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Factors affecting the ultrasonic disaggregation of powders

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Abstract. Using finely dispersed mineral particles of various origins and morphologies offers a promising strategy in controlling the structure formation in cement composites. However, the use of such additives is hampered because those additives proved to be prone to consolidation into rather dense aggregates. Fine dispersion and disaggregation of powders is possible with the aid of cavitation ultrasonic treatment. However, the optimal conditions for such processing can not be established without conducting simulation studies. The purpose of the present study was the identification of ultrasonic-action factors and conditions ensuring an efficient disaggregation of finely dispersed powders of various origins and particle morphologies. In our study, we used diopside, granulated blastfurnace slag (GBS), wollastonite, ash, and calcium carbonate powders. It is found that the process of ultrasonic treatment of aqueous suspensions is accompanied not only by the dispersion of initial particles and aggregates but, also, by simultaneous formation of new aggregates. That is why the observed variations of the specific surface area and the optical density of powders can be attributed to the variation of the fractional composition of dispersed phase. The activating capability of a mineral additive is due to the fraction of the particles less than 1 μm in size exerting a key influence on the variation of the specific surface area of the powder. Our estimate of the energy efficiency of the cavitation disaggregation of powders during an ultrasonic treatment shows that the most energy-favorable one is the ultrasonic treatment lasting for 1–5 minutes, i.e. during the period of the first half-wave of the variation of the particle fraction less than 1 μm in size.

1. Introduction

A promising method for improving properties of cement composites implies filling the cement-binder matrix with finely dispersed mineral particles of different origin and morphology. An analysis of literature data shows that, as modifiers in structural material science, not only finely dispersed natural materials but, also, various specially prepared powders can be used [1–5]. Introduction of both additives, natural minerals (quartz, granites, basalts and others) and technogenic wastes, promotes an increase of cement-stone density and stability, a reduction of water demand of mixtures and their segregability, an increase of water-retaining capacity and homogeneity of the composites, and shrinkage reduction.

However, the use of such additives is difficult due to their propensity for aggregation and inefficiency with respect to objects of this class of traditional methods of disaggregation by mechanical actions [6]. And, if it is not possible to destroy the aggregates, then the activating effect of the introduced additive decreases sharply, since the main advantage of a highly dispersed powder, the ability to form a large number of contacts with its very small content, is unrealized.

One of the promising research lines in the fine dispersion and disaggregation of dispersed phase is the use of ultrasonic cavitation. The cavitation process features a multi-factorial complexity of phenomena proceeding in the cavitation cavity [7–11]. Under the action of cavitation, on the local scale intense liquid microscopic flows and high-power local shock waves are generated; the shock waves ensure a rise of temperature to 5000 K and a rise of pressure to 100 MPa, both processes leading to mass-transfer

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intensification and to a substantial variation of the characteristics of treated medium [11]. Presently, well-substantiated concepts of the mechanism of ultrasonic dispersion are available. According to those concepts, both the shock waves and the acoustic flows having arisen due to the micro-explosions of cavitation bubbles can be factors causing the disintegration of particles and aggregates [6, 14]. Along with the dispersion, a dispersed-phase coagulation process develops in suspensions under the action of ultrasound. The mechanism of acoustic coagulation was attributed to the action of hydrodynamic forces, Bernoulli and Bierkness ones, on the particles. However, the expressions for the latter forces were derived for the case of hydrodynamic action of particles in a uniform steady-state flow of ideal incompressible liquid, whereas the liquid flow with a propagating sonic wave is non-uniform and unsteady. Considering the latter fact, many authors adhere to the opinion that the mechanism of particle coagulation process in a sonic wave fundamentally differs from the picture which the Bernoulli force produces in an incompressible liquid flow [16].

Also, the kinetics of dispersion and coagulation processes is influenced by the intensity and duration of ultrasonic action. In engineering practice, the ultrasound intensity can be evaluated from the oscillation amplitude [6, 7]. The time required for the dispersion of powders depends, first of all, on the intensity of the ultrasonic action, on the physico-mechanical properties of the material to be dispersed, and on the state of the surface of powder particles.

The efficiency of the dispersion process is largely defined by the erosion activity of the liquid, whose magnitude substantially depends on its physico-mechanical properties. Here, the most pronounced action is due to the saturated vapor pressure of the liquid because the micro-impact actions due to cavitation decrease sharply at a high value of the latter pressure inside cavitation bubbles. That is why it is recommended to disperse materials in aqueous solutions as the erosion activity of such solutions proved to be much higher than that of organic solutions [6].

During the ultrasonic treatment, the dispersed-medium temperature affects the cavitation intensity and, hence, the powder dispersion process. For each liquid, there exists a certain range of temperatures in which the intensity of the cavitation action is maximal; for water, this temperature range is 35 to 50 ˚C [8].

On the whole, the profound scope of performed theoretical and experimental studies has ensured rather wide a range of industrial applications of cavitation technologies [6–10, 17, 20]. Nonetheless, calculation-based identification of the conditions of ultrasonic treatment ensuring most efficient dispersion and disaggregation of powders of different origin and particle morphology is presently impossible. In this connection, there arises a necessity in experimental studies.

