Lightweight concrete with expanded aggregate from siliceous rocks

L V Zakrevskaya¹, I A Gandelsman¹, A A Gavrilenko¹ and P A Lubin¹

¹Department of Construction Industry, Vladimir State University, Vladimir, 600000, Russia
E-mail: lvzak@mail.ru

Abstract. The purpose of this study is to develop technology for the production of a new building composite made of silicon-containing rocks. An aggregate is foamed granules obtained on the basis of diatomite with the addition of caustic soda, dolomite, water and liquid glass. The material composition, materials processing, physical and mechanical properties have been observed. The problem of alkali-silicate interaction typical for traditional Portland cement and glass phase is solved through the use of magnesia binder. Samples on Portland cement and magnesia binder were obtained, the aggregate content was varied. Some aggregate fractions were used to obtain high packing density in the composite. Concrete was estimated according to density, compressive strength and tensile strength, water absorption and thermal conductivity. Analysis of the material structure was made. The results of the study of the microstructure of synthesized concrete indicate the presence of a cement matrix in which granules of various sizes are tightly packed. The results of X-ray phase analysis of synthesized concrete indicates that the cement matrix consists of forsterite crystals. Approximate savings in the use of concrete with the developed aggregate is about 10-20%. Such savings are possible due to the fact the surface occurrence of siliceous rocks allows them to be mined in an open way with minimal costs compared to other rocks, with a lower calcination temperature during material creation.

1. Introduction
The increased need for lightweight concrete sets the task of expanding its range on the basis of natural resources with improved thermal and structural properties.

The demand for high-quality building materials is growing every year. The most important performance indicators of building materials are strength, durability, environmental friendliness, reliability and cost.

In accordance with the Federal Law # 261-FZ “On Energy Saving and Increasing Energy Efficiency and Amendments to Certain Legislative Acts of the Russian Federation” special attention should be paid to thermal insulation of buildings.

In recent years monolithic construction in which lightweight concrete structures perform heat-insulating and enclosing functions is gaining popularity.

In modern literature this topic is represented by some studies that confirm the effectiveness of the use of diatomite rocks to create lightweight aggregates for concrete. Despite all the existing studies the potential of this rock is not fully disclosed and needs further study [1,2].

The main objectives of the study are: study of physical and mechanical properties of granular thermal insulation material, obtained on the basis of silica raw materials, and development of concrete compositions with high performance indicators.
Many technical and operational characteristics of concrete depend on the type and properties of the aggregate. Expanded clay is “the main field of activity” which worked out new approaches to the design of lightweight concrete mixtures on porous aggregates over the past decades [3].

Some scientific papers on this topic confirm the fact that the strength of concrete directly correlates with the strength of porous aggregate granules. However studies of the properties of the “aggregate-cement matrix” zone show that the nature of the bond between the aggregate and the cement matrix strongly affects both the strength and other properties of lightweight concrete [4,5,6].

Siliceous opal-cristobalite rocks, the reserves of which Russia ranks first in the world, belong to multi-purpose raw materials. The use of these rocks in construction as well as their origin and distribution has been studied in many papers.

Siliceous rocks are known to be light fine-pored powders composed of the smallest opal fragments of diatoms and cristobalite, as well as clay minerals [5,7].

The main and most common diatomite rocks are: diatomite and tripoli (Figure 1, 2) [7].

![Figure 1. Diatomite.](image1)

![Figure 2. Tripoli.](image2)

Diatomites and tripoli (celite, rock flour, diatomaceous earth, infusorial earth) are light fine-porous rocks. Diatomites are composed of the remains of diatomite algae (60-80%) and have a typically organogenic structure. They are loosely cemented, characterized by grayish-yellow color, very high porosity (20-84%) and low density (0.35-1.6 g/cm3). Sometimes they have a microlayered structure. The age of diatomites is the Paleogene-Neogene and Quaternary. In Russia large deposits of diatomites are found in the Volga region, the Urals and Transcaucasia. There is the possibility of using diatomite rocks as reactive amorphous silica in the Vladimir region.

Tripolis are similar to diatomites externally and in composition. In the wet state tripolis are easily rubbed with fingers. Opal (75-80%) (Figure 3), clayey matter, quartz grains, glauconite, carbonates and occasionally residues of diatoms are detected under a microscope. The content of fractions is smaller than 0.01 mm - 36.2 - 46.2%. The specific gravity is equal to 2.4 kPa; the density varies from 0.45 to 1.4 g/cm3; the porosity varies from 41 to 81%. High porosity (especially closed porosity) leads to low thermal conductivity. Diatomite rocks are used as hydraulic additives for cements, raw materials for the production of bricks and lightweight materials for backfilling [8, 9, 10].

