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Nadežda Ćalić, Ljubiša Andrić,
Igor Miljanović, Ivana Simović

MINING INSTITUTE BELGRADE

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Foreword

Practically, all human societies depend on the availability and use of mined products. Mining and mineral processing has played a vital part in the history and economy of the Balkans.

In the world, mineral processing was an art till the 1920s, when it started to become a science. The achievements of fundamental science enabled the explanation of phenomena in the processes of mineral processing, or they started from fundamental science to come to an appropriate solution in mineral processing. In many respects mineral processing becomes fundamental science.

Balkan countries have more or less rapidly accepted innovations in the field of mining and mineral processing.

Generations of professionals from Balkan trained on the tradition of mining schools, afterward universities, (Schemnitz established 1702, Jachimov 1716, Banska Štiavnica 1725, Jekatarinburg 1730, L' Ecole Polytechnique 1794 in Paris, Politehnika in Prague, and certainly the most famous Bergakademie Freiberg founded in 1765, and much later, universities in the United States and Soviet Union) contributed to today's level of development of mineral processing, and contributed to the quality of studies of mineral processing, both in the world, and so in the Balkans.

After the Second World War in the Balkans a large number of universities, faculty, institutes and laboratories of mining industry with special departments for mineral processing were opened. In many Balkan countries remarkable impact on development of mineral processing had Russian and American schools.

A great number of researchers and specialists in Balkan area were occupied for more decades by the research in mineral processing. The goal of this research was establishment of concentration process in industry, capacity enlargement, optimization of processes, increase the energy efficiency of processes and devices, introduction or construction of new machines. Based on those activities, Balkan mining has been evolving and continuously operates up to nowadays. As a result, in the Balkan countries appeared a significant number of successful researchers in the field of mineral processing. They founded the first Balkan mineral processing Committee (1973), and then the Balkan Academy of Mineral Technology.

Balkan Congress on Mineral Processing is beening held for 40 years. Participation in the work of the Committee of the Balkan mineral processing is a strong link between the development of the science and profession with global trends, and it provides the possibility of establishing direct contacts between researchers, designers, equipment manufacturers and investors from the region and around the world. It has already become tradition to hold every second year an international event, "Balkan Mineral Processing Congress," in which participate, not only Balkan experts, than experts from the whole world.

Maintenance XVI Balkan Congress on Mineral Processing in Belgrade from 16 to 21 June 2015 is held under the auspices of the Ministry of Mines and Energy of Serbia, with the financial assistance of the Ministry of Republic of Serbia. Incomparably greater financial support Congress had from sponsors who strongly support the mineral processing industry all over the world.



Prof. dr Nadežda Ćalić

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EFFECTS OF PHYSICAL PROPERTIES TO THERMAL DECOMPOSITION OF DOLOMITES

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Abstract This study has examined the effects of dolomite characteristics (chemical composition and physical properties; bulk density, porosity, water absorption rate) and calcination temperatures on the decomposition phenomena. Chemical, physical analysis and thermogravimetry technique have been performed in dolomite samples originating from different parts of Turkey, determining dolomite characteristic properties and quality. Differential thermal analysis (TG-DTA) curves, between 25-900°C, of dolomite shows endothermic peak, essentially due to decarbonation of dolomite, finishing at 800±3°C and concerning the decomposition peak are related to content of MgO ratio, impurities and microstructure and grinding time of dolomites and ambient gas type. At the calcination experiment, it was found that the calcination reaction begins at approximately 450°C and was completed at between 900-950°C. At temperatures higher than 950°C, recarbonisation of MgO and CaO started that increased the sample weight.

