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# Influence of sand drying and mixing sequence on the performance of mortars with fine recycled concrete aggregates

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ABSTRACT

Fine recycled concrete aggregates (fRCA, 0–4 mm) are produced from demolished concrete structures and consist of natural aggregates and old cement mortar. The presence of old cement mortar has detrimental effect on the fresh properties and strength of new concrete. This study aims to investigate the working mechanism and effectiveness of different methods for the optimization of mortar mixtures with fRCA. Three streams of fRCA were considered in the approach. As reference material, river sand was used. The river sand was replaced at 0 wt %, 25 wt% and 50 wt% with fRCA. The use of tailor-made superplasticizers (SP's), drying of fRCA, modified content of 0–0.250 mm, modified mixing sequence, increase of cement content were investigated. Once the mortar mixtures were optimized, the reaction kinetics was investigated with isothermal calorimetry. SP was applied to prevent use of additional water and to maintain mix consistency. When the river sand was replaced at 25 wt% with fRCA, no extra cement was needed. The air content of mortars with fRCA was up to 18 %, due to some unforeseen effects. The results indicated that using as received, agglomerated and unwashed fRCA may have a negative effect on the working mechanism of SP leading to high air content in the fresh mortars. Using dried fRCA has substantially decreased air content in mortars. In addition to drying of fRCA, change of mixing sequence has equal or even superior importance to reduction of air content. As a result, the compressive strength was comparable to reference mix with river sand at 25 wt% replacement level, however, the strength of mortars with 50 wt% fRCA was reduced despite that the cement paste content was increased. The use of fRCA did not affect the kinetics and degree of cement hydration in mortars with 25 wt% fRCA. The positive side of this is that the fRCA can be considered as non-reactive.

## 1. Introduction

In the Netherlands, yearly 33 Mton new concrete (roughly 15 million $m^3$) is produced while 12 Mton concrete recycled aggregates becomes available. Recycling of the yearly 12 Mton concrete results in roughly 7 Mton coarse recycled concrete aggregate and 5 Mton fine recycled concrete aggregates (fRCA). Currently, the fRCA are used in low-grade applications such as a substitute material for natural sand in cementitious renderings and masonry mortars [1-8], road constructions [9-12] and as a filling material for geosynthetic reinforced structures and soil stabilization [13]. Little knowledge and no guidelines or regulations exist to make optimal use of these characteristics of fRCA in order to make high quality concrete [14,15]. Various subfractions with their own gradings are possible within the 0–4 mm fraction of fRCA. Each of these subfractions have their own physico-chemical characteristics depending on the parent concrete, recycling technology as well as the storage of recycled material [16-18]. These physico-chemical characteristics have an influence on the performance of the fresh concrete mix as well as the hardened concrete [9,19-21].

The most influential property of fRCA for the concrete performance is their high water absorption (WA). Based on a comprehensive literature review, the WA of fRCA ranges from 3 to 14 %, while the WA of natural sand ranges from 0.3 to 4.0 % [14]. Higher WA of fRCA is caused by the presence of old cement mortar from the parent concrete. The water absorption of fRCA can be seen as the sum of the capillary absorption of both residual cement mortar and original natural aggregates [22]. High water absorption of fRCA reduces workability of the concrete mix which is often solved by increasing both water and cement contents

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to keep ensuring high quality and durability of the concrete. Therefore, large scale introduction and optimal use of fRCA in new concrete is hampered because the concrete performance is uncertain and increased cement content to compensate water demands of fRCA result in higher costs and $CO_2$ emissions. Consequently, it is a major challenge to simultaneously include fRCA as well as reduce cement content or at least keep it constant. A great deal of previous research into concrete with fRCA has focused on the workability. Several studies have explored the relationships between workability, water absorption kinetics and saturation degree of fRCA [23-26]. Other studies highlighted factors that are associated with the reduction of workability (reduced slump and slump-flow values):

- higher water demand of fRCA than the same concrete made with river sand. Porous surface of fRCA and increased amounts of fines (particles $<$ 0.250 mm) in fRCA increase the required amount of water to wet the particle surfaces adequately and to maintain a specified workability [24,27,28];
- angular shape and rough surface of fRCA which result in more frictional resistance at flow [29,30];
- decreased packing of the fRCA particles caused by diverse contact morphology of the particles [31].

Five strategies are often employed to optimize the behavior of fresh mortars/concretes with fRCA and performance. It should be noted that the presaturation of aggregates before the mixing is a general strategy of concrete producing.

