# USE OF DOLOMITE LIMESTONE IN THE DOMESTIC WASTEWATER TREATMENT

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**Abstract:** *The fields of dolomite limestone using are extremely varied as it can be employed in metallurgy, construction materials, chemical and glass industry, as well as multiple usages in agriculture and environmental protection. This paper focuses on the use of the dolomite from Budureasa area, Bihor County, in the process of purifying domestic wastewater from Glina Station near Bucharest, in order to reduce the content of nitrogen and phosphate ions in the raw domestic water below the limit permitted by the rules in force regarding their discharge into the emissary.*
**Key words:** *Dolomite limestone, brucitic dolomite limestone, domestic waste water, anionic ions, phosphate ions*

## 1. Introduction

Magnesium-bearing minerals, widespread on the surface of the globe, are very important and are used in multiple fields, representing a potential "savior" of the planet. As a result of processing operations that reveal reactive magnesium oxide (reactive magnesia), magnesium minerals can be a basic source in various fields of activity.

Magnesium is widely distributed in nature in the form of combinations, making up 2% of the Earth's crust. Thus, it is found in magnesite deposits (magnesium carbonate - $MgCO_3$) and especially dolomite (double calcium and magnesium carbonate, $MgCO_3 \cdot CaCO_3$). Very common are carnalite (potassium chloride and magnesium chloride - Mg, $Cl_2 \cdot KCl \cdot 6H_2O$), kieserite (magnesium sulphate - $MgSO_4 \cdot H_2O$), then different magnesium silicates - such as olivine (Mg, Fe) $SiO_4$], serpentine, talc, asbestos as well as aluminosilicates such as spinel, $MgO.Al_2O_3$ (V. Brana, 1967).

In soil, there are always combinations of calcium and magnesium coming from the disintegration of minerals. Many magnesium salts are dissolved in sea water or salty lakes, to which they give a bitter taste. Also, many mineral springs contain dissolved magnesium sulphate.

## 2. Areas of use of dolomitic limestone

Magnesite, dolomite and seawater are the most common sources for making metallic magnesium and its compounds, though brucite and olivine are also used.

Depending on the magnesia and magnesia content (magnesium oxide), the main industrial minerals are shown in table 1.

**Table 1** – *The main magnesium minerals with industrial value*

| Nr. | Mg, % | MgO, % | Mineral | Chemical formula |
|---|---|---|---|---|
| 1 | 41,68 | 69,12 | Brucite | $Mg(OH)_2$ |
| 2 | 34,5 | 57,3 | Forsterite (peridot series) | $Mg_2SiO_4$ |
| 3 | 30,0 | 49,7 | Peridot (Mg-Fe) | $(Mg,Fe)_2SiO_4$ |
| 4 | 28,8 | 47,8 | Magnesite | $MgCO_3$ |
| 5 | 26,3 | 43,6 | Serpentine | $3(Mg,Fe)O\ 2SiO_2\ 2H_2O$ |
| 6 | 11,96 | 19,83 | Bischofit | $MgCl_2 \cdot 6(H_2O)$ |
| 7 | 8,75 | 14,51 | Carnallite | $(K,Mg)Cl_3 \cdot 6(H_2O)$ |

As a result of the magnesia ore processing operations with significant economic value, there are several salable finished products that are used in different economic areas. The final products are usually obtained by thermal treatment applied to the rough magnesium ore at well-established temperature ranges, which give the calcined material different physical and chemical properties.

Magnesium oxide (or magnesia) resulting from the thermal treatment of various magnesium minerals, depending on the calcination temperature, may have different names (and different compositions), and the fields of use are different.

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The three magnesia forms obtained successively at increasing temperatures are: calcined caustic magnesia (CCA - caustic-calcined magnesia); burned or sintered magnesia (DBM); melted magnesia (FM - fused or electro-fused magnesia) (Schulze - Rettmer, R, 2010).

The main areas of use of these three basic products, available at variable prices, are:

- For CCM :

- Extraction of metallic magnesium;
- Production of molten magnesia;
- Production of special cements (eco-products);
- Electrical insulation;
- Paper industry;
- As a fertilizer in agriculture and animal feed;
- Stabilizer for the production of synthetic rubber;
- Uranium production;
- Protection of the environment (waste water treatment and desulphurization of combustion gases).