Table 1 shows the most important properties of diatomite rocks.

| Table 1. The physical properties of diatomite rocks. |
|-----------------------------------------------------|
| Density $\rho_0$, kg/m$^3$ | True density $\rho_{true}$, kg/m$^3$ | Porosity, % | Compressive strength, MPa | Mohs Hardness Scale | Thermal conductivity, W/(m·°C) |
| 350-1600 | 2000-2500 | 20-84 | 2-15 | 1-4 | 0.09-0.5 |
Some authors [9, 10, 11] state that the chemical composition of diatomites and tripolis is almost the same, but the microstructure is different. This is due to the difference in the geological age of the deposits. The chemical composition of diatomites is shown in Table 2.

**Table 2.** The chemical composition of diatomites and tripolis in the Vladimir region.

|          | SiO₂, mass% | Al₂O₃, mass% | Fe₂O₃, mass% | CaO, mass% | MgO, mass% | K₂O, mass% |
|----------|-------------|--------------|--------------|------------|------------|------------|
| Diatomites | 70 – 96     | 5 – 15       | 2 – 5        | 0,5 – 5    | 0,5 – 3    | 0,1 – 1,45 |
| Tripoli  | 66,2 – 84,7 | 2,7 – 18,6   | 0,2 – 5,6    | 0,24 – 22,1| 0,1 – 1,8  | 0,4 – 1,5  |

The phase composition of siliceous rocks is represented by three minerals: opal, cristobalite and quartz. The predominant mineral is opal, the content of which is usually 56-98%. In all siliceous rocks there is quartz, the content of which can vary from 5 to 35%. Cristobalite is almost absent in diatomites. In other varieties of siliceous rocks its content can reach 20%. Opal consists of extremely small particles or porous aggregates with the developed inner surface [12-15]. Scanning electron microscopy of the opal sample is shown in Figure 3 [16].

The aim of this study is to develop technologies for the production of a new building composite made of silicon-containing rocks. Low thermal conductivity and density with sufficient strength are the main advantages of the obtained aggregate. The problem of alkali-silicate interaction typical for traditional Portland cement and glass phase is solved through the use of magnesia binder.

### 2. Experimental

The following raw materials were used for the study:

- diatomite
- causticsoda
- water glass
- dolomite
• water

The obtained material received the working title “Diapen”.

“Diapen” is a building material of a new generation, synthesized from silica rocks with a porous, chemically stable structure and a raw material component for lightweight concrete.

Table 3 shows the composition of the raw material mixtures to obtain “Diapen”.

| Composition | Diatomite, mass% | Caustic soda, mass% | Waterglass, mass% | Water, mass% | Dolomite, mass% |
|-------------|------------------|---------------------|-------------------|-------------|----------------|
| C1          | 57               | 20                  | 12                | 5           | 6              |
| C2          | 60               | 20                  | 12                | 4           | 4              |
| C3          | 65               | 17                  | 9                 | 3           | 6              |
| C4          | 70               | 14                  | 6                 | 6           | 4              |
| C5          | 72               | 13                  | 5                 | 6           | 4              |

Preparation of raw materials and production of granules consisted of the following process steps:
• grinding diatomite
• dosage of main components
• adding caustic soda, water glass and water
• mixing ingredients
• production of granules by extruder
• swelling in the rotary kiln at temperature of 880 degrees

The appearance of the obtained aggregate granules is shown in Figure 4.

![Figure 4](image_url)

For the further research were chosen the composition C2 because of it has the less density and thermal conductivity in compare with the other ones.

Figure 5 shows the microstructure of “Diapen” granules, obtained by scanning electron microscopy. This microstructure allows to conclude that “Diapen” is a material with closed pores on the outer surface and developed porosity inside the granule.
Figure 5. The surface and internal structure of “Diapen” granules.

Mechanical and operational characteristics of “Diapen” were studied to assess the quality of the obtained material. The study of density, compressive strength in the cylinder and water absorption was carried out in accordance with GOST 9758-2012. Table 4 shows the measurement results of physical and mechanical properties of the porous aggregate.

