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Studies of the Relationship between Morphology and Pozzolanic Activity of Different Micro Fillers for HPC

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Abstract – **The given research is devoted to the studies of morphology, particle size and pozzolanic activity of different supplementary cementing materials (SCM) – thermally treated clays as well as waste products – coal ash, waste glass powder, timber and barley ashes. Different properties of SCM have been analysed: specific surface area, surface morphology, particle size distribution, chemical composition and lime-combining capacity in the reaction with $Ca(OH)_2$. The obtained results have indicated that micro additives with larger specific surface are more reactive.**

Keywords – **Cementing materials, micro fillers, morphology, pozzolanic activity, surface area.**

I. INTRODUCTION

Traditional concrete has four components: cement, water, fine aggregates, and coarse aggregates. For modern or advanced concrete technology, various mineral admixtures have been widely used in concrete constructions. The purposes of using these mineral admixtures in concrete are to gain economic benefits, protect the environment, improve the workability of fresh concrete, enhance strength, and especially the durability of hardened concrete, and decrease hydration heat [1]. Mineral admixtures are usually divided in siliceous and/or aluminous materials that are added to concrete during mixing. Many researchers also call them supplementary cementing materials (SCM). Four commonly used mineral admixtures are the following: silica fume, slag, fly ash, and metakaoline [1].

Metakaoline is produced by calcining Kaoline clay. The material is an aluminosilicate that reacts with free lime in a similar manner as silica fume [2]. When *clay* or shale is calcined at the temperature of 700 °C to 800 °C, the clay is dehydrated and its crystalline structure is totally disorganized. Silicon 28etrahedral becomes active so that it can react at ambient temperature with the lime liberated by the hydration of cement mineral alite or tricalcium silicate (C_3S) and tetracalcium aluminate ferrite (C_4AF) [3]. Metakaoline improves concrete performance by the packing effect and by reacting with calcium hydroxide, to form secondary calcium silicate hydrates (CSH) [1].

It is evident that particle surface texture has some influence on the strength of the aggregate-cement paste bond, but difficulty of measurement means that the property is rarely considered and almost never the subject of a specification requirement. The effects of particle shape and surface texture on workability are seemingly more pronounced for fine aggregates, in which particle geometry is more difficult to characterize [4].

II. EXPERIMENTAL PROCEDURE

A. Materials

The following supplementary cementing materials were studied: metakaoline – kaolinite clay, calcined at 700 °C (MK); Devonian clay, Liepa deposit, Latvia, calcined at the temperature of 700 °C (DC); borosilicate glass powder (BSG); lead silicate glass powder (LSG); coal ash (CA); wood ash (WA) and barley ash (BA).

In the current study, metakaoline (MK) was obtained in the laboratory furnace by the calcination of commercially available kaolinite clay at the temperature of 700 °C. The sintering process of MK used in this study lasted approximately 3 hours with temperature increase of 15 °C/min and holding of MK in the maximum temperature (700 °C) for 1 hour. After calcination MK was ground in the laboratory planetary ball mill Retsch PM 400 for 15 min, 20 min and 30 min at a speed of 300 rpm.

Devonian clay (DC) was also calcinated at the temperature of 700 °C, by using a slow calcination regime (heating rate of 10 °C/min, kept at the maximum temperature for 3 h). Already calcinated clay was ground in planetary ball mill Retsch PM400 for 15 min, 20 min and 30 min at a speed of 300 rpm.

The coal ash (CA), i.e., coal combustion bottom ash used in the research comes from a boiler house operating on coal. CA was ground for different grinding periods – 4 min, 15 min, 30 min and 45 min respectively.

Both applied glass types are the by-products and were selected for studies, due to the high content of amorphous SiO_2. Borosilicate glass powder (BSG) and lead silicate glass powder (LSG) were obtained from the local lamp recycling [5]. The chemical composition of both glass powders is given in Table 1.

Wood and barley ashes used in given experiments were taken from the local heating plant furnaces in Latvia. In order to obtain material with homogenous composition, ashes were ground in the already mentioned laboratory ball mill for the period of 4 min, at the grinding speed of 300 rpm.

The chemical compositions of each SCM are summarized in Table 1. In Table 2, the treatment (burning or grinding) along with designation is given.

