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Optimization of compositions and technology of production of small-clinker floured cements using the most effective fillers

Abstract. Considering the physical chemistry of grinding it is worth quoting the grinding of mineral building material as "the change of physical-chemical properties of finely ground materials can not only be due to the reducing the particle sizes, at mechanical grinding significant changes of the crystalline structure of their surface layers (thickness 15-20 microns) take place, in many cases the technological properties of fine powders are not so much due to dispersability but are namely due to the structure rupture", at that the energy costs for this are "significantly greater than for the exposal of surfaces with a clean cleavage". The speed of heterogeneous chemical processes involving fine powders is determined primarily not by the magnitude of their specific surface area, as commonly is believed, but by the decrease of energy of activation as the result of crystalline structure rupture and amorphization.

However, both specific surface area and energy demands to achieve are actual evaluation of the effectiveness of any material grinding at a particular unit.

The main factor of the production process of cements of low water demand is the grinding, characterized by grindability.

Key words: small-clinker floured cements, grindability, activation, phosphoric and blast furnace slags, energy resource-saving.

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Introduction

One of the urgent problems of cement production in the Republic of Kazakhstan is the absence of raw materials, because the considerable part of natural sources of raw materials in the immediate proximity is exhausted and has no real replacement. In the future, it will lead to the inevitable partial or complete replacement of natural resources by technogenic ones, the importance of which should increase steadily, including environmental reasons [1,2-6,13].

Nowadays, Kazakhstan has accumulated 10 million tons of electro-phosphoric slags (LLP "Kazphosphat", Taraz) and 85 million tons of metallurgical slags (LLP "IspatKarmet" Temirtau) and ash of the heat electric generation plant (HEGP) of Akmolinskaya oblast and North- Eastern regions of Kazakhstan. The richest deposits of carbonate, dolomite and dolomitic rocks are found in South Kazakhstan and Zhambyl oblasts [7-9].

The project aims to establish an industrial production of a new generation of high-tech small-clinker floured binders, the technology of which is based on joint grinding of the initial clinker or Portland cement without additions with carbonate rocks and wollastonite- containing rocks, as well as with blast furnace and electro -thermo- phosphoric slags and other technogenic wastes in the presence of superplasticizer (1 - 2%) in the Republic of Kazakhstan. Cement ratio: filler in the small-clinker floured cements (SCFC) varies from 20:80 (SCFC -20) to 80:20 (SCFC-80) [10-12]. The new type of cement has a low water demand, high brand strength from 300 to 1000, processibility, rapid rate of hardening in normal conditions. Its production is wasteless, environmentally full, allows the utilization of many technogenic materials. Construction from concretes on SCFC meets the principles of "green building".

Materials and methods

Portland cements manufactured atm Portland cement of LLP "Standartcement" PC500A0 according to GOST 10178 – Portland cement 500 free of additions, of normal hardening; Portland cements of LLP "Shymkentcement" were used as a binder.

Fields of carbonate -containing rocks (limestones), located in South Kazakhstan oblast, Tulkibasskyi area, LLP "Umit SD" and Zhambyl oblast, Sary- suskyi area, LLP "Kazphosphate" in the Republic of Kazakhstan have been used for study.

Blast-furnace and electro-thermo-phosphoric slags were the fillers in the study. Also, there was used a chemical additive in the form of a chemical (superplasticizer) - C-3 (specs TS 6-36- 0204229-625-90 *) of JSC "Polyplast" production.

The grinding equipment for research has been chosen on the basis of obtained results of grindability of SCFC - 100. The grindability has been studied in four types of laboratory mills, carrying out the grinding in different ways such as a ball one with abrasive and shock- gravitational grinding, vibratory -ball one (of "Consit" company) with abrasive and shock-vibratory grinding, a spring one with abrasive and splitting -compressing grinding, in centrifugal- planetary mill "Activator - 4m" with impact- abrasive grinding. Grindability of mineral components (PC and mineral additives) was evaluated by the specific surface area (S, cm^2/g), achieved in certain time intervals of grinding in one or another mill. The specific surface area of binders was determined at the PSH-12 (LLC "Hodakov" SPC). Operation of the device is based on the measurement of the specific surface area of powder materials by Kozeini- Karman method for air permeability and porosity of compacted powder layer and average sizes of particles corresponding to it [13].