Keywords: Dolomite, Dolomite characteristics, Thermal decomposition, MgO, CaO

INTRODUCTION

The original mineral name dolomite was given by N.T. Saussare, in 1792, in honor of the French geologist Deodat Guy de Dolomieu (1750–1801) and was first applied to rocks in the Tyrolean Alps (Bourrouilh-Le Jan, 2000). Dolomite, the rock, contains a large proportion of dolomite the mineral. Dolomite typically occurs as the major constituent of sedimentary formations in association with calcite. Carbonate rocks tend to be composed either mostly of calcite or mostly of dolomite (Fig. 1) (Blatt, 1992). Ideal dolomite has a crystal lattice consisting of alternating layers of Ca and Mg, separated by layers of CO_3 and is typically represented by a stoichiometric chemical composition of $CaMg(CO_3)_2$ where calcium and magnesium are present in equal proportions. The layer-type structure of dolomite is responsible for the strong anisotropy of the physical properties, including the thermal expansion. Theoretical composition of the dolomite is 21.86% MgO, 30.42% CaO and 47.73% CO_2. Varying amounts of impurities including SiO_2, Al_2O_3 and Fe_2O_3 are present in dolomite. The amounts and types of these impurities may have a large effect on the extent of densification. Dolomite occurs widely scattered in nature and is an important material for the metallurgical (as flux, as fettling material for the hearth of furnaces), pharmaceutical, paper and fertilizer industries (Leighton and Pendexter, 1962).

Thermal analysis might offer the means of defining the fraction of each mineral lattice in such mixtures and the concentration of each cation in each lattice. The correlation of the thermal data with the structural pattern should provide a broader understanding of these minerals in their natural occurrence. A general review of the literature on the decomposition of carbonates indicates that a great deal of variability exists in the reported values of the decomposition temperatures, activation energies and rates of decomposition. The decomposition of dolomite has been studied extensively because of its mineralogical interest and industrial importance (Powell and Searcy, 1978; Beruto et al., 2002).

The internal structure of a rock having open and closed pores in its texture affects its heat transfer. The changes in pore structure also play a significant role on the calcination mechanism and the reactivity of a calcined dolomite is strongly dependent on its physical and structural properties, which in turn are highly dependent on calcination conditions (Boynton, 1980).

Thermal decomposition of dolomite in air or in CO_2 has been the subject numerous studies (Otsuka, 1986; McIntosh, 1990; Samtani et al., 2002). Pure dolomite decomposes in two separate stages (eq. 1-2). The calcination reaction is reversible and occurs at an interface; the rate of CO_2 release is sensitive to the prevailing pressure of this product in the vicinity of the reaction interface. Both apparent kinetic parameters and reactivity are influenced by the prevailing CO_2 pressure.

$$MgCa(CO_3)_2 \rightarrow CaCO_3 + MgO + CO_2\uparrow \quad (1)$$

$$CaCO_3 \rightarrow CaO + CO_2\uparrow \quad (2)$$

The differential thermoanalytical (DTA) curve of dolomite show to definite endothermic peaks: the lower temperature peak is associated with the decomposition of dolomite structure and the formation of magnesia and calcite; the higher temperature peak with the decomposition of this calcite. The temperature of the second peak is shifted to a higher temperature with increasing the partial pressure of CO_2. The temperature of the first peak is decreased with increasing CO_2 pressure, so that the first and second endotherm peaks are shifted in the opposite direction with changing CO_2 pressure.

The first stage of the thermal decomposition of dolomite resulted in the formation of Mg-calcite [$(CaMg)CO_3$] and periclase (MgO), with the liberation of CO_2. It was further observed that under CO_2, dolomite decomposed directly to $CaCO_3$, accompanied by the formation of MgO between 550 and 765°C. Calcite decomposed to CaO between 900 and 960°C and under air, simultaneous formation of $CaCO_3$, CaO and MgO accompanied dolomite decomposition between 700 and 740–750°C. At the latter temperature, the calcite began to decompose even though a significant amount of dolomite was still present and simultaneous decomposition of the two carbonates was terminated at 780°C. Also, changes in decomposition rates of the various phases correlated with changes in the rate of weight loss determined by derivative thermo-gravimetric analysis (Maitra et al., 2005).