TABLE 1

CHEMICAL COMPOSITION OF SCM USED FOR STUDIES

Chemical composition, wt%	SCM							
	Metakaoline (MK), wt%	Devonian clay (DC), wt%	Coal ash (CA), wt%	Borosilicate glass powder (BSG), wt%	Lead silicate glass powder (LSG), wt%	Wood ashes (WA), wt%	Barley straw ashes (BA), wt%	St.dev., %
SiO_2	51.52	70.78	15.96	74.20	69.07	59.92	66.07	± 0.5
Al_2O_3	40.18	13.89	26.56	1.65	1.03	4.30	5.38	± 0.3–0.5
Fe_2O_3	1.23	4.00	0.86	0.16	0.19	1.60	1.78	± 0.03–0.5
CaO	2.00	1.32	10.30	2.09	1.39	19.89	6.06	± 0.2–0.5
MgO	0.12	0.67	2.17	-	-	2.32	1.68	± 0.2–0.5
K_2O	0.53	3.36	-	0.93	1.17	3.59	7.14	± 0.1–0.5
Na_2O	0.08	0.06	-	3.82	8.02	0.37	0.24	± 0.1–0.5
TiO_2	2.27	-	-	-	-	-	-	± 0.3–0.5
SO_3	0.00	-	0.50	-	-	-	-	± 0.1–0.5
C	-	-	29.40	-	-	-	-	± 0.5
PbO	-	-	-	-	20.02	-	-	± 0.5
B_2O_3	-	-	-	16.63	-	-	-	± 0.5
LOI	2.01	2.62	14.32	-	-	6.65	11.05	± 0.3
Total	99.94	96.70	100.00	99.48	100.00	98.64	99.39	

Regarding the chemical analysis, the attention was first of all paid to the content of SiO_2 and Al_2O_3, as those compounds are expected to play a main role in a pozzolanic reaction. DC, BSG and LSG exhibit the largest content of SiO_2 – around 70 wt%, also in wood and barley ashes it is rather high – 60 wt% – 66 wt%, the lowest is observed in CA – only around 16 wt%. MK has the highest content of Al_2O_3 – around 40 wt%, then CA follows with 27 wt% and DC with 14 wt%. For the rest of additives, content of Al_2O_3 is insignificantly low.

TABLE 2

DESIGNATION AND PREPARATION CONDITIONS OF SCM

Description of SCM	Designation
Kaolinite clay, burned at 700 °C, grinding time: 15 min	MK 15
grinding time: 20 min	MK 20
grinding time: 30 min	MK 30
Devonian clay, burned at 700 °C, grinding time: 15 min	DC 15
grinding time: 20 min	DC 20
grinding time: 30 min	DC 30
Borosilicate glass waste, grinding time: 30 min	BSG
Lead silicate glass waste, grinding time: 30 min	LSG
Coal ash, grinding time: 4 min	CA 4
grinding time: 15 min	CA 15
grinding time: 30 min	CA 30
grinding time: 45 min	CA 45
Wood ash	WA
Barley ash	BA

B. Methods

In order to characterize the relationship between particle morphology and their pozzolanic activity of selected SMC, data obtained by different methods were compared:

- particle shape and morphology – by scanning electron microscopy (SEM – TESCAN Mira\LMU Field-Emission-Gun);
- specific surface area of particles – by Porosimeter NOVA 1200E (0.35 nm – 200 nm) “Quantachrome Instruments”;
- content of reactive SiO_2, Al_2O_3 and Fe_2O_3 of particles – according to the Florentine method [6], [7];
- ability to react with $Ca(OH)_2$ (described below [8], [9]);
- measurements of particle size – by “90 Plus”, “Brookhaven Instr”.

The measurements of pozzolanic activity are based on the reaction between free lime and pozzolanic additive or SCM. During pozzolanic reaction, the free lime reacts with pozzolanic additive; thus, the simplest way how to follow the dynamics of pozzolanic reaction is either to measure the amount of calcium hydroxide or the amount of unreacted pozzolana. For the experiment, 1 g of SCM is mixed with 75 ml of saturated $Ca(OH)_2$. Then after different periods of time (in given case – after 3 and 24 hours), the amount of absorbed $Ca(OH)_2$ was measured by titration with 0.1 n HCl solution. The results are expressed as the amount of reactive CaO (wt%). Results, obtained by different methods are summarized in Tables 3–7.

