

Investigation of the influence of fine recycled sand on the setting behaviour of cement when used as supplementary cementitious material (SCM)

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Abstract. The building materials industry makes a major contribution to greenhouse gases emitted each year, particularly by the cement clinker production. Therefore, the aim should be to maintain an increased part of building material from demolition sites in the material cycle. The use of the fine material (< 2mm) from demolition waste in concrete has so far proved to be problematic due to the increased water demand and loss of compressive strength. One approach is the use of recycled concrete powder (RCP) as supplementary cementitious material (SCM). Demolition material used in this study has been obtained from discarded railroad sleepers and pre-crushed as sand (< 4 mm). The recycled sand was subjected to a mechanical and thermal activation process before use, then was ground to a particle size <63 μm and then fired at 4 different temperatures (750°C, 800°C, 850°C, 900°C). The aim was to convert parts of the hydrated C-S-H structure back into reactive silicate phases through firing process. They can contribute again to the hydration process when used as supplementary cementitious material. The ground and thermally treated material - called SCM - was examined for their physical and chemical properties. Subsequently, 10 and 20 Vol.-% were replaced by the SCM in a binder mixture, respectively. In a first step, the different water demand of the binders was documented. Ultrasonic methods were used to investigate the stiffening and setting behaviour of the binders. The decisive factor here was the proportion of chemically bound water in the binder mixtures. Finally, the mechanical properties of the binders were investigated in mortar tests. Acceptable compressive strengths were achieved compared to the reference mortar (mortar mixture without cement substitution). At first glance, it seems possible to use it as an SCM.

1 The use of recycled concrete powder (RCP) for cement replacement

In construction industry, the focus is currently on environmentally relevant aspects to conserve natural resources by improving the material cycle. One approach to keep materials in the material cycle is the use of fine recycled concrete components. These were so far scarcely used in reprocessing. In Germany, crushed sands with grain fractions < 2 mm have mainly been used in road construction - with correspondingly low binder contents [1]. Studies show, that mineral fine powders can certainly be used to improve the workability and increase the compressive strength via the packing density, e.g. [2]. However, research is currently being carried out of the thermal reactivation of hydrated cement constituents from recycled sands. The resulting hydraulically active phases can contribute to cement hydration again and thus be used as a cement additive. The primary goal of thermal reactivation is the generation of the C2S phase, which forms at relatively low temperatures. However, C2S is a polymorphic phase, so the heating and cooling process must be optimised [3]. Two modifications that have hydraulic properties are α' -C2S and β -C2S. Due to the

higher disorder state in the crystal lattice, α' -C2S is considered the more hydraulically active modification. These C2S polymorphs also have a smaller crystallite size and a higher specific surface area at lower firing temperatures [4]. β -C2S is formed predominantly at temperatures above 800°C [5; 6]. Serpell and Lopez [7] observe a maximum content of α' -C2S at 800°C, with a decreasing tendency at higher temperatures. In addition, a correlation between the α' -content and the subsequently determined compressive strength is described. An overview of the different approaches in terms of starting material, temperature parameters and water demand is presented in the review of Carrico et al.[8]. Here most of the cited authors use recycled cement paste as a precursor material.

In this paper, the question is if fine recycled concrete powder (RCP) can be used as a supplementary cementitious material without a previous separation process (separation of cementitious parts and fine aggregates). The aim of this study is to investigate whether the proportion of hydraulically active silicate phases generated by the firing process is sufficient to

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influence the water demand and the setting behaviour of the binder.

2 Process steps for creating the binder mixtures

2.1 Mineralogical composition of the untreated recycled sand

The mineralogical characterization of the precursor material and of the thermally treated materials was analyzed by X-ray diffraction (XRD) on a PANalytical Aeris diffractometer (Malvern Panalytical) with $\text{CuK}\alpha_1$ radiation. The samples were scanned in a range between $5\text{-}70^\circ 2\theta$, with a step size of 0.02° and a step time of 25.5s. The mineralogical composition of the recycled sand appears favourable for its use as a SCM. The material has a high quartzite content and contains feldspars, which indicates a natural sand aggregate. The presence of Portlandite and a low calcite content indicates low degree of carbonation (Figure 1). Based on the amorphous content, the potential to re-generate new calcium silicates from the C-S-H phases can be estimated.

