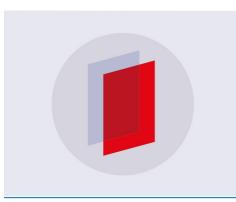
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Micro-Filler from Crushed Concrete Waste Influence on **Properties of New Concrete Made from Concrete Waste**

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Abstract. The micro-filler researched in the work was gotten by crushing concrete and reinforced concrete waste and sieving gotten particles. Researched crushed particle size was smaller than 0.125 mm. Main micro-filler characteristics were determined. After doing research according to the special research method and according to the gotten results it can be stated that additive (micro-filler) made from crushed concrete waste is active. It was determined that when using concrete waste for concrete mixture mixing a higher amount of water is needed since part of the water is absorbed by used waste filler moistening. Big amount of this excess water is probably absorbed by smaller mixture particles gotten during the crushing process. Due to this reason W/C ratio changes. Research was done that showed how the requirement of water is changed with smaller mixture particles of which size is 0-0.125 mm. 5 %, 10 %, 15 %, 20 %, 25 % and 30 % MFA was put in and mixed with water up to normal thickness paste. Research results showed that by changing the cement part with micro-fillers W/C ratio rises, normal thickness cement paste gotten when W/C is 0.27, and when gradually rising micro-filler amount the requirement for water rises proportionally. Wanting to evaluate micro-filler additive effect to self-binding material's hydration process calorimetry research was carried out. Cement and cement with micro-filler compositions' (85% CEM II+15% MFA) calorimetry curves were compared. When researching the hydration process electrical conductivity research was also carried out as well micro-filler influence on concrete strength properties. According to cement composition strength property results it can be stated that micro-fillers lower cement strength properties. After conducting research, it was determined that used filler from crushed concrete waste has to be sieved and separated from very small particles, which are smaller than 0.125 mm, since micro-particles that appear between large (4/16) and small (0.125/4) fraction in the mixture change the hardened concrete structure by rising the distance between fillers. Due to this even a small amount can significantly change gotten sample physical and mechanical characteristics (compression strength is lowered 38-42%, bending strength is lower 15 %) when comparing with mixture made using natural fillers.

1. Introduction

Independent from demolition building type and processing method, building demolition main stages are the same: first waste preparation, crushing, sorting, metal, which armoured reinforced concrete constructions, separation, first sieving, granulation, metal separation, sieving [1]. For building

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demolition usually excavators, hydraulic crushing scissors, metal separation aggregates are used. After demolition work, waste is processed using special granulation and sorting equipment. In these processes crushers, granulators, magnetic separators, sieves and air separators, which separate thermos-isolation, wallpaper and other impurities from concrete pieces, are used [2].

Other materials such as armature, timber, roofing and other materials should be removed from concrete rubble from building demolition waste during production, so that they couldn't contaminate gotten filler [3]. It would also be useful and rational, if wall concrete constructions would be separated from columns, ceiling. After thoroughly completing all rubble production stages we get various fraction bigger, smaller and micro-fillers [4]. Micro-fillers are fillers of which majority pour out through 0.063 mm pore sieve and which can be placed in building materials to give them certain properties (LST EN 12620:2003+A1:2008).

Micro-fillers according to specific surface area are classified to different size micro-fillers. To do so is appropriate due to the fact that in various material mixtures they play different roles [5]. Bigger micro-fillers, which particles are bigger than cement, can be useful in concrete and mortar, when big and or normal size sand is used, which have smallest fractions. These kinds of micro-fillers improve mixture filler granular-metric composition, lower their voids, allow saving binding materials, in concrete allow lowering W/C ratio, slightly rises concrete mixture mobility. Therefore, when there is the same mixture mobility, it is possible to get stronger concrete.

Average size micro-fillers, which particles are of cement size, are used when it is needed to rise cement paste amount in a mixture. Then the requirement for water rises and hardened cement stone strength lowers. These micro-fillers are used when producing smaller strength concrete with higher class binding materials, then this size micro-filler usage is rational. In mixtures the volume of binding materials paste increases, mixture mobility increases, products thicken better.

Especially smaller micro-filler particles are at least several times smaller than cement. These kinds of micro-filler particles fit between cement grains and because of this cement granular-metric composition improves. These micro-fillers are used for producing very strong concrete, plastics and putty. Very small micro-filler particles have special properties, due to the fact that their activated surface can better interact with binding material. Concrete strength rises due to the binding materials stone density increase.