- Presaturation before mixing (from 24 h up to 7 days [24]) or during mixing (for 10 min [25,32,33]). With presaturation of fRCA prior to mixing with other components, so-called water reservoirs are provided in the fRCA. A clear distinction has been made between the presaturation approaches: (1) by saturation of fRCA with precise water content calculated based on the WA of fRCA and (2) by full immersion. For instance, the mortar made with fRCA saturated with WA + 5% for 24 h had better workability than the one made with fully immersed fRCA for 24 h [33]. De Andrade et al. [34] used 80 % of the total water absorption capacity ($WA_{24h}$) of the fRCA to the mixture for saturating the fRCA during the mixing stage. This value was used for fRCA saturation based on the assumption that the recycled concrete aggregates would not absorb 100 % of their water absorption capacity during the short mixing process. The water absorption during casting and hardening was not considered. These findings [34] are in accordance with studies on workability of concretes with coarse recycled concrete aggregates [35,36]. The saturation of recycled concrete aggregates without any excess of water is a very complex task in the industrial manufacture process for ready mix concrete [22]. Also, according to previous research [24,28], replacing fine natural aggregates with fRCA and varying the degree of fRCA saturation had a negligible effect on the workability compared to the corresponding effect of the water-to-cement ratio.
- Mixing method. By now, modification of standard mixing method including a two-step mixing, four methods for mixing water, the increased mixing time and the delayed admixture addition time were used in concrete with coarse and fine recycled concrete aggregates in previous research work [37-43]. It should be noted that recycled concrete aggregates within the fresh concrete mix cannot absorb such an amount of water in a short period of time which is equal to the mixing time [44]. A higher absorption of the mixing water by the recycled aggregates was demonstrated by longer mixing time and adjusted water mixing sequence. This favors the greater removal of water from the fresh mortar mix, however, it reduces the consistency index [43]. The longer mixing time may also cause air entrapment, depending on the mixer type [45]. Effective mixing time and procedure needs to be adopted in the future for incorporating each component into the mix, to obtain the mortars and concretes with stable and reproducible properties.
- Drying. The drying of fRCA leads to the best workability of mortars due to the incomplete absorption of water by dried fRCA particles [33]. It leads to higher compressive strength, which was explained by better adhesion between dried aggregates and cement paste [25,33]. However, since the drying requires energy, as the inherent disadvantage of this strategy, makes it less suitable to be utilized for ready mix concrete compared with precast concrete.
- Chemical admixtures. The use of superplasticizers (SP's) maintains the workability, which for the same cement content allows reducing the water-to-cement ratio and improves the mechanical and durability properties of concrete with fRCA [46-48]. Furthermore, application of SP's was found to effectively reduce the drying shrinkage and creep of concrete with fRCA [49]. However, not all of the chemical admixtures that are widely used in concrete with natural sand work effectively in concrete with fRCA. For example, the use of some SP's was found to decrease the compressive strength and increase the drying shrinkage of concrete with fRCA [49]. Hence, suitable SP's for concrete with fRCA are needed.
- Treatment of fRCA. Treatment of fRCA particles was performed using different methods, which can be categorised in three main approaches: (i) improvement of the surface properties of fRCA by removing the detrimental materials (adhered mortar, light contaminants and organic matter, among others) [50-60]; (ii) improvement of the quality of the fRCA by enhancing their properties, either at surface or throughout the bulk aggregate volume [59], (iii) replacement of subfractions of particle size ranges of fRCA of same size range of river sand in mortar to provide a guidance on which particle size ranges of fRCA could be used as the replacement of sand in cement mortars for specific performance requirement [61]. Accelerated carbonation is most used approach (ii) to improve density of fRCA and decrease their water absorption [40,59,60,62,63]. The concrete with modified fRCA in general had reduced drying shrinkage, improved workability, mechanical properties, and durability. Although all studies led to improvement of properties of concrete with treated fRCA, the cost and upscaling possibilities are believed to be critical in future deployment of treatment methods. Considering the treatment nature and the cost, the treatment should be as robust as possible to allow for treatment of bigger amounts of fRCA and to decrease the fRCA cost, e.g. higher quality, higher price for the fRCA.

Although researchers have given a lot of reasonable explanations on complex flow behaviour through experiments there is no universal approach defined to obtain and maintain satisfactory workability of mortars/concretes with fRCA. Based on above discussed strategies and authors' comprehensive review of the concrete mix designs with fRCA from the current literature with their main characteristics [14], no step by step mix design procedure has been proposed that can be followed to find the optimum mix of concrete with fRCA for target slump, strength and durability. In addition, while focusing mainly on flow and evaluation of workability, less attention was given to other properties of mortars/concretes with fRCA, such as to air content and the impact of fRCA agglomeration on the air content. Minimum requirements for properties of fRCA and for properties of mortar/concrete with fRCA (agglomeration level, chloride and sulfate limits, the range of air content, strength) should be specified for each exposure class in the future.

This study aims to investigate the working mechanism and effectiveness of different methods for the optimization of mortar mixtures with fRCA. The clarification of the mechanisms is required not only for a better understanding of the fresh behaviour of mortars with fRCA, but also for the development of reliable treatment procedure that can be used in practice for concrete mix design with fRCA. The use of tailor-made superplasticizers (SP's), drying of fRCA, modified content of 0–0.250 mm, modified mixing sequence, increase of cement content

