

PRODUCTION VARIABLES OF FINELY DISPERSED CEMENT SYSTEMS

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ABSTRACT

This article discusses production variables of finely dispersed cement systems for decorative coatings. The influence of fine grinding and modification on properties of colloidal systems is considered. It is demonstrated that intensive increase in specific surface area occurs in the grinding interval from 10 min to 50 min, then the kinetics of increase in specific surface area becomes slower. It is found that instead of natural sands it is possible to apply screenings of concrete scrap. It is determined that natural sands and screenings of concrete scrap are characterized by good grindability. Grinding variables for sand and screenings of concrete scrap are identical. It is established that particle size distribution of finely ground cement systems varies upon dispergation, and content of finer particle sizes in total bulk of ground substance increases with increase in specific surface area, thus facilitating elimination of stresses in solidified cement stone. It is mentioned that upon increase in filling ratio of cement from 30% to 50% water requirement of finely ground cement mixtures increases. It is established that the use of preliminary regrinding of fillers and subsequent combined grinding of dry constituents makes it possible to obtain homogeneous fine cement mixture with preset particle size distribution. It is demonstrated that combined activation together with modifiers facilitates obtaining of required strength properties of colloidal cement composites, thus providing decorative coatings on the basis of colloidal cement materials.

INTRODUCTION

Now-a-days the problem of long-term protection of facades and structures is very important. In this regard, high attention is paid to decorative coatings on the basis of colloidal cement systems characterized by wide range of application and protection, capable to provide long-term service and architectural expression for buildings and structures.

Production of beautiful and efficient decorative coatings, resistant against atmospheric impacts, depends on processing of initial materials, maximum homogeneity of solid phase and high dispersity of composite material, selection and optimization of compositions and properties of colloidal cement systems, providing extraordinary decorative

properties of coatings in combination with long-term lifetime.

In order to obtain decorative coatings, it is necessary to study peculiarities of crystalline structure of near-surface layers of solids and major regularities of generation of colloidal systems with their properties. Efficiency of decorative coatings on the basis of colloidal cement system can be improved by integrated application of mineral fillers and modifiers (Ur'ev, 2013; Lopanchenko and Kosukhin, 2006; Gerchin and Temnikov, 2003; Kaprielov and Karpenko, 2006).

In order to obtain reasonable application of raw stuff it would be interesting to use industrial wastes as fillers, including fine screenings of concrete scrap, which is not widely used at present (Bazhenov and

Myrtazaev, 2008; Belykh and Fadeeva, 2008; Krivtsov, *et al.*, 2010). In this regard, it is required to study the influence of properties of concrete scrap screening on possibility to produce fine cement systems. Preliminary studies demonstrated that secondary raw stuff on the basis of concrete scrap screening can be used not only in concretes but also for production of constructing composites and mixed binders on its basis.

High decorative properties of colloidal materials are provided by improvement of homogeneity, adhesion properties, color retention and long-term lifetime as a consequence of generation of dense homogeneous structure, establishment of optimum ratio of components, improvement of technological properties of filled cement pastes (Kaprielov and Karpenko, 2006).

Optimization of structure and properties of composite fine cement systems is an urgent task, its solution would provide for improvement of cost efficiency, reliability, and long-term lifetime of decorative coatings on their basis (Gerchin and Temnikov, 2003; Kaprielov and Karpenko, 2006; Ur'ev and Dubinin, 1980).

Mechanical activation of components makes it possible to obtain fine cement systems with improved viscoplastic properties, improved placeability, intensive structure formation in early times of solidification; herewith, improved dispersity and physicochemical activity of their components permits significant improvement of structural and mechanical, rheological and engineering properties (Krasovskii, 2012). Up-to-date grinding technologies, additives and modifiers applied for processing of technogenic raw stuff provide new possibilities for production of efficient construction materials and items on their basis.

Thus, this work studies into the influence of grinding, mechanical activation, and modification on dispersity, activity, and technological properties of fine disperse cement systems intended for production of decorative coatings.