Micro-filler surface activity is related to surface accumulated energy, surface layer coarseness and chemical composition. Surface energy is higher the smaller particles and the bigger surface area is. Just produced micro-fillers have higher surface energy, higher adhesion and absorption with other materials; they better encrust with macro-molecular film of water, and then micro-filler resistance to outer normal and shear tensions lowers. To moisten sharp-edged particle surface it requires more water and the mixture gotten is not as mobile. When micro-filler particles are round their surface area is smaller, surface energy is smaller as well. Such particles more compactly arrange in mixture, thicken easier, less water is needed to use for mixture moistening. Micro-filler particles bigger than 0.063 mm have relatively small surface area, way lower surface energy, due to which these particles are assigned to the inert fillers.

In this work we provide research, during which research was done on micro-filler, gotten crushed concrete and reinforced concrete waste and sieved gotten particles, results. Researched crushed particle size was smaller than 0.0125 mm. Main micro-filler characteristics, their activeness, and other properties that show how mixtures with such micro-fillers mixing requires higher water amount were determined. Also, such micro-filler usage recommendations are presented.

2. Research methods and raw material characteristics

Rentgenographic concrete waste research was performed with diffractometer DRON-7. (Cu anode, Ni filter, monochromator, gaps 1:8:0.5 mm). Diffractometer tube mode during work: U=30 kV, I=10 mA. Written diffraction pattern was encrypted comparing gotten experimental inter-planar distance d and line relative integral intensity I/I0 values with respective values ASTM file.

Device Mettel toledo Education line EL20 was used for binding material solution and suspension pH research, Mettel toledo Education line EL30 was used for electrical conductivity research. Calorimetric research was carried out with device ToniCal III (Toni Technick GmBh).

Portland cement and Portland cement with MFA additive paste normal thickness and beginning of setting was determined according to LST EN 196-3:2005+A1:2009. Cement with MFA additive bending and compression strength was determined according to LST EN 196-1:2007.

Cement and MFA specific surface area was determined with Blain machine according to standard LST EN 196-6:2010. Cement and micro-filler from concrete waste bulk density was determined according to LST EN 1097-3:2002. Cement particle and micro-filler particle density was determined according to LST 1476.2:1997.

Cement, concrete waste and prepared sample micro-structure was determined with scanning electron microscope (SEM EVO LS 25, Zeiss Germany). Raw material chemical (elemental) composition was determined with analyser OXFORD Instruments INCA Penta FET×3.

According to research method, described in literature (Martusevičius et. al 2002), MFA activity was determined. Mineral additive activity – that is additive characteristic in room temperature to react with calcium hydroxide. Micro-filler paste with hydrated lime (4:1) is kept for 7 days in air (Figure 1 left), afterwards it is kept for 3 days in water (Figure 1 centre). Mineral additive is held active when its paste with hydrated lime after experiments doesn't spill out (Figure 1 right).



Figure 1. View of filler aggregate samples during the activity detection: filler aggregate and paste of hydrated lime putty in an air (left); filler aggregate and paste of hydrated lime putty in water (centre); filler aggregate and paste of hydrated lime putty after investigations (right)

Active SiO₂ amount in micro-filler was determined by dissolving SiO₂ in potash (KOH) according to LST 1577.1:1999. Active SiO₂ amount by % is calculated according to this formula:

$$SiO_2 = \frac{m_1}{m_2} \cdot 100,\%$$
 (1)

where: m_1 – sample mass before experiment, g; m_2 – sample mass after experiment, g.

Cement, smaller filler standard sand and micro-filler from crushed concrete waste were used for experiments. Cement: composite limestone Portland cement CEM II/A-LL 42,5 N, which meets LST EN 197-1:2011/P:2013 standard requirements. Smaller filler: natural sand meeting LST EN 197-2 requirements. Micro-filler from crushed concrete waste was gotten by sieving crushed concrete structures.

Cement and micro-filler chemical composition is presented in Table 1, and properties in Table 2.

	Chemical composition, %						
	CaO	SiO_2	SO_3	FeO	Al ₂ O ₃	MgO	K ₂ O
Cement	67.81	14.67	5.72	3.57	3.33	3.07	1.83
Micro-filler	47.08	36.62	1.16	3.58	5.09	4.43	2.04

Table 1. Chemical composition of materials.

Table 2. Properties of materials.							
	Bulk density g/cm ³	Particle density g/cm ³	Specific surface area cm ² /g				
Cement	1.02	2.75	3200				
Micro-filler	0.96	2.50	2910				

After researching micro-fillers it was determined that without calcium hydro-silicate, which is in micro-filler, this raw material's main minerals are these (Figure 2): quartz (Q), calcite (K), dolomite (D), feldspar (F), also portlandite (P) Ca(OH)₂, illite (I). According to micro-filler rentgenografic research results it can be determined that MFA mineral composition depends on starting material's, used for crushing, cement stone composition and partially on bigger filler type that was in concrete.