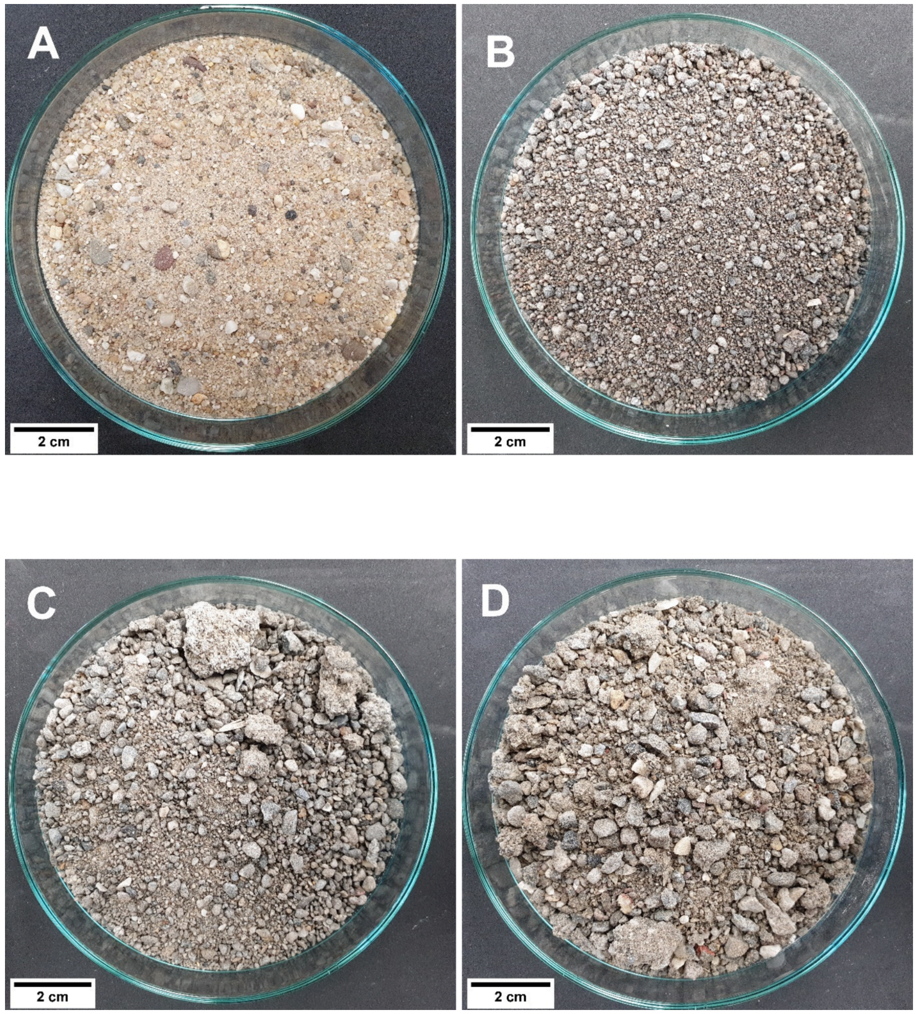


**Fig. 1.** River sand (A) and as received unwashed fRCA with different origins (B, C, D). As received fRCA C and D are considerably agglomerated due to outdoor storage conditions, compared to sand B which was not stored after production.

**Table 1**
Chemical and physical properties of studied sands [16].

| | A | B | C | D |
|---|---|---|---|---|
| Water soluble sulphates (wt.%) (EN 1744–1 § 10.2) | 0 | 0.16 | 0.15 | 0.10 |
| Acid soluble chlorides (wt.%) (EN 1744–5) | 0 | 0.04 | 0.04 | 0.04 |
| Old cement paste (wt.%) (EN 1744–5) | 0 | 19 | 25.8 | 16.2 |
| Quartz content (wt.%) (QXRD) | 94.1 | 57.7 | 62.7 | 72.1 |
| Calcite content (wt.%) (QXRD) | 0 | 8.7 | 7.6 | 3.8 |
| Amorphous content (wt.%) (QXRD) | 0 | 25.1 | 21.9 | 19 |
| Reactivity (J/g of sand) (TAM Air, Thermometric) | 0 | 0.71 | 0.72 | 0.51 |
| Content of 0–0.063 mm (wt.%) (EN 933–1: 2012) | 0 | 0.75 | 1.4 | 2.0 |
| Content of 0–0.250 mm (wt.%) (EN 933–1: 2012) | 5 | 5 | 12 | 17 |
| BET Surface area ($m^2/g$) of 0–0.250 mm fraction ($N_2$ adsorption) | 0.7 | 8.9 | 6.4 | 7.8 |
| Moisture (wt.%) of as received sand (EN 1097–5) | 3.6 | 10.8 | 9.4 | 8.2 |
| Water absorption (wt.%) (EN 1097–5) | 0.4 | 7 | 7 | 6.4 |
| Density ($g/cm^3$) (Helium Pycnometer) | 2.65 | 2.54 | 2.53 | 2.51 |

were investigated to optimize and improve mortar mix design. The flow, air content and compressive strength are investigated to evaluate the suitability of different methods to design mortars with fRCA. Once the mortar mixtures are optimized with regard to air content and strength, the reaction kinetics is investigated by means of isothermal calorimetry.

## 2. Experimental program

### 2.1. Materials

#### 2.1.1. Cement

The cement used in all experiments was an ordinary Portland cement, CEM I 42.5 N (ENCI, the Netherlands). The composition of the cement, measured by X-ray fluorescence (XRF) was the following (by mass): CaO 63.8%, $SiO_2$ 20%, $Al_2O_3$ 4.8% $Fe_2O_3$ 3.3%, MgO 2.0%, $SO_3$ 3.1%, $K_2O$ 0.5%, $Na_2O$ 0.2%.

#### 2.1.2. Sands

As reference material, river sand (A) was used in this study. Three different fRCA (B, C, D) were used. The as received fRCA appearances are shown in Fig. 1. The parent concrete is unknown for the fRCA. The recycling technique was rotor crusher (sand B) or jaw crusher combined with a cone crusher (sands C, D). Type of storage was ‘no storage (‘fresh’) for sand B and outdoor for sands C and D. Washing of fRCA was not performed. The chemical and physical properties of the sands are given in Table 1. Fig. 2 and Fig. 3 present particle size distributions for the subfraction 0–0.350 mm and total fraction 0–4 mm.