METHODS

This work investigated into processing procedures of initial raw stock using mechanical and chemical activation and modification enabling production of highly concentrated dispersed colloidal systems with low apparent viscosity and superior operating properties. The required dispersity of components was achieved using efficient grinding facilities. Homogeneous distribution of highly dispersed

components in dispersion medium is provided by high-speed agitators.

Grinding fineness of components was determined by variations of specific surface area (S_{spec}). The specific surface area of powdered materials was determined by a T-3 pneumatic meter. Dispersity of fine fractions of filled cement systems was studied using laser analyzer.

The influence of mechanical activation and modification was estimated by variation of major physicochemical and engineering properties of finely dispersed cement systems. Average density of cement stone was determined using cubic samples with the dimensions of 20×20×20 mm, that of cement-sand mortar – using bar samples with the dimensions of 40×40×160 mm, dried to constant weight at 105°C.

Optimum particle size distribution of fine filler for mortar was determined on the basis of sand fraction ratio providing for the densest particle packing, the criterion of packing density was maximum value of bulk density of filler.

Major properties of ingredients mixed with water, such as mobility, average density, segregation, water retaining capacity, water gain from mortar mixture, ultimate compression strength (hereinafter: strength), expansion upon breakage, expansion upon bending, shrinkage, moisture content, water absorption, freeze resistance of solidified mortar were determined in accordance with the Russian standard GOST 5802 “Mortars, test methods”.

Samples after compaction were held prior to demolding in normal storage chamber at $(20 \pm 2)^\circ\text{C}$ and relative air humidity of 95-100%, then released from molds in (24 ± 2) h after placement of mortar mixture. After demolding the samples were held in the first three days in normal storage chamber at relative air humidity of 95-100%, and then, before testing, in a room at relative air humidity of $(65 \pm 10)\%$.

RESULTS

The influence of grinding variables on specific surface area, particle morphology and reactivity upon interaction with Portland cement (C) was studied with the aim of comparison by the example of filler (F) obtained upon grinding of cement scrap screening and natural quartz sand in ball mill and vibratory mill.

Specific surface area of sand, obtained upon grinding in vibratory mill from 10 to 90 min, increases from 320 to 990 m^2/kg , that of cement scrap from 380 to 970 m^2/kg , respectively; upon grinding in ball mill – from 150 to 340 m^2/kg , respectively.

Vibratory mill is the most efficient grinding facility for production of colloidal cement material in terms of uniform particle size distribution, reasonable specific surface area including power consumption, as well as the optimum structure imperfection of particles, determining the interaction degree with cement binder. In this work binders intended for improvement of viscous and rheological properties of fine cement systems were ground in vibratory high-speed mills.

The main components for production of colloidal mixtures were Portland cements, natural sands, various fillers (cement scrap screening, metakaolin, and others), additives and pigments. Wide range of materials used in the studies is attributed to the necessity to examine possibility of practical application of various compositions for decorative coatings on the basis of colloidal cement mortar.

Portland cement, grade PTs500-D0, Bukhtarminskaya cement company, was used in laboratory studies. Natural sands from Nikolaev and Baiserke deposits, Almaty oblast, were used as fillers for colloidal cement materials.

In terms of processing and costs of screening separation of sand into particle size fraction of 2.5-1.25, 1.25-0.63, 0.63-0.315, 0.315-0.16 mm is the most efficient (Krasovskii 2012).

Screening of secondary concrete scrap was also used as filler. Cement scrap, obtained from Portland cement, grade PTs400-D0 according to the Russian standard GOST 10178, was ground and screened to obtain particle size fraction of 5 mm and below. Table 1 summarizes particle size distribution of sand from concrete scrap screening.

According to chemical and mineralogical analysis the concrete scrap screening meets the requirements to filler, the sand fineness modulus (M_{fine}) is 2.8,

in terms of total screen residue, 0.63 and 0.16, it is classified as coarse.

Selection of rational fraction composition of sand obtained from concrete scrap for mortar mixtures was based on obtained minimum intergranular porosity. In order to obtain steady results for mixtures intended for decorative coatings the sand of concrete scrap screening was fractionated.