In this study, the effects of dolomite characteristics to the decomposition temperature of dolomite at various temperatures studied by differential thermal analysis and with calcination experiments, heated in a muffle furnace under air atmosphere.

MATERIAL AND METHOD

The dolomites were taken from different large commercial deposits: Mersin and Erdemli (Mersin) region and Antalya region (Alanya) in Turkey. During sampling, rock types having no bedding planes were selected to eliminate any anisotropic effects in the measurements of the samples. The dolomite samples present macroscopically light and dark grey coloured comprising discrete and tiny crystals. Microcracks were not present throughout the mass of Alanya and Mersin dolomite samples; but microcracks, dense vein system and schistosity were observed throughout the mass of Erdemli dolomite samples.

Analyses were performed on dolomite samples by using the following analytical procedure. In the chemical analysis and thermal experiments (TG-DTA analysis and calnination procedure) ground -0.5 mm size dolomites were used. Cubic specimens of 3 cm mean edges were prepared for the physical experiments.

- XRF (Siemens SRS 300 X-ray Flouresans Spectrometer) was used to determine the chemical compositions of dolomite samples.
- X-ray diffraction (XRD) analysis of finely pulverized limestone samples for the identification of the presented crystalline compounds. The analyses performed with a Rigaku DMAX III C XRD. The diffraction interval was between 2θ-20°-60° with a step of 0.02°.
- Physical properties (the bulk density, effective porosity, water absorption rate) of the limestones were determined using saturation and buoyancy techniques, as recomended by ISRM and TSE (TS 699, 2009).
- Differential thermal and thermogravimetric analyses (simultaneous TG/DTA, SII Exstar 6000 DTA-TG) were carried out to determine quantitatively and qualitatively the various compounds presented in samples. Analyses were performed in samples of dolomite at a temperature range of 25-900°C and gradient of 10°C/min.

RESULTS AND DISCUSSION

Dolomite characteristics

The chemical analyses results of dolomite samples are presented in Table 1. It was found that the studied dolomites are MgO higher than 20%, the impurities (Fe_2O_3, Al_2O_3, SiO_2 and MnO) are very low. Since the $MgCO_3/CaCO_3$ ratio varies with the type of dolomitic limestone, the decomposition temperature does not remain constant.

The mineralogical composition of limestone was obtained by XRD analysis. The main component proved to be calcite ($CaCO_3$) and dolomite ($CaMgCO_3$) for all samples, although this does not preclude the possibility of the presence of small quantity (SiO_2).

Table 1, Chemical composition of dolomite samples

Sample	Fe_2O_3 (%)	Al_2O_3 (%)	SiO_2 (%)	MnO (%)	CaO (%)	MgO (%)	LOI (%)
Mersin	0.071	0.11	0.28	0.027	30.80	21.84	46.872
Erdemli	0.050	0.43	0.31	0.050	29.01	21.52	48.630
Alanya	0.046	0.06	0.16	0.041	30.52	20.50	48.673

Table 2, Physical properties of the dolomites

Samples	Unit weight (g/cm^3)	Water absorption ratio (%)	Porosity (%)
Mersin	2.87	0.50	2.80
Erdemli	2.81	0.74	3.10
Alanya	2.84	0.62	2.40

The analysis results of physical properties of the dolomite samples are shown Table 2. All samples indicated medium void volume values in porosity (2.5-5%) (Tarhan, 1989). The value of unit weight (2.8-2.9 g/cm^3) was found to be characteristic for a dolomite.

Calcination of the Dolomite

In the calcination experiments, firstly, -0.5 mm size and amount of 10 g powder dolomite samples were heated in a muffle furnace (Nabertherm) with thermal control under air atmosphere at different temperatures (450°C, 500°C, 550°C, 600°C, 650°C, 700°C, 750°C, 800°C, 850°C, 900°C, 950°C, 1000°C) for 1 h in each temperature, to determine on behavior of calcination. The previous study (Kılıç, 2006) shown that 1 hour heating time is enough for calcination amount of 10 g powder samples in a muffle furnace.