2.2 Processing of the material for use as SCM

The sand was ground in a ball mill for 8-10 minutes at 400 rpm to a particle size smaller than $63\ \mu\text{m}$.

The ground RCP were thermally treated at four different firing temperatures T: 750°C , 800°C , 850°C as well as 900°C , respectively. They were poured into refractory crucibles and then placed in a muffle furnace. After reaching the target temperature (T) after 1.5 hours, this temperature was maintained for 2 hours. To achieve rapid

cooling to room temperature, the powders were immediately removed from the furnace at the end of the firing time and cooled to room temperature. The specific gravity of the powders was determined by helium pycnometer. Using Blaine equipment, the specific surface area of the RCPs was determined according to DIN EN 196-6 [9].

3 Testing setup of the binder admixtures

3.1 Determination of water demand and solidification behaviour

The standard stiffness of the reference, as well as that of the mixtures, was determined according to DIN EN 196-3 [10]. Subsequently, the w/c ratio and the w/b ratio were calculated for each binder mixture. The w/b-value describes the ratio of water to the sum of cement and SCM, the w/c-value only the ratio of water to cement. The test was carried out when 10 and 20 Vol.-% of the cement was replaced by the thermally treated RCP in comparison to the reference (pure cement specimen).

The determination of the setting point is also regulated by DIN EN 196-3 [10]. The setting behaviour in the first 24 hours after preparation of the mortar mixtures was investigated with the aid of the Vikasonic ultrasonic measuring device from *Schleibinger GmbH* to get more information than just only one single result. The cement paste was placed in a Vicat ring clamped between an ultrasonic transmitter and an ultrasonic transducer. A temperature sensor for recording the heat development was also inserted into the mixture. The entire setup can be seen in Figure 2. Sound waves were sent through the cement paste at a sound frequency of 54 kHz, an excitation voltage of 1000 V, a pulse rate of 1.00 seconds, at an interval of 120 seconds.