#### 2.1.3. Superplasticizer

For mixes without SP, the consistency decreased after 15 min (Fig. 4a). A study was performed to determine type and suitable percentage of SP for mortars with fRCA in order to maintain consistency for

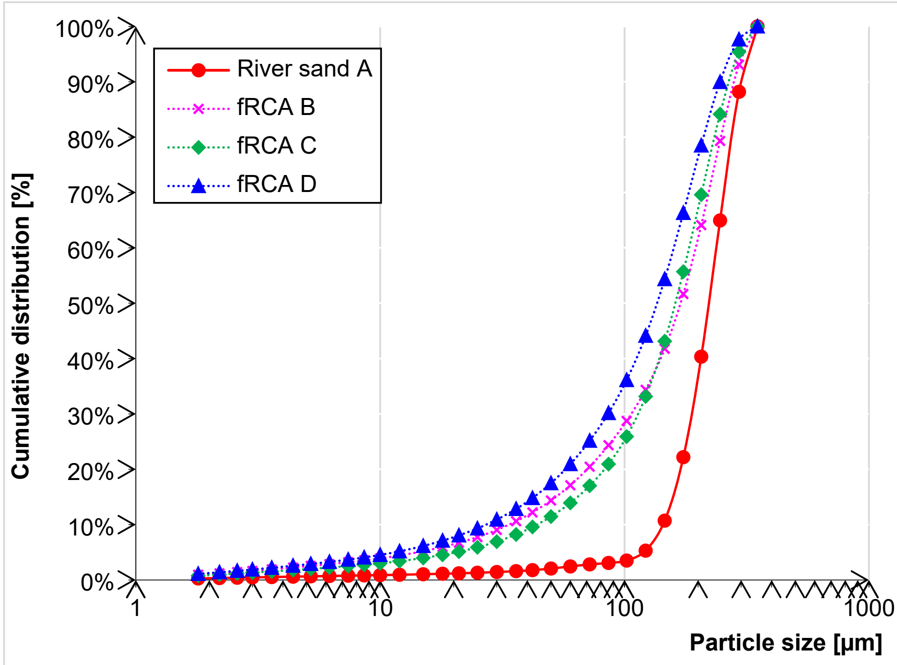


**Fig. 2.** Particle size distribution of the subfraction 0–0.350 mm in river sand and fRCA (measured by laser diffraction method).

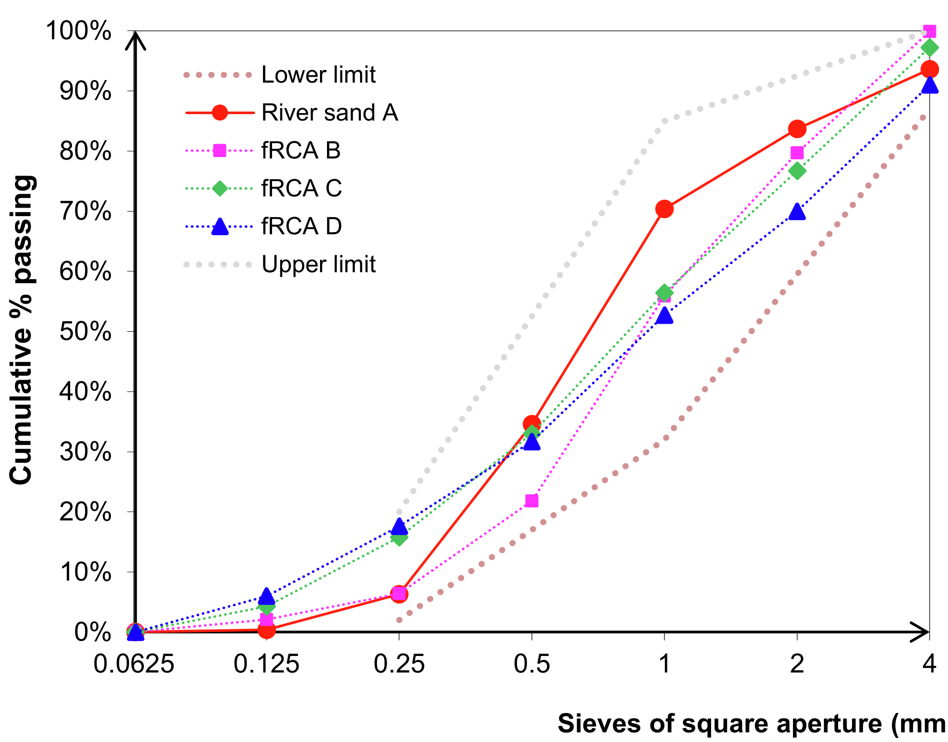


**Fig. 3.** Particle size distribution of the total fraction in river sand and fRCA (obtained by dry sieving method, EN 933–1: 2012), upper and lower limits according to EN 12620. Sand B has particle size distribution 0–4 mm, while particle size in sands C and D ranges from 0 to 6 mm.

longer time. CUGLA SP based on polycarboxylate ether in combination with defoamer was used. The admixture was supplied as a brown suspension with 20 % of solid content and a density of 1090 kg·m$^{-3}$. It is known that SP can entrain air in the fresh mix [64]. This is why most commercial formulations of SP contain defoamer to prevent excessive air entrapment [65,66].

The consistency of mortars was measured by a mini-slump test after each 15 min. Fluidity of mixes with SP amounts 0.2 wt% and 0.4 wt% by cement were investigated (Fig. 4b, c). Fig. 5 shows the flow of mortars (25 wt% fRCA C) for different SP amounts.