The purpose of the present study was to identify factors and conditions of the ultrasonic action providing for a most efficient disaggregation of finely dispersed powders of different origin and particle morphology.

To achieve this goal, the following tasks were solved:

– to establish the nature of the change in the content of particles smaller than 1 μm, depending on the specific surface area of the initial powders and the duration of the ultrasonic treatment;

– to substantiate the energy-preferable duration of processing of powders by ultrasound, which provides a maximum content of particles smaller than 1 μm.

2. Materials and Methods

In the present study, as the carrier medium we used distilled water and as the dispersed phase, powders of heating-plant ash, granulated blastfurnace slag, diopside, wollastonite, and calcium carbonate. The particle size study of the powders was performed on an FSKh-6 photosidementer, whose work is based on the sedimentary Stokes law and the law of radiation attenuation in the turbid media of Lambert-Berra. The granulometric composition of initial powders is shown in Figure 1. It should be noted that for methods based on light scattering there is a characteristic description of the hydrodynamic diameter of particles that can be both primary particles and their aggregates and agglomerates [12, 13]

The optical density of suspensions was measured on a KFK-2MP photocolorimeter (λ = 470 nm). The test compositions were prepared by mixing powders in distilled water. The cavitation treatment of specimens was implemented using a UZG Volna-M device generating up to 1000-W ultrasound (model UZTA 1.0/22). The working compounds were placed in a transparent cylindrical vessel, where it was sonicated at a frequency of 22 kHz for different duration and power of ultrasound.