The particle size distribution (PSD) of SCFC - powders was performed at the laser analyzer "Analisette 22 Nanotech" (Germany) with a measuring range 0,1-2000 microns and "Horiba LA-950" (Japan) with a measuring range 0,01-3000 microns[14-17] .

Results and discussion

Specific surface area (Ssa) of Portland cement and the time required to achieve it were adopted as the grindability criterion. Specifically, of the experiment there were obtained kinetic curves of Ssa dependence from the time and grinding conditions. Specifications of the mills are presented in Table 1.

Table 1

Specifications of mills

Name of the indicator	Types of mills			
	Spring one	Vibratory- ball one of LLC "Consit"	Ball one	Centrifugal planetary mill
1	2	3	4	5
Engine power, kW	1,5	0,55	0,25	15
The volume of the grinding chamber, 1	4,1	5	9,4	1300(x4)
Mass of grinding bodies(balls), kg	-	10-14	15-20	0,6 - 2,0

Diameter of bodies being ground (mm) share in %	-	10-20% 12-60% 14-20%	10-20% 12-60% 14-20%	5 - 20% 10-60% 15-20%
Weight of the material being crushed , kg:				
- max	0,5	1,8	4,0	0,4
- min	0,1	0,7	1,0	0,05

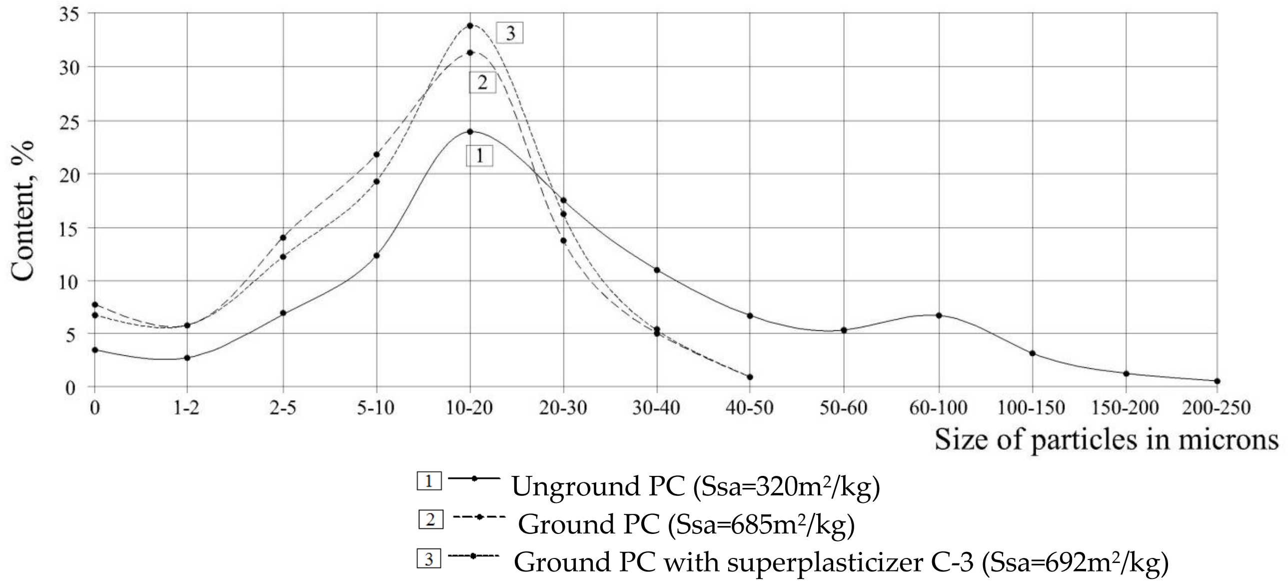

Figure 1. Distribution of the particles of the initial and ground Portland cement according to the sizes