Real density of sand of concrete scrap screening with the particle size fraction of 2.5-0.16 was 2640 kg/m³, that of 1.25-0.16 – 2560 kg/m³. Bulk density of sand of particle size fraction of 2.5-0.16 was 1586 kg/m³, that of 1.25-0.16 – 1594 kg/m³. The sand porosity is 39.6% for particle size fraction of 2.5-0.16 and 38.5% for particle size fraction of 1.25-0.16. It was found that intergranular porosity of rationally selected mixture is achieved for sand particle sizes of 1.25-2.5 and 0.16-0.315 at the fraction ratio 2.3:1.

Grindability of components used as fillers was determined on the basis of grinding kinetics of natural sand and concrete scrap screening. Taking into account that initial particle size distributions of quartz sand and concrete scrap screening are different, their grindability was determined with variation of grinding time. Preliminary quartz sand and concrete scrap screening were reground to specific surface area of cement, since initial cement is finer and can be readily ground. Grindability and specific surface area as a function of grinding time of quartz sand and concrete scrap screening in laboratory vibratory mill with steel milling balls (10-20 mm in diameter, vibrator moment 22 kg cm) are summarized in Table 2.

Dispersity of fine fractions of filled cement systems was studied using a laser analyzer. Particle size distribution in fine powder was determined. Cement/filler mixtures were prepared in the ratios of 70:30

Table 1. Particle size distribution of sand from concrete scrap screening

Oversize, %	Screen mesh, mm					Undersize 0.16, %	Fineness modulus, M_{fine}
	2.5	1.25	0.63	0.315	0.16		
Particular	23.35	12.78	15.60	29.82	10.73	7.72	2.8
Complete	23.35	36.13	51.73	81.55	92.28		

Table 2. Specific surface area of ground product as a function of grinding time

Description of material	Grinding time, min									
	10	20	30	40	50	60	70	80	90	100
	Specific surface area, m ² /kg									
Sand, Nikolaev deposit	320	472	650	720	790	860	915	970	1001	1080
Sand, Baiserke deposit	310	465	635	710	785	850	910	955	970	1000
Concrete scrap screening	395	440	690	770	810	890	945	970	990	1060
Cement	410	540	750	820	-	-	-	-	-	-

(composition 1), 60:40 (composition 2), and 50:50 (composition 3). Table 3 summarizes correspondence of dispersity of colloidal cement systems with various fillers. Achievement of maximum dispersity of fillers is usually positive not only for its properties but also for properties of fine cement systems.

In addition, the obtained materials are characterized by wide particle size range, which predetermines decreased water requirement in terms of normal consistency of cement paste (NC). This is confirmed in (Kaprielov and Karpenko, 2006; Plugin, *et al.*, 2003) where it is stated that addition of correct fillers results in decrease of water requirement with retention of appropriate rheological and strength properties and simultaneous decrease in shrinkage deformations of cement stone.

For fine cement systems with sufficiently high specific surface area the water requirement for obtaining of normal consistency increases proportionally to its content on cement bulk. With cement filling ratio of 30-50% the water requirement of fine cement mixtures varies in the range of 26.5-45.1%.

Water requirement of compositions with regard to filled cement (C+F), that is, in terms of $W/(C+F)$, at equal filling ratio can be adjusted by specific surface area in wide ranges. Addition of filler with specific surface area of 300 m²/kg in amount of 30% to cement with specific surface area of 450 m²/kg and above promotes increase in water requirement which is 29.4%, and at filling ratio of 50% the water requirement is 38.7% (Fig. 1).

If a filler with specific surface area of 860 m²/kg is used, the same composition will have higher water requirement of 31.3-42.8% depending on cement filling ratio. Water requirement of cement systems with various filling ratio is 27.6-34.3% at filler specific surface area of 300 m²/kg, 26.5-31.5% at filler specific surface area of 560 m²/kg, and 28.3-38.3% at filler specific surface area of 860 m²/kg.