- For DBM: obtaining basic refractory bricks;
- For FM: thermal insulation, electrical insulation (electric furnaces and installations)

### *2.1. Domains of use in the industrial field*

*a) In metalurgy* limestone is widely used and is one of the necessary technological products that participate in most ore extraction processes. Calcium is used as a foundry in the production of iron and steel, and dolomite for the production of refractory bricks used in furnaces or for the production of refractory mixtures and refractories which are resistant to high temperatures. The limestone introduced into the furnace also has the role of iron ore desulphurization.

*b)* Large quantities of limestone or lime are used in *the manufacture of cement*, which, depending on the manufacturing recipe, is included in the composition of different types of cements.

*c) The production of lime* is an extremely important industrial field, because many types of activity use the calcium and magnesium source under this form of product, resulting from the thermal processing of different types of limestone associated with dolomite or brucite.

### *2.2. The use of dolomite limestone in agriculture*

In agriculture, dolomite limestone are used as fertilizers in acid soil detoxification and deoxidation, as mineral mixtures in animal feed (nutritional supplement) or as Ca and Mg in some crops of important vegetables, fruit trees and vines.

It is important to note that just a few years ago (2013), a Romanian product called DelCaMag (or DEL-CA-MAG) was launched on the market, which produces amazing results in Romanian agriculture and beyond. This product is dolomite by Delniţa (Harghita County), which has a special peculiarity that makes it unique (at least at European level) (Ecological Farming Magazine "Eco Ferma", 2014).

Delniţa Dolomite has an amorphous structure, translated by the fact that between calcium and magnesium ions there is a much weaker connection than in crystalline dolomite, which favors dissolution processes in the soil or gastric juice of animals, favoring a much faster assimilation of them.

The beneficial effects of Delniţa dolomite were evaluated by the team of researchers from the National Research and Development Institute for Pedology, Agro-Chemistry and Environmental Protection - ICPA Bucharest. According to them, using DELCAMAG increases crop yield by 30%. The study refers to fruit trees, potatoes, vines, linen or celery.

"The documentary study on dolomite deposit at Delniţa, Harghita County, in connection with its importance for agriculture" by ICPA, shows that using dolomite is a 100% natural solution that can be used to increase productivity for a number of important crops for the economy such as fruit trees, potatoes or vines.

According to the study, the use of dolomite as a calcium and magnesium for soil cultivated with plants gives them resistance to breakage and dropping, owing to their essential role in the development of the root system and strains. Dolomite does not exert phytotoxic effects on plants, even in larger quantities (being considered as the only ion with this property) and its absence influences negatively the absorption of phosphoric ions and nitrates. "Mineral maintains calcium pectin time longer in the pulp of fruit, ensuring greater resistance to conservation and induction of positive effects on plant resistance to disease and pest attack," says study (www.eco-ferma.ro, 2014).

Dolomite is used as a dietary supplement in farmed animals (cattle, sheep, poultry), calcium and magnesium intake favoring the absorption of nutrients in feed by altering the permeability of cell membranes and having an important role in developing the bone system and increasing resistance to various diseases.

### *2.3. The use of dolomite limestone in the environmental protection*

In the protection of the environment, dolomite limestone or their derivatives are used in the:

- Treatment of combustion gases from thermal power plants (desulphurisation)
- Neutralizing acid waters
- Treatment of household waste water
- Recovery of heavy metals from industrial waste water

At the level of 1973, there were 42 FDG units (Flue Gas Desulfurization) applied to thermo-power plants in Japan and the USA. In 2000, 278 economically developed countries operated 678 FDG units, operating in thermo-electric plants producing approximately 229 GigaWatt (Rubin E., Yeh S. et al., 2004).

An alternative to removing sulfur from the combustion gases is removing sulfur from the fuel before or during combustion. The hydrodesulphurization of the fuel before use is carried out by a fluidized bed of lime during combustion. The lime reacts with SO2, causing it to form sulfates, which become part of the ash. This elemental sulfur is then separated and the ash can subsequently be used, for example, for the production of agricultural products. Safety is one of the greatest benefits of this method, as the whole process takes place at atmospheric pressure and ambient temperature (Official Paqell website).