In order to obtain the target flow of 140–150 mm, higher amount of SP than 0.2 wt% by cement was needed for mortars with fRCA. The increase of SP from 0.2 wt% to 0.4 wt% by cement resulted in significantly larger flow of mortars (Fig. 4b, c). It can be seen that the use of SP increased the flow of mortars regardless of the type of fRCA used. The consistency of mortars with fRCA C and D was longer maintained than of

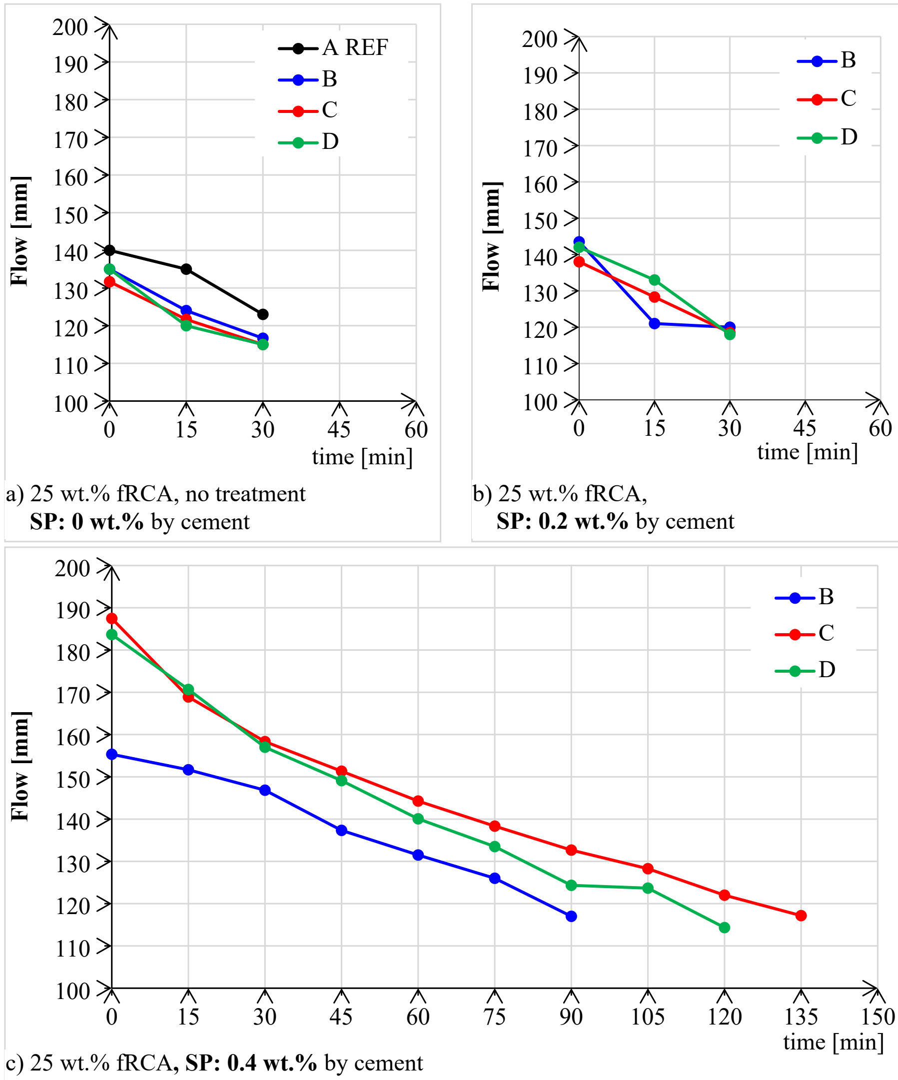


**Fig. 4.** Flow evolution over time for mortars without and with SP.

(a) (b)

**Fig. 5.** Flow after 15 S, mortars with 25 wt% replacement, fRCA C, (a) SP: 0.2 wt% by cement (flow: 138 mm), (b) SP: 0.4 wt% by cement (flow: 187.5 mm).

mortar with fRCA B (Fig. 4c).

For the target flow of 140–150 mm, it was decided to use the dosage of 0.3 wt% SP in the following experiments. The water content has been corrected relative to the water present in the SP.

## 2.2. Mortar series and mortar mixing

### 2.2.1. Mortar series

The overview of mix design optimization is given in Fig. 6. Table 2 presents an overview of the mortars mix series. All mortars had the same effective water-to-cement ratio of 0.5 and the cement-to-sand ratio (1:3), this ratio ecxludes the water added for WA of aggregates. The effective water-to-cement ratio was kept the same for all mixes in order to observe

