*Acta Polytechnica* **57**(1):49–57, 2017
doi:10.14311/AP.2017.57.0049


# PROPERTIES AND MICROSTRUCTURE OF CEMENT PASTE INCLUDING RECYCLED CONCRETE POWDER

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Abstract. The disposal and further recycling of concrete is being investigated worldwide, because the issue of complete recycling has not yet been fully resolved. A fundamental difficulty faced by researchers is the reuse of the recycled concrete fines which are very small ($< 1$ mm). Currently, full recycling of such waste fine fractions is highly energy intensive and resulting in production of $CO_2$. Because of this, the only recycling methods that can be considered as sustainable and environmentally friendly are those which involve recycled concrete powder (RCP) in its raw form. This article investigates the performance of RCP with the grain size $< 0.25$ mm as a potential binder replacement, and also as a microfiller in cement-based composites. Here, the RCP properties are assessed, including how mechanical properties and the microstructure are influenced by increasing the amount of the RCP in a cement paste ($\leq 25$ wt%).

Keywords: recycled concrete; recycled concrete powder; recycled concrete fines; mechanical properties; cement replacement.

## 1. Introduction

The disposal and further recycling of concrete is being investigated worldwide, because the issue of complete recycling has not yet been fully resolved [1]. Because concrete consists mostly of aggregates, current research is primarily focused on how to process and add coarse or fine recycled aggregates to new concrete and mortar mixtures [2–6]. However, reuse of the fine powder ($< 1$ mm; largely consisting of a cement paste) produced during the recycling process is still complicated and often expensive. The research presented here investigates how recycled concrete powder (RCP) modified by a high-speed mill (grain size $< 0.25$ mm) performs as a potential binder replacement, and also as a microfiller in cement-based composites.

Concrete is composed of 65 to 70 % coarse and fine aggregates, with the remainder consisting of a cement paste [7]. In recycled concrete aggregates, the part containing the hydrated cement paste causes water absorption and thus can be problematic [8]. The fine fraction of recycled concrete is largely composed of a hardened cement paste, the amount of which depends on the used recycling technology. Additional RCP processing stages (e.g., crushing or grinding) can break grains down to a finer level, to a point at which water absorption no longer plays a significant role. Such RCP modification is conducted according to what specific purpose the material must fulfill. This paper discusses the RCP produced from recycled concrete that serves as a partial binder replacement or as a raw material for cement production.

### 1.1. The RCB as a binder

When reintroducing concrete powder into a mixture, clinker must be recreated with the same conventional burning process as for Portland cement clinker in a rotary kiln [1, 9]. This process requires large amounts of energy and produces high $CO_2$ release, although less than in the case of conventional cement clinker.

Hydrated cement paste can be dehydrated with a heat treatment. Shui et al. [10] investigated this and observed that hardened cement paste treated at 500 °C is composed mainly of dehydrated C-S-H gel, CaO, and part of the original calcium hydroxide ($CaOH_2$). After mixing with water, hydration products, such as C-S-H gel, ettringite and $CaOH_2$ were re-established. Shui et al. [10–12] have demonstrated that dehydrated cement paste is capable of recreating the original hydration products upon contact with water, showing that the mechanical properties of rehydrated cement paste depend primarily on the temperature at which dehydration proceeds and also that the rehydration process can create the alkaline environment that is necessary for recreating fly ash [12]. CaO, produced by the decomposition of $CaCO_3$ and $CaOH_2$, can react with water and recreates $CaOH_2$ and eventually the C-S-H gel with the presence of other compounds. This rehydration process is very similar to the Portland cement hydration; non-hydrated cement grains, which were contained in the cement paste before heat treatment, were also found to react after a contact with water.

Florea et al. [13] explored the influence of dehydration on recycling the concrete powder at different temperatures and possible applications for mortar mixtures, concluding that the very fine fraction of recycled concrete treated at 800 °C can be used to replace 20 wt% of Portland cement CEM I 42.5 in mortars without significant loss of strength, equivalent to replacing the cement with fly ash. The fine fraction of recycled concrete treated at 500 °C was not so efficient and 20 wt% replacement of cement led to a significant decrease in the mortar strength. Pozzolanic activity was observed at both temperatures when the fine fraction was combined with ground granulated slag. When replacing 10 wt% of mixed cement (containing 70 % slag) with a heat-treated concrete powder, the strength of the mortar cured for 28 days improved by 15 to 20 %, probably due to a high lime content. Most of the samples containing the RCP had a higher compressive strength than the observed reference samples. These results suggest that the RCP can contribute to the enhancement of the mechanical properties in mixtures, which, apart from binding constituents, may also function as a filler.