Table 4. Properties of the porous aggregate.

| Fraction of the aggregate, mm | 1 – 5   | 5 – 20  |
|-------------------------------|---------|---------|
| Bulk density, kg/m³           | 295,10  | 191,90  |
| Compressive strength, MPa     | 3,77    | 2,66    |
| Water absorption, mass%       | 4,2     | 6,3     |
| Water absorption, volume%     | 6,2     | 6,5     |
| Thermal conductivity W/(m°C)  | 0,085   | 0,070   |

It is recommended to use magnesia binders because the obtained aggregate has a high content of silicates in its composition, representing the danger of alkali-silicate interaction in concrete with this aggregate[17-20]. To test the theoretical assumptions experimental samples of lightweight concrete were made on traditional Portland cement and magnesite cement. Table 5 shows the composition of lightweight concrete with different binders.
Table 5. Composition of lightweight concrete.

|                | Magnesia cement, mass% | Aggregate, mass% | Sand, mass% | Grouting fluid, mass% |
|----------------|------------------------|------------------|-------------|-----------------------|
| LB-C/M-1       | 13,43                  | 23,58            | 52,25       | 10,74                 |
| LB-C/M-2       | 16,44                  | 24,36            | 48,35       | 10,85                 |
| LB-C/M-3       | 19,49                  | 25,12            | 44,50       | 10,89                 |
|                |                        |                  |             |                       |
|                | Portland cement, mass% | Aggregate, mass% | Sand, mass% | Water, mass%          |
| LB-C/P-1       | 11,82                  | 23,58            | 38,48       | 26,12                 |
| LB-C/P-2       | 13,50                  | 24,36            | 40,00       | 22,14                 |
| LB-C/P-3       | 14,20                  | 25,12            | 42,50       | 18,18                 |

Concrete samples were taken from specially prepared laboratory mixes of concrete mix and tested. Samples were solidified for 28 dys at the air temperature of 20°C and relative humidity of 95% in accordance with GOST 18105-2010. Samples were tested and stored after hardening at the air temperature of 20°C and relative humidity of 55%.

The sand used in to produce concrete was middle-sized with a density of 1670 kg/m³. The binder was portland cement B42,5.

The density of concrete was determined in the air-dry state and according to GOST 12730.1. Compressive strength, flexural strength and tensile strength of samples were determined according to GOST 10180-2012. Determination of water absorption was carried out in accordance with GOST 12730.3-78. Determination of thermal conductivity was carried out in accordance with GOST 30256-94. Samples were tested using the MIT-1 device under normal conditions.

3. Results and discussion
Comparative characteristics of concrete on Portland cement and magnesia cement are shown in the Table 6.

Table 6. Results of property testing.

|                | LB-C/M-1 | LB-C/M-2 | LB-C/M-3 | LB-C/P-1 | LB-C/P-2 | LB-C/P-3 |
|----------------|----------|----------|----------|----------|----------|----------|
| Density, kg/m³ | 692      | 690      | 690      | 664,60   | 660,5    | 656,0    |
| Compressive strength, MPa | 8,76     | 9,00     | 9,00     | 5,15     | 5,20     | 5,15     |
| Water absorption, volume% | 10,8     | 10,0     | 10,5     | 11,3     | 8,7      | 8,9      |
| Thermal conductivity W/(m*C) | 0,315    | 0,320    | 0,328    | 0,291    | 0,25     | 0,24     |

The sample LB-C/M-2 was selected for the study of the structure and phase analysis. The results of the study of the microstructure of synthesized concrete shown in Figure 6, 7 indicate the presence of a cement matrix in which granules of various sizes are tightly packed. X-ray phase analysis of the sample is shown in Figure 8.
Figure 6. Cement matrix with forsterite crystals.

Figure 7. Small and large fractions, the densest packing.
4. Conclusion
One of the reasons for the high mechanical strength of “Diapen” and concrete with “Diapen” is the formation of needle-like crystals that create reinforcement cages throughout the concrete volume. We can see it in the study of the structure using scanning electron microscopy and X-ray phase analysis.

Test results convincingly showed that the developed material has good thermal insulation and strength properties that allow to produce high-quality material for enclosing and thermal insulation structures, especially for actively developing monolithic-frame and low-rise construction, as well as using the developed aggregate as loose-fill insulation.

It should be noted that the extraction of siliceous rocks in the Vladimir region is about 2 times cheaper than the extraction of raw materials for the production of expanded clay. Its cost (coupled with the lower firing temperature in the process of creating) is lower than the cost of production of expanded clay. The surface occurrence of siliceous rocks allows to be mined in an open way with minimal costs compared to other rocks used for similar purposes. Approximate savings in the use of concrete with the developed aggregate will be about 10-20%.