At the experiments, the evaluation of the dissociation completion was performed by measuring the mass loss of the samples before and after calcination. The mass loss corresponds to the released amount of CO_2 during the calcination process. The measured percentage of mass loss is around 47% (eq. 1-2). Therefore, the dissociation of dolomite and calcite and the decomposition of dolomite are successfully accomplished. It was also found that the calcination reaction begins at approximately 450°C and was completed at between 900-950°C. At temperatures higher than 950°C, recarbonisation of MgO and CaO started that increased the sample weight (Fig. 1).

Secondly, thermal analysis was performed in a simultaneous TG–DTA. The experimental conditions were: (i) continuous heating from room temperature to 900°C at a heating rate of 10ºC /min, TG and DTA curves were obtained. The following data was obtained by thermal analysis: (i) reaction peak temperature and main effect (endothermic or exothermic) and (ii) content of bound water, which is the weight loss in the temperature range 450–800±3°C and content of CO_2 released during the decomposition of magnesium carbonate phase (Fig. 2).

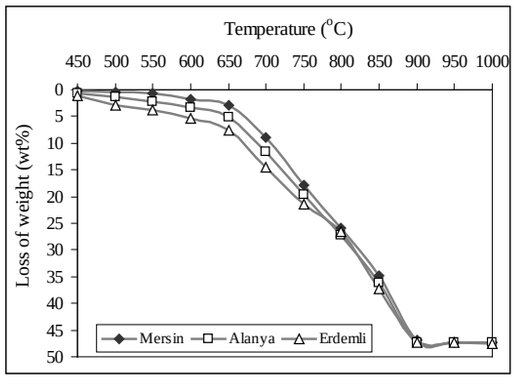
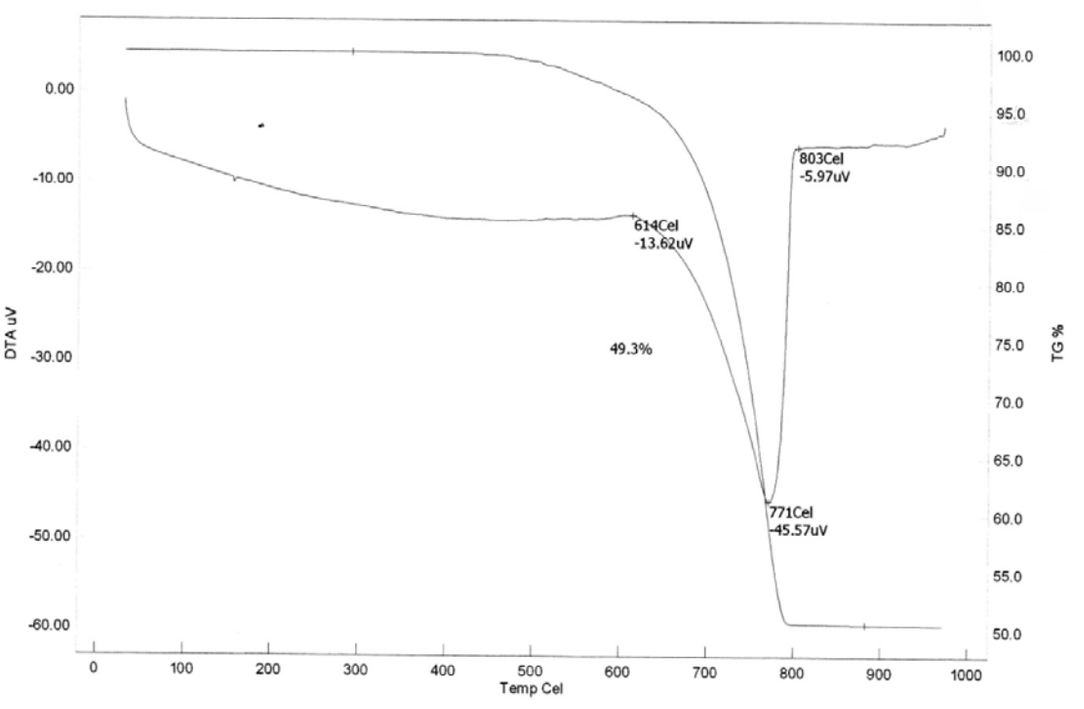
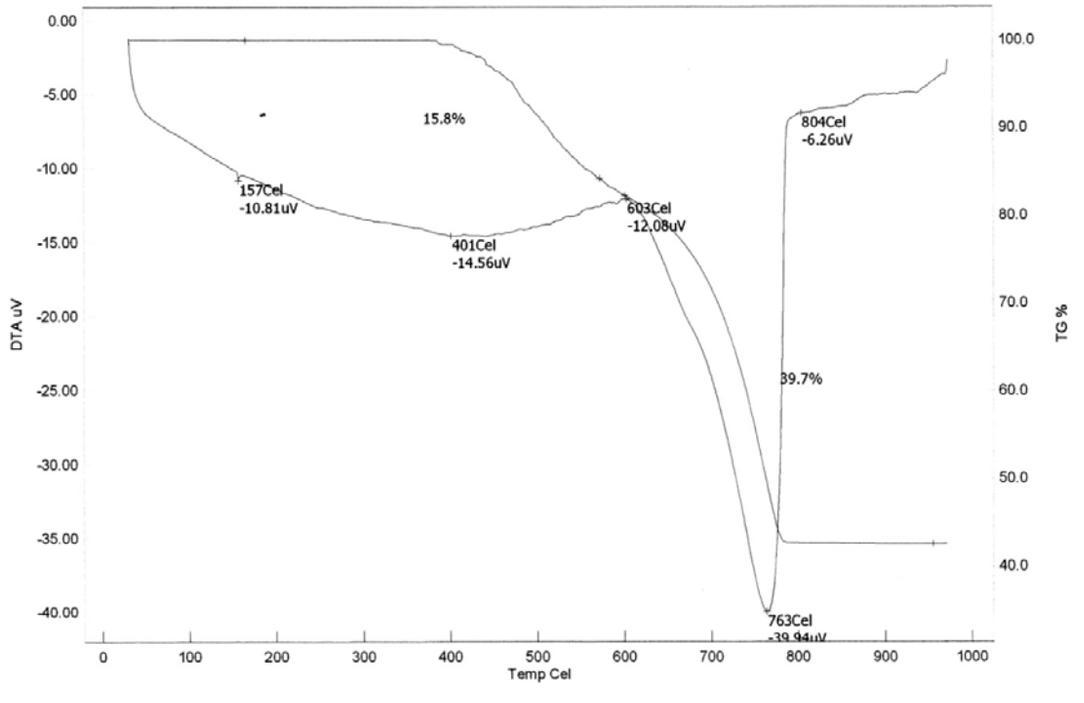