Schoon et al. [14, 15] explored the possibility of using the very fine fraction of recycled concrete as an alternative material for producing the Portland cement clinker, analyzing the RCP samples from various recycling facilities to examine whether particular production processes as well as the sources of old concrete used for obtaining the RCP had an effect on the properties of clinker. The results of this study indicated that neither process nor the source had a significant effect on the mineralogy of the resulting clinker.

Other studies [16, 17] have shown that it is possible to use the RCP as an alternative to conventional materials, primarily as a source of $SiO_2$ and in some cases, because of high content of CaO, as an alternative to limestone. The homogenization phase must be adjusted to the specific chemical composition of any RCP, since homogenization directly affects the production rate, particle size, and the chemical composition of cement [14].

Gastaldi et al. [16] produced a cement clinker made by combining the RCP with limestone and schist. They concluded that the mineralogical composition and a subsequent lack of $C_3S$ prevents the use of the RCP on its own, because of its inappropriate proportions of CaO and $SiO_2$. Because of the high $SiO_2$ content, it is more suitable to use it as a substitute for natural sand in cement mixtures, and any mixture for clinker should contain only 20 to 40 % of the RCP. At higher levels, clinker cannot be considered to be Portland clinker but rather a supplementary cementitious material. Kwon et al. [17] confirmed these findings, suggesting that 30 % of the RCP should be the limit for producing high quality clinker.

Ahmari et al. [19] developed a geopolymer based binder using the RCP, together with fly ash. As a source of silicon and calcium, the RCP, at a maximum quantity of 50 wt%, contributes to a solid bond between the new aggregate and the binder, resulting in a greater compressive strength of the geopolymer, which is in the form of a poly-sialate-disiloxo, and therefore forms a stronger bond than a geopolymer created using fly ash alone.

### 1.2. The RCP as a microfiller and potential binder

In mixtures based on cement, microfillers are used to displace excess water between grains of cement and aggregates. Microfiller grains are smaller than cement grains and ideally should have a spherical shape [20]. The RCP can potentially serve as a microfiller, but prior research into this topic is limited.

Kim et al. [21] investigated the properties of cement pastes and mortars by partially replacing cement with the RCP, observing a workability decrease as the RCP levels increased, with hydration delayed by almost two hours compared with reference cement. In the case of mortar containing 45 % of the RCP, compressive strength decreased by 70 % and water absorption increased by approximately 70 %. Because of this a maximum of 15 % of the RCP was proposed.

Lidmila and Šeps [22–24], who used a very fine fraction of recycled concrete from old railway sleepers, concluded that reactive properties can be achieved by milling, which may expose non-hydrated grains. Because reactivity is low due to the small content of non-hydrated grains, the fine fraction could be used in larger quantities to fortify railway structure subsoils [23], or in small quantities for cementitious composite materials.

Liu et al. [25] focused on the pozzolanic properties of a hybrid powder composed of concrete and brick demolition waste which was analyzed for microstructure and chemical composition. The hybrid powder significantly influenced the microstructure of the cement paste and most of the pozzolanic activity involved particles obtained from crushed bricks. A level of approximately 30 % hybrid power was recommended as a replacement for cement in concrete mixtures.

The assumption regarding exposure of non-hydrated cement grains during milling process as investigated by Lidmila and Šeps [22, 23] was used as a basis for the research described in this paper. In the first stage of the research, the potential reactivity and level of milling for the RCP were explored. RCP particle size was measured to determine if milling the RCP to a level similar to cement exposed non-hydrated grains. RCP hydration heat and mechanical properties were also measurement, in order to investigate the impact of increasing RCP levels on the properties of cement pastes.

## 2. Materials and samples

Samples used in this research were made of the RCP milled by LAVARIS Ltd. at high speed and Portland cement CEM I 42.5 (Radotín, Czech Republic). The RCP was obtained from the PB2 and the SB8 railway

| Mixture | Cement (CEM I 42.5 R) [g] | RCP [g] | Water/binder ratio [–] | Slump flow test [mm] |
|---|---|---|---|---|
| CEM (ref) | 1000 | – | 0.350 | $130 \pm 5$ |
| RCP 5 (5%) | 950 | 50 | 0.350 | $130 \pm 5$ |
| RCP 10 (10%) | 900 | 100 | 0.354 | $130 \pm 5$ |
| RCP 15 (15%) | 850 | 150 | 0.358 | $130 \pm 5$ |
| RCP 20 (20%) | 800 | 200 | 0.361 | $130 \pm 5$ |
| RCP 25 (25%) | 750 | 250 | 0.365 | $130 \pm 5$ |

