# Determination of size properties of the organomineral insulation nanofiller based on the wood matrix

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**Abstract**. This article provides a comparative analysis of the results obtained in determining the particle size of the basalt-based wood-mineral insulation nanofiller using photon correlation spectroscopy in various dispersion media after mechanical milling of a raw material. Aqueous solutions with various concentrations of glycerine were used as diluents. The studies demonstrated that glycerine-based basalt suspensions exhibit sufficient sedimentation stability that produces reliable data on the nanofiller size properties. This is achieved due to such dispersion medium properties as density and permittivity. Furthermore, determinations of the zeta potential of the basalt particle surface in glycerine showed that this dispersion was electrostatically stabilized and thus would be aggregation-stable.

## 1. Introduction

Economic efficiency of individual processes and production as a whole in various industries greatly depends on the thermal insulation of process equipment. Due to this fact, thermal insulation is widely used, for example, in mechanical engineering, metallurgy, chemical and petrochemical industry, oil refining, pharmaceutical and food industry, etc [1-2]. It is worth noting that the organomineral composite produced on the basis of the wood matrix is currently one of the most efficient insulation materials due to its unique natural properties. Studies [3-5] demonstrated the possibility of producing a thermal insulation material on the basis of the composition consisting of the wood machining waste (sawdust, bark), pre-crushed up to a 1-2 μm size, with its pore volume saturated with basalt nanoparticles. The studies [5] found that the use of basalt nanoparticles as fillers of the wood polymer matrix allowed improving its performance properties. At a filling ratio of around 30-40%, heat resistance, mechanical and heat-conducting properties of a material are improved significantly.

One of the major problems in producing such nanocomposites is provision of interaction between the polymer and the filler. The effect of the nanodispersed filler on the polymer properties strongly depends on the distribution of particles in the polymer matrix. In the initial state, basalt particles may represent agglomerates ranging in size from a few microns to a few nanometers. In order to effectively modify polymer properties, it is necessary to provide dissolution of agglomerates and regular distribution of mineral nanoparticles in the polymer matrix. For this purpose, it is required to obtain reliable data on the modifier size properties at the first stage of composite production.

Determination of the particle size in finely-dispersed systems uses various methods [6, 7]. The most common ones include photon correlation spectroscopy based on the Rayleigh scattering law [8]. In this case, while experimentally implementing this method, it is important to analyze size properties

of finely-dispersed systems exhibiting sufficient sedimentation stability in a utilized dispersion medium. Consequently, when determining size properties of finely-dispersed basalt samples, account must be taken of not only the time factor, but also of the dispersion medium properties (density, permittivity) which affect sedimentation stability of analyzed objects.

This study aims to conduct a comparative analysis of results obtained in determining the basalt particle size using photon correlation spectroscopy in various dispersion media after mechanical milling of the raw material.

Sedimentation stability of solid particles in the suspensions analyzed was evaluated by the particle deposition rate ($\omega_0$, m/s) calculated on the basis of Stokes' law by the following expression [9]:

$$\omega_0 = \frac{d^2(\gamma - \gamma_1)g}{18\mu}, \quad (1)$$

where $d$ – a diameter of a deposited particle, m;
$\gamma$ – density of a deposited particle, kg/m$^3$;
$\gamma_1$ – density of a medium, kg/m$^3$;
$g$ – acceleration of gravity, m/s$^2$;
$\mu$ – dynamic viscosity of a medium, Pa·s.

## 2. Materials and methods

Basalt from the Myandukha deposit (Arkhangelsk region, Russia) was selected as the object of the study. This material has high strength, thermophysical and fire-resisting properties, and it is also very susceptible to mechanical dispersion with reproducible determinations of the particle size. In our opinion, aqueous solutions of glycerine are the most optimal choice among various diluents. This is due to the possibility of varying the density value in a water-glycerine system (from 1 to 1.26 g/cm$^3$) by only changing the component ratio. Control over the quantitative component ratio is realized by the refraction index through the refractometric method. Besides, glycerine is nonvolatile, optically inactive in the visible and ultraviolet spectrum, and highly miscible with water.

Basalt dispersing was carried out by mechanical wet milling in a planetary ball mill 'Retsch PM100', using large grinding bodies of 2 cm in diameter at a rotor speed of 420 rev/min for 30 min. The size and zeta potential of particles was determined with the analyzer 'Delsa Nano Series Zeta Potential' and 'Submicron Particle Size Analyzers (Delsa Nano)' through the dynamic light scattering measuring method. The true density of basalt particles was determined by the pycnometric method. The density of aqueous glycerine solutions was determined by the areometric method. The refraction index was determined by the refractometric method with the XRD unit. Dynamic viscosity was determined with the Brookfield viscometer.