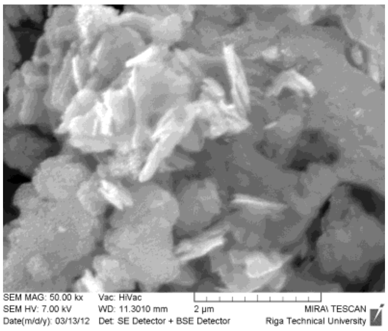
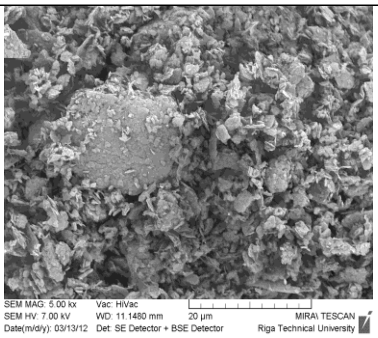
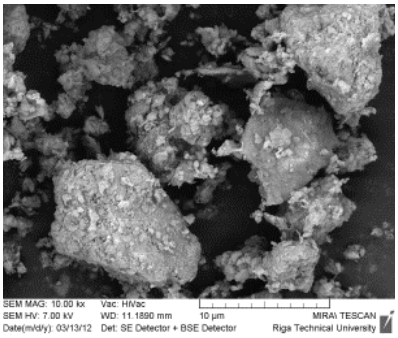
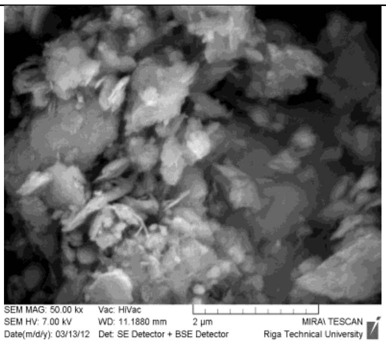
III. RESULTS

A. Particle Shape and Morphology (SEM)

Images, characterizing the particle shape and morphology obtained by SEM, are depicted in Table 3.