Table 1. Composition of tested samples.

sleepers. In the first stage of the testing, a cement paste without the RCP, the (the reference sample), was used to investigate a potential reactivity and the level of milling. Second stage samples contained a combination of 50 % of cement and 50 % of the RCP. Both samples had the same water-binder (w/b) ratio of 0.35 and were used for calorimetric measurement. Measurement of particle size distribution and specific surface measurements were subsequently performed on both sample types.

In the second stage, cement paste samples with varying RCP percentages were tested for their mechanical properties. Six mixtures with 0 %, 5 %, 10 %, 15 %, 20 % and 25 % of the RCP by weight were prepared (Table 1). Because cement and the RCP behave differently when mixed with water, the w/b ratios ranged from 0.35 (cement) to 0.365 (RCP 25 %) and varied according to the RCP levels. The unifying parameter for these mixtures was workability defined by the slump flow test. Each set contained 6 prismatic samples with dimensions of $40 \times 40 \times 160$ mm. The samples were removed from casts after 2 days and shrinkage measurements were carried out. The samples were then cured for 28 days under laboratory conditions at $21 \pm 2$ °C with relative humidity $50 \pm 5$ %.

For microscope examination, only the reference cement paste and 25 wt% of the RCP samples were used. The tested cylindrical specimens were 30 mm in diameter and 50 mm in length. After curing, the samples were cut into 5 mm-thick slices using a diamond saw and polished with silicon carbide grinding papers #300, #500, #1200, #2400 and #4000. Finally, the specimens were polished by an emulsion with 0.25 µm nanodiamonds. Technical alcohol was used for cleaning samples in an ultrasonic bath in-between the individual polishing steps.

## 3. Measurement methods

Calorimetric measurements were conducted on an isothermal TAM Air calorimeter for accurate measurement of heat flow and hydration heat production. Mixtures were tested for 7 days at a constant temperature of 20 °C and were stored in sealable plastic containers, each containing from 32 g to 37 g of mixture. Based on the weight of each sample (measured before testing) the results of the heat flow and hydration heat were related to a 1 g of cement for a better comparison between the individual RCPs.

A slump flow control test was performed on all mixtures. The mould, in the shape of a truncated cone (base diameter 80 mm, top diameter 70 mm, 40 mm high), was placed on a shaking table, filled to the brim, and compacted. After filling, the mould was removed and 20 shake cycles were performed. Two perpendicular dimensions were measured after shaking. The slump flow value was defined as an average of both measurements rounded to the closest 10 mm.

RCP particle size distribution and determination of middle grain size were performed using the Fritsch ANALYSETTE 22 MicroTec plus laser device. A Matest E009 device was used to measure the specific surface area using the Blain method.

The length of each box in the mould was measured before the mixture was added to it and the length of samples was measured after 2 days, immediately after the removal from the casts. The resulting sample shrinkage was related to the length of 1 m and was only preliminary measurement carried out to verify the influence of increasing w/c ratios for RCP samples.

Dynamic Young's modulus and shear modulus were monitored using the resonance method based on measuring the natural frequency for the prismatic $40 \times 40 \times 160$ mm samples. The samples were supported during measurement with a soft elastic pad at nodal points. Measurements were conducted on 6 samples for each set prior to flexural and compressive strength tests. Resonance detection was performed using a Brüel & Kjær assembly consisting of a type 3560-B-120 measurement station for recording the excitation and response signals; type 4519-003 acceleration transducers; a type 8206 impact hammer type; and a computer. This method required a knowledge of the dimensions and the weight of each sample prior to testing. Excitation and response signals were transformed from the time domain to the frequency domain using the Fast Fourier Transform (FFT). Frequency Response Function (FRF) was calculated as the ratio of the response and the excitation force in the frequency domain. The appropriate basic natural frequencies were evaluated using the FRFs after which the dynamic Young's Modulus and shear modulus were evaluated [26, 27].

