MICROSCOPIC PERFORMANCE OF CEMENTITIOUS COMPOSITE WITH RECYCLED CONCRETE ADMIXTURE

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Abstract

The topic of emission reduction and recycling in civil engineering is highly discussed topic over last years. With respect to construction waste production, it is obvious to reuse materials such as concrete. Coarse aggregate recycling and reinforcement rebars separation are the most used procedure in this field so far. Recently, methods including recycling of concrete cementitious matrix were introduced to the market. As such, the concrete matrix consists of matured hydration products of the cement (calcium hydroxide, calcium silica hydrate, etc.) as well as non-hydrated Portland cement particles (clinker), which can represent up to 5 % of the matrix. The focus of this article is placed on microscopic level of cementitious composite with admixture of finely grounded recycled concrete. The impact of recycled addition on micro-structure of the composite, namely the percentage representation of individual material phases, is investigated by image analysis of micro-graphs obtained with scanning electron microscopy. The micro-mechanical performance of each composite is examined with displacement driven grid indentation on several levels of the sample. In consideration of the complexity of the cementitious composite with addition of finely grounded concrete, additional testing of input materials and referential cement paste sample is indispensable to properly distinguish mechanical properties of each composite phase. Young's modulus of individual phases is thus determined from spectral deconvolution of obtained indentation data with respect to the composite structure.

Keywords: Recycled concrete, phase, nano-indentation, SEM, spectral deconvolution, image analysis

1. INTRODUCTION

The construction and demolition waste (CDW), as the largest portion of stored waste material, became worldwide problem in both environmental and economic way. For example, according to data collected by Czech Statistical Office over the year 2016, 14.3 million tons of construction and demolition waste was produced in Czech Republic. Even though the long-term monitoring indicates decrease of CDW production, the amount of land-filled construction and demolition waste increases approx. 4.5 % annually. This projected trend underlines the potential development of much efficient and thrifty CDW re-cycling methods, that could reduce waste deposition while saving natural sources and environment. One of economic problems of CDW recycled materials (compare to traditional raw materials), can be paradoxically higher price due to the transportation costs [1, 2]. In general, concrete is 30 to 60 % made of coarse aggregate which is typically reused. In other words, 70 to 40 % of the concrete is considered as secondary waste product. Absolute recycling of ordinary concrete, when high-speed milling is used, transforms the waste material into very fine powder. Such recycling processing opens material ability for potential use as micro-filler (for non-reactive hydration products and aggregate) and partial cement substitute in case of non-hydrated clinker in cement paste [3].

The potential of recycled finely grinded concrete as substitute for pulverized limestone in asphalt mixture was shown in M. Chen study [4]. Compare to pulverized limestone (based on XRD SEM analysis), recycled concrete powder particles have rougher surface and show higher content of silicon dioxide (SiO_2). Mechanical properties of this component depend on the environment thermal conditions, as shown in this study. The results indicate increased strength and fatigue persistence in higher temperatures. Features of cement paste with partial replacement of a binder by fine grinded recycled concrete are discussed in [5]. As corroborated by the results, the replacement impacts the mixture in fresh state due to decreased workability and prolonged hardening time (delay up to 2 hours). The study recommendation inclines to 15 % limitation of the replacement, as mechanical results of samples with higher fine grinded recycled concrete content (45 % cement replacement) show reduction of compressive strength up to 30 %. The potential pozzolanic activity of pulverized recycled concrete from railway sleepers due to presence of non-hydrated clinkers, likely unfolded by fine milling [6,7]. The recycled material,as the level of non-hydrated clinker in raw concrete is low, is thus feasible for subsoil under railway structures strengthening [8]. Other potential small scale utilization can be found in cementitious composite material.

This study is focused on micro-mechanical performance of the recycled concrete drain gutter, in form of finely grinded powder (0 - 180 μm particles), used as partial replacement in cementitious composite. To ensure adequate interpretation of measured data, both referential cement paste and original concrete drain gutter were also subjected to micro-mechanical and micro-structural testing.