In Figure 1 the granulometric composition of the particles of the initial cement (320 m^2/kg) has a bimodal character of distribution with maxima in the sizes range of 10-20 microns and 63-100 microns. At this the right-hand branch of the curve “stretches” to 200- 250 microns. Grinding PC with 1% C-3 and without it increases the ‘height” of the main maximum (range 10-20 microns) with 29% to 34 and 32%, respectively, due to the re- grinding of larger fractions, at this the role of C-3 in the grinding is low (over the entire range.) Grinding in both cases increases the share of all fractions smaller than 10-20 microns by 10-15%, which should lead to a high speed and depth of the cement binder hydration. Average geometric size of particles of the initial, ground “dry” and with C-3 composes respectively 17.3 microns; 7.8 microns and 8.5 microns [10-12,18-21].

As seen in Figure 2 the character of the curves of PSD of ground sand refers to a bimodal one with maxima in the range of 10 ... 20 microns (25%) and 2 ... 5 microns (17.5%). The total share of average particles 2 ... 20 microns reaches 58%, thin ones (<2 microns) - 25% , large ones (> 20 microns) - 17%. Introduction of SP C-3 at sand grinding leads to the shift of the curve toward thin (28%) and medium-sized particles (70%), fractions over 20-30 microns disappear. At this the content of these particles has considerably reduced (up to 2%).

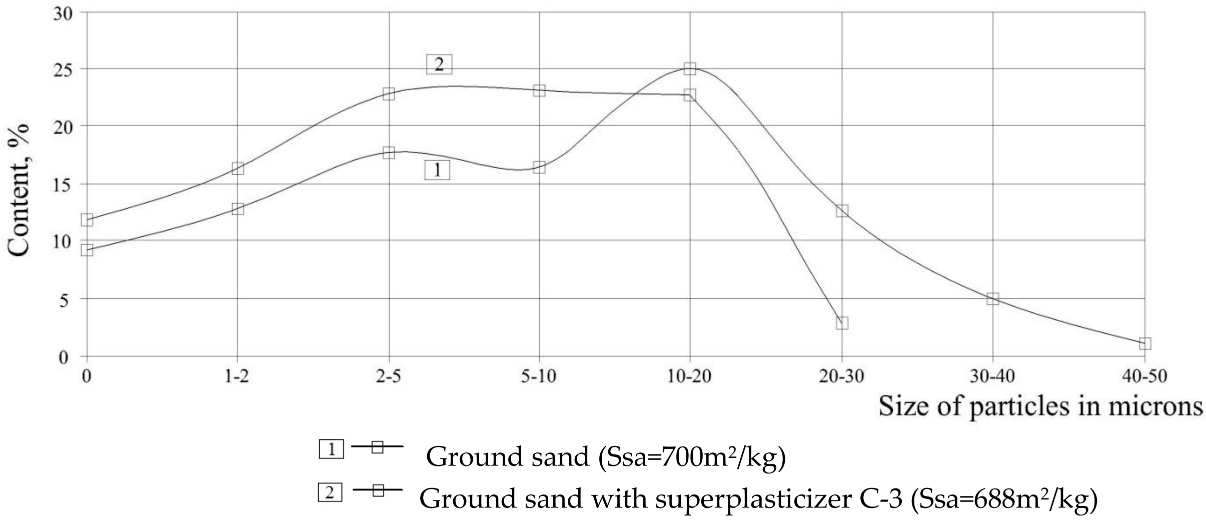

Figure 2. The particle size distribution of ground quartz sand

Comparing the curves of PSD of ground PCs and quartz sand, then at equal Ssa, they differ not only in smaller average geometric sizes of ground sand (4.13 microns and 6.20 microns, without and with C-3 respectively), but also in the lack of particles 30-40 and 40-50 microns of the latter and, in general, by greater influence of superplasticizer C-3 on grinding the material. Character of PSD of limestone (Figure 8) is also described by a bimodal curve, but its peculiarity is in the small maximum (rather a "shoulder") in the range - 10-20 microns. A strongly pronounced maximum of interval of 2-5 microns - 22% dominates, but the share of finer particles 0-2 microns is also high - 33%. On average, their interrelationship with the particles in the range of 2-20 microns is exactly 1:1. Grinding with the addition of C-3 increases the share of fine particles (up to 5 microns) 65%, the share of large and medium particles reduces.