Flexural and compressive strength were determined

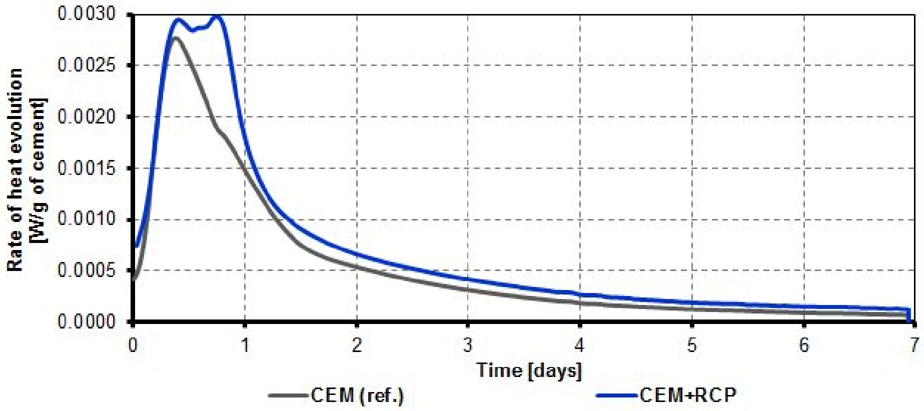


Figure 1. Rate of heat evolution for reference cement and cement with 50 % of the RCP.

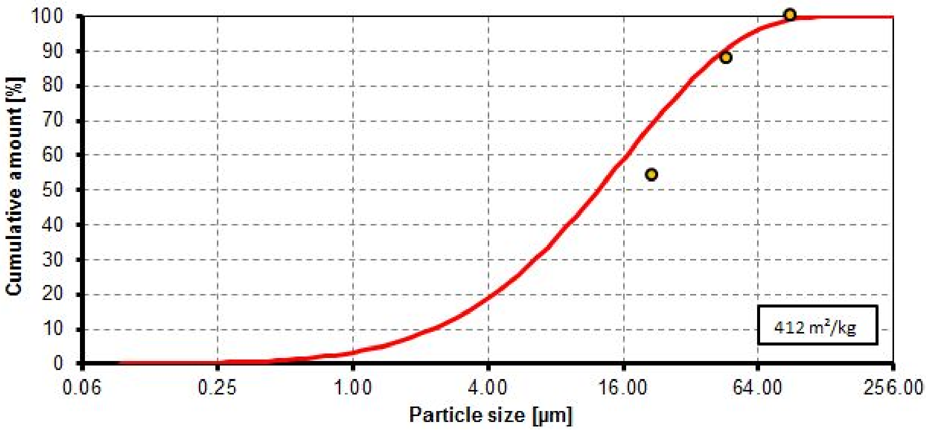


Figure 2. The particle size distribution and specific surface area of the RCP. Dots represent particle size distribution for CEM I 42.5 R.

on 28-day old samples using a model FP100 Heckert device. The testing was a displacement controlled at a constant rate of 0.1 mm/s in the case of three-point bending and 0.3 mm/s during the compression test. The distance between supports during the three-point bending test was 100 mm. A uniaxial compressive test was performed on the broken specimen halves with effective dimensions $40 \times 40 \times \sim 80$ mm [28, 29].

The microstructure of the composites was examined using optical and scanning electron microscopes. A Neophot 21 optical metallurgical microscope was used to obtain images at 100× magnification for the surface quality evaluation and preliminary pore distribution. A Philips XL30 ESEM-TMP FEI scanning electron microscope was used for an identification of individual phases and their levels at 100× magnification. A scanning electron microscopy (SEM) was used in BSE (backscattered electrons) mode at a low pressure (10–20 Pa) and at an accelerating voltage of 30 kV.

## 4. Experimental results

Figure 1 illustrates the heat evolution. By relating heat flow to a 1 g of cement and not an entire RCP mixture it was possible to highlight the differences in the hydration process caused by the RCP. After 8 hours, there is an obvious increase in heat flow for almost all mixtures containing the RCP versus reference cements, due to the $C_3S$ reaction. After 19 to 26 hours, when $C_3A$ reacts, an increase in heat flow for the RCP mixtures was also evident. After 48 hours and until the end of monitoring, $C_2S$ reacts and the heat flow for the RCP samples was also slightly higher than in the reference samples. Hydration heat exhibited by the RCP samples during the entire 7-day measuring period was 440.105 J/g and 352.023 J/g for the reference cement.

Figure 2 is a particle size distribution diagram. The red line represents the RCP. 34 % of the RCP particles were larger than 20 µm and only 10 % were larger than 45 µm, with the largest grain size being 130 µm and an average grain size of 12 µm. The measured specific surface area for the RCP was 412 $m^2$/kg. The orange dots in the diagram represent CEM I 42.5 R cement reference samples. 45 % of the reference particles were larger than 20 µm and 12 % of the grains were larger than 45 µm, with the largest grain size being 90 µm and the average grain size, 18 µm. The specific surface area of the reference cement was 351 $m^2$/kg.