In the process of neutralization of acidic waste water, but also for the recovery of heavy metals from different types of industrial waste water, limestone and especially dolomite limestone with brucite are very used, and there are some clear advantages of the presence of brucite, Mg (OH)2 in limestone material. (Simandl, G.J., Paradis, S., & Irvine, M.,2007).

At present, magnesium as a metal or in various compounds that may result from dolomite or dolomite limestone calcination with brucite has a very wide range of uses and is becoming a highly sought after and profitable product to be produced and marketed.

The cessation of the non-ferrous ore extraction activity at national level has turned the attention of this area to the resources indispensable to the sector with the highest growth in recent years, namely: construction.

According to the National Mineral Resources Agency, the most valuable mineral resources are the limestone deposits, limestone being the raw material necessary for the production of cement and lime, silico-calco-sodic glass, the mosaic for ornamental works, decorative concrete, as well as aggregates used in road construction.

Starting from the many fields of use and resources existing in our country, all researches aiming at a superior valorization of magnesium-bearing minerals is of particular economic importance.

## 3. Research regarding the use of Dolomite limestone with brucite from Budureasa area, Bihor County, in the domestic residual waters treatment

The most valuable deposits that can provide a viable source of reactive magnesium oxide that can be recovered are the dolomite limestone [Mg (OH) $_2$], which exist in Romania.

In our country, at the beginning of the nineteenth century, brucite appeared as a substance almost unknown both in terms of its economic and of mineralogical valorization. Then, the only deposits on the territory of Romania, the ones in the Bihor Mountains, the Budureasa - Pietroasa area, were highlighted. (Panaiotu C.E., 2005).

As a mineral, brucite is relatively easy to identify optically, the minerals it includes (moulder, gibbsite, gypsum, talc) appear in different types of rocks or mineral associations. The pure brucite has the appearance of a micaceous, uniaxial and local appearance of fibrotic development. The variety of ferrobrucit (formerly, "amakinit") has a brownish-yellow color, showing the same birchiness as pure brucite, as can be seen in figure 1.

Generally, brucite develops on dolomite (Ca and Mg carbonate), periclaz (Mg oxide) and forsterit (Mg silicate), in conditions of increased water activity. In turn, it transforms relatively easily into hydrogenous and less in serpentine minerals (Petrulian N., 1973).

**Fig.1.** *Brucite*

### *3.1. Physic-chemical characteristics of the Dolomite limestone with brucite, from Budureasa area, Bihor County*

For laboratory tests regarding the use of dolomite limestone in household waste water treatment, which consists in the reduction of the nutrient content (nitrates and phosphates), bruciticdolomite limestone was used, having the chemical composition presented in tables 2 and 3.

**Table 2** *The results of the general chemical analysis on the brucite limestone sample (macroelements)*

| ELEMENT | LD | VALUES (%) |
|---|---|---|
| $SiO_2$ | 0,01 | 0,42 |
| $TiO_2$ | 0,01 | 0,01 |
| $Al_2O_3$ | 0,01 | 0,03 |
| MnO | 0,01 | 0,02 |
| $Fe_2O_3$ | 0,01 | 0,13 |
| **MgO** | **0,01** | **23,18** |
| **CaO** | **0,01** | **33,12** |
| $Na_2O$ | 0,01 | 0,09 |
| $K_2O$ | 0,01 | 0,06 |
| $P_2O_5$ | 0,05 | 0,18 |
| P.C. | 0,10 | 42,28 |