TABLE 3

SEM MICROGRAPHS OF SCM

	
MK 15	MK 30
	
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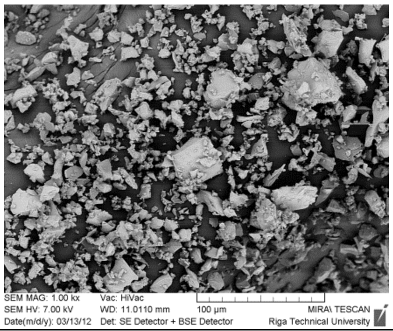
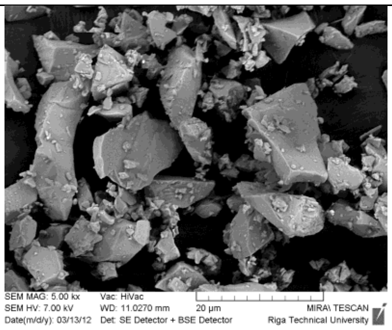
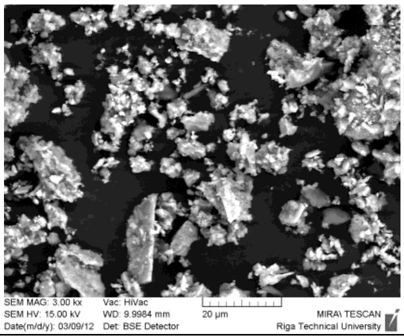
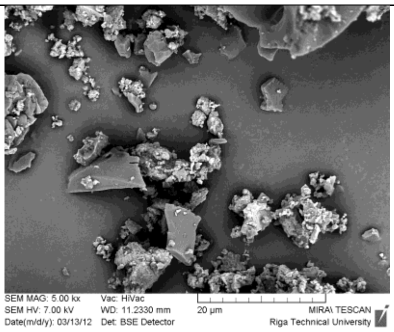
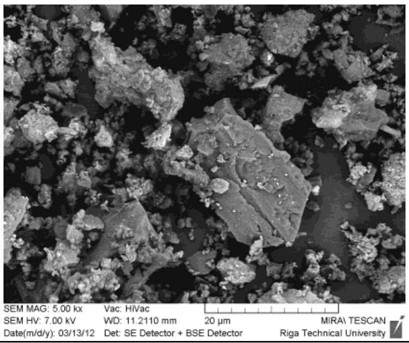
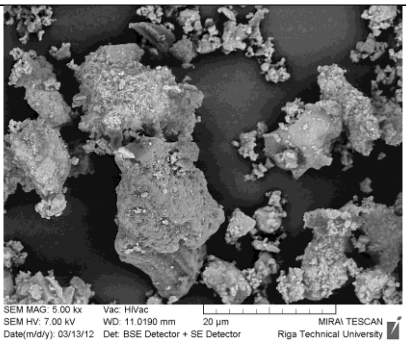
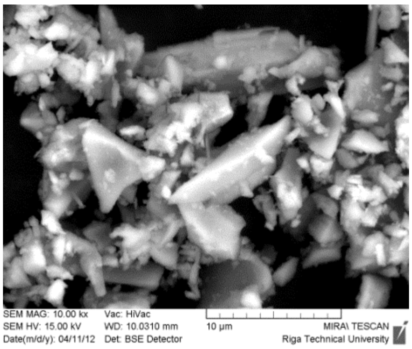
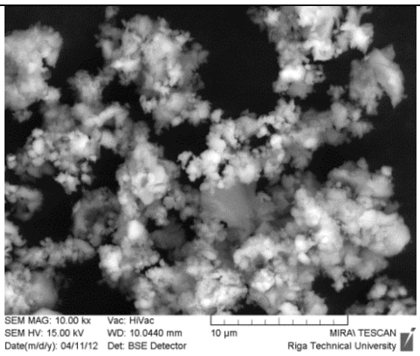
	
BSG	LSG
	
CA 4	CA 15

SEM

	
CA 30	CA 45
	
WA	BA

The shape of both clay particles – metakaoline (MK) and Devonian clay (DC) – is rather similar. Both have a lamellar shape characteristic of clay materials. For Devonian clay, the margins of plates are little bit more rounded than for kaolinite particles. For the particles obtained from waste glass, the shape is irregular, sharp-edged with smooth surface (glass powder BSG and LSG). Shape of CA particles is rather similar independently of the treatment parameters – irregular, angular, with porous surface. Regarding wood and barley ashes (bio ashes), the shape of particles could be compared with glass powder particles, as it is irregular and angular.

B. Pozzolanic Activity and Content of Reactive SiO_2 and R_2O_3

Content of reactive SiO_2 and R_2O_3 (common amount of Al_2O_3, Fe_2O_3 and TiO_2), detected by chemical analysis as well the pozzolanic activity, measured after 3 and 24 hours in reaction with $Ca(OH)_2$, expressed as the amount of reactive CaO (wt%) is summarized in Table 4.

C. Particle Size and Specific Surface Area

The specific surface area of studied SCM, measured by BET, and the particle size, measured by DSL (as min/max and average) are summarized in Table 5.

TABLE 4

CONTENT OF REACTIVE SiO_2 AND R_2O_3, AS WELL THE POZZOLANIC ACTIVITY, MEASURED IN REACTION WITH $Ca(OH)_2$

SCM	Content of reactive SiO_2/R_2O_3, wt%	Pozzolanic activity after 3 h (amount of CaO, wt%)	Pozzolanic activity after 24 h (amount of CaO, wt%)
MK 15	1.10 / 32.76	0.0392	0.0483
MK 30	1.91 / 32.47	0.0336	0.0483
DC 15	1.02 / 6.73	0.0319	0.0332
DC 30	1.05 / 7.79	0.0430	0.0357
BSG	0.50 / 0.36	0.0269	0.0095
LSG	-	0.0157	0.0157
CA 4	1.51 / 6.35	0.0402	0.0220
CA 45	2.13 / 7.37	0.0340	0.0203
WA	4.88 / 4.04	0.0084	0.0500
BA	0.56 / 2.56	0.0179	0.0078