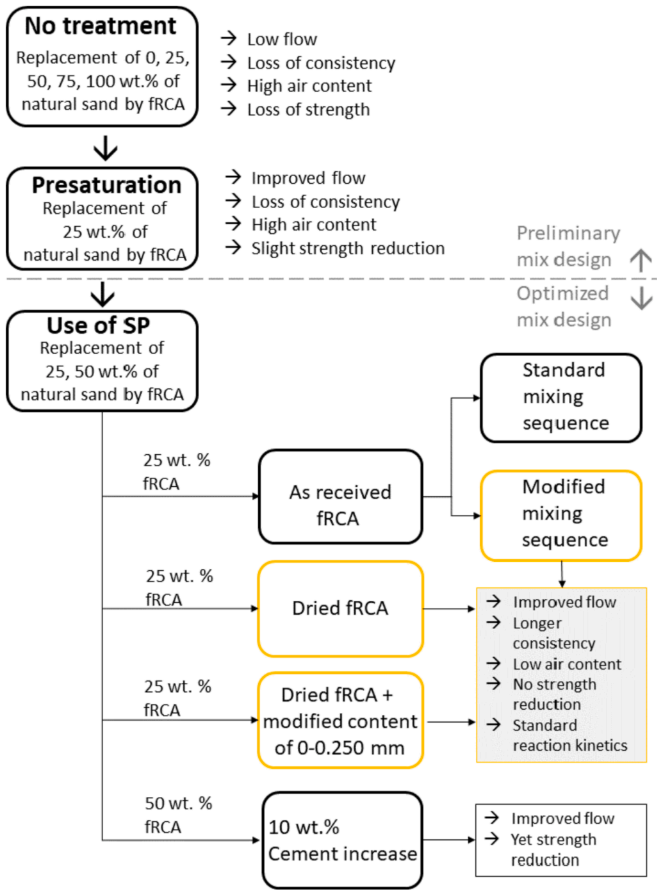


**Fig. 6.** Mix design optimization flow. Yellow colour highlights the most effective optimization strategies in this study. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

the influence of the fRCA type and quality on the properties of the mortars.

In the preliminary mix design two strategies were applied: no treatment of fRCA and presaturation of fRCA.

In the mixes with no treatment of fRCA, five replacement levels were studied: 0 wt%, 25 wt%, 50 wt%, 75 wt%, and 100 wt%. Mortars with 50 wt%, 75 wt%, 100 wt% fRCA had no flow, while mortars with 25 wt% fRCA had flow, irrespective of the fRCA source, but quick loss of consistency. Significant amount of air bubbles were observed in the mortars with all replacement levels.

In the mixes with presaturation of fRCA, the replacement was fixed on 25 wt% of natural sand by fRCA. The procedure for presaturation was as follows: first sand was added in the mixing bowl. Afterwards, water was added to fRCA which was equal to water absorption (24 h) of fRCA plus mixing water. The presaturation of fRCA was for 10 min. Then reference sand and cement were added at the last stage. The mixing was first at the low speed for 2 min and then 0.5 min at the high speed. Mixes with sand presaturation had lower flow compared to the mixes without sand presaturation and also quick loss of consistency (after 15 min). The 28 days compressive strength of mortars with fRCA decreased compared to reference mortar (A100).

Based on the preliminary mix design observations, it was decided to use SP for the final mortars mix design optimization and replacement levels of 0 wt%, 25 wt% and 50 wt% of natural sand by fRCA. Besides use of SP, also different treatments of fRCA were applied. The overview of optimized mortar mix designs is given in Table 2. Four series of mortars are investigated and compared to the performance of mortars with reference river sand:

**Table 2**
Mortars mix series.

| Series 1 (S1) Drying of sand (standard mixing sequence) | |
|---|---|
| S1i_A100 (as received sand, SP, 5 wt% 0–0.25 mm) | S1ii.A100 (dried sand, SP, 5 wt% 0–0.25 mm) |
| S1i_B25 (as received sand, SP, 5 wt% 0–0.25 mm) | S1ii.B25 (dried sand, SP, 5 wt% 0–0.25 mm) |
| S1i_C25 (as received sand, SP, 12 wt% 0–0.25 mm) | S1ii.C25 (dried sand, SP, 12 wt% 0–0.25 mm) |
| S1i_D25 (as received sand, SP, 17 wt% 0–0.25 mm) | S1ii.D25 (dried sand, SP, 17 wt% 0–0.25 mm) |
| Series 2 (S2) Modified content of 0–0.250 mm | |
| | S2ii.C25 (dried sand, SP, 5 wt% 0–0.25 mm) |
| Series 3 (S3) Modified mixing sequence | |
| S3i_A100 (as received sand, SP, modified mixing) | |
| S3i_B25 (as received sand, SP, modified mixing) | |
| S3i_C25 (as received sand, SP, modified mixing) | |
| S3i_D25 (as received sand, SP, modified mixing) | |
| Series 4 (S4) Cement increase | |
| S4i_A100 (as received sand, SP, 10 wt% cement increase) | S4ii_A100 (dried sand, SP, 10 wt% cement increase) |
| S4i_B50 (as received sand, SP, 10 wt% cement increase) | S4ii_B50 (dried sand, SP, 10 wt% cement increase) |
| S4i_C50 (as received sand, SP, 10 wt% cement increase) | S4ii_C50 (dried sand, SP, 10 wt% cement increase) |
| S4i_D50 (as received sand, SP, 10 wt% cement increase) | S4ii_D50 (dried sand, SP, 10 wt% cement increase) |

- **series 1 (S1)** (25 wt% fRCA): mortars with SP and as received and dried fRCA:

i. as received fRCA with its original particle size distribution (0–4 mm) in all mixes,

ii. dried fRCA with its original particle size distribution (0–4 mm) in all mixes (drying of the sand was performed at 105 °C in the standard oven until constant mass);

- **series 2 (S2)** (25 wt% fRCA): mortars with SP and modified content of 0–0.250 mm subfraction (dried sand), so that in each fRCA this content was the same as illustrated in Fig. 7. Samples were first dried at 105 °C and 0–0.250 mm content was adjusted by sieveing. Compared to B (~6 wt%), C and D sands have multiple times higher percentages of subfraction 0–0.250 mm (15–17 wt%). In order to evaluate contribution of the content of subfraction 0–0.250 mm to the air content in mortars, mortar mix C25 was selected and two designs were tested (i) C25 mix with 5 wt% of subfraction 0–0.250 mm and (ii) C25 mix with 12.5 wt% of subfraction 0–0.250 mm.
- **series 3 (S3)** (25 wt% fRCA): mortars with SP and modified mixing sequence (as received sand and dried sand). The modified mixing sequence is explained in details in Section 2.2.2.
- **series 4 (S4)** (50 wt% fRCA): mortars with SP and increase of cement (as received sand and dried sand).