Optimum concentration and filler type should provide minimization of internal stresses due to structure arrangement of composite; prevent cracking in solidifying stone of cement disperse systems (Korolev, *et al.*, 2004). It is established that rational dispersity of fine cement systems (in terms of grain specific surface area) is 500-550 m²/kg. At coarser grinding water segregation is observed accompanied by decrease in stone strength. At finer grinding (860 m²/kg) water requirement of fine cement binder increases significantly. Further studies were performed with fillers with dispersity of 550 m²/kg.

Water requirement for variation of normal consistency of binder as a function of filling ratio of cement systems was determined in the experiments (Fig. 2). It is established that addition of fillers (natural sand and concrete scrap screening) slightly decreases water requirement of fine cement systems. Thus, upon addition of up to 30% of fillers minimum normal consistency is observed: for sand 26.4%, for concrete scrap screening 26.7%, further content increase actually does not influence on water requirement of the system, but leads to slight water segregation.

High quality colloidal cement systems with improved efficiency of decorative coatings can be possibly obtained by their complex modification due to addition of metakaolin in combination with highly efficient additive and superplasticizing agent (Korolev, *et al.*, 2004; Caladrone, *et al.*, 1994; Engelhard Corp. High Reactivity Metakaolin MetaMax, 2002; Yuhnevskii, 2012; Batrakov, 2006).

The main task of modification of colloidal cement systems is to use reasonably the features of separate components influencing on processes occurring upon hydration of cement systems aiming at achievement of high multifunctional effect. The properties of filled fine cement systems depend considerably on

Table 3. Specific surface area and particle size distribution of finely ground cement systems

Description of material	Specific surface area, m ² /kg	Content of particle size, μm, %						
		0-5	5-10	10-20	20-30	30-40	40-60	>100
Portland cement without additives								
Portland cement	310	1.03	4.96	15.87	32.94	21.05	15.59	8.56
Filler: Natural sand								
Composition 1	550	3.97	35.14	38.42	12.52	9.38	0.57	-
Composition 2	550	3.52	29.47	34.46	20.26	12.04	0.25	-
Composition 3	550	3.47	25.60	36.49	20.43	13.81	0.20	-
Filler: Cement scrap screening								
Composition 1	550	4.97	31.90	36.12	14.86	11.53	0.62	-
Composition 2	550	3.92	30.21	32.43	13.09	19.68	0.67	-
Composition 3	550	3.64	29.97	31.67	20.61	13.46	0.65	-

the structure of dispersed fillers and additives in their composition (Skripnikova and Sazonova, 2014; Chulkova and Berdov, 2009).

Metakaolin, superplasticizing agent, and redispersed polymer powder, added upon grinding to optimum specific surface area, were used as components of modifier of colloidal cement material in order to obtain cement stone with superior operation properties.

Metakaolin ($Al_2O_3 \cdot 2SiO_2$) is an active mineral additive, one of the most efficient pozzolanic additions for cement systems. The use of metakaolin would improve the structure of cement stone due to reaction with $Ca(OH)_2$ and generation of secondary

silicate hydrates and calcium aluminates, as well as decrease in efflorescence and destruction as a consequence of silicate alkali reaction (Caladrone, *et al.*, 1994; Engelhard Corp. High Reactivity Metakaolin MetaMax, 2002).

It is known that metakaolin acts as catalyst upon cement hydration which leads to increase in reaction rate and increase in cumulative heat released in early hydration period, as well as to decrease in porosity and variation of pore size ratio to lower values (Caladrone, *et al.*, 1994).

Plasticizing effect of metakaolin can be attributed to the fact that its particle size distribution supplements the particle size distribution of colloidal cement material. Kaolin is able to bind alkalis (K, Na, Li) into insoluble compounds with chemical compositions similar to those of zeolites and field spars, which in its turn provide more reliable protection of decorative coatings on the basis of colloidal cement glue against efflorescence and destruction as a consequence of silicate alkali reaction (Caladrone, *et al.*, 1994; Engelhard Corp. High Reactivity Metakaolin MetaMax, 2002).