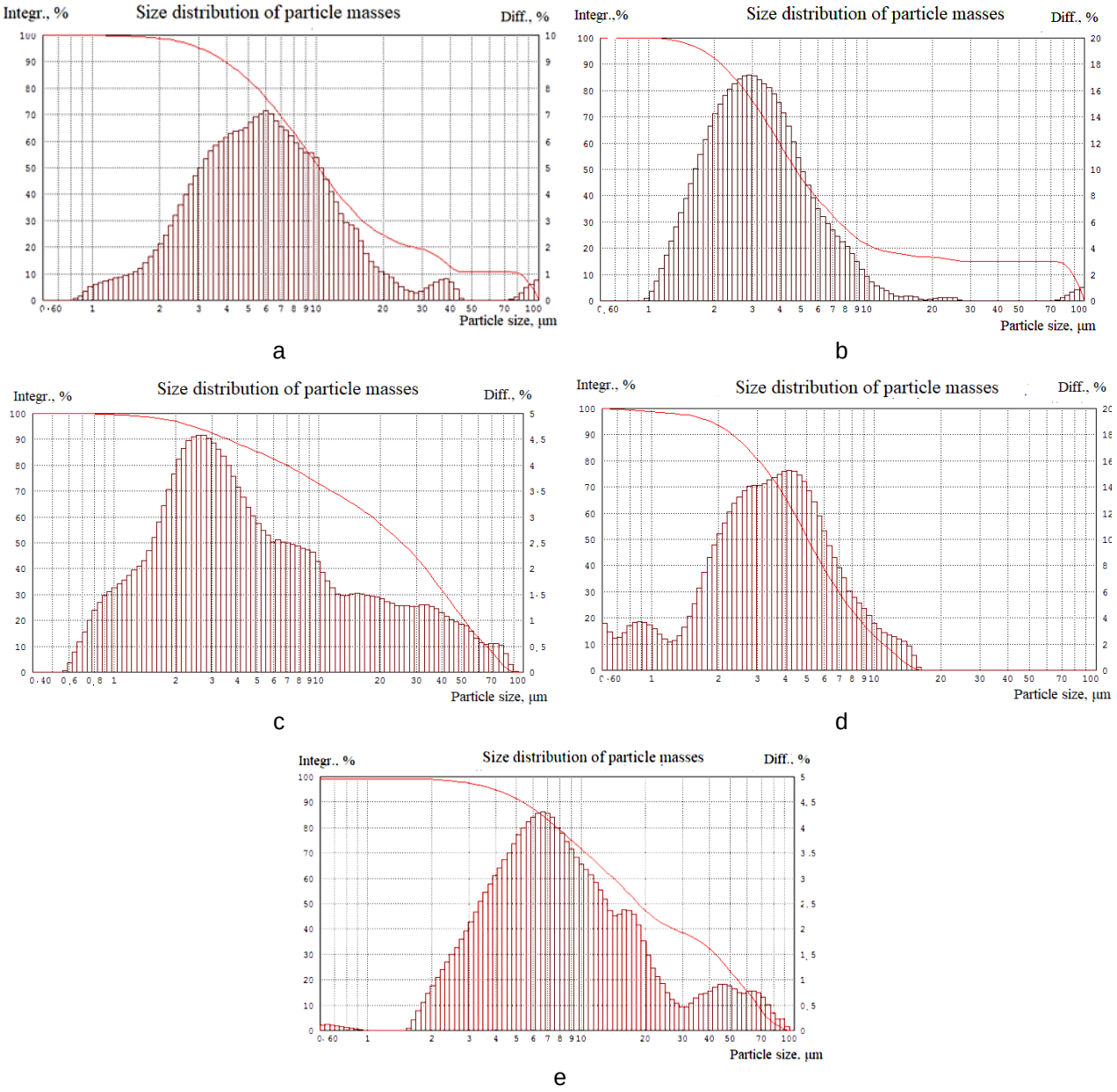

Figure 1. Granulometric composition of virgin (non-processed) powders: a – ash (S_{sp} = 2840 cm²/g), b – granulated blastfurnace slag (S_{sp} = 4186 cm²/g), c – diopside (S_{sp} = 1840 cm²/g), d – wollastonite (S_{sp} = 5226 cm²/g), e – calcium carbonate $CaCO_3$ (S_{sp} = 2327 cm²/g).

3. Results and Discussion

Treatment with ultrasound of aqueous suspensions of powders of various origins and particle morphologies is accompanied with a change of specific surface area, an integral characteristic characterizing the degree of dispersion and aggregation of powders (Figure 2). The obtained results on the variation of specific surface area proved to be well correlating with the data on the variation of optical density at the passage of light through ultrasonically processed suspensions (Figure 3). The variation of the granulometric composition of dispersed phase can be judged considering the data on the variation of root-mean-square particle diameter (d_m) versus the duration of ultrasonic treatment (Figure 4). For all the powders, the variation of d_m exhibits an oscillating behavior being a result of the dispersion and coagulation processes. During the initial period of ultrasonic treatment (0–5 min), the impact action due to the ultrasonic waves, the intense motions of particles, and the inter-particle collisions all cause the rupturing of bonds between dispersed-phase particles, this rupturing leading to a profound increase of free surface energy (by a factor in excess of 10) [6]. The molecular forces thus increased hamper the further rupturing of particles and aggregates. That is why the rate of dispersion decreases in value during further treatment of the suspension, and the particle aggregation process begins. It should be noted here that, due to coagulation, no restoration of the poly-dispersed system to its initial (pre-dispersion) state occurs since, as a rule, fragments of aggregates and coarse particles never restore into an entity by absorbing water and by forming, due to a higher specific surface energy, a solvate shell with a monolayer of heavily bonded water that will not allow the van der Waals forces to draw together the fine solid particles [14].

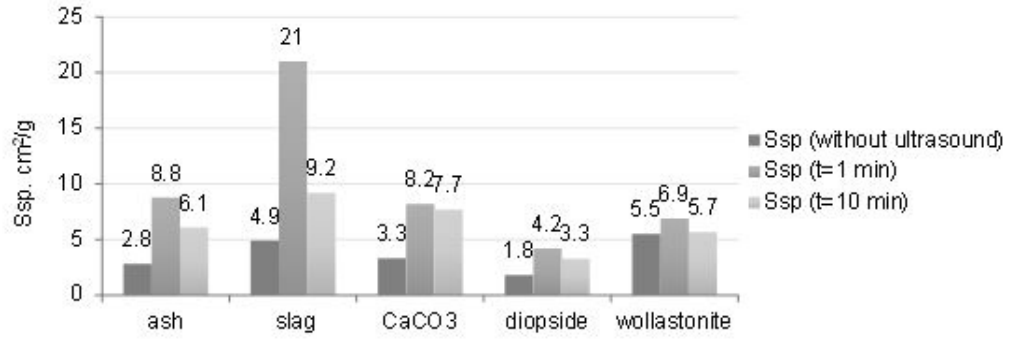

Figure 2. Variation of the specific surface area of powders versus the duration of ultrasonic treatment.

Thus, the ultrasonic treatment of aqueous suspensions is accompanied, first, by the disaggregation of initial particles and aggregates and, second, by the simultaneous formation of new aggregates. That is why the observed variations of specific surface area and optical density are defined by the variation of the fractional composition of dispersed phase. In all the powders, the behaviors exhibited by the fine (sized less than 1 μm) and coarse (sizes greater than 10 μm) particle fractions exhibit an oscillating character being a consequence of the simultaneously proceeding dispersion and coagulation processes. Since, as a rule, at short durations of ultrasonic treatment (less than 5 minutes) the amount of particles smaller than 1 μm in size increases, and the amount of particles greater than 10 μm in size, decreases in magnitude, then the dispersion process at those times prevails over the aggregation process. On the other hand, with increasing the duration of ultrasonic treatment, the amount of particles smaller in size than 1 μm decreases, and the amount of particles greater than 10 μm in size normally increases in magnitude, this finding being indicative of the prevalence of the coagulation process (Figure 4).