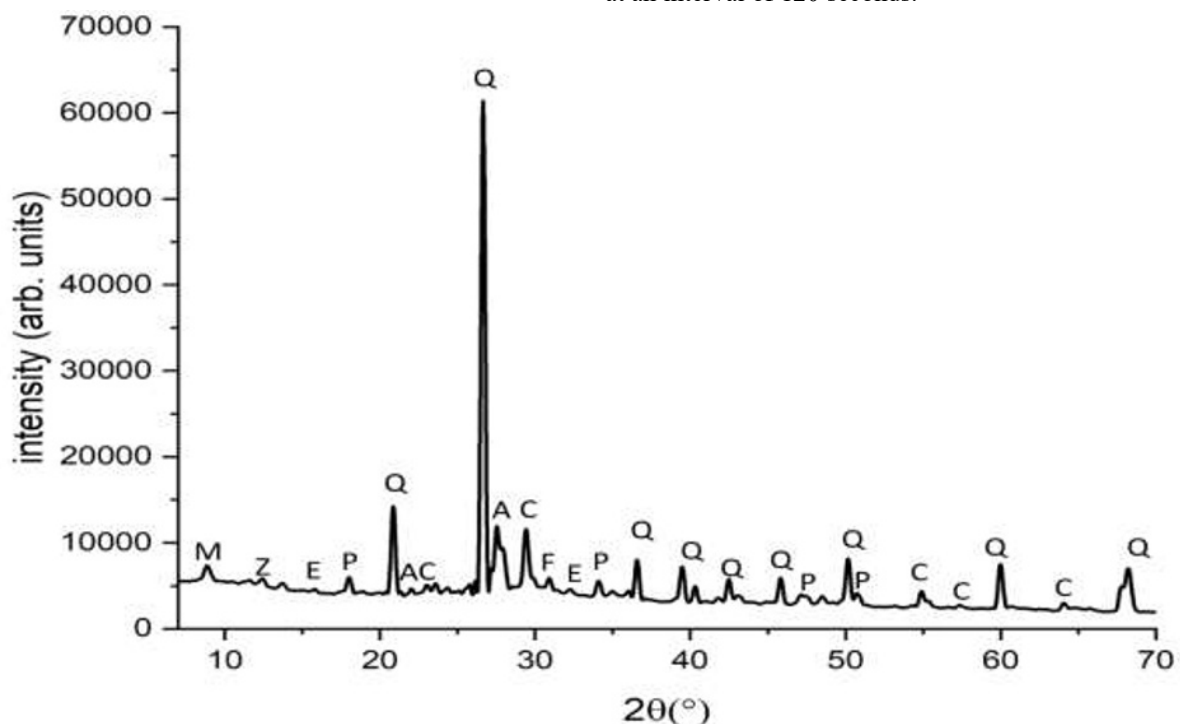


Figure 1: XRD pattern of the thermally untreated RCP; Q=Quartz; C=Carbonate; P=Portlandite; A=Albite; M=Muscovite; Z= Zeolithe; F= Feldspar;



Figure 2 : Experimental setup of the Vikasonic ultrasonic device.

There are publications in which the Vikasonic measuring system for monitoring the setting behaviour of cement and mortar has been tested [11–13]. The authors describe the good agreement - depending on the cement type - between the setting times determined by ultrasound and the cement manufacturer's specifications. Thus, for each type of binder, a bending relationship can be determined between the penetration depth according to EN 196-3 [10] and the examination by means of the Vikasonic measuring device [11]. The manufacturer refers to the good reproducibility and the small deviations of the measurement results compared to the needle penetration method. Nevertheless, the correlation between the penetration depth of the Vicat needle and the ultrasound transit time should be determined [14].

The Vikasonic measures the time of velocity to penetrate the sample. Using the measured sound velocity, the Young's modulus curve in the first 24 hours of the fresh mortar has also been recorded every five minutes. Due to the exothermic reaction of cement with water, the setting behaviour can be observed closely via temperature tracking, which is thus an important indicator of setting [15]. For the formulation, the w/b values were used with which the respective mixtures achieved the standard stiffness (Table 4).

The determination of the start of solidification was carried out by determining the time of the lowest temperature after the start of recording. However, the temperature value here is only a qualitative value since the system was

not set up in an adiabatic state. The end of solidification was determined at the point of the highest slope of the curve of the velocity after the start of solidification. For this purpose, the derivative of the function of the velocity was formed and the inflection point of this was determined. The velocity is included in the formula of the Young's modulus (formula 1):

$$E_{mod} = (\rho \cdot v^2)/1000 \text{ [N/mm}^2\text{]} \quad (1)$$

Where E_{mod} is the dynamic Young's modulus [N/mm²], ρ is the density of the sample [g/cm³] and v is the speed of ultrasonic waves [m/s]. The velocity curve was used since it offers the same extreme points with respect to time as the Young's modulus due to the formula. Using the Origin data analysis and plotting program, the curves were first smoothed and then these relevant markers were determined (Figure 3).

3.2 Use of RCP as SCM in mortar Prisms - Compressive strengths

In order to carry out tests on hardened mortar samples, one formwork of each mixture was filled with three prisms according to DIN EN 196-1 [16]. These were stored under water and sampled 28 days after production. The standardized mortar recipe served as a reference and consisted of 450g cement, 225g water and 1350g standard sand. For the mixtures with RC proportions, the water content was the same and only the cement was replaced by 10 or 20 Vol.-% with thermally treated RCP.

The mortar formulations can be found in Table 1. The prism halves were tested for compressive strength according to DIN EN 196-1 [16].