The A100 mortar mix is used as reference mix, with 100 wt% of river sand. Mixes with 25 wt% and 50 wt% replacement of river sand by fRCA were labeled writing the type of sand (B, C, D) and incorporation percentage together.

### 2.2.2. *Mixing sequences*

A Hobart mixer was used for mixing 3 L batches. For mortar series 1, 2, 3, and 4 standard mixing sequence was applied as follows:

1) premixing river sand and fRCA for 1 min at stand I;
2) adding cement to river sand and fRCA and mixing for other 1 min at stand I;

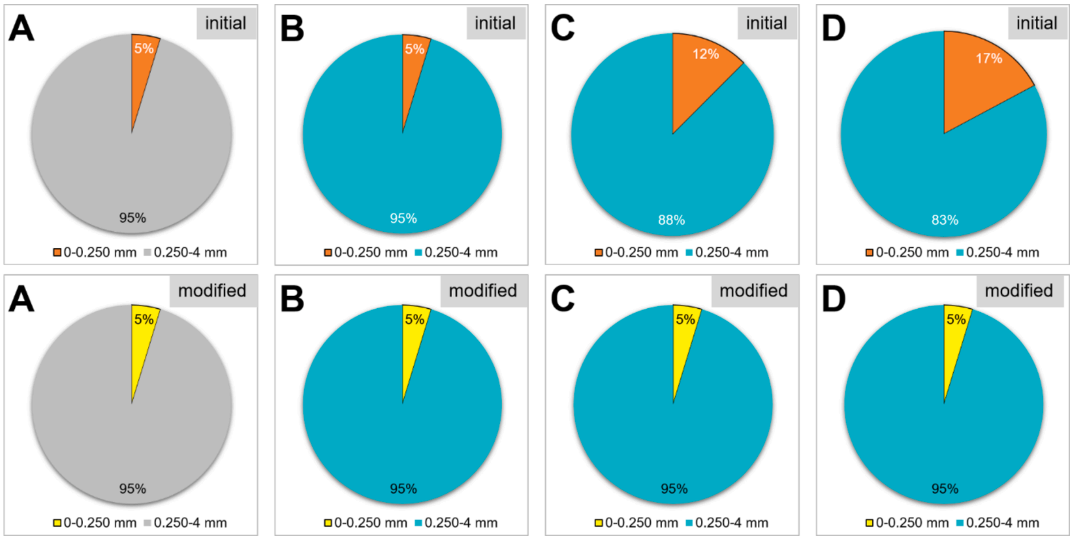


**Fig. 7.** Modified content of subfraction 0–0.250 mm towards the use of same content of 0–0.250 mm in all mixes.

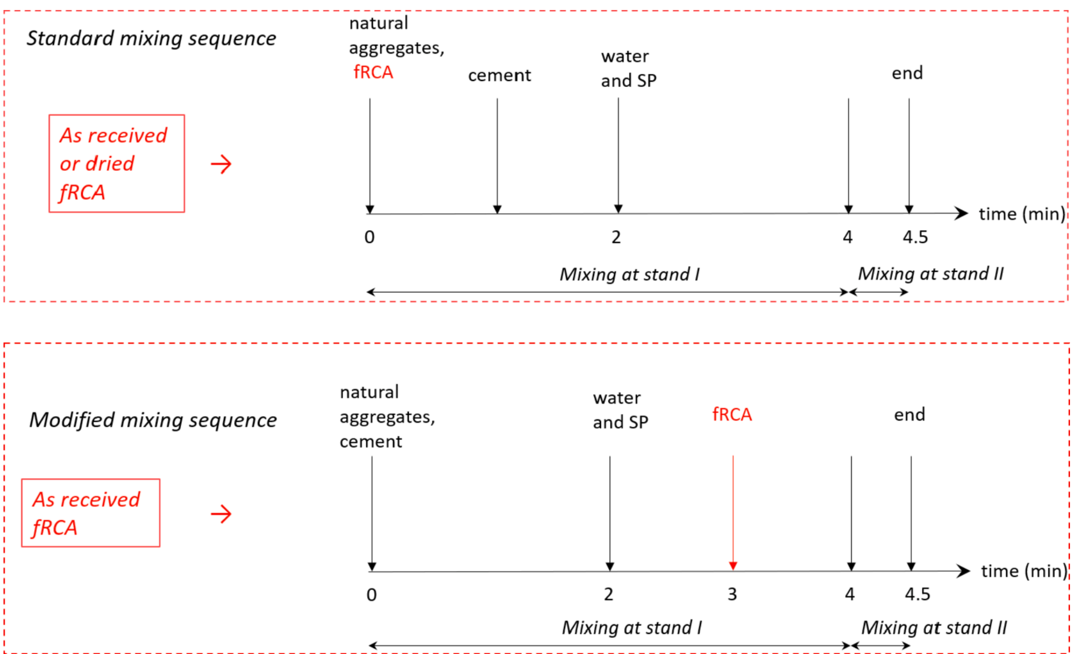


**Fig. 8.** Standard and modified mixing sequences for mortars with fRCA.

3) SP was added to the tap water and the resulting suspension was poured onto the raw materials in the mixer. Mixing continued at at stand I for 2 min and for 0.5 min at stand II.