## 3. Comparative analysis of results obtained in basalt particle size determination

According to the results of true density determination of finely-dispersed basalt test samples, the value of 2700 kg/m$^3$ was obtained, which was in good agreement with literature data.

Table 1 shows the results obtained in determining the properties of the dispersion medium, size and zeta potential of disperse phase particles.

Figures 1 and 2 show the graph of basalt particle size dependence on the time in utilized dispersion media. Deposition rate values calculated by equation (1) allowed obtaining a graph of dependence of the basalt particles deposition rate on their size (shown in Figure 3).

**Table 1.** Results of determining the glycerine aqueous solutions properties, size and zeta potential of basalt particles (at 20 °C)

| Glycerine % by weight | Density, g/cm$^3$ | Refraction Index | Viscosity, cP | Permittivity [10] | Zeta-potential, mV | Average particle size, nm |
|---|---|---|---|---|---|---|
| 0 | 1.000 | 1.333 | 1.005 | 80.37 | -2.25 | 207.7±56.7 |
| 50 | 1.130 | 1.398 | 6.00 | 65.63 | 298.34 | 108.5±28.7 |
| 100 | 1.260 | 1.474 | 1410 | 41.14 | 601.62 | 34.7±8.5 |

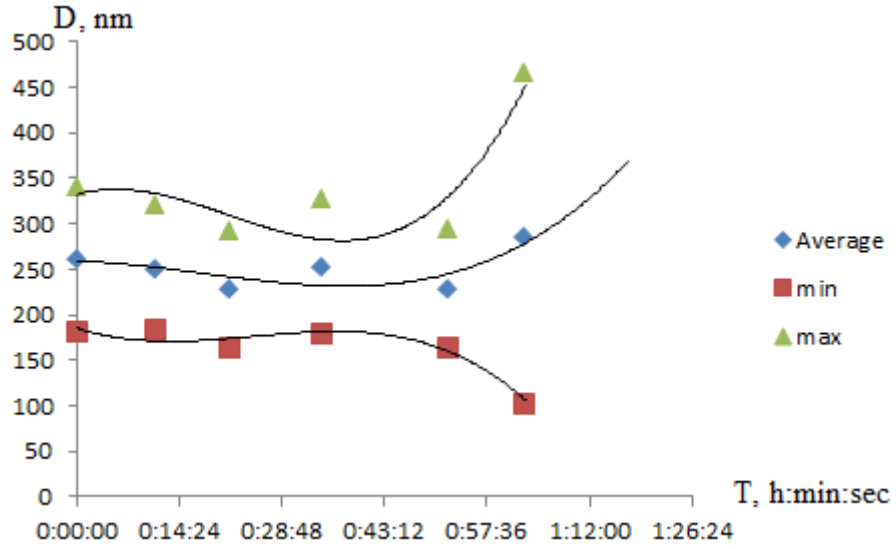


**Figure 1.** The graph of basalt particle size dependence on time in water

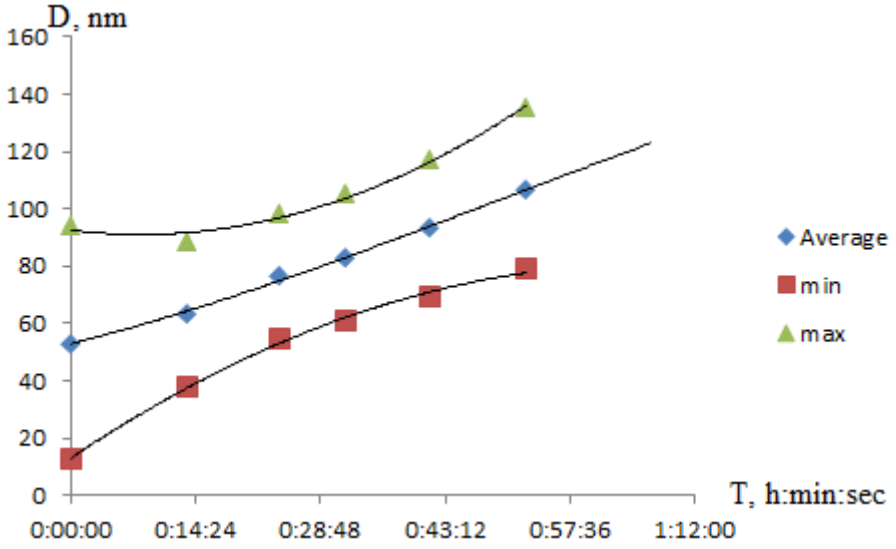


**Figure 2.** The graph of basalt particle size dependence on time in glycerine

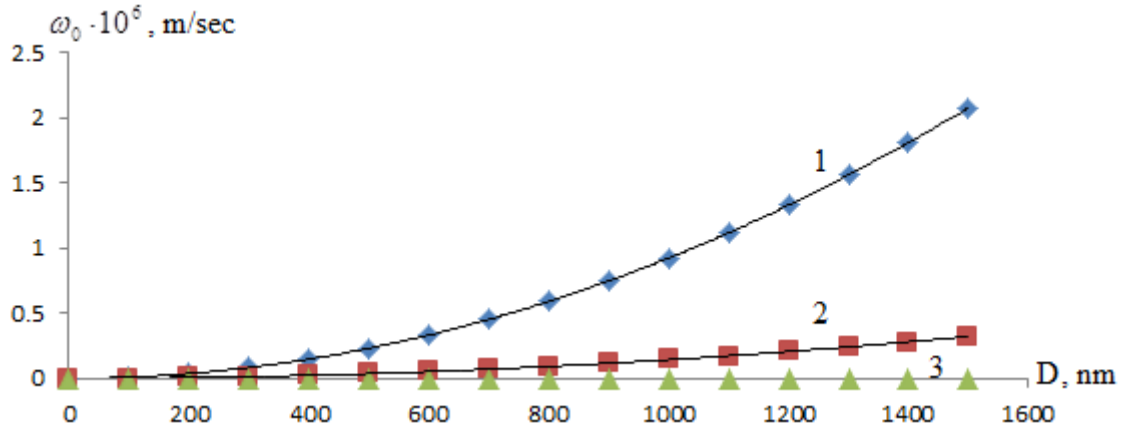


**Figure 3.** The graph of basalt particles deposition rate dependence on the diameter: 1 - in water; 2 – in the 50% aqueous solution of glycerine; 3 – in glycerine

When comparing the results of studies on the basalt particle size determination in various diluents, the following provisions can be distinguished:

1. Results of determining the basalt particles surface zeta potential in glycerine demonstrated that this dispersion was electrostatically stabilized ($\zeta >> 30$ mV), and thus would be aggregation-stable. In contrast, the basalt aqueous suspension is extremely unstable ($-30 < \zeta < 30$ mV), and, as a consequence, the solid phase tends to coagulate and subsequently sediment in it. Besides, having compared the data from Table 1 and Figures 1 and 2, one can come to a conclusion that with the increase in permittivity which characterizes the polarity of the solvent, the aggregate and sedimentation stability drops significantly.