TABLE 5

PARTICLE SIZE AND SPECIFIC SURFACE AREA, m^2/g

SCM	Surface area, m^2/g	Min / max particle size, nm	Dominating particle size (50 %), nm
MK 15	16.52	739 / 923	840
MK 30	15.86	476 / 600	541
DC 15	21.95	177 / 697	542
DC 30	21.42	702 / 886	799
BSG	1.126	-	-
LSG	0.252	538 / 678	612
CA 4	11.64	1832 / 2312	2084
CA 45	9.849	776 / 979	883
WA	0.293	1364 / 3190	2464
BA	0.480	914 / 1153	1039

IV. DISCUSSION

The shape of both clay particles – metakaoline (MK) and Devonian clay (DC) – is rather similar. Both have a lamellar shape characteristic of clay materials. The values of specific surface are also rather close – around 16 for MK and 21 m^2/g – 22 m^2/g for DC. However, the longer grinding time in both cases does not provide the larger specific surface (see Table 5).

As the specific surface area for DC is slightly larger than for MK, it could be expected that the Devonian clay would be more effective as pozzolanic additive than metakaoline, however the chemical analysis indicates higher values of reactive SiO_2 and R_2O_3 in metakaoline, if to compare with calcinated Devonian clay. Respectively, if the amount of reactive SiO_2 in metakaoline is 1.10 %...1.85 % and R_2O_3 is 32.19 % ... 32.76 %, then in Devonian clay the numbers are lower – SiO_2 – 1.05 % ... 1.11 %, but R_2O_3 6.75 % ... 7.79 %. It should be mentioned that in both clays the amounts of reactive SiO_2 are very close – around 1 %; however, the amount of reactive Al_2O_3 differs significantly.

According to Table 4, after 3 hours of reaction with $Ca(OH)_2$, MK 15 is more active than MK 30, the pozzolanic activity for both is 0.0392 (MK 15) and 0.0336 (MK 30), respectively. Although the values are rather close, it could indicate that longer time of grinding does not improve the activity. At the same time, after 24 h of reaction with $Ca(OH)_2$, activities are still close and both MK 15 and MK 30 have similar values – 0.0483 that is more than after 3 h, and it indicates that after 24 h the reaction has occurred more completely.

Activity after 3 h for DC 15 is lower than for DC 30 (0.0319 and 0.0430), which probably means that longer time of grinding has improved the activity of DC. Activity of DC 15 after 24 hours of reaction is 0.0332, i.e., slightly higher than after 3 h, but for DC 30, on the contrary, it has decreased to 0.0357. Although in this case the values are close, it could indicate that for DC the reactions with lime more intensively occur during the first hours. At the same time, it could be mentioned that despite the fact that all obtained values are rather close, MK 15 seems to be more active from both clays after 24 hours of reaction.

The shape of CA particles is rather similar independently of the treatment parameters – irregular, angular, with porous surface, but the specific surface area of particles is 11.64 m^2/g and 9.849 m^2/g, respectively for particles ground for 4 min and 45 min, i.e., after longer grinding time, the specific surface area decreases, in contrast to what was expected. Obviously, the optimal grinding time has been exceeded and particles have started to form agglomerates. This also explains why the pozzolanic activity for CA 45 is lower than that for CA 4, despite the fact that the amount of reactive SiO_2/R_2O_3 is higher for CA 45 particles. Respectively, for the values of specific surface area of 11.64 m^2/g and 9.849 m^2/g, the corresponding values of reactive SiO_2/R_2O_3 are 1.51%/6.35% and 2.13 %/7.37 %, while for the particles ground for 4 min or 45 min, the pozzolanic activity – 0.0402 %/0.0220 % and 0.0340 %/0.0203 % (see Tables 4 and 5).

For the particles obtained from glass powder, the shape is irregular, sharp-edged with smooth surface (glass powder BSG and LSG), but in this case, particles with a larger specific surface area are more active, e.g., if for BSG glass particles, the specific surface area is 1.126 m^2/g, but pozzolanic activity after 1 h and 3 h is 0.02688 and 0.00952, then for LSG glass particles with surface area of 0.0252 m^2/g, pozzolanic activity is 0.01568 both after 3 h and 24 h. Consequently, although both types of glass have a particular pozzolanic activity, it is possible that in concrete mix they would act first of all as a micro additive for filler.