2. METHODOLOGY AND DATA ANALYSIS

For purpose of this study, referential cement paste and cementitious composite with grinded recycled drain gutter inclusion were prepared and left to mature for 28 days. The referential specimen (REF) was made of CEM II 42.5 R cement with water to cement ratio of 0.35. In case of second specimen (REC30), 30 % of cement weight was replaced with finely grinded recycled drain gutter. In addition, third sample of original concrete drain gutter (10 years old, further denoted as ORG) was added for proper identification of the recycled powder inclusion effect on the paste. All three samples were embedded in epoxy-resin and their surface grinded and polished, avoiding further hydration and selective abrasion, for purposes of nano-indentation and scanning electron microscopy.

2.1. Indentation and spectral deconvolution

The micro-scale elastic properties of individual phases present in each material were evaluated based on nano-indentation results (Ti 750 series, Hysitron Inc.). In general, indentation technique reflects th dependency of material resistance to the probe propagation into the indented half-space. Indentation modulus (and/or other elastic mechanical properties) of indented region are determined from unloading part of the record. Incorporated errors of the measurement (creep, visco-elasticity etc.) can be avoided by appropriate setup of measurement [9 - 13]. In all cases of testing, the displacement driven load function was selected, where both loading and unloading lasted 5 seconds each with an in-between 60 seconds segment with constantly held maximum displacement of 150 nm. The measurement was performed on different levels of the sample, based on specimen heterogeneity and expected number of present phases. Histograms of measured data were further processed with spectral deconvolution, where micro-mechanical features of material individual phases are determined with respect to possible interference of main, soft, phase with other, tougher, phases. The maximum allowed error between indentation and processed data and structural representation did not exceed 10 %.

The referential specimen (cement paste) indentation was performed in three separate locations. In each position, rectangular grid constructed of 21 by 21 indents with mutual separation of 10 μm was implemented. In case of original concrete drain gutter, two material levels were selected for indentation - area of cementitious matrix (without aggregate present) and inter-facial transition zone (ITZ) between matrix and aggregate (presence of clinker not expected). On both material levels, two indentation grids (similar to grids imposed on referential specimen) were implemented. The composite with 30 % recycled pulverized gutter replacement was also tested via indentation on two material levels. 12 locations indicating presence of inclusion were subjected to grid indentation similar to previous samples (REF and ORG). 4 random positions, capturing mostly cement matrix, were indented in 51 by 51 indents pattern, mutually separated by 15 µm. The exponentially increased number of measurement data inevitable for proper mechanical features identification of most individual phases present in the composite.

2.2. SEM and image analysis

The scanning electron microscopy (SEM) investigation was performed in MIRA II LMU (Tescan corp., Brno) on polished specimens coated with thin layer of carbon necessary to ensure proper conductivity of the surface. Chemical composition (in weight percentage) of each phase was determined using EPMA based on the detection of the X-rays by energy dispersive X-ray detector (EDX, Bruker corp., Berlin) on several point of each position. Due to the time-demands and statistical insufficiency of EPMA phase determination, image analysis of SEM BSE micrographs was used to obtain phase volumetric representations in material. In general, energy of back-scattered electron is dependent on atomic number of the element which is relevant to brightness of the phase in BSE diagram. Based on estimated number of phases and their gray-scale range from EDX measurements of each sample, it is possible numerically quantify percentage representation of each phase from several BSE SEM graphs by image analysis.

The EDX SEM investigation of cement paste (REF sample) was applied in 20 points of interest from which basic phases of the matrix were identify, including their brightness range in gray-scale. Furthermore, 20 SEM BSE diagrams were analyzed providing average description specimen structure. In case of original concrete drain gutter sample, image analyses of two magnification levels was performed in order to adequately and effectively determine all present phases. Close magnification BSE diagrams image analyses (identical to REF specimen) determined average representation of cement matrix phases. Image analysis of SEM BSE diagrams (12 images) capturing outlying structure of the specimen served for aggregate - matrix ratio evaluation. Unfortunately, identical procedure of micro-structure investigation of composite specimen (REC30) gave not conclusive information of phases representation due to very close ranges of brightness in gray-scale of chemically equal phases.