The evaluation of concrete with “Diapen” properties according to GOST 9758-86 and their comparison with analogues shows that the obtained material is not inferior to traditional building materials and can be recommended as a highly effective thermal insulation and thermal insulation structural material.

References
[1] Qian T, Li J, Ma H and Yang J 2016 Adjustable thermal property of polyethylene glycol/diatomite shape-stabilized composite phase change material Polym. Compos. 37, 854–860
[2] Karaman S, Karaçekli A, Sari A and Biger A 2011 Polyethylene glycol (PEG)/diatomite composite as a novel shape-stabilized phase change material for thermal energy
storage Sol. Energy Mater. Sol. Cells 95 1647–1653

[3] Zhang D, Zhou J, Wu K and Li Z 2005 Granular phase change composites for thermal energy storage Sol. Energy 78 351–480

[4] Yu S, Wang X and Wu D 2014 Microencapsulation of n-octadecane phase change material with calcium carbonate shell for enhancement of thermal conductivity and serving durability: synthesis, microstructure, and performance evaluation Appl. Energy 114 632–643

[5] Ling T and Poon C 2013 Use of phase change materials for thermal energy storage in concrete: an overview Constr. Build. Mater. 46 55–62

[6] Al-Deqs Y, Khraisheh M A M and Tutunji M 2001 Sorption of lead ions on diatomite and manganese oxides modified diatomite Water Res. 35 3724–3728

[7] Hadjar H 2008 Elaboration and characterisation of new mesoporous materials from diatomite and charcoal Micropor. Mesopor. Mat. 107 219–226

[8] Garderen N, Clemens F, Mezzomo M, Bergmann C and Graule T 2011 Investigation of clay content and sintering temperature on attrition resistance of highly porous diatomite based material Appl. Clay Sci. 52 115–121

[9] Li M, Kao H, Wu Z and Tan J 2011 Study on preparation and thermal property of binary fatty acid and the binary fatty acids/diatomite composite phase change materials Appl. Energy 88 1606–1612

[10] Xu B and Li Z 2013 Paraffin/diatomite composite phase change material incorporated cement-based composite for thermal energy storage Appl. Energy 105 229–237

[11] Wen R, Zhang X, Huang Z, Fang M, Liu Y, Wu X, Min X, Gao W and Huang S 2018 Preparation and Thermal Properties of Fatty acid/diatomite Form-Stable Composite Phase Change Material for Thermal Energy Storage Solar Energy Materials and Solar Cells 178 273-279

[12] Letelier V, Tarela E, Muñoz P and G Moriconi 2016 Assessment of the Mechanical Properties of a Concrete made by Reusing both: Brewery Spent Diatomite and Recycled Aggregates Constr. Building Mater. 114 492-498

[13] Wang R, Ren M, Gao X and Qin L 2018 Preparation and Properties of Fatty Acids Based Thermal Energy Storage Aggregate Concrete Constr. Building Mater. 165 1-10

[14] Qian T and J 2018 Octadecane/C-Decorated Diatomite Composite Phase Change Material with Enhanced Thermal Conductivity as Aggregate for Developing structural–functional Integrated Cement for Thermal Energy Storage. Energy 142 234-249

[15] Zhang G 2013 Microstructural modification of diatomite by acid treatment, high-speed shear, and ultrasound Micropor. Mesopor. Mater. 165, 106–112

[16] Jung K, Jang D and Ahn K 2014 A novel approach for improvement of purity and porosity in diatomite (diatomaceous earth) by applying an electric field Int. J. Miner. Process. 131 7–11

[17] Sun Z, Yang X, Zhang G, Zheng S and Frost R 2013 A novel method for purification of low grade diatomite powders in centrifugal fields Int. J. Miner. Process 125 18–26

[18] Zhang J, Ping Q W, Niu M, Shi H and Li N 2013 Kinetics and equilibrium studies from the methylene blue adsorption on diatomite treated with sodium hydroxide Appl. Clay Sci. 83–84 12–16

[19] Qin Y 2015 Sodium sulfate–diatomite composite materials for high temperature thermal energy storage Powder Technol. 282 37–42

[20] Cao L, Tang Y and Fang G 2015 Preparation and properties of shape-stabilized phase change materials based on fatty acid eutectics and cellulose composites for thermal energy storage Energy 80 98-103