm	-0,10 mm
--	As received	12.21	40.21	26.73	13.82	4.95	2.08
0.1 T	NMP (product)	7.09	54.34	33.67	4.85	0.04	0.01
1.8 T	II.sep, II.NMP	5.94	51.13	38.34	4.56	0.01	0.01
	II. sep. MP	9.98	58.09	27.25	4.58	0.09	0.01

In order to evaluate the wet magnetic processing, the chemical composition, the thermal behavior and the X-ray diffraction patterns of the magnetic and the non-magnetic fractions of the processed samples were analyzed and compared with the as-received sample. From the chemical composition point, there are big differences in iron content of the processed samples. It is very low in the nonmagnetic portion after the first pass and is the lowest level in the second passé of the non-magnetic fraction. When compared with the thermal behavior of the samples, the degree of the weight loss are changing depending of the loss ignition content of them which are related with the carbonization of the phases in the samples.

In Table 3, the second and the third lines show the beneficiation results of the starting sample after the high intensity wet magnetic separation process. Any processing of minerals is determined from physical and chemical properties. The chemical composition results of the magnetic and the nonmagnetic portion of the process were compared with the as-received sample. From the chemical composition point of view, there are big differences in iron content of all samples. The recovery of magnesite or the removal of iron bearing minerals from the magnesite tailing was successfully done and resulted considerable differences in the chemical compositions. Iron content in the nonmagnetic fraction that is called product is very low, deceased from 3.14% to 0.38%, but it was increased from 3.14% to 6.98% in the magnetic fraction. Unfortunately, iron content of magnesite is not zero value because of it is in the spinel form of lizardite structures (1M ($\text{Mg}_3(\text{Si}_2\text{O}_5)(\text{OH})_4$), 1T (trigonal pyramids, $\text{Mg}_{2.82}\text{Fe}_{0.07}\text{Al}_{0.09}(\text{Si}_{1.94}\text{Al}_{0.06})\text{O}_5(\text{OH})_4$), and 2H (hexagonal plates, $\text{Mg}_{2.83}\text{Fe}_{0.05}\text{Al}_{0.10}(\text{Si}_{1.93}\text{Al}_{0.07})\text{O}_5(\text{OH})_4$) as seen consist of Al, Mg, Fe and Si. Lizardite is kaolinite-serpentine group mineral and displays poly types. The differences between lizardite phases come from octahedral and tetrahedral sheets structure. Al, Mg, Fe and Si control chemical composition when Fe where shows two valance states, involves the structure of lizardite sheets changes and becomes more complex. At higher magnetic intensity lizardite is separated from magnesite ore depending on iron in lizardite.

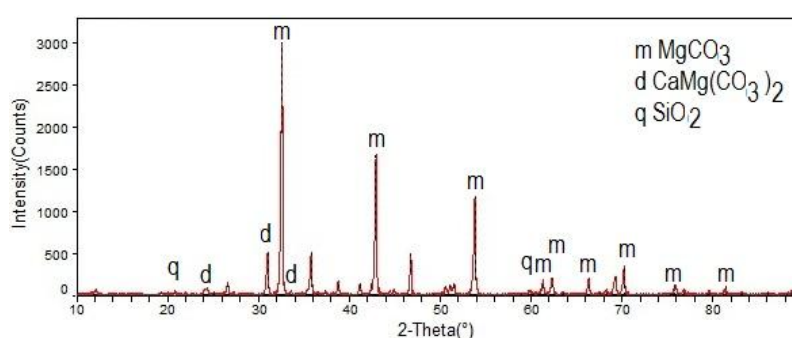


Figure 4. X-ray diffraction of the product (nonmagnetic portion at 1.8T).

The X-ray diffraction pattern of the nonmagnetic portion of the high intensity wet magnetic separation after the one pass at 1.8T. As is shown in the identified peaks of the figure, all peaks

belong to Mg related oxides and minerals such as MgCO_3 , MgO , $(\text{Ca}, \text{Mg})\text{CO}_3$. On the other hand, as a waste of the process, the magnetic portion of the separator was analyzed by the X-ray diffraction method that is presented in Figure 4. As is noted, the non and magnetic samples displays so different patterns and peaks on them. As seen in Figure 5, the magnetic portion contains different lizardite mineral forms that was iron bearing minerals in the magnesite tailing sample. The chemical analysis of the samples support the X-ray diffraction results on the base of iron oxide content. As it expected, the iron oxide content of the magnetic portion was the highest level in the three of the results. There was also color differences between the processed samples that the magnetic portion was in dark but the nonmagnetic portion was white-creamy.