3. RESULTS AND DISCUSSION

The results of spectral deconvolution and histograms, from which individual phases features were derived, are summarized in **Table 1** and **Figure 1**. Direct indentation modulus comparison between can be made due to incorporation of mutual influence of phases with different stiffness. For example, integration of recycled LD CSH into the matrix of new composite seams to increase its modulus by approx. 12 %, compare to features of same phase present in original concrete gutter. On the other hand, the incorporation and interaction of recycled HD CSH phase with matrix with significantly lower stiffness results in decrease of its indentation modulus by approx. 6 %. In case of portlandite evaluation in REC30 sample, newly formed and recycled inclusion of this phase indentation modulus are too close and could not be separated. Presented results can be described as average (homogenized) value of both. Non-hydrated clinker and aggregate inclusion, given their stiffness and possible measurement error, remain intact, i.e. with same value of indentation modulus.

Table 1 Results of individual phases micro-mechanical features (indentation modulus E_r [GPa])

Sample	ITZ	LD CSH	R-LD CSH	HD CSH	R-HD CSH	Portlandite	Clinker	Aggreg.
REF	nonex.	17.1 ± 5.6		40.1 ± 6.1		77.7 ± 18.6	125.7 ± 6.6	nonex.
ORG	16.7 ± 3.6		22.0 ± 2.1		46.2 ± 4.6	72.6 ± 9.3	127.5 ± 3.8	106.1 ± 8.5
REC30	not det.	16.2 ± 3.4	24.7 ± 6.2	39.7 ± 2.4	43.7 ± 4.0	75.5 ± 12.2	122.6 ± 7.2	103.7 ± 7.7