Laboratory tests revealed that 7% can be considered as optimum amount (the ratio of cement/filler/metakaolin is 70:23:7), since further increase in metakaolin portion in fine cement systems leads to decrease in their activity.

Fine fillers and polymer additives facilitate adjustment of plastic properties of fine cement systems and mortars on their basis, creating possibility of plastic consistency (Morozov and Morozova, 2008). Herewith, it is necessary to consider for the fact that polymer additives influence on hydration kinetics of cement clinker minerals, thus predetermining the rate of formation of cement stone structure and its parameters (porosity, composition, and stability of hydrate phases, propensity to aging and cracking).

Mechanical activation in combination with modifiers using optimum processes makes it possible to achieve required properties of disperse cement systems and

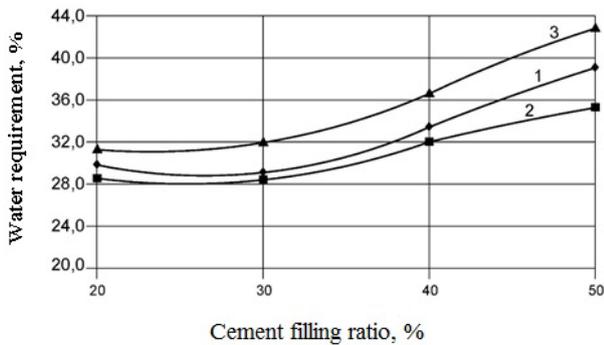


Fig. 1 Water requirement of finely ground cement systems as a function of cement filling ratio at cement specific surface area of 450 m²/kg and higher: 1 - Filler specific surface area - 300 m²/kg; 2 - 550 m²/kg; 3 - 860 m²/kg.

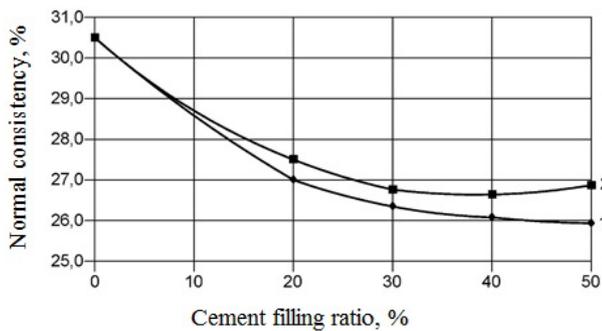


Fig. 2 Normal consistency of cement paste as a function of cement filling ratio: 1 - Natural sand; 2 - Concrete scrap screening.

Table 4. Influence of processing of initial components on physicochemical properties of products

Processing	W/C	Compression ultimate strength, MPa, at the age of, days			
		1	3	7	28
Filler: Natural quartz sand					
Combined activation	0.5	11.5	23.4	32.6	58.3
Separate activation of cement and filler up to $S_{sp}=550$ m ² /kg	0.5	7.3	17.9	29.7	52.7
Filler: Concrete scrap screening					
Combined activation	0.5	10.3	24.1	31.8	57.9
Separate activation of cement and filler up to $S_{sp}=550$ m ² /kg	0.5	8.0	16.1	24.8	50.0

targeted structure formation, and, as a consequence, improved stability of protecting decorative coatings on the basis of colloidal cement glue (Kaprielov and Karpenko, 2006).

Targeted structure formation with increase in specific surface area can be achieved by means of uniform grain distribution without formation of aggregates. It is known that with increase in particle fineness and in surface energy the propensity to aggregation increases which significantly varies upon addition of various modifiers in the course of mechanical activation which prevent coalescing of cement particles during grinding. Addition of various modifiers in the course of mechanical activation makes it possible to obtain chemically modified material and enables control of physicochemical properties and processes of structure formation.