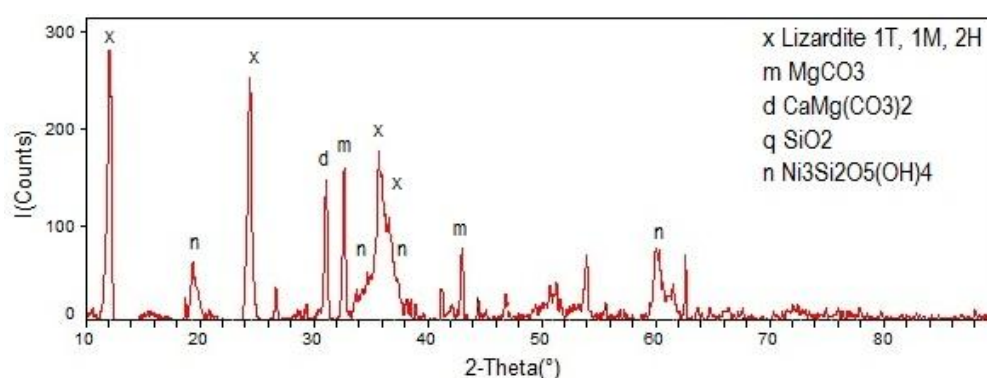


Figure 5. X-ray diffraction of the waste (the magnetic portion after the HIWMS process).

An inverse relationship exists between MgO and iron bearing minerals. When the MgO content is high, the iron content is decreased. The variation between the magnetic portions of the processed samples proves that the magnesite ore is referred to as a carbonate containing ore. The presence of iron oxide in the raw and the magnetic portions is associated with traces of iron containing minerals of lizardite.

The chemical compositions of both the magnetized and the non-magnetized portions of the samples were given in Table 1. As seen from these columns, the chemical compositions of the processed samples are quite different from the original sample. Iron content of magnesite sample can be removed by a number of chemical, physical and physicochemical methods. But magnetic separation is environment friendly method to remove any ferrous oxides from magnesite tailing because it is physical method without using any chemical agents. The

magnetic portions have a high iron oxide content. MgCO_3 content of the processed sample was increased up to 91.03% after the wet magnetic operation but it was 62.46% the waste of the process.

3.3. Calcination and calcination behavior

When compared with the thermal behavior of the samples, the degree of the ignition loss are important criteria for minerals that depending on organic content of them. The loss ignition of was calculated on the received, magnetic and the nonmagnetic portion of the processed samples, given in Table 4. As is expected, the magnetic portion was shown the lowest value in the samples that indicates the carbonate content of it is low. On the other side, the product sample has high loss ignition value which is related with the removal of carbonate content of the magnesite.

Table 4. The loss of ignition content of the samples.

Wt. %	T(Magnesite waste)	W (Magnetic)	P (Magnesite)
Moisture	4.67	5.05	3.31
Loss of ignition	37.01	17.02	48.00

The thermal behavior of the magnesite ore sample was investigated using thermal analysis method. TG/DTA curves of the sample was presented in Figure 6. Thermogravimetric mass loss changes of the sample was determined by DTA and TGA. The sample displays four various weight loss zones depending whether the sample contains carbonate minerals. It was noticed that the first zone was 25-436 °C that of 1.75% weight lost observed, but the huge amount of weight lost was occurred in the second zone at temperature between 436-650 °C that was 44.51% of the total. The third one was between at temperature 650-826 °C was 2.4% of weight loss was observed. The final zone was 826-1200 °C with a slow continuing loss was 0.22%.

As seen from the TG curve, the received sample starts to decompose at 436 °C and continue until 826 °C. The mass loss between these temperatures was 47%. After this temperature and up to 1200 °C total mass loss was 0.22%. The total weight loss of the magnesite sample was found to be 48.91% of total at temperature between 25-1200 °C.

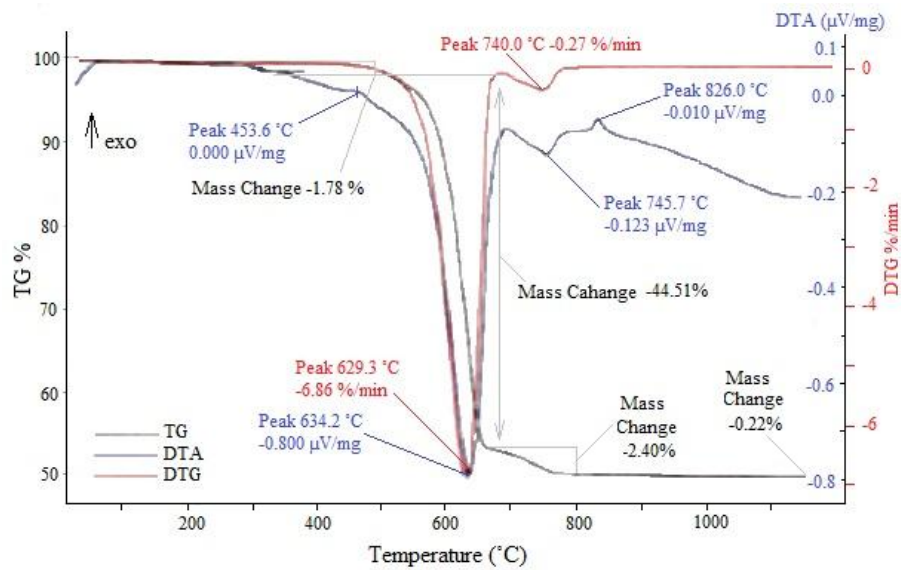


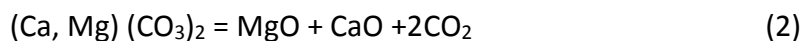
Figure 6. TG/DTA of the received magnesite ore sample.