LD – detection limit

**Table 3** *The results of the general chemical analysis on the brucite limestone sample (microelements)*

| ELEMENT | LD | VALUES ($\%*10^{-4}$) |
|---|---|---|
| Li | 0,2 | 0 |
| Be | 0,2 | 0 |
| V | 1,0 | 5 |
| Cr | 0,1 | 1,9 |
| **Co** | **0,5** | **29,3** |
| **Ni** | **0,5** | **2,7** |
| **Cu** | **0,1** | **14,2** |
| **Zn** | **1,0** | **42** |
| As | 0,1 | 0 |
| Rb | 0,1 | 0,2 |
| Sr | 1,0 | 86 |
| Y | 0,1 | 1,1 |
| Zr | 1,0 | 11 |
| Nb | 0,1 | 0,6 |
| Mo | 0,1 | 0,3 |
| Cd | 0,1 | 0,4 |
| Sn | 0,4 | 3,6 |
| Sb | 0,5 | 0,5 |
| Te | 0,01 | 0 |
| Cs | 0,1 | 0 |
| Ba | 1,0 | 10 |
| W | 0,25 | 0 |
| Pb | 1,0 | 15 |
| Bi | 0,1 | 0,1 |
| Th | 0,1 | 0,4 |
| U | 0,1 | 0,4 |
| Tl | 0,2 | 0 |
| Ga | 0,1 | 0 |
| **Se** | **0,1** | **0,1** |

Analyzing the results of the general chemical analysis, it can be seen that, as expected, the largest share is held by calcium and magnesium oxide.

*Thermo-differential and thermo-gravimetric analysis* was performed on a sample of material (figure 2) in the furnace, at 1000ºC with a heating rate of 10ºC / min.

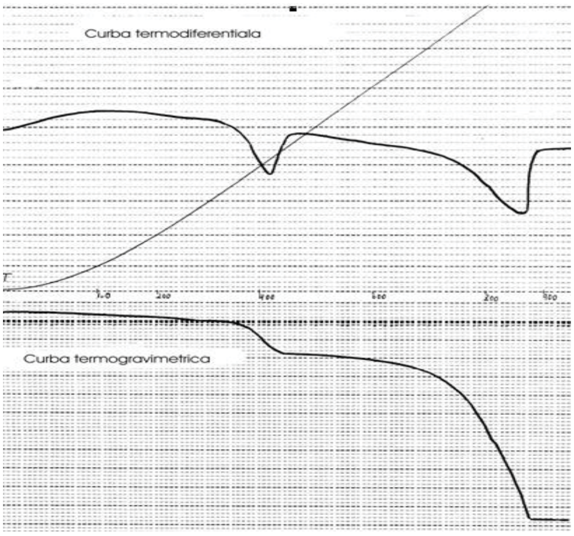


**Fig.2.** *Thermo-differential and thermo-gravimetric curves*

The weight loss recorded by the TG curve corresponds to the enthalpy variations recorded by the DTA curve through the endothermic effects characteristic of brucite, calcite and dolomite traces. The endothermic effect at 420°C represents the dehydroxylation of brucite by the loss of a water molecule marked by the first weight loss step on the TG curve shown in the figure. The endothermic effect at 880°C indicates the decomposition of calcium carbonate - calcite and the elimination of carbon dioxide through the second weight loss step on the thermo-gravimetric curve. The endothermic effect at 800°C indicates the presence of dolomite (traces).

The results of this type of analysis indicate the presence of these minerals in the following proportions: Brucite – 23.2%; Calcite - 42.7%.

Knowing the decomposition temperatures of the component minerals is particularly important for the processing steps when it is desired to highlight a particular mineral in order to recover it through various technological processes.

*Aspectul general al calcarului dolomitic la micro The general appearance of the dolomite limestone on the electron microscope presented on different scales (200, 100, 20 and 5 microns) in figure 3 (a, b, c, d) reveals that the main component minerals are dissociated at this level of shredding of the material. In the figure denoted by (d) a 20-micron particle of brucite is observed, white patches representing ultrafine calcine or dust particles, but not particle-bound, they change their position during "bombardment" X-ray beam sample (INCDMRR Bucharest, 2005).*