Figure 3 is a shrinkage diagram. It is obvious that

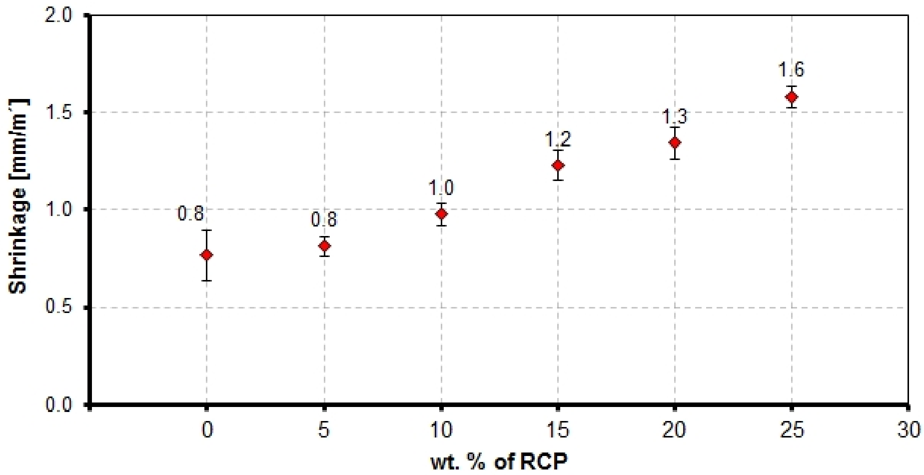


Figure 3. Shrinkage of samples containing increasing RCP percentages after 2 days.

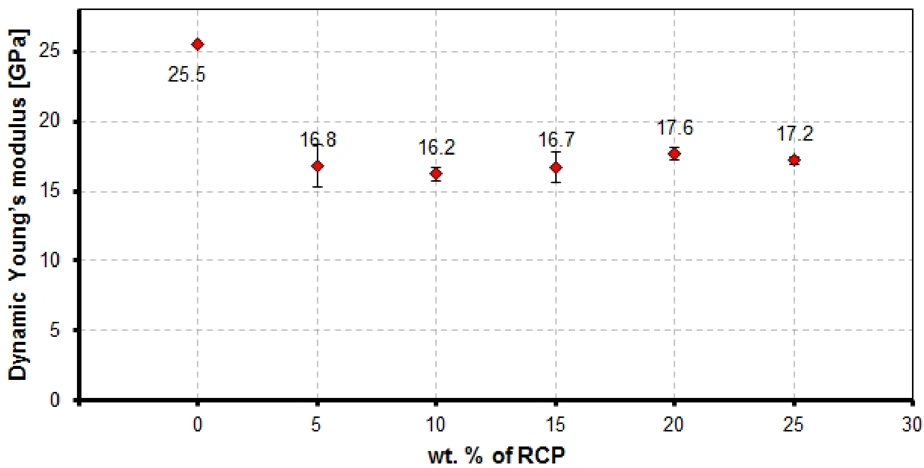


Figure 4. Dynamic Young's Modulus for the samples with increasing RCP percentages.

increasing levels of the RCP negatively influenced the shrinkage. There is almost no difference between reference cement and samples with 5 wt% of the RCP. However, between 5 and 25 wt% of the RCP, shrinkage increased linearly. Samples with 25 wt% of the RCP showed twice as much shrinkage as the control samples.

Figure 4 shows the dynamic Young's modulus for specimens with increasing RCP content. 5 wt% of the RCP samples exhibited a decrease in the dynamic Young's modulus by approximately 35 % when compared with the reference samples. Higher RCP levels, up to 25 %, had no further impact. A similar trend was evident in the case of the dynamic shear modulus (Fig. 5). A 32 % decrease of dynamic shear modulus between the reference sample and the RCP samples was observed and the percentage of the RCP had a minimal impact on dynamic shear modulus (Fig. 5).

Figure 6 shows the flexural strength for samples with increasing RCP percentages. Unexpectedly, samples with 5 wt% of the RCP exhibited higher flexural strength - approximately 7 % - than the reference samples. Flexural strength was also higher for samples with 15 wt% of the RCP, approximately 25 % higher than reference strength. However, increasing the RCP content to 20 wt% resulted in decreased flexural strength.