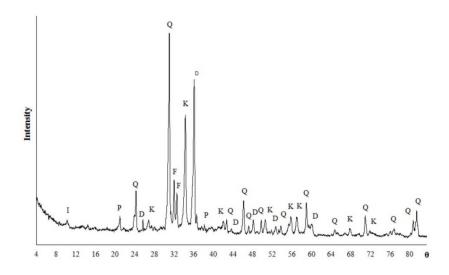


Figure 2. X-ray pattern of filler aggregate: Q – quartz; K – calcite; D – dolomite; F – feldspars; P – portlandite; I - illite

Researched cement and MFA surface image is shown in Figure 3. Gotten micro-filler particles are of uneven shape, particle surface, like that of cement particles, is with sharp, not rounded corners. MFA particle size reaches 0.5-100 µm, cement powder biggest particle size reaches 50 µm.

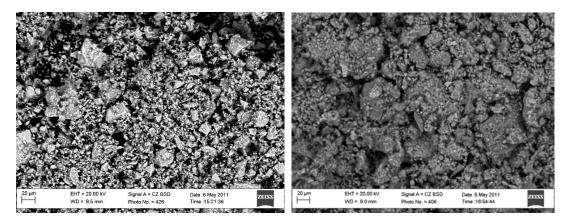


Figure 3. Surface view of the particles of the cement (left) and filler MFA aggregate (right) (SEM picture)

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Performing activity detection of micro-filler MFA according to LST 1577.1:1999 it was determined that MFA has 30 % active SiO₂, due to which it can be attributed to active micro-filler, in which SiO₂ amount can be 5–50 %. MFA activity can be determined according to (Martusevičius et al. 2002) research method and according to gotten results it can be said that the additive is active. However, additive effect on cement paste's hardening was additionally researched and it was determined what kind of influence it had on hardening process' characteristics.

3. Results and discussions

Research was done that shows how requirement for water in binding material's mass is changed with smallest concrete mixture particles – MFA. 5 %, 10 %, 15 %, 20 %, 25 % and 30 % MFA was put in and mixed with water till paste was of normal thickness. Figure 4 shows water and solid particle ratio dependency on MFA amount and determination of beginning of setting results.

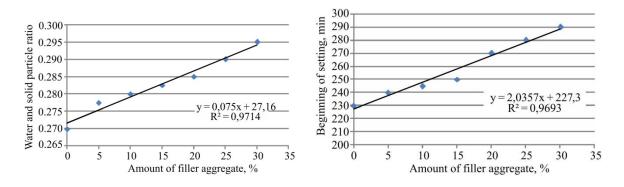


Figure 4. Water and solid particle dependence on the amount of filler aggregate in cement paste and dependence of initial set of Portland cement on the amount of filler aggregate in the paste

Research results have shown that changing mixture cement part MFA raises water and solid particle ratio. We can see that without waste normal thickness paste is gotten when ratio is 0.27 and when gradually rising MFA amount the requirement for water rises proportionally and is 0.295 when MFA amount in mortar is 30 %. We get a similar dependency when determining binding materials with MFA beginning of setting. When putting MFA in binding material the beginning of setting time increases by even 60 min.

Calorimetric research was done to evaluate MFA additive effect on binding material's hydration process. When cement hydrates, heat is emitted and in different cement hydration stages this amount of warmth is different. Cement (marking 100% CEM) and cement and micro-filler compositions (85% CEM+15% MFA) calorimetric curves were compared. Since in MFA can stay non-hydrated cement minerals and formed portlandite analogical research was done with pure MFA (100% MBA). Emitted heat speed and amount change during hydration process is shown in Figure 5.

Mixing binding materials with water sudden emission of heat happens in compositions. Cement mixed with water and moistening its particles mineral crystals start to melt. During first 30 min cement emission of heat speed reaches about 14 W/kg h, however by putting MFA into cement the speed lowers to 8 W/kg h. Later, in cement as well as in cement with micro-filler compositions this process slows down; however, after 4 h second sudden heat emission stage starts, during this period cement hydration crystallization is happening and after 10 h after mixing materials with water starting cement and micro-filler compositions heat emission speed becomes close to cement hydration speed. In Figure 5 we can see that mixing micro-filler with water heat barely released. During first 3 min heat emission speed reached only 1 W/kg h, afterwards lowered and there was no second heat emission stage.