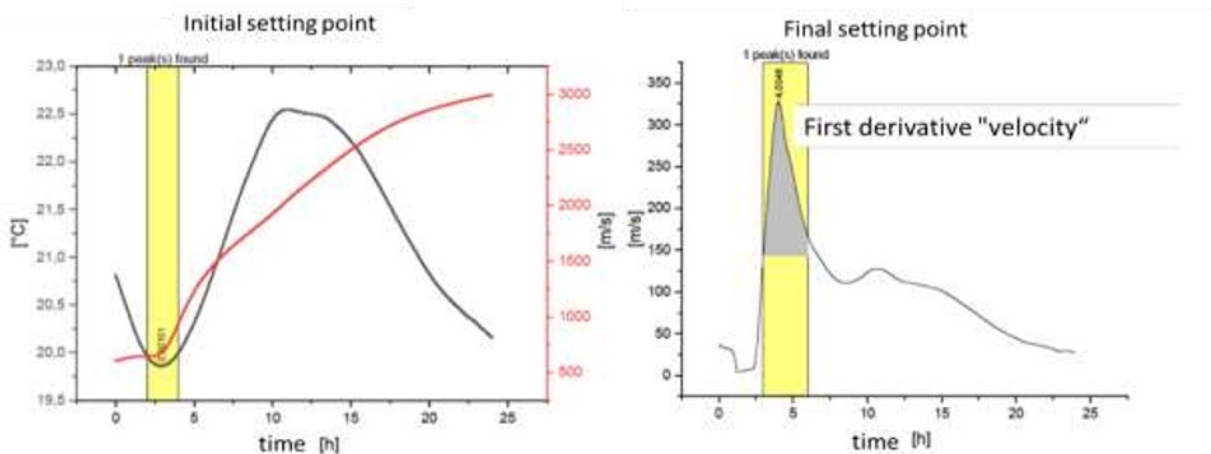


Figure 3 : Method for determining the initial setting point and the final setting point : Left: Temperature and velocity curve; right: First derivative of the velocity to determine the end of solidification.

Table 1 : Weights for compressive strength testing on mortar prisms.

Specimen	C [g]	SCM [g]	W [g]	Agg [g]	w/b
Reference	450		225	1350	0.50
750_10	405	34.2	225	1350	0.51
800_10	405	34.1	225	1350	0.51
850_10	405	34.0	225	1350	0.51
900_10	405	35.2	225	1350	0.51
750_20	360	68.4	225	1350	0.53
800_20	360	68.3	225	1350	0.53
850_20	360	67.9	225	1350	0.53
900_20	360	70.3	225	1350	0.52

4. Results

4.1 Change in mineral composition after the firing process

During the thermal treatment process, the strength-building phases of the old cement are decomposed and transformed into new calcium silicate phases. The XRD investigations show that at predominantly α' -C2S is formed at temperatures up to 800 °C. This modification exhibits a higher disorder state in the crystal lattice compared to β -C2S and can thus be considered as more reactive. At temperatures above 850 °C, only β -C2S is present (Table 2). It can be assumed that the α' modification is formed because of the dehydration of the C-S-H phases.

Table 2: Resulting C2S modifications depending on treatment temperature.

T [°C]	750	800	850	900
Phase 1	α' -C2S	α' -C2S	α' C2S	β -C2S
Phase		β -C2S	β -C2S	

In addition, SEM revealed that new crystalline phases formed from amorphous particles at temperatures around 800 °C (Figure 4). The order state in the crystalline lattice

thus increases from temperatures above 800 °C and the reactivity of the SCM decreases.

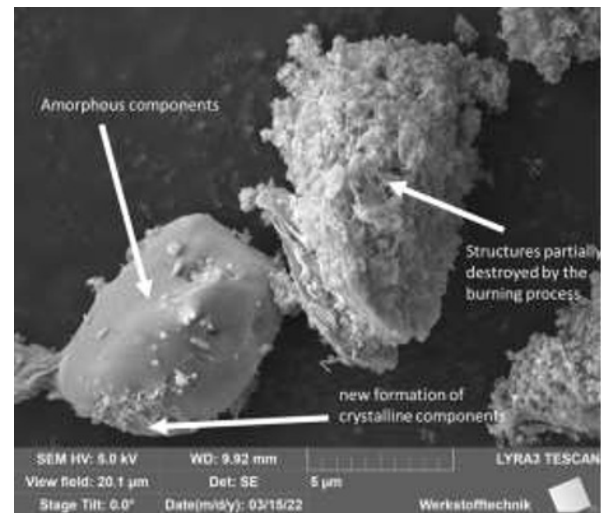


Figure 4 : SEM image of the RCP thermally treated at 800°C.