Figure 7 presents compression testing results, which exhibited similar patterns as the results for flexural strength. Compression strength for 5 wt% of the RCP samples showed an increase of almost 10 % when compared to reference samples. Compression strength for 25 wt% of the RCP samples was still comparable to the reference samples, with a slight decrease in strength of only 4 %.

An optical microscope was used to take images (Figure 8) of microstructures in order to assess the surface quality and pore distribution. The replacement of the RCP in samples negatively affected the cohesion of mixture matrices, resulting in deterioration of the polished surface. The optical microscope images showed minimal differences in the number of pores, but, as seen in Figure 9, SEM images reveal a higher number of pores. The distribution of the various phases within the samples can also be detected in the SEM images. The RCP aggregate can be seen as the dark grey spots

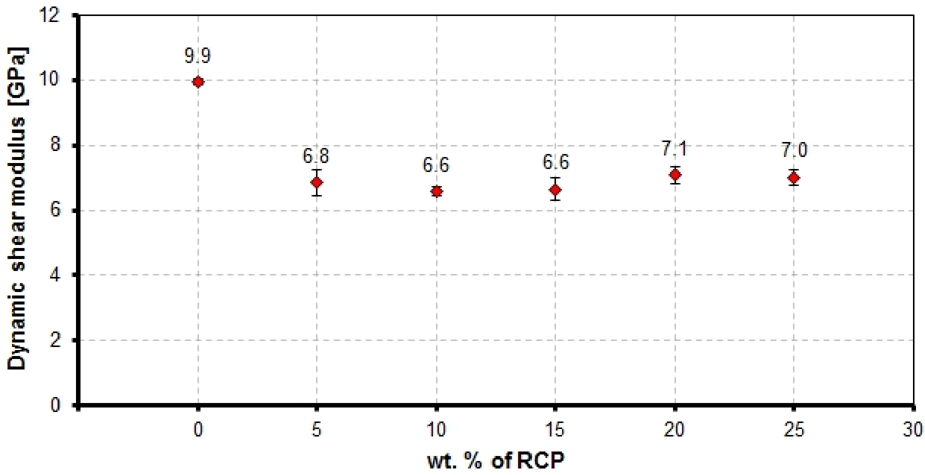


FIGURE 5. Dynamic shear modulus for the samples with increasing RCP percentages.

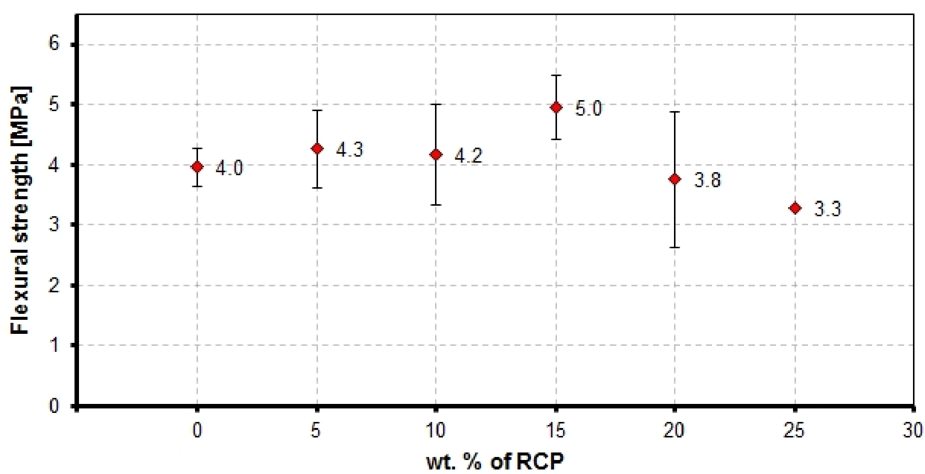


FIGURE 6. Flexural strength for samples with increasing RCP percentages.

in the RCP 25 sample. The decrease of non-hydrated cement grains (white spots) is obvious in case of RCP samples versus the reference samples. Given the ratio of cement and the RCP and the presumption that the RCP contains only a fraction of non-hydrated cement grains when compared to reference samples, an RCP 25 sample should contain approximately 25 % fewer non-hydrated cement grains than the reference samples. However, the observed difference was almost 53 %.

## 5. Discussion

Calorimetric measurement results support the assumption that cement grains in the RCP are potentially reactive and can be exposed through very fine grinding [30] and are therefore recommended for future investigations of the RCP properties.