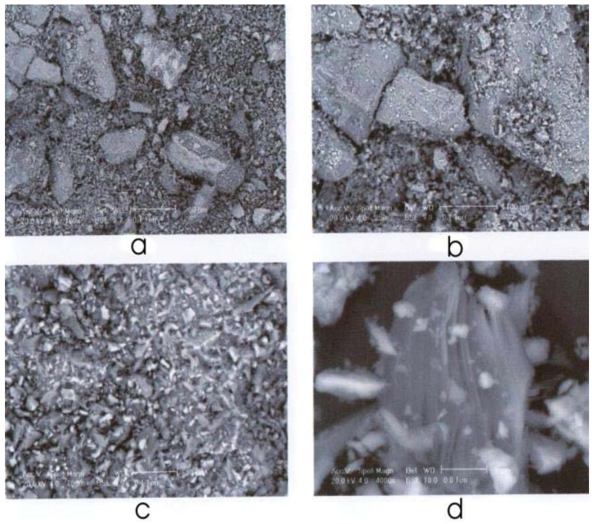


**Fig.3.** *The general aspect of the sample on the electron microscope*

The results of the granulometric analysis after three shredding operations (two successive crushing with jaw crushers and a chopping up to 2-3 mm in the disk mill) are presented in Table 4.

From the cumulative rejection curve shown in figure 4 it can be seen that the weight of intermediate material (0.4-0.04 mm) is relatively reduced after the three shredding operations, the material being composed of a homogeneous mixture of coarse granules with ultra-fine particles.

The lower hardness and density of brucite compared to other mineral species leads from the outset to the idea of its selective crushing, since as found in the magnesium oxide content on granulometric classes, the brucite concentrates in the ultrafine fraction (below 0, 04 mm), the MgO content in this class being 28.56%, 6% higher than the average content of the entire sample that is 22.56%.

The distribution of brucite on grain-size classes is very interesting in that the whole material can be divided into three granulometric layers with the average MgO content distributed as follows: the material between 0.44-2 mm has an average content of 21.84% MgO, the material between 0.04-0.4 mm has an average content of 17.24% MgO and the ultra fine material -0.04 mm has a MgO content of 28.56%.

**Table 4** *Granulometric analysis of the fine material*

| Granulometric class (mm) | Rejections, $q_i$ | | Cumulated rejections $\Sigma q_{i}$, % | Content MgO, % |
|---|---|---|---|---|
| | grams | % | | |
| +2 | 20 | 1,61 | 1,61 | **21,514** |
| 2-1,6 | 33 | 2,65 | 4,26 | **21,742** |
| 1,6-1,25 | 104 | 8,37 | 12,63 | **22,265** |
| 1,25-0,8 | 251 | 20,22 | 32,85 | **22,216** |
| 0,8-0,63 | 106 | 8,53 | 41,38 | **21,476** |
| 0,63-0,4 | 129 | 10,38 | 51,76 | **21,717** |
| 0,4-0,2 | 117 | 9,43 | 61,19 | **19,646** |
| 0,2-0,16 | 28 | 2,25 | 63,44 | **17,796** |
| 0,16-0,125 | 28 | 2,25 | 65,69 | **17,466** |
| 0,125-0,074 | 79 | 6,36 | 72,05 | **14,594** |
| 0,074-0,040 | 25 | 2,01 | 74,06 | **13,462** |
| -0,040 | 322 | 25,94 | 100,00 | **28,566** |
| Total/average | | 100,00 | | **22,56** |

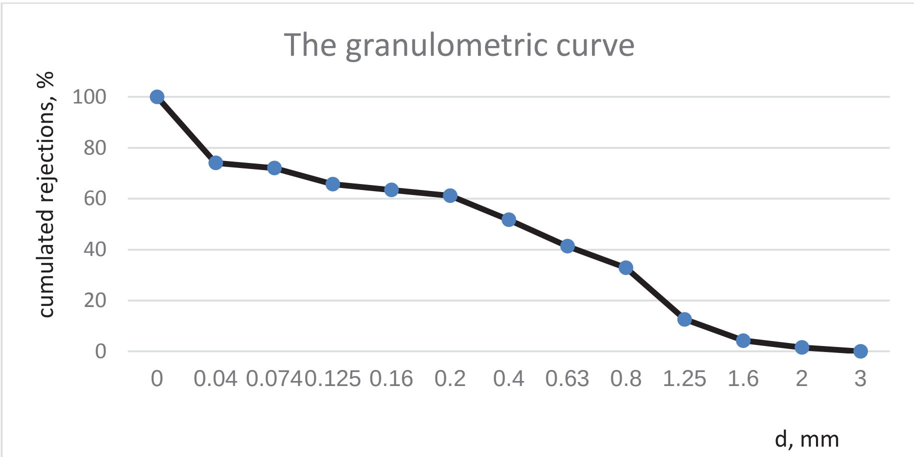


**Fig.4.** *Granulometric curve on a representative sample*

### *3.2. Laboratory research regarding the use of dolomite limestone in the household waste water treatment*

For the laboratory waste water treatment trials, three types of dolomite limestone were used: dolomite crude limestone, calcined dolomite limestone and magnesium oxide-rich concentrate. The granulometry of the material differed according to the type of material, as follows:

- The crude dolomitic limestone was crushed under 10 mm;
- Calcined dolomite limestone was crushed under 3 mm;
- Impure magnesium oxide was obtained by carbon dioxide solubilization of calcined dolomite limestone, having micronized dimensions. The content of magnesium and calcium oxides that will react with nitrate and phosphate ions in domestic waste water are increasing after calcination and $CO_2$ solubilization operations (fig.5).

Magnesium required for precipitation should not be added as a precipitating reagent. Research done at the University of Petrosani aims to use dolomite ($CaMgCO_3$) to generate the necessary magnesium ion. Magnesium dissolving occurs only to a small extent (R. Sârbu, 2015)

The solubility product allows a magnesium solubility of 0.005 mol / l. Magnesium dissolves in greater proportion as a result of the reaction with carbon dioxide:

$$Ps = [Mg^{2+}]\cdot[CO_3^{+2}] = 2{,}6\cdot10^{-5}$$

$$Mg(CO_3) + CO_2 + H_2O = Mg(HCO_3)_2$$

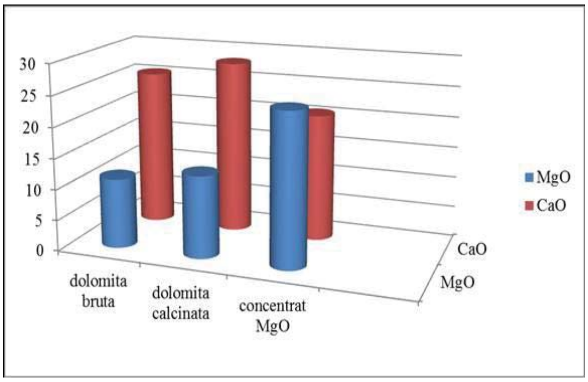


**Fig.5.** *The variation of MgO and CaO (%) contents from the three material types*

The struvite formed in this reaction, in the case it is not contaminated by heavy metals, can be used successfully as a chemical fertilizer with phosphorus and ammonium.

Two types of household waste water taken from the 2nd stage effluent from the Glina Waste Water Treatment Plant were used for rinsing tests.

The conditioning of the domestic waste water with the three types of magnesium mineral component was done at 10 minutes, after which the two products were obtained by filtration: purified water and solid material. After the pre-conditioning phase, the solutions obtained were filtered and then subjected to chemical analysis.

Besides the above-mentioned aspect, through conditioning, with magnesium minerals, the noticeable odor of the domestic waste water predominantly from the nitrogen-containing nutrient content has been significantly reduced.

Laboratory tests were performed according to the scheme shown in figure 6. The results of the chemical analyzes performed on the used and obtained products are summarized in table 5.