It could be expected that comparing different particles with a similar shape – irregular, angular and with close values of a specific surface area, e.g., CA 4 and BSG (with a surface area of 11.64 g m^2/g and 11.20 g m^2/g), also the pozzolanic properties will be close; however, the content of reactive SiO_2/R_2O_3 differs significantly – for CA 4 it is 1.51 %/6.35 % that is much more than in BSG powder – 0.50 % and 0.36 %. This could be related to the surface morphology of coal ash particles, which is more porous, as well as to a different chemical composition.

Regarding wood and barley ashes (bio ashes), the shape is irregular and angular; however, the values of specific surface area are significantly lower, i.e., the particles of bio ashes are larger. If to compare both ashes, a specific surface area is larger for barley, although the content of reactive SiO_2/R_2O_3 % is significantly lower, at the same time the pozzolanic activity for barley ashes is higher than for wood ashes. Thus, it could be concluded that also for this group of SCM particles with a larger specific surface will be more pozzolanically active. In other words, for particles of similar origin and chemical composition, the pozzolanic activity will be determined by a specific surface area as well as the surface morphology.

If to compare the values of specific surface area and pozzolanic properties for particles with similar size, e.g., for CA 45 with dominant particle size of 800 nm – 900 nm and DC 30 with average particle size of 800 nm (see Table 6), it could be concluded that the specific surface area depends both on the shape and the surface morphology of particles.

TABLE 6

COMPARISON OF SPECIFIC SURFACE AREA AND CONTENT OF REACTIVE SiO_2/R_2O_3 FOR PARTICLES WITH A SIMILAR SIZE

SMC	Dominant particle size, nm (obtained by DSL)	Specific surface area, m^2/g (BET)	Content of SiO_2/R_2O_3, wt%
DC 30	798.6	21.42	1.05 / 7.79
MK 15	840.3	16.52	1.10 / 32.76
CA 45	882.5	9.849	2.13 / 7.37

Placing SCM consecutively from particles with smaller size to larger (see Table 7), no interconnection with corresponding surface area values could be observed that once again confirms that for particles with similar size, the specific surface could be significantly different and, thus, also the pozzolanic activity.

TABLE 7

COMPARISON OF PARTICLE SIZE AND SPECIFIC SURFACE AREA OF DIFFERENT SCM

SCM	Dominant particle size, nm (obtained by DSL)	Specific surface area, m^2/g (BET)
MK 20	522.8	19.54
MK 30	541.1	15.86
DC 15	541.8	21.96
DC 20	723.8	22.17
DC 30	798.6	21.42
MK 15	840.3	16.52
CA 45	882	9.849
CA 4	2083.9	11.64

V. CONCLUSIONS

If to compare the particles with similar chemical composition, i.e., with similar content of reactive SiO_2 and R_2O_3, the particles with a larger specific surface area will be more pozzolanically active, but if to compare the particles with equal or similar shape, the particles containing larger amount of reactive SiO_2 and R_2O_3 will be more active. The higher the values of specific surface are, the better the pozzolanic properties of material are.

The surface area for particles with similar size could be different that indicates the different morphology. At the same time, the pozzolanic activity for particles with similar size could be different, if the morphology is different, independently of the similarities in shape.

Metakaoline and Devonian clay, coal ashes, both waste glass powders, wood and barley ashes, could be used as an additive or micro filler for concrete aggregate as well as the supplementary cementing material, improving the properties of high performance concrete.

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Linda Krāģe, Diāna Bajāre, Aleksandrs Korjakins, Janīna Sētiņa, Inna Juhņeviča, Inta Kiriloviča. Pētījumi par sakarību starp dažādu HPC mikropildvielu morfoloģiju un pucolānu aktivitāti

Saistoties portlandcementa minerāliem − trikalcija silikātam (C_3S) un tetrakalcija alumoferītam (C_4AF) − hidrolīzes reakcijās, papildus hidrauliskajiem savienojumiem kalcija hidrosilikātiem vai aluminātiem (CHS vai CHA), veidojas arī portlandīts $Ca(OH)_2$, kam nav hidraulisku īpašību. Tāpēc, bieži tiek izmantotas aktīvās minerālās piedevas, kas reaģē ar šo $Ca(OH)_2$, veidojot sekundāros kalcija hidrosilikātus vai aluminātus.

