Prospects for Effective Use of Dolomite in Concrete Compositions

Viktor Vaganov¹, Andrey Kireev², Sergey Avdeev³, Genādijs Šahmenko⁴, Māris Šinka⁵

¹ Institute of Structural Macrokineatics and Materials Science RAS
², ³ Vladimir State University, ⁴, ⁵ Riga Technical University

Abstract – The article discusses possibilities for creating sustainable concrete compositions from local dolomite raw materials. In the first part of the article, practical examples of the use of dolomite quarry by-products (siftings) as a component of concrete mixes are summarized. The second part of article presents experimental study on obtaining caustic dolomite. This method allows obtaining the product with higher added value. Local dolomite raw materials are used and appropriate regimes of thermal treatment have been tested.

Keywords – Dolomite, magnesia cement, sustainable binding material, X-ray test.

I. Introduction

Dolomite is a widespread sedimentary rock in the flatland part of Europe. It is commonly used in road and building construction for foundation bases and as an aggregate for cement and asphalt concrete. It is considered that dolomite has reduced mechanical properties and reduced resistance against freeze-thaw cycles, comparing to magmatic rock. Therefore, building regulations considerably restrict the use of dolomite rock for concrete exposed to freezing and thawing cycles. At the same time, dolomite aggregate has some benefits. A lot of studies confirm the formation of better contact zone cement paste – aggregate in the case of carbonate aggregate.

Monteiro and Mehta concluded that a hydrated calcium carbonate-calcium hydroxide compound is formed at the interface and formation of smaller calcium hydroxide crystals appears to be responsible for the strengthening of the transition zone [1].

The authors [2] studied the contact zone between cement paste and major concrete minerals (field spar, calcite, quartz, opal and chaledony). It was found that all the mentioned minerals, except for quartz, begin interacting chemically with the cement paste, which causes weakening of the contact zone of cement paste.

Kayyali [3] confirmed formation of good contact zone between cement paste and carbonate rock.

The benefits of the use of magmatic rocks as concrete aggregate are arguable. The main problem of magmatic rock is alkali–silica reactions between silica aggregate and alcalines presented in cement [4].

Dolomite is a cheap and locally available material in the regions where magmatic rocks are unavailable. A lot of experimental and practical results confirm possibilities to produce excellent concrete compositions from locally available dolomite.

Other perspective application of dolomite rock is obtaining dolomite lime and magnesium binder (caustic dolomite) as binding materials for mortar and concrete. Magnesium binder has a lot of advantages, specifically it provides good contact zone wits organic natural fiber aggregate.

The first part of this article presents a review of the works devoted to the use of local dolomite resources as filler in different concrete mixtures as well as to the application of dolomite lime in natural fiber composites. The second part of the article discusses application potential of dolomite lime and some experimental results on technology of producing magnesium cement from local dolomite rocks. Thereby, the objectives of this research are to find the most effective and appropriate ways for utilizing local dolomite.

II. Review of Use of Local Dolomite as Component of Concrete Mixtures

There is a significant amount of small dolomite fractions remaining as the secondary product of dolomite quarries. The problem of its rational utilisation has not been solved yet. The secondary product of dolomite quarries is a multi-fractional mix (particle size range from 0.1 mm up to 8 mm) – see Fig. 1. It may contain up to 30 % of fine and ultra-fine particles (<100 μm), which may be used as a micro-filler. It should be noted that micro-filler is an important component of modern concrete mixtures. The first function of micro filler is preventing segregation in highly workable mixes [5]. The second function is filling micro voids creating more dense microstructure and increasing impermeability [6].

Fig. 1. Unfractionated dolomite by-product (left) and fractions after sieve size analysis (right).

There have been a lot of efforts to incorporate secondary dolomite raw materials in the self-compacting concrete (SCC) as a micro-filler. Positive results were obtained by researchers Rudžionis et al. [7]. Previous studies done by authors [8] show that replacing traditional quartz sand by dolomite sand slightly increases water absorption, but in spite of this fact, resistance against freeze-thaw cycles up to F150 is achieved in all cases.

Also dolomite by-product has been tested in the lightweight aggregate concrete (LWAC) compositions. This type of LWAC compositions is presented in cement [4].