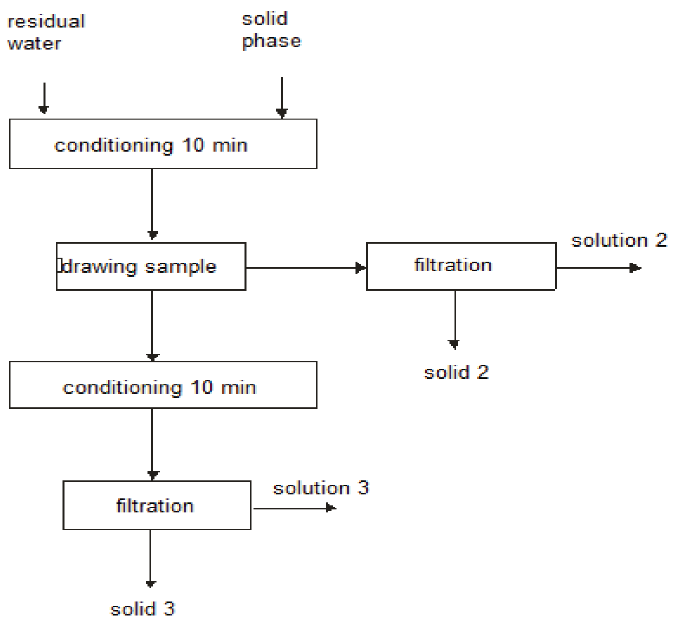


**Fig.6.** *The flow sheet of the laboratory research*

**Table 5.** *Results using raw, roasted dolomite and magnesia*

| **Mineral: raw dolomite** | | | | | |
|---|---|---|---|---|---|
| Contents, % | Mg | Ca | $NO_3^-$ | $PO_4^{-3}$ | CCOMn |
| Solution feed (S1) | 2,12 | 39,83 | 1,2 | 23,77 | 14,147 |
| Solution 2 (S 2) | 19,5 | 29,1 | sld | 9,1 | |
| Solution 3 (S3) | 22,4 | 22,84 | sld | 7,85 | |
| | | | | | |
| Contents, % | Mg | MgO | Ca | CaO | |
| Solid feed (1) | 6,83 | 11,32 | 25,46 | 35,62 | |
| Solid (2) | 19,92 | 33,02 | 15,9 | 22,24 | |
| Solid (3) | 18,87 | 31,28 | 16,38 | 22,91 | |
| **Mineral: roasted dolomite** | | | | | |
| Contents, % | Mg | Ca | $NO_3^-$ | $PO_4^{-3}$ | CCOMn |
| Solution feed (S1) | 2,12 | 39,83 | 1,2 | 23,77 | 14,147 |
| Solution 2 (S 2) | 24,85 | 17,17 | sld | 6,8 | |
| Solution 3 (S3) | 7,1 | 16,1 | sld | 3,1 | |
| | | | | | |
| Contents, % | Mg | MgO | Ca | CaO | |
| Solid feed (1) | 8,03 | 13,31 | 28,13 | 39,36 | |
| Solid (2) | 18,45 | 30,58 | 16,26 | 22,75 | |
| Solid (3) | 18,25 | 30,25 | 15,87 | 22,2 | |

| Mineral: magnesia | | | | | |
|---|---|---|---|---|---|
| Contents, % | Mg | Ca | $NO_3^-$ | $PO_4^{-3}$ | CCOMn |
| Solution feed (S1) | 2,12 | 39,83 | 1,2 | 23,77 | 14,147 |
| Solution 2 (S 2) | 0,03 | 130,5 | sld | 4,13 | |
| Solution 3 (S3) | 0,13 | 13,63 | sld | 2,78 | |
| | | | | | |
| Contents, % | Mg | MgO | Ca | CaO | |
| Solid feed (1) | 14,9 | 24,7 | 20,65 | 28,89 | |
| Solid (2) | 15,08 | 25 | 17,28 | 24,18 | |
| Solid (3) | 10,53 | 17,45 | 20 | 27,98 | |

From the results of the chemical analyzes carried out on the solutions and products of the mineral materials used, the following facts can be concluded:

1. The content of nitrogenous and phosphate ions in the household raw water decreases significantly after 10 minutes (solution 2), through conditioning it with the magnesia purification products, as can be seen in figures 7 and 8;
2. Significant differences are found between the three types of mineral materials used, dolomite limestone calcined at 650oC and MgO-rich concentrate resulting in higher net results over the same time period of use. It is obvious that the Mg ions are released by the calcination operation to reduce the phosphate and nitrate ions to the limit for the evacuated effluent 5 mg / l for PO43- and 1 mg / l for NO3-.