4.2 Physical properties of the recycled sand after the grinding and the firing process

The values of density determinations of all single RCP are shown in Table 3. With increasing treatment temperature, the density of SCM increases. The scatter of all values was consistently around 0.060 g/cm³. Only at 750°C there was a single value of 2.50 g/cm³ and a resulting deviation of 0.133 g/cm³. Compared to the cement (3.100 g/cm³), the densities of all RCPs are lower. The specific surface area results show an effect of temperature on the RCP texture. The thermally untreated RCP is 3006.76 cm²/g. There is initially an increase up to the first firing stage of 750 °C, as can be seen in Table 3. Subsequently, the surface area of the SCM decreases until 850 °C and increases again slightly to 2788.07 cm²/g at 900 °C firing temperature.

Table 3 : Blaine values and densities of the reference and all used recycling powders (SCM)

material	Blaine value	density [g/cm ³]
CEM I 42,5 N	4000	3.10
RCP untreated	3007	2.17
SCM 750 °C	4140	2.37
SCM 800 °C	3513	2.36
SCM 850 °C	2672	2.36
SCM 900 °C	2788	2.43

4.3 Influence of cement substitution by SCM on the water demand of binders

4.3.1 Water demand determined by standard stiffness test

By determining the standard stiffness, it was possible to determine the w/c as well as the w/b value of the respective samples, see Table 4. For all mixes with replacement of the cement by SCM, it was found that the w/c value increased compared to the reference without cement replacement. The w/b value, on the other hand, first dropped when replacing with untreated RCP, and when thermally treated RCP, the water consumption increased again. At 10 Vol.- % replacement of SCM the w/c and w/b values reached their maxima when the 750°C SCM was used. Thereafter, they dropped with further increase of the firing temperature.

Table 4 : Overview of the w/c and w/b values determines as standard stiffness according to DIN EN 196:3 [10] ; Cement substitution rate: 10 Vol.- %.

material	Specimen name	w/c	w/b
CEM I 42,5 N	reference	0.28	
RCP untreated	untreated	0.28	0.26
SCM 750 °C	750_10	0.31	0.29
SCM 800 °C	800_10	0.31	0.28
SCM 850 °C	850_10	0.31	0.28
SCM 900 °C	900_10	0.31	0.28

At 20 Vol.- % replacement by SCM, Table 5, the w/c value increased significantly more than still at 10 Vol.-% replacement. The values reach their maximum at 850 C and remain approximately the same at 900 C

Table 5 : Overview of the w/c and w/b values for which the standard stiffness was determined according to DIN EN 196:3[10]; Cement substitution rate: 20 Vol.-%.

material	Specimen name	w/c	w/b
CEM I 42,5 N	reference	0.28	
RCP untreated	untreated	0.32	0.27
SCM 750 °C	750_20	0.34	0.29
SCM 800 °C	800_20	0.34	0.28
SCM 850 °C	850_20	0.35	0.29
SCM 900 °C	900_20	0.35	0.29

4.3.2 Influence on the setting points of the binder mixtures

When RCP is used as SCM, an earlier initial setting point is observed in Figure 5 with a 10 Vol.- % replacement in comparison to the reference specimen. The earliest setting point is marked by the sample with 850 °C SCM. With 20 Vol.-% replaced, the initial setting point only occurs earlier with the use of unfired or 900 °C fired RCP than with the reference. The remaining samples are in a similar time range to the reference.