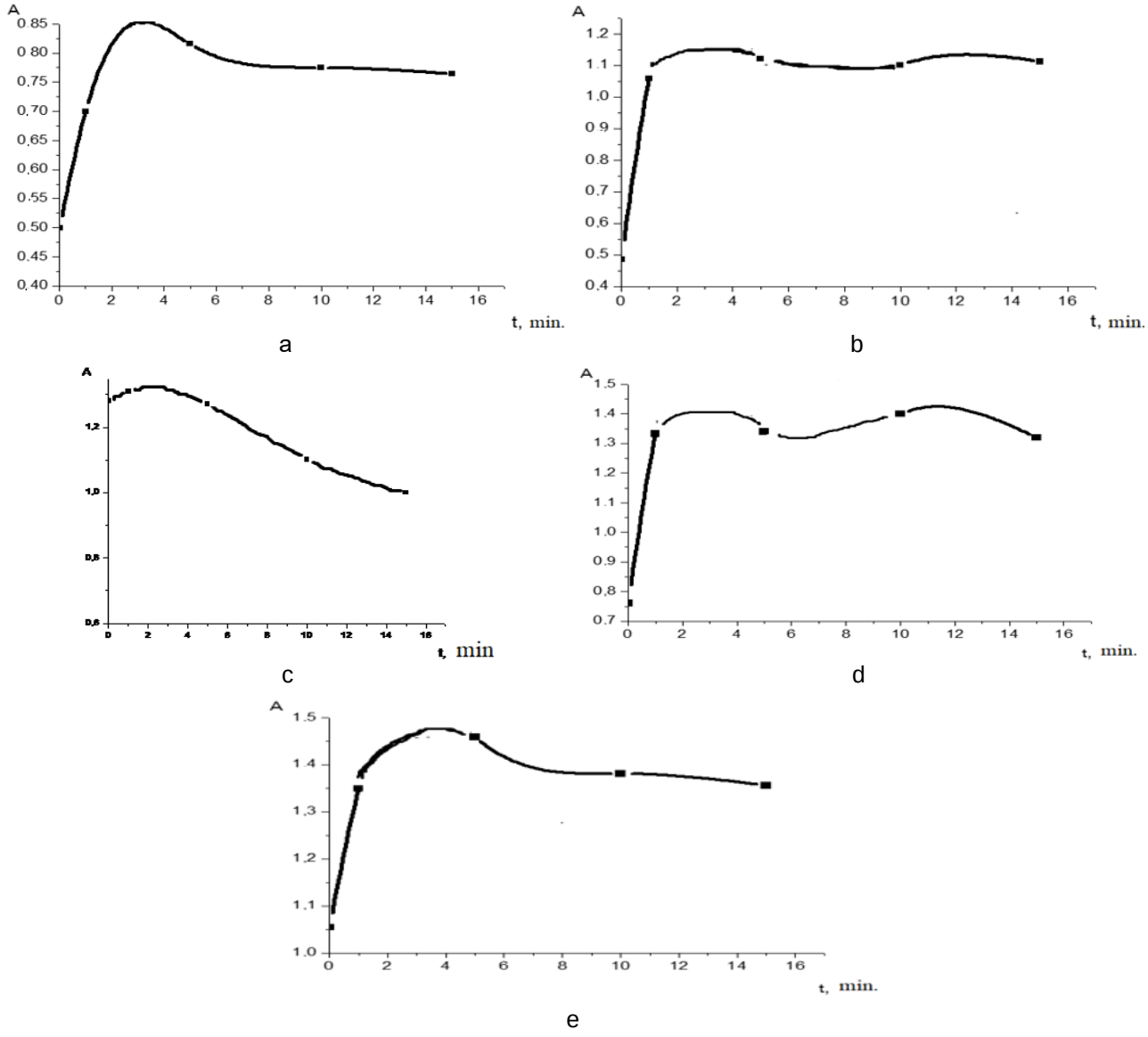

Figure 3. Variation of the optical density of suspension versus the duration of ultrasonic treatment: a – ash; b – diopside; c – $CaCO_3$; d – granulated blastfurnace slag; e – wollastonite.