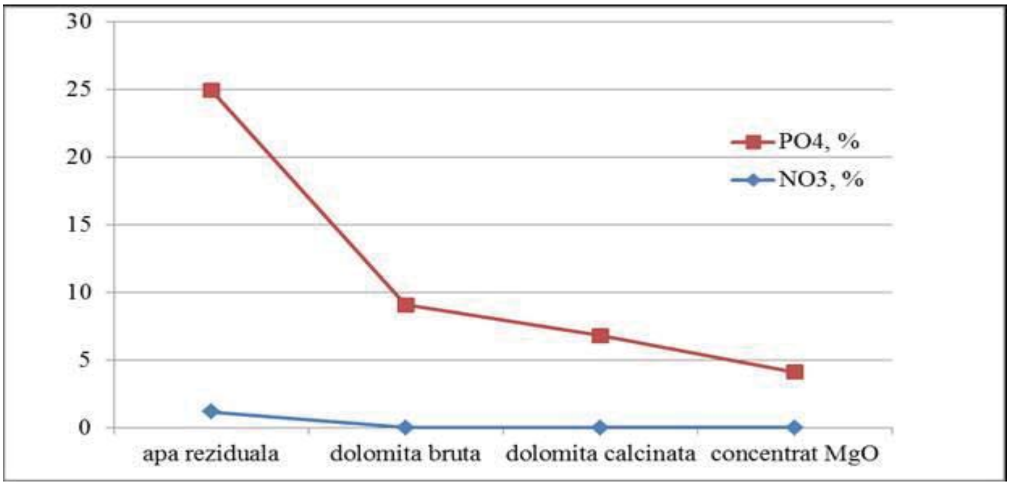


**Fig.7.** *Variation of nitrate and phosphate ion contents in waste water (mg / l) treated with different types of magnesium mineral substances (solution 2)*

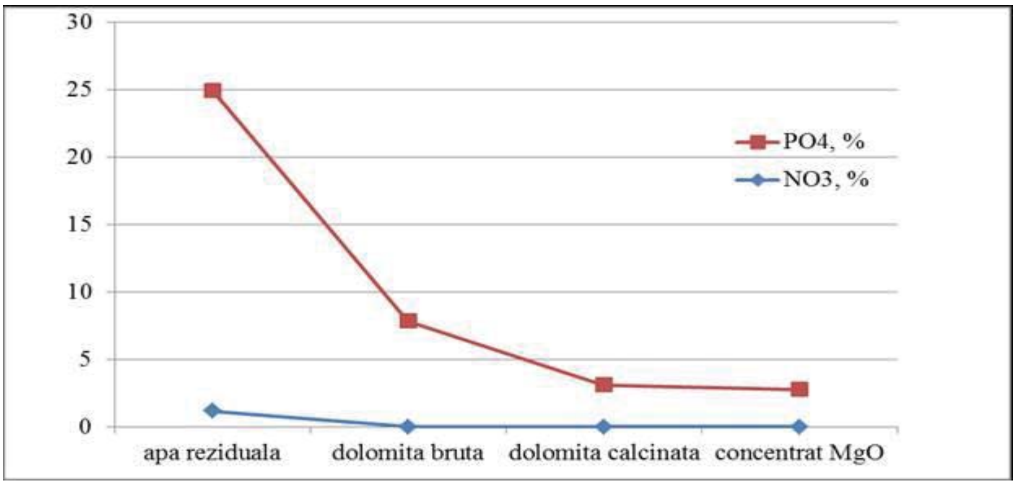


**Fig.8.** *Variation of nitrate and phosphate ion contents in wastewater (mg / l) treated with different types of magnesium mineral substances (solution 3)*

## 4. Conclusions

Dolomite, in all its natural forms of presentation in nature, is an economically attractive mineral, because the multiple areas of use impose it on the market, and the recovery technologies are not very complicated and require no major investments, unless extraction is desired of dolomite of metallic magnesium.

Dolomite is enriched by the presence of brucite (magnesium hydroxide), which, due to its special properties, leads to an increase in the content of MgO reactive, which determines the widening of the range of uses, both in industry, but also in agriculture or environmental protection, turning dolomite with brucite into a universal panacea to protect the environment.

Budureasa, Bihor County displays the largest limestone quarry in Europe, estimated at about 110 million tons, with MgO content ranging between 20-25%, which is the subject of this research.

Following the household wastewater treatment tests from Glina Station near Bucharest, using three limestone varieties, it was found that the nitrogen and phosphate ions in the raw domestic water fall below the permissible limit for discharge into the emissary.

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