The sharp shape of RCP particles negatively influences water demand and our results indicate that w/b ratio increased as percentages of the RCP increased, which led to a higher shrinkage in the RCP samples than in the control samples. Additionally, because the RCP is comprised of hydrated particles that have a lower density than cement particles and thus a lower modulus of elasticity, the RCP-containing samples exhibited greater shrinkage while drying. In future research, this negative effect might be reduced by introducing superplasticizers into mixtures [31] and improving the initial 2 day curing process (e.g. by covering molds with foil to reduce water evaporation).

A decrease of dynamic Young's and shear moduli for RCP samples was primarily caused by the lower density of old cement paste in the RCP and also impacted by higher water content. This caused the formation of higher numbers of pores in the RCP-containing samples.

Our study illustrated that the substitution of cement with the RCP positively affected flexural strength for up to 15 wt% of the RCP. This increase in flexural strength is likely caused by a combination of the binder effect for non-hydrated cement grains (proven with hydration heat measurement, Figure 1) and the microfiller effect of old cement paste (achieved because of the milling process, Figure 2) as well as by the aggregate in the RCP, all of which contributed to

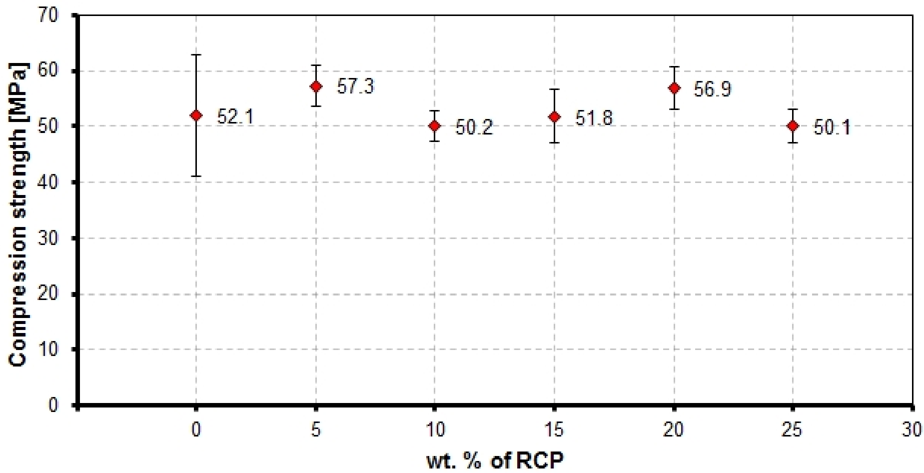


Figure 7. Compressive strength of samples with increasing RCP percentages.

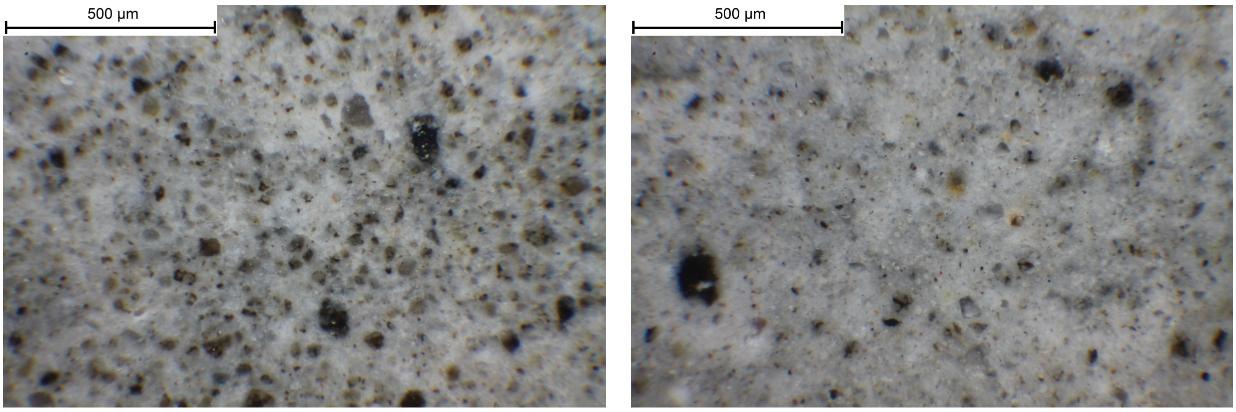


Figure 8. Optical microscopy images at 100× magnification of (left) reference sample and (right) sample with 25 wt% of the RCP.

a reduction in cohesiveness of the cement paste. It is presumed that higher percentages of the RCP in samples reduced flexural strength due to a weakening in the bonds between the RCP and cement particles as in the case of the transition zone between aggregates and cement in concrete.