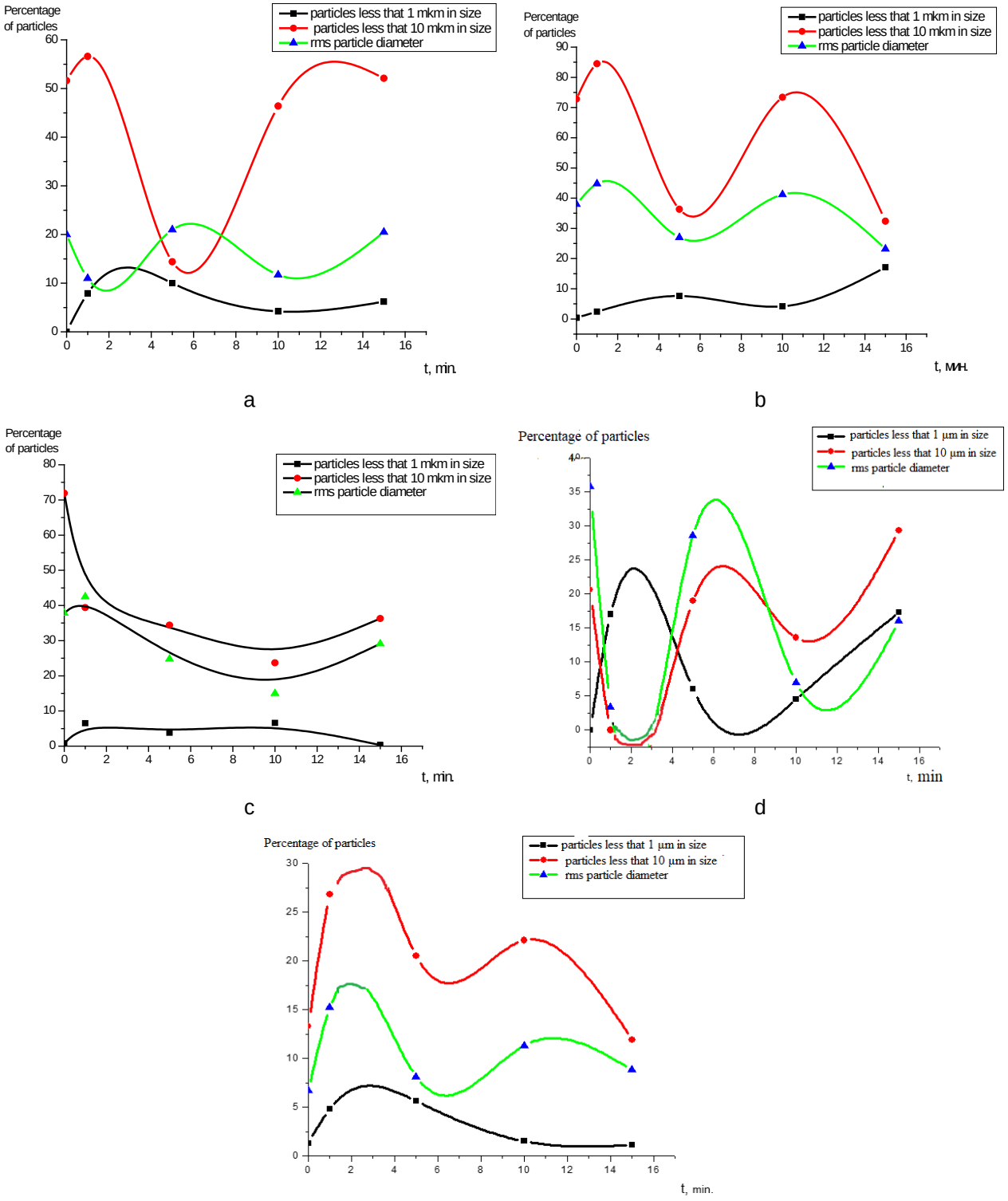

Figure 4. The content of particles versus the duration of ultrasonic treatment: a – ash; b – diopside; c – $CaCO_3$; d – granulated blastfurnace slag; e – wollastonite.

Under identical conditions of the ultrasonic treatment, the amount of particles of the newly formed fraction sized less than 1 μm exerts a predominant action on the behavior of the specific surface area of particle, a parameter largely determining the activating capacity of the mineral additive (Figure 5). Since the latter regularity is exhibited by powders of various origins and particle morphologies, then the fraction of the particles sized less than 1 μm can be considered as a criterion of the efficiency of the ultrasonic dispersion and disaggregation of mineral additives, whose maximization will promote the enhancement of the reactivity of the mineral additive.

The data in Figures 6–8 show that, along with the duration of supersonic treatment, the intensity of the ultrasonic action and the specific surface area (S_i) of initial powders can be recognized as factors influencing the formation of the maximum amount (fraction) of the particles of the size fraction less than 1 μm in a suspension treated with ultrasound.

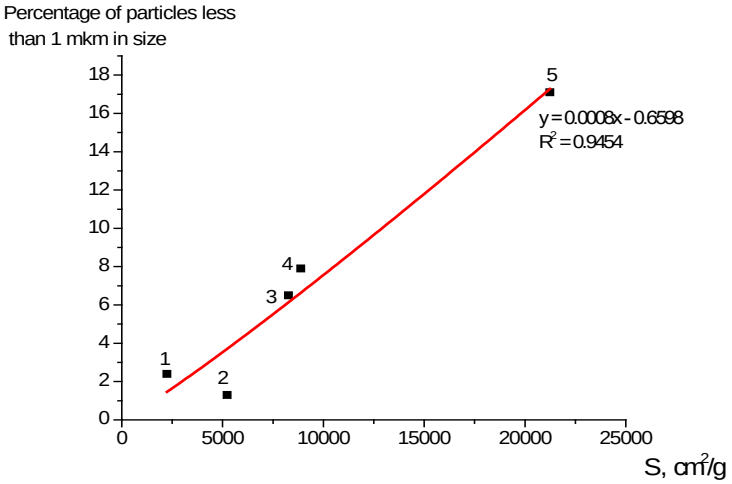

Figure 5. Contents of particles of various size fractions versus the specific surface area of powder for 1-minute duration of ultrasonic treatment.