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consists of cement, silica sand and expanded clay (exclay aggregate), the most popular lightweight aggregate. In these experiments natural sand has been replaced by dolomite sand containing fractions smaller than 8 mm and ultra-fine particles <0.1 mm. LWAC based on dolomite sand insures the same mechanical properties and water impermeability as LWAC prepared with natural sand. Compressive strength close to 25 MPa is achieved for both compositions. Furthermore, the incorporation of dolomite by-products improved the workability and stability of the produced mixes [9].

Lightweight aggregate concrete blocks find wide application in modern building construction, mainly as a wall material. The most popular material is exclay blocks, which are characterized by highly porous structure and are produced using vibro-pressing technology. In accordance with [10], silica sand may be replaced by secondary dolomite sand in vibro-pressed LWAC blocks. The obtained blocks with dolomite sand are characterized by the same compressive strength; density reduction effect up to 45 kg/m³ was achieved.

Summarizing the above mentioned research, it may be noted that angular shape and porous surface of secondary dolomite aggregate particles is a limiting factor regarding mix flowability. It may be compensated by the use of appropriate plasticizers and silicate fume admixtures. Another serious drawback of the secondary dolomite aggregate is heterogeneity of the mix and particle agglomeration during exposed storing. It may be compensated using intensive mixing technology. Other method of dolomite deep treatment included its thermal processing and further grinding.

III. Dolomite Lime and Magnesia as Binding Agents (Review)

Producing a binding agent is an alternative way for rational use of dolomite resources. This method envisions thermal treatment of dolomite raw materials. Depending on the temperature of thermal treatment, different kinds of binding agents may be obtained.

Thermal decomposition of dolomite takes place in the temperature range of 700 – 900 °C, and thermograms show two peaks of endothermic effects, consequently, the process takes place in two stages. In the first stage of dissociation MgO and CaCO₃ are formed:

$$\text{CaMg(CO}_3\text{)}_2 = \text{MgO} + \text{CaCO}_3 + \text{CO}_2 - Q \quad (1)$$

and CaO in the second stage:

$$\text{CaCO}_3 = \text{CaO} + \text{CO}_2 - Q \quad (2)$$

Moreover, contradictory data on dolomite calcining reported in the literature appears to refer mainly to different compositions of raw materials and modes of calcining. According to [11], endothermic effect corresponding to decomposition of magnesia component appears within the temperature range 720 – 760 °C, and of calcium one – within the range 895 – 910 °C. Based on [12], the main peaks of dolomite decomposition endoeffects are observed at 810 °C and 900 °C, in addition, partial overlapping of dissociation processes was recorded.

Dolomite lime production process takes place at the temperature close to 1000 °C, providing full decomposition and formation of calcium and magnesium oxides. Dolomite lime hardening is based mainly on carbonation of calcium hydroxide, like in the natural lime. Magnesium oxide in this case is over-burned and non-reactive (so called “dead” magnesia oxide). At the same time, some kinds of dolomite lime show much better strength properties comparing to lime. This effect may be explained by the presence of hydraulic admixtures when coal fuel is used.

In the previous experiments, compressive strength of dolomite lime mortar samples has been compared to various types of air lime [13], summarized in Table I. To enhance the compressive strength and to make the limes more hydraulic a pozzolanic additive metakaolin was added. Experiments were carried out on mortar samples when sufficient amount of amorphous SiO₂ material was added to hydrated lime. In presence of water it creates calcium silicate hydrate or C-S-H:

$$\text{Ca(OH)}_2 + \text{H}_2\text{SiO}_4 \rightarrow \text{CaH}_2\text{SiO}_4 + 2 \text{H}_2\text{O} \quad (3)$$

Mix compositions of lime with metakaolin are summarized in Table II. For each type of lime, four different proportions of metakaolin were added – 0 %, 5 %, 10 % and 20 %. and ratio is 1 : 2.