Comparative curves of PSD of Portland cement and of the "second" components of SCFC - fillers are presented in figure 9. They imply that PSD of Portland cement differs significantly from PSD of fillers (with almost equal Ssp ≈ 700m²/kg) at dominance of the fraction 10-20 microns (about 35%) with nearly symmetrical change to the right and to the left. PSD curves of carbonates (limestone and marble) are shifted toward smaller factions and differ

To quantify the efficiency of grinding we introduced the following criteria:

1) grindability factor: $GF = (S_2 – S_1) / \Delta\tau$ (m^2/kg •min),

Where S_1 and S_2 - specific surface area of the material (m^2/ kg) before and after grinding during the time $\Delta\tau$ (min);

2) specific energy costs on the increase of the specific surface area of the material $\Delta S = 100$ m^2/kg

$E = N * \Delta\tau / (\Delta S * 10^{-2})$ (kWh * kg/m^2);

3) efficiency coefficient of SAM at grinding F_{SAM} is equal to the ratio of the GF of grinding with SAM to GF when grinding without SAM: F_{SAM} - GF_{SAM}/GF_o

Results of calculations of the obtained experimental data are presented in Table 2.

Table 2

Design criteria of Portland cement grindability

No.	Indicators	Type of mills							
		Ball one		Consit		Activator		Spring one	
		w/a	C-3 2%	w/a	C-3 2%	w/a	C-3 2%	w/a	C-3 2%
1	2	3	4	5	6	7	8	9	10
1	Specific surface area, m²/kg - initial S_1 - calculated S2	320 380	320 425	320 460	320 600	280 600	300 600	320 500	320 600
2	Grinding time Δτ minutes to S2 (calculated)	480	480	480	360	**6**	4	2	5
3	Coefficient of grindability GC (m²/kg* min)	0,125	0,218	0,29	0,78	53.3	75	90	56
4	Specific energy costs E (S = 100 m²/ kg) * kWh kg/m²	6,66	3,8	3,14	1,17	0,467	0,33	0,028	0,043
5	Efficiency coefficient of SAM at grinding of K_{SAM}	1,74	2,69	1,4	0,62				

In Table 2 specific energy costs, grindability coefficient and grinding time increase in the series: spring, activator, vibration - ball, ball one. Whereas the efficiency coefficient of SAM at grinding increases in the series: spring, activator, ball and vibratory- ball one. In the spring mill SAM is not the grinding intensificator, on the contrary, it slows it down, the reason is probably in a different mechanism of grinding in the spring mill (cracking) when the surface energy effects less on strength than in the vibration one (shock - abrasive mechanism of grinding).

In general superplasticizer C-3 accelerates the grinding from 1.5 to 2 times and reduces energy costs by 40-90%.

Thus, for further studies mills that have industrial analogs have been selected - vibratory- ball and planetary - centrifugal ones. Refusal from a spring mill is connected with the absence of such ones in an industrial version.

Specific surface area of powders - Ssa is an integral one, more precisely, an averaged characteristic of their dispersability, though being widely used. The quantitative granulometric composition or disperse composition which affects the technological characteristics of the powder, as such, and on the properties of the cured binder, in particular cement stone, seems much more informative and meaningful for binders. Therefore, grinding efficiency of PC and its modifying components (quartz sand - as control composition, limestone, etc.) should be assessed precisely by disperse composition - the particle size distribution (PSD), which was done on the laser analyzer “Analisette 22 Nanotech” in the laboratory “CSRIgeolnerud” (Kazan). Grinding of materials was performed in the vibratory- ball mill “Consit”.

There are presented the PSD curves of the initial unground Portland cement and ground “dry” and with C-3 to Ssp about 700 m^2/kg in figures 6-8.

Conclusion

There was found out dependence of Portland cement grindability, depending on the type of grinding equipment.