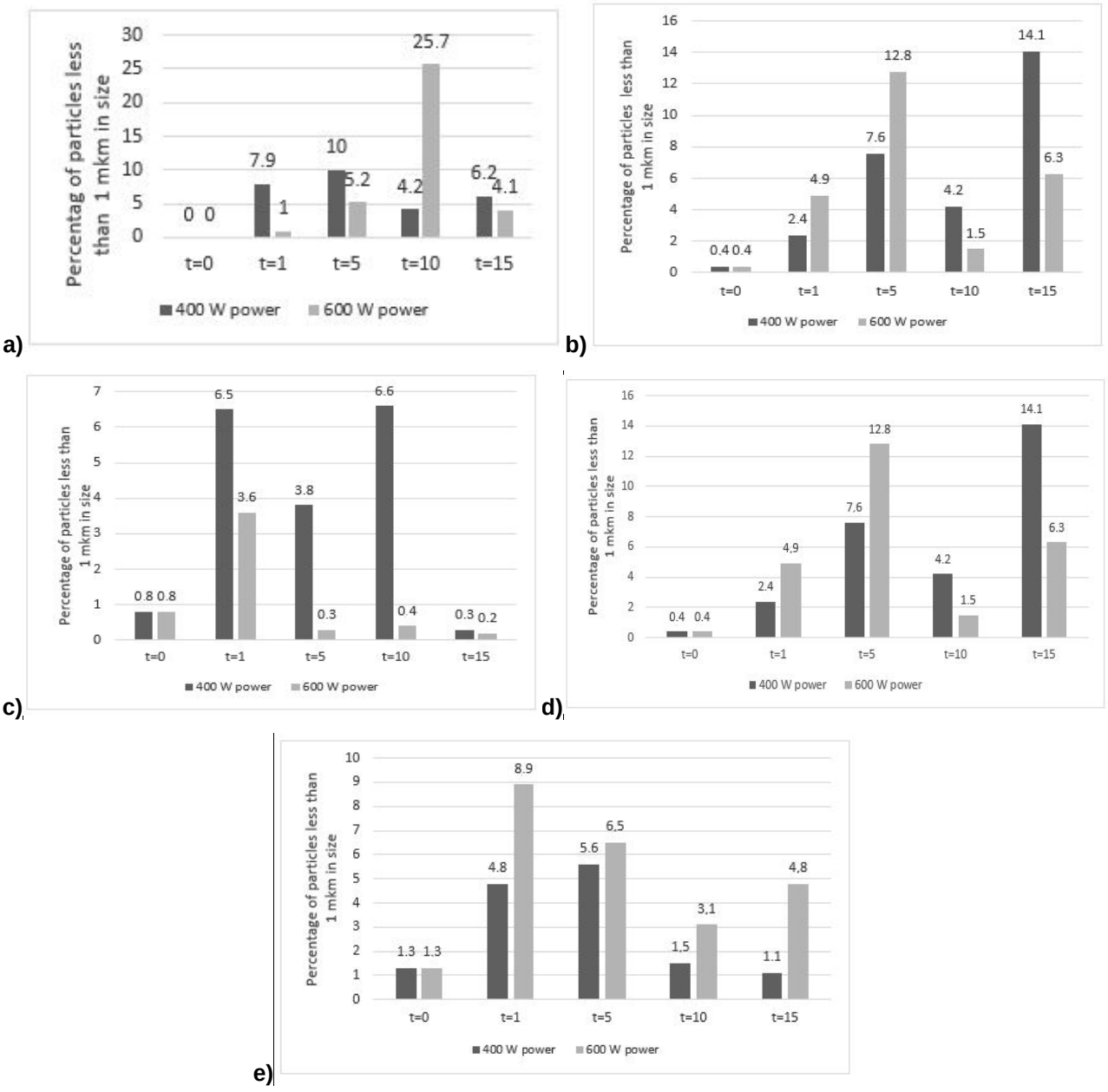

Figure 6. Variation of the contents of particles of various size fractions at different durations and intensities of ultrasonic treatment: a – ash; b – diopside; c – $CaCO_3$; d – granulated blastfurnace slag; e – wollastonite.

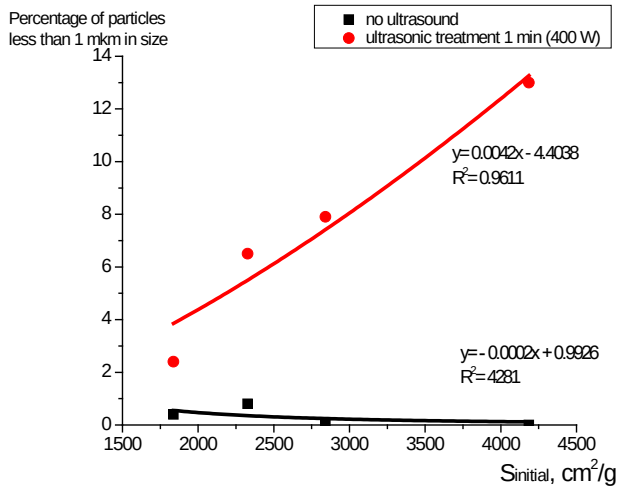

Figure 7. Dependence particle fraction content of less than 1 mm from the original surface area of powders of different genesis and morphology.