The final setting point (Figure 6), on the other hand, is subject to less variation than the initial setting point. But also here, the compounds with 10 Vol.-% SCM replacement show an earlier solidification end than those with 20 Vol.-%. Both curves are nearly parallel and have their minimum with the recipes using 850 °C fired RCP. The biggest difference to the reference with 238 minutes are 750_20 and 900_20 with 286 and 288 minutes.

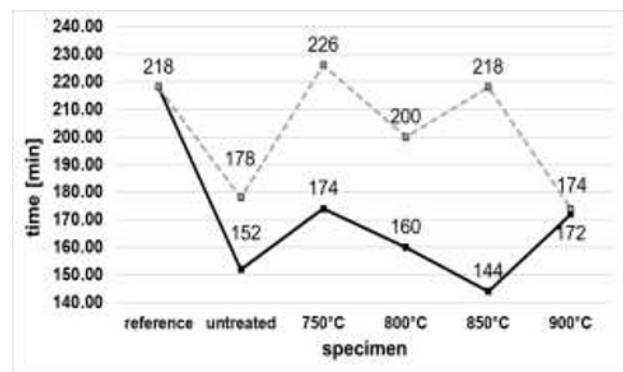


Figure 5 : Initial setting point in cement substitution with RCP as a function of firing temperature: grey: 20 Vol.-% substitution, black 10 Vol.-% substitution

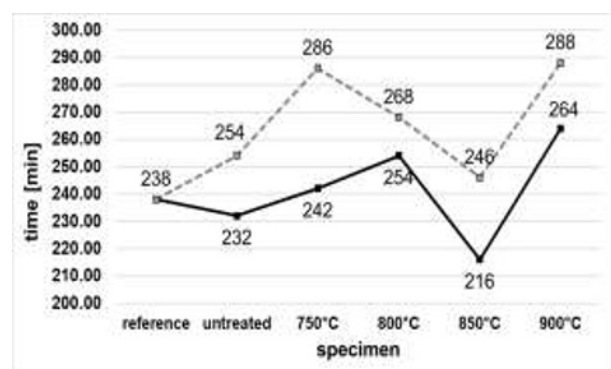


Figure 6 : final setting point in cement substitution RCP as a function of firing temperature: grey: 20 Vol.-% substitution, black 10 Vol.-% substitution.

4.4 Compressive strength

The values of the compressive strength of the mortar prisms indicate that the reference shows the highest value of 45.67 N/mm². The mixtures with 10 Vol.-% replacement are relatively close to each other in the range

from 91.64 % (800_10) to 84.04 % (850_10) compared to the reference. The range of cement substitution with 20 Vol. % SCM is much wider with 85.57 % (750_10) as the highest to 64.33 % (850_20) as minimum. For both, it is noticeable that with the use of the 850 °C SCM, the compressive strength has the lowest values (Figure 7).

5. Discussion

5.1 Evaluation of the standard stiffness results

5.1.1 Cement substitution by 10 Vol.- % SCM

The present results of the tests carried out in this work show only a moderate increase in water demand of the SCM blends compared to the reference. One explanation for this is that in this series of tests the cement was exchanged in Vol.- %, and not in M.- %. This results in a lower total mass of powder content (cement + SCM), since the cement was replaced with an SCM that has a lower bulk density. It can therefore be assumed that the effect of the additional consumption of water due to the use of RC materials is compensated by the reduction in the mass fraction of powder (cement + SCM). The difference between thermally treated and untreated RCP suggests that the thermal treatment creates a more reactive material, which can thus chemically bind more water. Another reason could be that water absorption is higher for materials with lower density [17].

5.1.2 Cement substitution by 20 Vol.- % SCM

The water consumption of the mixtures with 20 Vol.- % cement replacement are slightly higher compared to the

samples with 10 Vol.- % (Table 5). This can be explained by the increase in water consumption when cement is substituted by SCM. However, the increase is not particularly large, which can also be explained by the further reduction in the mass of binders.