Modifier was added using two procedures: with mixing water and upon combined grinding of components. In accordance with the adopted procedures the modifier consumption is 7.9% of cement weight. Using the combined activated mixture of colloidal cement material in preset ratio of 70:30 and the mixture of separately activated and mixed cement with fillers cement mortar was prepared with subsequent preparation of samples. The influence of technological procedure, type of processing (combined and separate grinding of components) on strength kinetics was determined on samples of cement paste of normal consistency.

Initial components were loaded into preliminary wetted chamber of bladed mixer in the following sequence: sand, water, colloidal cement material with modifier. The stuff was mixed in 120 s. The ultimate compression strength was determined using preformed cement bar samples with the size of 40×40×160 mm (cement/sand in the weight ratio of 1:3), water/cement ratio: $W/C = 0.4$. Table 4 summarizes the experimental results of laboratory samples.

DISCUSSION

Analysis of increase in specific surface area of various filler materials as a function of grinding time, determined by a T-3 meter on the basis of air permeability approach, demonstrated that intensive increase in specific surface area occurs in the grinding time interval from 10 to 50 min, then the kinetics of increase in specific surface area becomes slower. It is established that sands and concrete scrap screenings are characterized by good grindability, and in 90 min specific surface area of sand increases up to 1001 m²/kg, and that of concrete scrap screening up

to 990 m²/kg. It can be concluded that the grinding time necessary for achievement of required specific surface area is nearly the same both for sand and concrete scrap screening.

Good grindability of concrete scrap screening can be attributed to lower hardness of included minerals in comparison with quartz sand as well as by the fact that the screening contains mortar portion which has lower strength.

Investigation into particle size distribution of fine cement systems revealed that with increase in specific surface area of powder the content of finer fraction in total bulk of ground material increases, that they contain particles of micro- and nanosizes, which eliminate occurrence of stresses in solidified cement stone which could lead to microcracking and other defects. Portland cement without additives and fillers is characterized by negligible content of fine fractions of 0-10 μm (slightly above 6%). According to the obtained results the colloidal cement glue of optimum composition on the basis of various cements is a fine dispersed powder with dominating particle size in the range from 0 to 40 μm. Herewith, the size of more than 90% of powder particles is from 0 to 30 μm. Exactly these particles predetermine high specific surface area and activity of fine cement systems.

Water requirement of dispersed cement systems varies as a function specific surface area of the components. (Fig. 1) illustrates that with increase in specific surface area up to 600 m²/kg at the same filling ratio the water requirement of cement paste increases even higher and equals from 31 to 45.1% at various filling ratio. Probably, maturity of particle surface and high specific surface area promote increase in water requirement. Decrease in water requirement of fine cement systems upon addition of fillers is probably related with denser internal structure of particles.

Modifier addition to fine cement systems by conventional mixing resulted in the fact that samples upon similar conditions of solidification are characterized by various strengths due to non-uniform distribution of the additive in total bulk of the mixture. Modifier addition upon mechanical activation promotes more uniform distribution of ingredients and uniform distribution of the additive over the surface of cement and filler particles. The data in Table 4 demonstrate the advantage of combined activation of components of colloidal cement material with modifier. Strength of the samples after combined activation increased at all

stages of solidification, especially in the first day (by about 1.5-fold).

CONCLUSIONS

The following conclusions can be drawn from the above discussion:

1. The possibility of application of concrete scrap screening instead of natural sand upon production of fine cement system is proven.
2. Intensive increase in specific surface area of components occurs in the milling time range from 10 to 50 min, then the kinetics of increase in specific surface area becomes slower.
3. It is determined that natural sands and screenings of concrete scrap are characterized by good grindability.
4. With increase in specific surface area of fine mixtures the content of finer fraction in total bulk of ground substance increases, it contains particles of micro- and nano-sizes, which promotes prevention of stresses in solidified cement stone capable to induce microcracking and other defects.
5. Addition of fillers (natural sand and concrete scrap screening) slightly decreases water requirement of fine cement systems.
6. Application of preliminary regrinding of filler to specific surface area of cement and combined regrinding of components of colloidal cement material with modifier to optimum specific surface area promotes increase in strength at all stages of solidification.

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