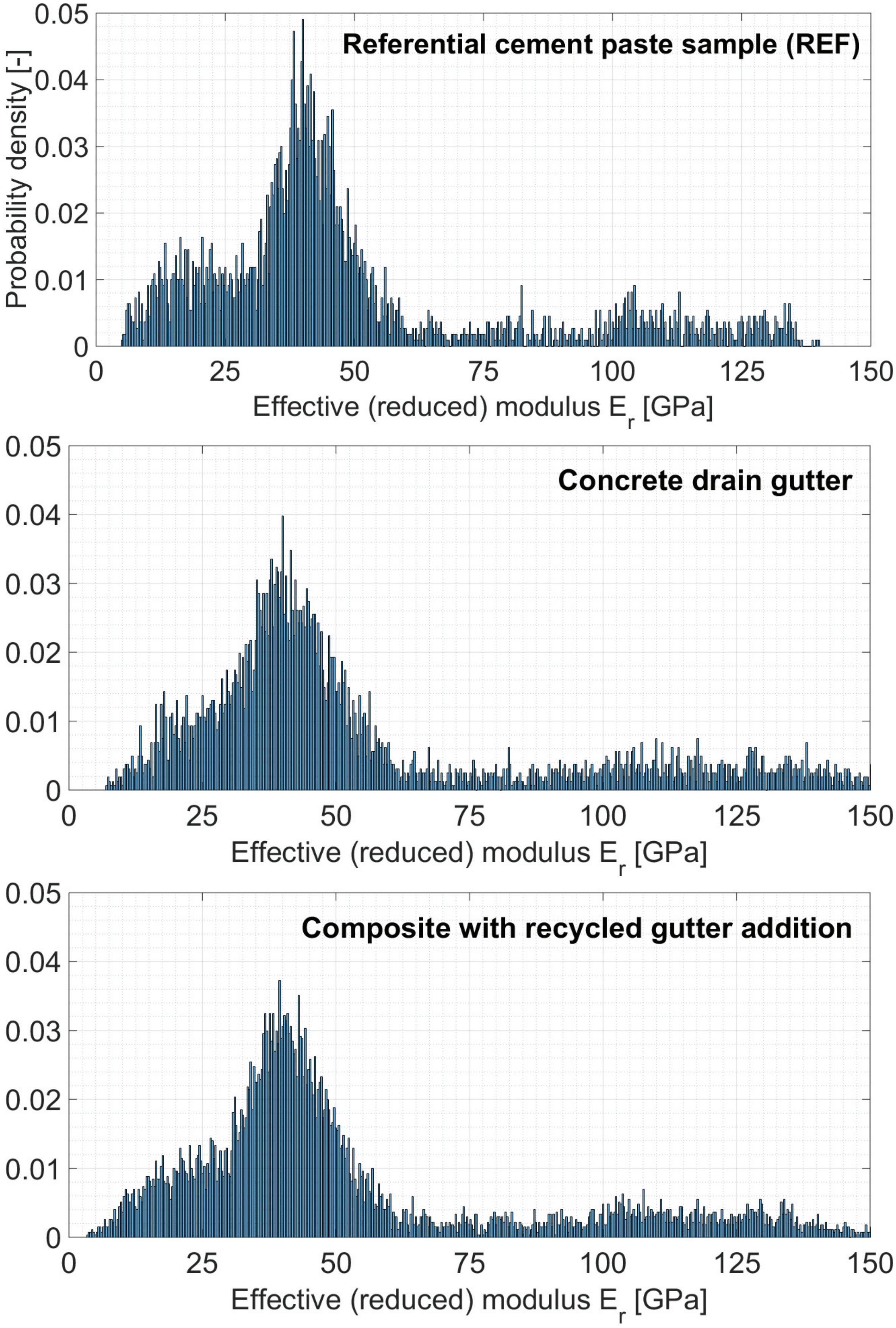

Figure 1 Histograms of indentation results

Micro-structural results of referential and original drain gutter samples are shown in **Table 2.** The REC30 sample, due to its complex structure (possible 9 present phases) and almost identical gray-scale brightness range of several phases, could not be properly evaluated with respect to number of measurements (data, diagrams). Even so, results of phase percentage representation were used for in spectral deconvolution procedure (with respect to used pulverized recycled gutter amount and its individual phases representation) as an error indicator.

Table 2 Results of average individual phases percentage representation [%]

	Pores	**LD CSH**	**HD CSH**	**Portlandite**	**Clinker**	**Aggregate**
Cement paste	10.39 ± 0.82	44.33 ± 3.73	18.20 ± 1.46	18.50 ± 1.73	9.72 ± 0.93	nonexisting
Concrete drain gutter	1.83 ± 0.12	13.92 ± 0.97	20.45 ± 1.22	6.53 ± 0.30	4.91 ± 0.18	52.36 ± 3.54

4. CONCLUSION

The nano-indentation technique in combination with SEM structure observation was use in the study of cementitious composite with 30 % cement replacement with pulverized recycled concrete gutter. Based on the results of post-processed measured data, following can be stated:

- The addition of recycled concrete, in newly produced cementitious composite, does not impact the micro-mechanical features of newly formed matrix phases.
- Incorporated particles of recycled pulverized concrete drain gutter function in the composite as micro- and nano- filler, i.e. impact mainly mechanical features of the matrix on the meso- and macro-scale.
- The combination of indentation technique and SEM proved essential in complex composite investigation.
- Detailed investigation of composite’s inclusions was essential for proper identification of micro-mechanical features of individual phases.
- The number of indentation data necessary for investigation exponentially increases with number of expected phases in the material, leading to very time-demanding testing and measurement evaluation.
- The micro-structural investigation of cementitious composite with 30 % cement replacement with pulverized recycled concrete gutter via SEM and image analysis was not conclusive. Closer investigation and higher number of diagrams is recommended for in order to describe composite’s structure.
- The presumption of reactive clinker involvement in hydration process of cement can be also corroborated with further comparison of composite structure and clinker phase representation of referential and original material.

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