Darbā pētītas dažādu smalki dispersu materiālu īpašības. Šie materiāli ir potenciālas aktīvas minerālās piedevas – termiski apstrādāti māli (importēts kaolīns un Latvijas devona māli (Liepas atradne), apdedzināti 700 °C), tādi atkritumprodukti kā akmeņogļu pelni, atkritumu stikla pulveri, koksnes pelni un miežu pelni. Analizētas sekojošas piedevu īpašības: īpatnējās virsmas laukums (noteikts, izmantojot BET metodi), virsmas morfoloģija (SEM), daļiņu izmēru sadalījums, ķīmiskais sastāvs (aktīvā SiO_2 un R_2O_3 (kopējais Al_2O_3, Fe_2O_3 un TiO_2) saturs, kā arī pucolānu aktivitāte jeb kaļķa saistīšanas spēja, reaģējot ar $Ca(OH)_2$. Iegūtie rezultāti rāda, ka daļiņu smalkumam ir būtiska nozīme kaļķa-pucolāna reakcijā, aktīvākas ir mikropiedevas ar lielāku īpatnējo virsmu. Tomēr šis pieņēmums ir spēkā līdz optimālajam daļiņu smalkumam, un tālāka daļiņu malšana nesniedz apmierinošus rezultātus, jo malšanas laikā daļiņas veido aglomerātus, kurus nepieciešams disperģēt šķīdumā labākai reakcijai ar kaļķi. Vienāda vai līdzīga izmēra daļiņām atšķirīgas morfoloģijas dēļ var būt atšķirīgas īpatnējā virsmas laukuma vērtības, kas būtiski ietekmē arī pucolāna īpašības. Noteikts, ka pētītās piedevas izmantojamas gan kā papildus cementējošs materiāls – aktīvā minerālā piedeva, gan kā mikropildviela jeb piedeva betona pildvielai.

Линда Краге, Диана Баяре, Александрс Корякинс, Янина Сетиня, Инна Юхневича, Инта Кириловича. Изучение взаимосвязи между морфологией и пуццоллановой активностью различных микронаполнителей для HPC

Связываясь между собой, минералы портландцемента – трикальций силикат (C_3S) и тетракальций алюмоферрит (C_4AF), в результате гидролиза в дополнение к гидравлическим соединениям – гидросиликату или алюминату кальция, образуют также портландит $Ca(OH)_2$, который не обладает гидравлическими свойствами. Поэтому часто используют активные минеральные добавки, которые реагируют с $Ca(OH)_2$, образуя вторичные гидросиликаты или алюминаты кальция.

В работе исследованы свойства различных мелкодисперсных материалов или потенциальных активных минеральных добавок – термически обработанной глины (импортированный каолинит и девонская глина латвийского происхождения (месторождение Лиепа), обожженные при 700 °C), а так же продуктов отходов – угольной золы, порошка стекла, древесного и ячменного пепла. Анализированы следующие свойства: удельная площадь поверхности (БЕТ), морфология поверхности (СЭМ), распределение размеров частиц, химический состав (содержание реакционноспособных SiO_2 и R_2O_3 (общее количество Al_2O_3, Fe_2O_3 и TiO_2)), а так же пуццоллановая активность или реакционная способность извести и $Ca(OH)_2$. Полученные результаты показали, что размер частиц имеет огромное влияние на известково-пуццоллановую реакцию, более реактивными являются микродобавки с большей удельной поверхностью. Однако это предположение действительно до какого-то оптимального размера частиц, так как дальнейшее уменьшение размера частиц не дает удовлетварительных результатов и указывает на формирование агломератов в процессе помола, которые нужно диспергировать в растворе для лучшей реакции с известью.

Частицы идентичного или аналогичного размера из-за различной морфологии могут различаться значениям площади удельной поверхности, что значительно влияет на пуццоллановые свойства. Определено, что исследованные добавки могут быть использованы как дополнительный цементирующий материал – активная минеральная добавка, а также как микронаполнитель или добавка к наполнителю бетона.