$$\text{C}_0\text{L} 90\text{C}_0\text{L} 90\text{C}_0\text{L} 90\text{C}_0\text{L} 90$$

| Sample name | Lime type | Lime, g | Meta, g | Sand, g | Water, g | Hydr. Add, % |
|-------------|-----------|---------|---------|---------|----------|-------------|
| C0 | CL90 | 300 | 0 | 600 | 266 | 0 % |
| C1 | CL90 | 285 | 15 | 600 | 266 | 5.0 % |
| C2 | CL90 | 270 | 30 | 600 | 266 | 10.0 % |
| C3 | CL90 | 240 | 60 | 600 | 266 | 20.0 % |
| C4 | CL80 | 300 | 0 | 600 | 300 | 0 % |
| C5 | CL80 | 285 | 15 | 600 | 300 | 5.0 % |
| C6 | CL80 | 270 | 30 | 600 | 300 | 10.0 % |
| C7 | CL80 | 240 | 60 | 600 | 300 | 20.0 % |
| C8 | DL60 | 300 | 0 | 600 | 200 | 0 % |
| C9 | DL60 | 285 | 15 | 600 | 200 | 5.0 % |
| C10 | DL60 | 270 | 30 | 600 | 200 | 10.0 % |
| C11 | DL60 | 240 | 60 | 600 | 200 | 20.0 % |
The results seen in Fig. 2 show that addition of metakaolin significantly increases compressive strength of all types of lime. For CL90 20 % of metakaolin increased the strength by 3.37 MPa, for CL80 – by 1.72 MPa and for DL60 – by 4.27 MPa. Initial strength is also higher for DL60, which is explained by the admixtures from the coal fuel used.

Fig. 2. Strength results of various air limes with metakaolin.

In modern construction, such limes with enhanced strength are used as binders for natural fiber composites, mainly for lime-hemp concrete (LHC). These materials are made from hemp shives that are by-products of hemp-fibre production; they act as a filler and hydraulic lime as a binder. They are used as self bearing wall insulation materials that are used together with load-bearing timber frame. They have good thermal insulation capabilities (around 0.07 W/m·K) [14], high water vapour permeability, good acoustical and fire resistance properties [15].

LHC materials also have negative CO₂ balance [16], which means that these materials sequester more CO₂ than is released in its production. There are two main contributors to this CO₂ absorption – hemp stalks sequester carbon dioxide through photosynthesis and hydrated lime in the binder hardens by carbonisation [17]. This is one of the reasons why lime and dolomitic lime are used in these materials. Lime also contributes to water vapour permeability, fire resistance properties and improves the resistance to bio-degradation.

IV. Production of Magnesia Binder from Dolomite

It is known that dolomite decomposition takes place within the temperature range of 700 – 900 °C, where thermograms normally show two peaks of endothermic effects, consequently the process takes place in two stages. In the first stage of dissociation MgO and CaCO₃ are formed:

$$\text{CaMg(CO}_3\text{)}_2 = \text{MgO} + \text{CaCO}_3 + \text{CO}_2 - Q$$

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Moreover, contradictory data on dolomite calcining reported in the literature appears to refer mainly to different compositions of raw materials and modes of calcining. According to [11], endothermic effect corresponding to decomposition of magnesia component appears within the temperature range 720 – 760 °C, and of calcium one – within the range 895 – 910 °C. Based on [12], the main peaks of dolomite decomposition endoeffects are observed at 810 °C and 900 °C, in addition, partial overlapping of dissociation processes was recorded.

Multiple subsequent studies [18], [19], [20], [21] report that the main problem of binder production from dolomite is absence of sharp boundary between decomposition of CaCO₃ and MgCO₃. All authors agree upon negative effect of calcium oxide, leading to decrease of strength and water resistance of the forming rock. Thus, to obtain caustic dolomite, calcining should be performed so that the product would contain as much MgO as possible and not more than 1 – 2 % CaO, which is highly complicated.

Existing literature data analysis allows concluding that:

- it is possible to produce a high-strength binder from dolomite rocks. At the same time, widespread occurrence of dolomite rocks that allows avoiding significant transportation costs and comparatively low temperature of calcining make production of binder from dolomite a promising solution for construction industry;
- it is necessary to search for opportunities to control calcining processes to ensure that minimal decomposition of calcium component occurs at most complete decarbonization of magnesia component. Analysis of literature shows that depending on rock deposits, due to differences in chemical composition, original rock impurities, dolomite crystallization, calcined rock size distribution and other factors, both calcining temperature range and the needed isothermal exposure times vary.