Up to the level of 20 wt% of the RCP, compressive strength was increased and primarily affected by the RCP microfiller effect, which causes the filling of small pores with the fine particles of aggregates and old cement paste contained in the RCP and a slight binder effect.

Findings for flexural strength approximately match those in the prior research [30]. But there was found an improvement in case of 10 % to 20 % amount of the RCP. Also, the influence of the RCP on compressive strength was similar to prior research [30].

On the microstructure images, the higher number of pores in RCP samples can be seen. The lower number of non-hydrated cement grains in RCP samples is visible on SEM images and likely caused by the fact that low density RCP did not hinder access of water to the cement grains and thus a larger amount of cement could hydrate when compared with the reference samples. However, this might have been the result of an inaccurate image analysis or statistic deviation. Lower differences in the number of non-hydrated cement grains can likely be expected in future studies including larger numbers of samples.

## 6. Conclusions

This paper examined the RCP as a cement replacement at levels of 5 to 25 wt%, comparing results with a control sample and measuring properties and microstructures. Findings include:

- Cement paste with 50 % cement and 50 % old concrete milled at a similar level as cement produces higher hydration heat than cement alone, probably due to exposed non-hydrated cement grains.
- Partial replacement of cement with the RCP causes higher shrinkage than that observed for paste containing only cement (2 times in the case of 25 wt% of the RCP samples) due to RCP's lower density and resultant lower modulus of elasticity. This negative effect could likely be reduced by using superplasticizers and improving the curing process.
- A decrease of dynamic Young´s and shear moduli for RCP samples (approximately 35 %) was primar-

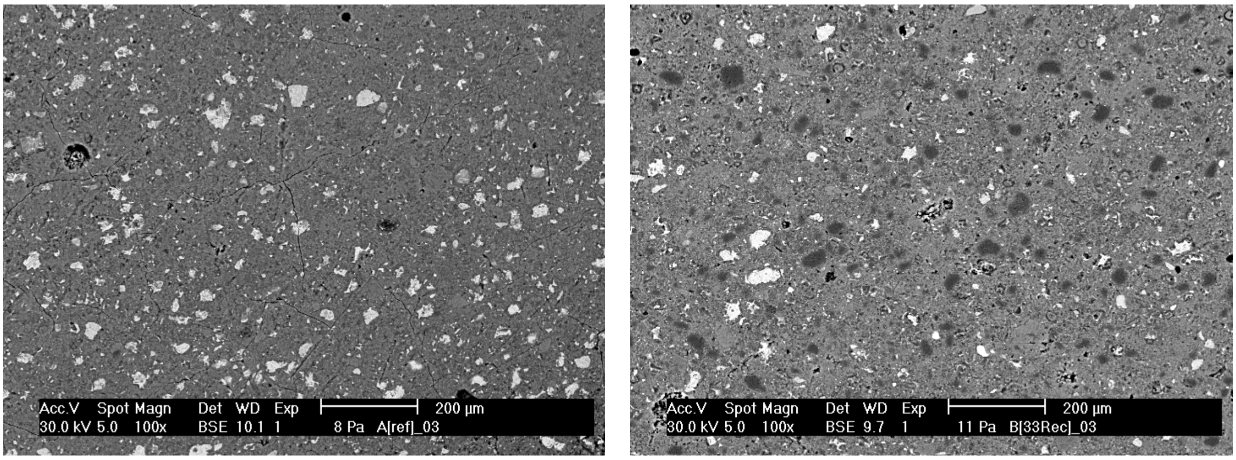


Figure 9. SEM images at 100× magnification of (left) reference sample and (right) sample with 25 wt% of the RCP.

ily caused by the lower density of old cement paste in the RCP and slightly impacted by higher water content. This led to the formation of a higher number of pores in samples containing RCP.

- For 15 wt% of the RCP, flexural strength increased by approximately 7 %. For 5 wt% RCP, compressive strength increased by approximately 10 %, likely due to its reactivity and microfiller properties.


## Acknowledgements

The authors wish to express their gratitude to the Faculty of Civil Engineering, Czech Technical University (CTU) in Prague (SGS project SGS16/201/OHK1/3T/11 and TAČR project TA04031256) as well as the Center for Nanotechnology in Civil Engineering at the Faculty of Civil Engineering, CTU in Prague. The authors also thank to Czech National Library of Technology, Prague, namely to Stephanie Krueger.


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