Fig. 1, Loss of weight of the samples at different temperatures

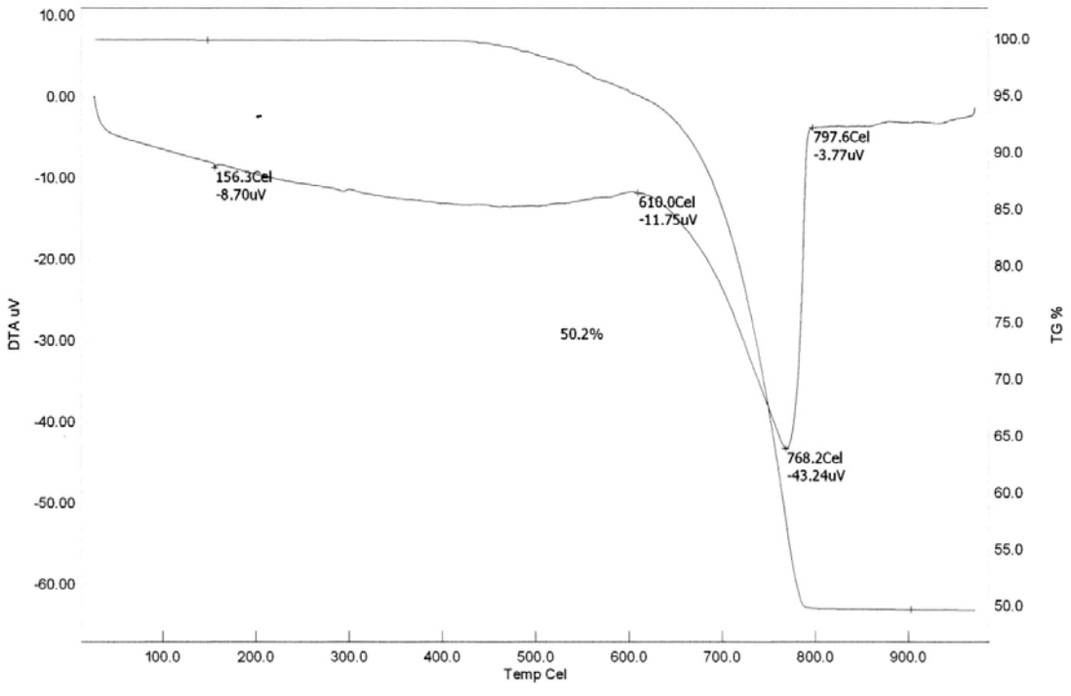
The endothermic peak at 195°C on DTA curve is attributed to loss of water. The weight loss above 450°C, measured by DTA-TG (Fig. 3), is attributed to the CO_2 from the decomposition of magnesium carbonate. Comparing the percentage values of CO_2 measured by DTA/TG, it is evident that the values are very high and similar to each other. When comparing these results, it is found that the peak is decomposition of dolomite maximum occurs at 800±3°C. Furthermore, the second peak, the peak is decomposition of calcium carbonate phase, it is not observed at a temperature range of 25-900°C. If the samples were ground long time (1-5 h) (Caceres and Attiogbe,1997) approximately at 900°C, the decomposion peak of calcium carbonate can be observed. In the crystal structure of dolomite it is implicit that it must be treated as a definite compound when considering the thermal decomposition.


DTA uV
TG %
Temp Cel
614Cel
-13.62uV
803Cel
-5.97uV
771Cel
-45.57uV
49.3%

(a)


DTA uV
TG %
Temp Cel
157Cel
-10.81uV
401Cel
-14.56uV
603Cel
-12.08uV
804Cel
-6.26uV
763Cel
15.8%
39.7%

(b)



(c)

Fig. 2, Thermograms of decomposition of dolomite (a) Mersin, b) Erdemli, c) Alanya)

CONCLUSION

This study investigated the effect of dolomite characteristics to the decomposition temperature of dolomite at various temperatures studied by differential thermal analysis and with calcination experiments. The samples can be characterised as limestone-dolomite with low content of impurities (Fe_2O_3, Al_2O_3, SiO_2 and MnO) and MgO higher than 20%. The impurities could function as catalysts owing to their influence in the crystalline structure. Further investigation should be accomplished regarding the effect of dolomite texture on reactivity.

In this study, it was found that all the samples indicated medium void volume values in porosity (2.5-5%). The value of unit weight (2.8-2.9 g/cm^3) was found to be characteristic for a dolomite. These findings show the fact that these dolomites have compact texture and this type of stones are preferred in the production of dolomite lime. It will have a good quality.

Decomposion of dolomite (TG-DTA), finishing at 800±3°C and concerning the decomposition peak are related to content of MgO ratio, impurities and microstructure and grinding time of dolomites and ambient gas type. At the calcination experiment, it was found that the calcination reaction begins at approximately 450°C and was completed at between 900-950°C. At temperatures higher than 950°C, recarbonisation of MgO and CaO started that increased the sample weight.

The rate of temperature increase may be an additional parameter for the evaluation of product reactivity which is augmented by increasing the product reactivity.

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