V. Materials and Methods

The mineralogical composition was determined by X ray diffraction methods using “D8 Advance” diffractometer (Bruker AXS, Germany) under the following survey conditions: an X-ray tube – copper (Cu Kα-radiation), with a nickel filter. The X-ray tube voltage is 40 kV, current – 40 mA, exposure – 1.1 hour, sample diameter – 10 mm; goniometer rotation rate – 4 deg/min; time constant – 1.0 sec; impulse counting rate – 1·10⁴ imp./sec.

The scope of this research included comparative analysis of specifics of magnesia binder production from dolomite residues of two quarries in the Vladimir Region – Andreyevsky (the Sudogodsk Region) and Melehovsky (The Kovrov Region) further referred to as (A) and (K) respectively.

The mineralogical composition of the raw material of two quarries is given in Table III.

Technical magnesia chloride (bishofite MgCl₂·6H₂O) was used as the grouting fluid.

To obtain magnesia binder and to determine optimal calcining temperatures that provide a maximum yield of MgO phase, at the minimum yield of CaO phase, dolomite powder was calcined during 1 hour in the kiln at the temperature of 600 °C – 900 °C. Also the effect of calcining duration was investigated at the temperatures 780 °C, 820 °C and 840 °C. An example of phase composition of the calcined dolomite is presented in Fig. 3.
TABLE III
MINERALOGICAL COMPOSITION OF USED DOLOMITE SAMPLES

| Compound name | Chemical formula | Space group | Y-Scale (%) | S-Q (%) |
|---------------|------------------|-------------|-------------|---------|
| Dolomite      | CaMg(CO₃)₂       | R-3 (148)   | 89.83       | 98.51   |
| Quartz        | SiO₂             | P3221 (154) | 4.64        | 0.99    |
| Calcite       | CaCO₃            | R-3c (167)  | –           | 3.91    |

The summarized X-ray test results for calcined samples at various temperatures and exposure times are presented in Table IV.

Based on the data presented in Table IV, the following conclusions can be made:

• the maximum possible yield of MgO phase that forms during calcining of the dolomite taken directly from different quarries (crushing residues) does not exceed 30–35%.
• the calcining temperature range letting MgO form most actively lies in the range 800 – 900 °C for the tested materials;
• the maximum amount of CaO phase is formed in the same temperature range, the maximum yield is recorded in the range around 900 °C;
• fineness of pre-ground dolomite does not have significant influence on the temperature range producing the maximum MgO yields.

TABLE IV
INFLUENCE OF CALCINING REGIME ON BINDER CHEMICAL COMPOSITION (SUMMARY)

|       | MgO  | CaO  | CaCO₃ | SiO₂  | CaMg(CO₃)₂ |
|-------|------|------|-------|-------|------------|
| 600 °C|      |      |       |       |            |
| 1 hour|      |      |       |       |            |
| coarse| 0.74 | 1.16 | 98.10 |
| middle| 0.37 | 2.35 | 97.28 |
| fine  | 0.46 | 1.76 | 97.45 |
| 700 °C|      |      |       |       |            |
| coarse| 3.6  | 5.7  | 89.57 |
| fine  | 5.26 | 3.41 | 88.04 |
| 800 °C|      |      |       |       |            |
| coarse| 30.02| 39.77| 7.45  |
| fine  | 30.45| 46.72| 4.09  |
| 900 °C|      |      |       |       |            |
| coarse| 33.89| 64.21| 1.9   |
| fine  | 33.74| 59.06| 1.89  |

| 750 °C|      |      |       |       |            |
| 10 min.| 4.16 | 2.66 | 92.56 |
| 20 min.| 5.31 | 2.22 | 91.24 |
| 30 min.| 5.85 | 2.21 | 90.75 |

| 780 °C|      |      |       |       |            |
| 30 min.| 15.26| 4.16 | 64.09 |
| 60 min.| 8.34 | 11.05| 64.18 |
| 80 min.| 25.52| 25.89| 49.14 |
| 820 °C|      |      |       |       |            |
| 10 min.| 15.63| 22.67| 35.3  |
| 30 min.| 25.52| 25.89| 49.14 |
| 60 min.| 33.78| 29.03| 64.57 |
| 840 °C|      |      |       |       |            |
| 30 min.| 29.79| 28.1 | 64.77 |

Fig. 3. Results of X-Ray analysis of dolomite sample (No. A) calcined at 800 °C during 60 minutes.
To determine calcining temperature intervals more accurately, thermal tests with use of thermal analysis techniques were conducted: thermogravimetric analysis, differential scanning calorimetry and differential thermal analysis.