5.2 Determination of the solidification behaviour of the binders

The results of the ultrasonic investigation to determine the solidification period with the use of RCP as SCM show an earlier onset of solidification compared to the reference at both 10 Vol.- % and 20 Vol.- % replacement. It is noticeable that the initial setting points 20 Vol.- % substitution occur significantly earlier than the setting point of the reference cement, except for sample 750_20 (Figure 5). The thermally untreated RCP can also stimulate an earlier onset of hydration through its cement phases. The same effect has also been observed in [18], but there only thermally untreated RCP was used. This could be caused by a dilution effect at higher replacement rates.

The final setting point of the sample series with 10 Vol.- % substitution is approximately in the range of the reference, the mixtures with 20 Vol.- % substitution show a slightly later point in time. The total solidification time thus increases in comparison with the solidification time of the reference. The exception here are the samples with 850 °C SCM: these reach their solidification end earlier than the other mixes with SCM substitution. This could be explained by a peak in the formation of β -C2S at 850 °C and the increased transformation at 900 °C of highly active C-A-S into inactive γ -compounds [4; 19]. The general increase in solidification time may be due to

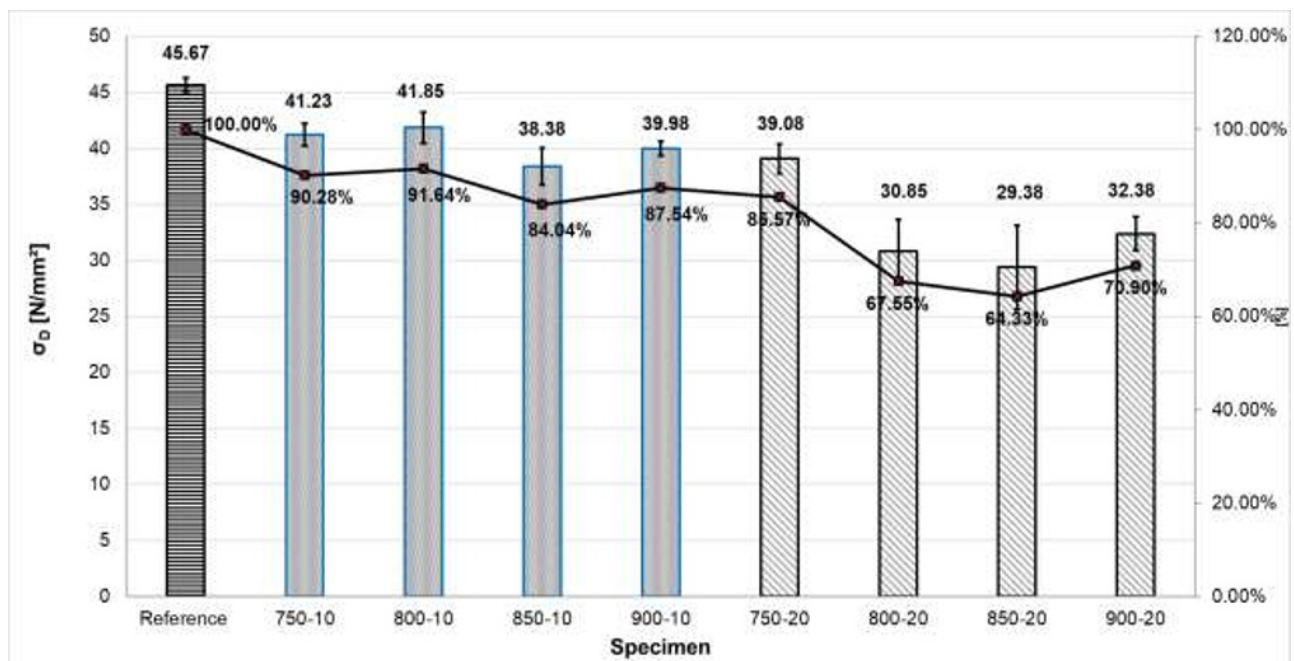


Figure 7 Values of compressive strength and standard deviation determined on mortar prisms with cement substitution by SCM, compared to the reference specimen, which only contains CEM I 42.5 N as a binder.

a slowed formation of hydration products. A reduction in specific surface area due to the addition of the RCP is also a crucial factor, as fineness directly affects the rate of hydration. The fact that this is not the case in the mixes using the RCP fired at 750 °C can be explained by a reduced reactivity of the RCP compared to pure cement. Although the surface area of this RCP is very high and additionally contains less C₃A [20].