2. Results of the experimental determination of the size of basalt particles through time (Figure 1, 2) demonstrated that the slow growth of the particle size at a constant rate could be observed in glycerine, while the range of size properties remained virtually the same (a polydispersity index of 0.056). This indicates that by stabilizing particles aggregatively, glycerine not only acts as a diluent, but also as a surface-active agent (surfactant). At the same time, coarsening of particles in water occurs at different rates, whereas the range of size properties increases through time (a polydispersity index of 0.388).

3. Comparison of basalt deposition rates in various diluents (Figure 3) demonstrated that for nano- and especially micro-sized particles, there was a substantial probability of deposition in water in the process of the particle size determination using dynamic light scattering. Consequently, these sedimented particles may be overlooked in the analysis results, which does not provide a complete and accurate information on size properties and fractional distribution in the suspension.

## 4. Conclusion

After conducted studies of the particle size of the basalt-based wood-mineral insulation nanofiller using photon correlation spectroscopy it is possible to conclude that nanodispersed basalt exhibit sufficient aggregate and sedimentation stability in a glycerine-based dispersion medium.

It is shown that in the aqueous dispersion medium the sedimentation process of basalt particles proceeds intensively. This fact causes significant errors in the assessment of the particle size characteristics of basalt by dynamic light scattering.

It was found that in glycerol nano- and micro-sized particles of basalt electrostatically stabilized and have sufficient aggregate stability for measurements of their sizes. This makes the use of this organic solvent preferred as the diluent.

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