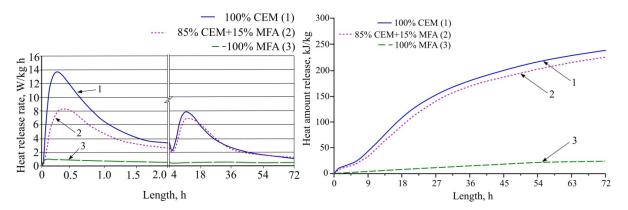


Figure 5. The heat release rate during the hydration of test materials and their compositions (left) and heat amount released during the hydration from the tested materials and their compositions (right)

Comparing cement and cement with micro-filler compositions emitted heat amounts (Figure 5) we can see that heat emission depends on cement amount in compositions. We can also see that emitted heat amount from micro-filler is very small (Figure 5, curve 3).

When researching hydration process electrical conductivity research was done (Figure 6). From presented data in figure it can be noted that from cement and MFA ions are emitted into suspension and suspension is electrically conductive. Cement electrical conductivity during 3 min raised up to 15 μ S/cm, while that of micro-filler – up to 2,85 μ S/cm. Cement electrical conductivity continued to rise and after 180 min from mixing with water start reached 18 μ S/cm, and after 220 min ion emission from cement suspension started to lower. During this period cement beginning of setting started and ion amount started to decrease. MFA after 3 min electrical conductivity gradually lowered. Electrical conductivity decrease in suspension with micro-filler shows that ion exchange between particles and suspensions' liquid phase happens weakly.

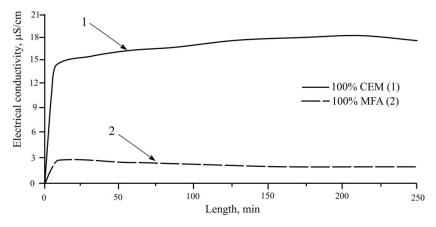


Figure 6. Variation of electric conductivity of the cement and filler aggregate during the hydration process

After conducting research, it can be noted that emitted heat amount from MFA slightly rises (Figure 5) and from MFA into suspension ions are emitted (Figure 6), which can mean that MFA has a certain amount of active materials: free lime amount or part of non-hydrated cement.

When researching MFA additive effect on cement's strength characteristics 5 standard mortar mixtures were prepared by changing Portland cement part with micro-filler: 0 % (B0), 5 % (B5), 10 % (B10), 20 % (B20), 30 % (B30) and determining mortar bending and compression strengths after 28-day hardening.

Figure 7 shows hardened sample bending strength values. After determining sample bending strengths, as it could have been expected, we saw that biggest strength after 28-day hardening was achieved by B0, which didn't contain any MFA. It can be noted that sample in which cement part was replaced by 5 % MFA (B5) bending strength is quite close to sample's B0, which has no MFA, strength. In composition where cement 10 % part was changed by MFA bending strength lowers by 5 %, while rising micro-filler amount up to 30 % bending strength lowers by even 15 % when compared to sample B0 strength values.

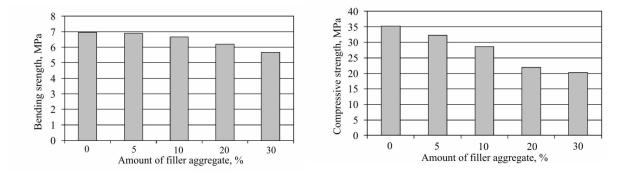


Figure 7. Dependence of samples' bending strength (left) and compressive (right) on the amount of filler aggregate in compositions investigated

According to Figure 7 shown cement sample compression strengths after 28-day hardening results it can be noted that depending on MFA amount cement composition compression strength lowers. However, it can be noted that samples which have a composition of 20 % and 30 % cement replaced with MFA compression strength values after 28-day hardening lowered by even from 38 and 42 % when comparing to sample B0 strength values. According to cement composition strength properties results it can be said that MFA lowers cement strength properties.

4. Conclusions

Carried out research showed that micro-filler gotten from crushed concrete waste changes the requirement of water for getting normal paste. When putting in bigger micro-filler amounts into cement normal thickness paste was gotten with bigger water amount, due to this when mixing mixtures which have micro-fillers from crushed concrete waste overall water requirement in mixture should grow because of micro-filler influence too.

Even though carried out research showed that the additive is active, however, by changing part of cement with micro-filler from concrete waste lower amount of heat emission speed and its amount in mixture is obtained. It is believed that due to the inactive ion emission from micro-filler not only cement hardening will get slower but also hardened sample mechanical strength will also lower.

After carrying out research it was determined that used filler from crushed concrete waste has to be sieved and separated from very small particles, which size is smaller than 0.125 mm, since micro-particles that appear between bigger (4/16) and smaller (0,125/4) fractions in mixture change hardened concrete structure, raise distance between fillers. Due to this even a small amount of them considerably changes gotten sample physical and mechanical characteristics (compression strength lowers 38-42%, bending strength 15%).

Acknowledgment

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