Also detectable is a higher duration with the addition of 10 Vol.- % RCP compared to 20 Vol.-%. This is initially contradictory since the mixes with 10 Vol.- % SCM contain a higher fineness as well as more cement. This can be explained by the earlier initial set point compared to a substitution by 20 Vol.- % SCM and the lower density of the mortar mixture. This is because by substituting cement with SCM by Vol. % instead of mass %, the total mass of the mixture is lower, due to the lower density of the RC material. Consequently, fewer starting materials are present for hydration and thus the reaction is shorter in overall duration.

5.3 Compressive strength

The results of the compressive strength test in Figure 7 show an increase with the use of SCM as cement replacement. In [19], the highest compressive strength was determined for RCP with 750 °C firing temperature by using RCP as SCM. This is explained by the dehydration of the C-S-H phases and the formation of hydraulically reactive calcium silicate phases. It can be seen consistently that the specimens with the SCM fired at 750 °C exhibit the highest compressive strength. This is clearly shown at 20 Vol.- % substitution SCM, while at 10 Vol.- % substitution by SCM the values between 750 °C and 800 °C SCM are very close. This is confirmed in the literature [4; 7] and can be explained by the fact that the content of reactive α' -C₂S is decisive for the long-term strength. The formation of a less dense C-S-H matrix above a treatment temperature of 800 °C is another possible explanation [21].

6 Summary and conclusion

This paper describes an attempt to describe the influence of thermally reactivated RCP on the setting behaviour of the binder when used as SCM. A temperature range between 750 °C and 900 °C was tested. This range was selected because reactive C₂S phases can form, which can contribute to cement hydration. The material at hand proved to be favourable in its mineral composition to be used as SCM. Aggregate of natural sand and a low calcite content (low loss on ignition), and a relatively high binder content are included. The XRD analysis confirms the decomposition of the C-S-H phases during the thermal treatment. Between 750 °C and 800 °C the α' -polymorph was the main occurring modification, while further increasing of temperature mainly formed β -C₂S. The particle density increased with treatment temperature, while the specific surface area decreased. This could be

caused by the onset of agglomeration of particles at temperatures above 750 °C.

The ground and thermally activated RCP were used as SCM in cement and mortar. Despite the presumed dilution effect due to the lower densities of the SCM, the water demand of the binder mixtures increased with increasing degree of substitution.

After water demand of each binder mixture was determined, the setting behaviour of the binders was traced by means of ultrasonic testing. It was found that with a cement replacement by SCM of 10 Vol.-%, an earlier initial setting point was reached. However, the final setting points of all mixtures showed only minor deviations compared to the reference cement. This is since the cement substitution by the SCM results in fewer phases that contribute to high early strengths. However, the total duration of the setting process was longer for all mixtures. A degree of substitution of 20 Vol.-% leads to a reduction of the setting duration time compared to a degree of substitution of 10 Vol.-%. At a treatment temperature of 750 °C, lowest influences of the degree of substitution on the setting duration were observed.

Also, in terms of mortar compressive strength, the SCM, which was thermally treated at 750 °C, proved to be promising. At a substitution level of 10 Vol.- %, over 90 % of the strength of the reference mortar was achieved. Even with further cement substitution of 20 Vol.-%, over 85 % of the reference compressive strength was achieved. This represents the smallest drop in mortar compressive strength with increasing cement substitution by the SCM. This agrees well with the XRD investigations. Predominantly the hydraulically more active α' -C₂S phase was determined at a treatment temperature of 750 °C.

The investigated material thus offers an approach to find a possible application as SCM, even without complex separation processes of cement and aggregate. Nevertheless, the SCM can still be optimised by increasing the proportion of reactive components.

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