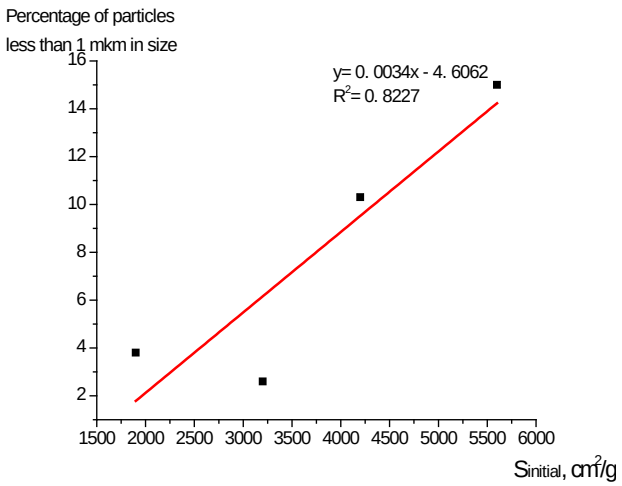

Figure 8. Variation of the content of particles of size fraction less than 1 μm in granulated blastfurnace slag powders of different specific surface areas.

Ultrasonic dispersion and disaggregation can both be classed to the mechanical dynamic activation due to cavitation and due to the mutual friction of rapidly moving and colliding particles. Since the center of a collapsing cavitation bubble is normally situated at some distance from the surface of the solid particles, then, on the adoption of a value of 10^2–10^3 MPa [14] for the pressure produced by cavitation-bubble explosion, one can arrive at a conclusion that the stress applied to a powder particle will be two or three orders smaller than the theoretical strength of particle material as determined by the Griffith formula, 10^4 to 10^5 MPa [16]. That is why, in spite of the fact that, because of structural imperfections, the strength of real materials is much lower than its theoretical value, the energy of a cavitation micro-explosion will always be insufficient for the fracture of fine particles containing a low amount of defects. In this connection, we believe that the main process defining the variation of the granulometric composition is, first of all, the disaggregation of powder particles bonded together by autohesion forces and having much a lower strength in comparison with homogeneous particles.

The effectiveness and kinetics of the fracture of particles and their aggregates under conditions of an ultrasonic treatment depends on the intensity of the shock wave and on the velocity of the acoustic flows generated in the medium during the micro-explosion of cavitation bubbles. Presently, two models for cavitation dispersion are known, the fracture of solid particles under the action of shock waves and that under the action of acoustic flows. According to the first model, high-intensity local shock waves ensuring the rise of temperature to 5000 K and the rise of pressure to 100 MPa are generated during micro-explosions of cavitation bubbles. Here, the pressure pulse exerts a most pronounced disturbing action on the particles floating on the surface of cavitation bubbles. That is why a necessary condition for the dispersion of particles in this model is a spherical shape of the gas bubbles at the time of collapsing. The latter is only possible when the diameter of such bubbles will substantially exceed the sizes of the particles and aggregates to be dispersed [14–15, 19].

According to the second model, the dispersion process proceeds due to the collisions of the particles being accelerated, by the cavitation-induced micro-jets, to velocities reaching tens and hundreds meters per second. Previously, calculations performed for powder particles sized 0.1, 0.01, and 0.001 mm [19] have proved the kinetic energy to be sufficient for the fracture of the particles during the collisions; indeed, the generated stresses were in the range from 10 to 10^3 MPa, this values being comparable with the pressure 10^2–10^3 MPa produced by a collapsing cavitation bubble.

It should be noted here that the previously obtained data [14–16, 19] show that the cavitation action can simultaneously obey both models. However, depending on the size of dispersed-phase particles and the parameters of the ultrasonic treatment (amplitude and frequency), one of the two models will appear prevailing. For instance, coarser particles will be dispersed due to the cavitation-explosion-induced acoustic flows while the finer particles, due to the intense shock waves (since the pressure pulse due to these shock waves exerts a most pronounced disturbing action only when the dispersed-phase particles float on the surface of the cavitation bubble, the latter in turn being only possible when the sizes of such a bubble will be greater than the particle sizes [20]). In our opinion, an aggregate formed by particles smaller is size than the voids in such an aggregate can be considered as a version of the latter ultrasonic dispersion scheme. In the latter case, the gas-bubble size will be defined by the void size. Accordingly, the ultrasonic-field parameters must ensure cavitation explosions of gas bubbles contained in the voids of such aggregates. Thus, the intensity of the ultrasonic action is to be chosen considering the possibility of ensuring the cavitation of gas bubbles of radius $R_{bubble} = R_{void}$.

With due regard for the fact that the fraction of the particles smaller than 1 μm plays a determining role in the formation of dispersed-phase surface, the choice of the optimum duration of ultrasonic treatment can be made considering an estimate of the energy efficiency of the cavitation disaggregation of powders treated with ultrasound. This efficiency can be evaluated considering the value of the coefficient of the energy efficiency of cavitation dispersion ($K_{en.eff.}$), which can be determined from the formula

$$K_{en.eff.} = E_d / E_{cav.} 100\ \%,$$

where E_d is the energy spent for increasing the specific surface area of the particles smaller than 1 μm during the time t;

$E_{cav.}$ is the energy consumed for the production of cavitation.