PC grindability in the presence of C-3 increases in the series: ball, vibratory- ball, spring and activator, whereas the specific energy costs, grindability coefficient and grinding time increase in the series: spring, activator, vibration - ball, ball. At this coefficient of efficiency of SAM in grinding increases in the series: spring, activator, ball and vibratory- ball. In spring mill SAM is not the grinding intensificator , but on contrary, it slows it down, the reason is probably in a different grinding mechanism in the spring mill (cracking) when the surface energy less effects on strength than in the vibration one (shock - abrasive mechanism of grinding).

The opportunity to intensify the process of grinding the fillers -quartz sand, carbonate rock (limestone) and slags have been found out.

So, superplasticizer C-3 has practically no influence on the grinding of sand. Another pattern is observed when grinding it with limestone. Grinding the limestone with additive of C-3 increases the share of fine particles (up to 5 microns) 65% and reduces share of large and medium particles. Such a difference in PSD has a positive topological aspect when combining PC powders and fillers. Particles of the latter ones will fill the volume “gaps” between the PC particles, increasing the density of their total packaging.

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Ең тиімді толтырғыштарды қолдана отырып, ұсақ клинкерлі цементтерді алудың құрамы мен технологиясын оңтайландыру

Аңдатпа. Ұнтақтаудың физикалық химиясын ескере отырып, минералды құрылыс материалдарының ұнтақталуын келтірген жөн: «ұсақ ұнтақталған материалдардың физикалық-химиялық қасиеттерінің өзгеруі тек бөлшектердің кішіреюіне байланысты емес, механикалық ұнтақтауда кристалды құрылымның айтарлықтай өзгеруі олардың беткі қабаттарында (қалыңдығы 15-20 микрон) орын алады. Көптеген жағдайларда ұсақ ұнтақтардың технологиялық қасиеттері дисперстілікке байланысты емес, дәлірек айтқанда, құрылымның жарылуымен байланысты, бұл үшін энергия шығындары «айтарлықтай таза бөлшектелген беттердің экспозициясынан гөрі үлкен». Ұсақ ұнтақтар қатысатын гетерогенді химиялық процестердің жылдамдығы, ең алдымен, олардың белгілі бір беткі қабатының шамасымен емес, көбінесе, кристалдық құрылымның жарылуы мен аморфизациясы нәтижесінде активтену энергиясының төмендеуімен анықталады.

Дегенмен, нақты беткі қабат пен оған қол жеткізудің энергиялық қажеттілігі - бұл белгілі бір қондырғыдағы кез-келген материалды ұнтақтау тиімділігінің нақты бағасы.

Суға төмен сұранысқа ие цементтерді өндіру процесінің негізгі факторы ұсақталумен сипатталатын ұнтақтау болып табылады.

Түйін сөздер: ұсақ ұнтақталған цемент, ұнтақталу, активация, фосфорлы және домна пештерінің шлактары, энергияны үнемдеу.

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Оптимизация состава и технологии производства мелкоклинкерных цементов с использованием наиболее эффективных наполнителей

Аннотация. Рассматривая физическую химию измельчения, стоит отметить измельчение минеральных строительных материалов: «изменение физико-химических свойств тонкоизмельченных материалов может происходить не только за счет уменьшения размеров частиц, при механическом измельчении значительные изменения кристаллической структуры их поверхностных слоев (толщиной 15-20 мкм) во многих случаях технологические свойства тонкодисперсных порошков обусловлены не столько диспергируемостью, сколько разрушением структуры», при этом затраты энергии на это «значительно больше, чем для обнажения поверхностей с чистым сколом».

Скорость гетерогенных химических процессов с участием тонкодисперсных порошков определяется в первую очередь не величиной их удельной поверхности, как принято считать, а уменьшением энергии активации в результате разрыва кристаллической структуры и аморфизации.

Однако удельная площадь поверхности и потребность в энергии для ее достижения являются реальной оценкой эффективности измельчения любого материала на конкретном агрегате.

Основным фактором производственного процесса маловодных цементов является измельчение, характеризующееся измельчаемостью.

Ключевые слова: цементы мелкоклинкерные, измельчаемость, активация, фосфорные и доменные шлаки, энергоресурсосбережение.

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