In the DTA curve of the sample, there were three endothermic. It noticed that the first peak of the DTA curve belongs to a small endothermic peak at approximately at 454 °C temperature probably due to dehydration of (OH) from the sample. The second one was sharp and main peak at 634.2 °C and the final endothermic was observed at temperature around 745.7 °C. It should be noted that there was one exothermic peak at 650 and 826 °C.

With carbonate, as a major structure, with little gangue minerals, the reactions will be in dissociation of carbonate of magnesite and dolomite. The peak at 629 °C is attributed to the decomposition of magnesite;



Pure MgCO_3 dissociates at this temperature to form 47.8% MgO and 52.2% CO_2 . When heated sufficiently magnesia creates a crystal structure identical with natural periclase. As explained pervious parts, various calcinated magnesia are produced in different density and structure. The second peak at 634 °C is belong to the decomposition of dolomite;



Solid Ca/MgCO_3 undergo thermal decomposition to give a solid magnesite or calcia powder and CO_2 according to reactions 1 and 2. CaCO_3 has to be calcinated at higher temperature than MgCO_3 .

The endothermic peak at 950 °C, can be explained by the dehydration of lizardite ($3\text{MgO}\cdot 4\text{SiO}_2\cdot \text{H}_2\text{O}$) or clinocristotile ($\text{Mg}_3\text{Si}_2\text{O}_5\cdot (\text{OH})_4$) the magnesite with the following reaction;

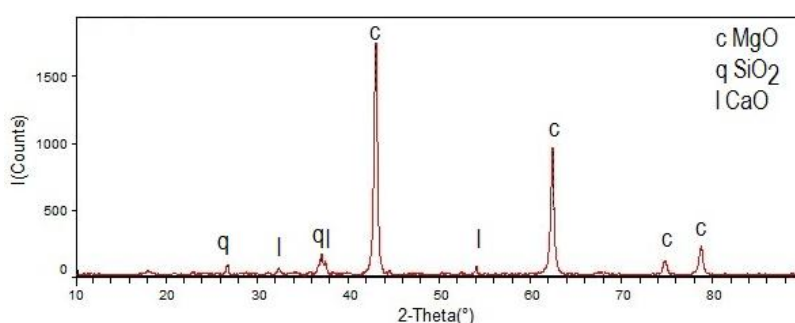
$$3\text{MgO}\cdot 4\text{SiO}_2\cdot \text{H}_2\text{O} = 3\text{MgSiO}_3 + \text{SiO}_2 + \text{H}_2\text{O} \quad (3)$$


Figure 7. X-ray diffraction of the product (the non magnetic fraction), calcinated at 1000 °C.

After the high intensity magnetic separation of the magnesite ore at 1.8T, the non-magnetic fraction of the obtained sample is called product that was heat treated at 1000 °C in a muffle furnace in air atmosphere for 4h. in an alumina crucible. XRD pattern of the calcinated magnesite was given in Figure 7. The identified phase was only magnesite (MgO) and small amount of trace phases such as quartz (SiO_2) and lime (CaO). As seen from Figure 7, any other impurity phases were not found.

Table 5. Chemical analysis of the calcinated magnesite.

$\text{MgO},\%$	$\text{SiO}_2,\%$	$\text{CaO},\%$	$\text{Fe}_2\text{O}_3,\%$	$\text{Al}_2\text{O}_3,\%$	$\text{Cr}_2\text{O}_3,\%$	$\text{MnO},\%$
85.39	7.72	6.14	0.63	0.09	0.63	0.02

The chemical analysis of the calcinated magnesite sample was presented in Table 5. Magnesite content has been increased significantly from 65.84% to 85.39% with decrease in amount of iron oxide from 5.58% to 0.63%. Silica ratio to CaO is around 1.12 % in the calcinated magnesite. This product is suitable for chemical and metallurgical applications[3, 4,18].

4. Conclusions

This paper reviews basic characteristic and beneficiation methods of Kumaş magnesite ore waste by the wet-high intensity magnetic separation and calcination were investigated in laboratory conditions. It is shown that magnesite tailing consists of 77.69% MgCO_3 with a 3.14% of Fe_2O_3 . After the process, MgCO_3 content was increased 91.03% and Fe_2O_3 content was decreased to 0.38% as well as silica, alumina. The results of the characterization studies showed that the iron bearing mineral, lizardite, was the main impurity in the sample. It was successfully separated at the magnetic field of 1.8 T from the magnesite portion. There are differences in chemical compositions, phases even on the colors of the processed samples. The obtained magnesite was subjected to calcination process at 1000 °C that that showed pure XRD pattern and 85.39% MgO content was in it.

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