The calculated curve of the energy efficiency factor versus the duration of ultrasonic treatment shown in Figure 9 indicates that the long-term action due to the cavitation leads to a decreased value of the energy efficiency factor. Most probably, the reduction of the efficiency of the ultrasonic treatment is due to the fact that the ultrasonic-curing process of aqueous suspensions is accompanied not only by the dispersion of initial powders and aggregates but, also, by the concomitant formation of new aggregates; on the other hand, according to the definition of the energy efficiency factor, the energy spent for coagulation will pertain to the losses.

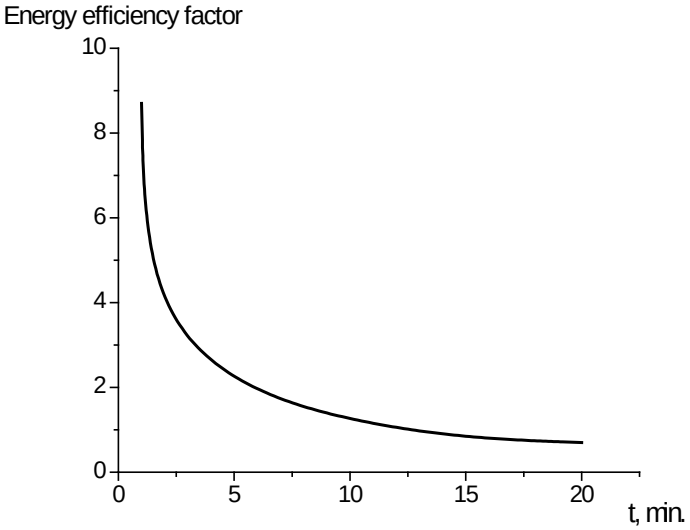

Figure 9. Energy efficiency factor versus the duration of ultrasonic treatment.

Thus, at a relatively small difference between the contents of particles with sizes smaller than 1 μm, the most efficient treatment is the treatment with ultrasound lasting for 1–5 minutes, that is, for the period of the first half-wave of variation of the amount (fraction) of particles smaller than 1 μm in size.

4. Conclusions

The research results showed that, regardless of the genesis and morphology of the particles, by varying the duration of cavitation treatment in powders, it is possible to initiate both a dispersing and aggregating effect. In this regard, time optimization of the processing of powders by ultrasound according to the criterion of maximum disaggregation will increase the potential of such powders as additives to improve the properties of cement composites.

1. Regardless of the time of ultrasonic treatment, the fraction of particles of the most active fraction (less than 1 micron) of wollastonite, chalk, ash, diopside powders, blast furnace granulated slag does not exceed 25 %. Therefore, from the point of view of energy efficiency, it is more preferable to treat these powders with ultrasound within 1–5 minutes, that is, during the first half-wave of a change in the fraction of particles of the fraction <1 μm.

2. The ultrasound processing time optimal for a particular powder will depend on the parameters determining the power (intensity) of the ultrasound effect: amplitude, frequency, and volume of the working chamber of the ultrasonic unit.

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Факторы, влияющие на ультразвуковую дезагрегацию порошков

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Ключевые слова: ультразвуковой, кавитация, ультрадисперсная добавка, дезагрегация, измельчение, цемент, строительство

Аннотация. Использование высокодисперсных минеральных частиц различного генезиса и морфологии является перспективным направлением управления структурообразованием цементных композитов. Вместе с тем, использование таких добавок затруднено тем, что они, как правило, консолидированы в достаточно плотные агрегаты. Тонкое диспергирование и дезагрегация порошков возможно с помощью кавитационной обработки ультразвуком. Однако оптимальные условия такой обработки не могут быть установлены без проведения имитационных исследований. Целью исследования является определение факторов и условий ультразвукового воздействия, обеспечивающие эффективную дезагрегацию высокодисперсных порошков разного генезиса с различной морфологией частиц. Для проведения исследований использовались порошки диопсида, ДГШ, волластонита, золы ТЭЦ, карбоната кальция. Установлено, что процесс ультразвуковой обработки водных суспензий сопровождается не только разрушением исходных частиц и агрегатов, но и одновременным образованием новых агрегатов. Поэтому, имеющие место изменения удельной поверхности, оптической плотности в данном случае обусловлены изменением фракционного состава дисперсной фазы. Активирующую способность минеральной добавки оказывает фракция частиц размером менее 1 мкм, оказывающую ключевое влияние на изменение удельной поверхности порошка. В результате оценки энергетической эффективности кавитационной дезагрегации порошков в процессе их обработки ультразвуком показано, что энергетически более предпочтительным является обработка ультразвуком в пределах 1–5 мин, т. е. в период первой полуволны изменения доли частиц размером менее 1 мкм.

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