Fig. 4 and Fig. 5 show thermal analysis data for dolomite samples from two quarries, which demonstrate that decomposition temperatures of magnesium and calcium components are found to be located close (811 and 841 °C for the Kovrov quarry and coincide to those of the Andreyev quarry (810 °C).

![Fig. 4. K Sample TGA. Green curve – TG. Brown curve – DTG. Residual mass 55.2 % masses.](image1)

![Fig. 5. A Sample TGA. Green curve – TG. Red curve – DTG. Residual mass 57.8 % masses.](image2)

Two peaks at 453 and 509 °C possibly refer to sample dehydration. Furthermore, there is a chance of the main TG curve drift due to structural heterogeneity of the sample. The sample is represented by a set of hard fine particles (not powder) and that is why primary decomposition of these particles is accompanied by mechanic destruction and mass loss curve drift.

The study of the microstructure of the forming cement paste during hydration was done with help of raster electron microscopy equipped with local chemical analysis add-on.

Fig. 6 (a) shows typical structure, resulting from hydration of magnesia binder, consisting of individual micro-sized particles of Mg(OH)2 that form dense conglomerates, their zoomed image (arrow-marked) is shown in Fig. 6 (b). Furthermore, in certain areas that make 30–40 % of the total amount acicular crystals of magnesia oxychlorides have been identified.

![Fig. 6 (a). Typical micro structure of hydrated magnesium cement.](image3)

![Fig. 6 (b). Acicular crystals of magnesia oxychlorides have been identified.](image4)

Sedimentary rock dolomite nowadays is the basic source of coarse aggregate in building construction. Complex approach to rational use of dolomite resources provides for complete use of by-products of dolomite quarries. The first observed direction is the use of dolomite fines as a fine component in modern concrete, it is confirmed that by-products of dolomite quarry (fine fractions) may be successfully used as micro-filler in self-compacted concrete, allowing to achieve high (up to S3 flow class) workable mix. They can be also used as effective micro-filler in lightweight aggregate concrete and exlay concrete wall blocks.

The second observed direction is obtaining caustic dolomite for binding agent in magnesium binders. It has been concluded that calcining duration and temperature are important factors of dolomite preparation.

Rational use of dolomite resources very closely corresponds to the conception of sustainability and life cycle of building materials.

VI. CONCLUSION
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Viktor Vaganov, Doctor of Technical Sciences, senior research associate at the Institute of Structural Macrometals and Materials Science of the Russian Academy of Sciences (2013), Director of the Centre of Carbon Nanomaterials of the Vladimir State University (2008), Author of more than 100 research papers. Field of research: structural materials science. Address: 87 Gorkogo Street, Vladimir, Russia E-mail: vaganov.viktor-1953@yandex.ru

Andrey Kireev, research engineer of the Vladimir State University, research associate at the laboratory “X-ray Diagnostics of Materials” of VLGU. Graduate of the Vladimir State University (2009). Author and coauthor of 30 scientific works. Fields of research: anode materials, protection against corrosion, x-ray methods of analysis of materials. Address: 87 Gorkogo Street, Vladimir, Russia E-mail: ariant-tp@gmail.ru

Sergey Avdeev, Candidate of Technical Sciences, Vladimir State University, Director of the Institute of Construction Architecture and Power Engineering (2016). Author of 20 scientific works. Field of research: Efficient building constructions. Address: 87 Gorkogo Street, Vladimir, Russia E-mail: asf_vlgui@mail.ru

Genadijs Šahmenko, Dr. sc. ing., Leading Researcher, Institute of Materials and Structures, Riga Technical University. Fields of research: High performance concrete, ecological building materials, foam concrete, non-destructive testing. Address: Kalju iela 1, Riga, LV-1658, Latvia E-mail: genadijs.sahmenko@rtu.lv

Māris Šinka, PhD student, Institute of Materials and Structures, Riga Technical University. Fields of research: Natural and ecological building materials, life cycle. Address: Kalju iela 1, Riga, LV-1658, Latvia E-mail: marris.sinka@rtu.lv