For mortar series 3, a modified mixing sequence was used:

1) premixing river sand and cement for 2 min at at stand I;
2) SP was added to the tap water and the resulting suspension was poured onto the raw materials in the mixer. Mixing continued at at stand I for other 1 min.
3) adding as received fRCA to materials in the mixer. Mixing continued at stand I for 1 min and for 0.5 min at stand II.

The illustration of the mixing sequences is given in Fig. 8.

### 2.3. Methods

#### 2.3.1. Flow measurements

The standard mixing sequence was followed for the slump-flow tests. The flow was tested by the mini-slump spread test to determine the flow values of the mortar mixtures according to EN 1015–3:1999. A truncated conical mold was prepared and filled with fresh mortar mixtures (diameter 70-100 mm and height 60 mm). The spreading table was always wiped and wet first and then the flow test would be performed. By following this procedure, it was ensured that there is no friction between the fresh mortar and the spreading table during the flow tests. The introduction of fresh mortar was divided into two layers, and each layer was compacted with a wooden tamper for 10 S to ensure uniform filling of the mold. Subsequently, the mold was lifted smoothly in vertical direction and the slump-flow value was measured after 15 S of free falling from a height of 10 mm on the flow table. The fresh mortar for this test had a minimum volume of 1.5 l. The flow value was obtained as the

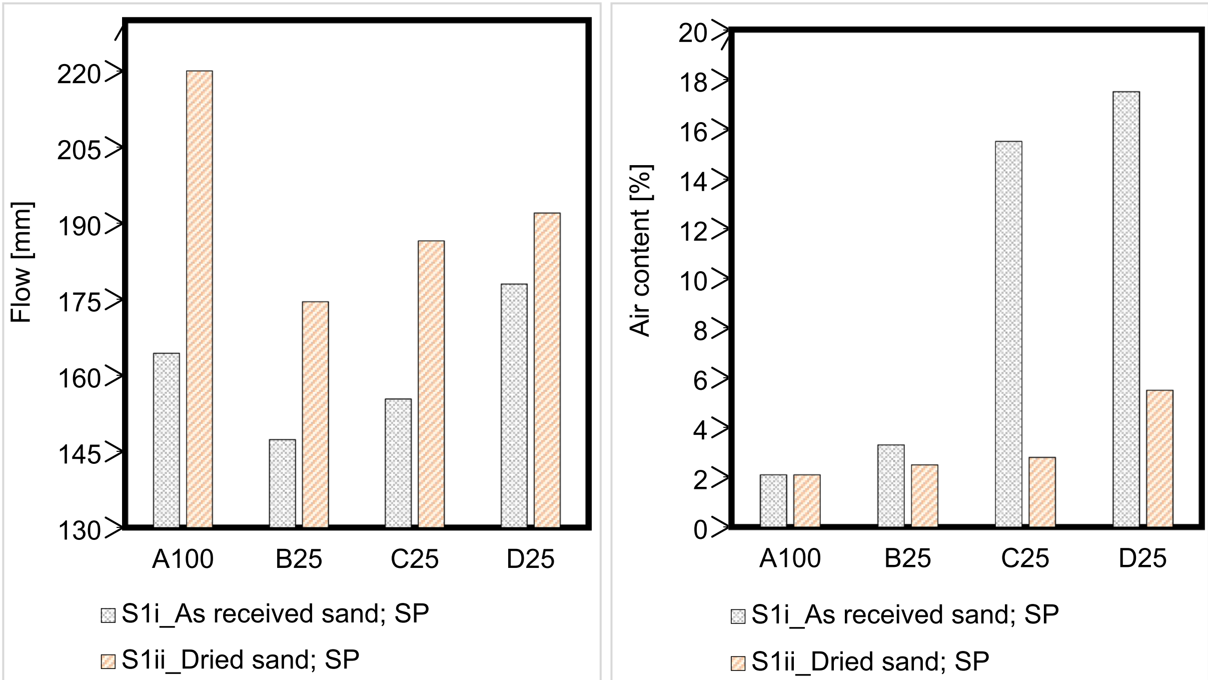


**Fig. 9.** Flow and air content of mortars.

mean value of the two measurements.

#### 2.3.2. *Air content*

The standard and modified mixing sequences were followed for the air content tests. Air content evaluation of fresh mortars was performed with standardized pressurized gauge method in accordance with EN 1015–7:1998. The reading of apparent air content percentage is taken with the accuracy of 0.1 % and ± 1 % of measuring error. The fresh mortar for this test had a minimum volume of 1.5 l. The air content is calculated as the mean value from the two individual values of each mortar test sample.

#### 2.3.3. *Compressive strength*

The standard and modified mixing sequences were followed for the compressive strength tests. The fresh mixtures were cast in prisms moulds (40 × 40 × 160 mm$^3$). The fresh mixtures were filled in two steps on compaction table. Each step was followed by compaction for 15 s in order to remove entrapped air. Samples were demolded 24 h after production and stored at > 95 % relative humidity, at room temperature until testing.

First, the three-point flexural bending test was performed according to the EN 196–1:2016. Three specimens were tested per age. Two halves of the specimen (cubic 40 × 40 × 40 mm$^3$) were then used for testing the compressive strength. The compressive strength was calculated as an average value of six samples. Then the standard deviations were

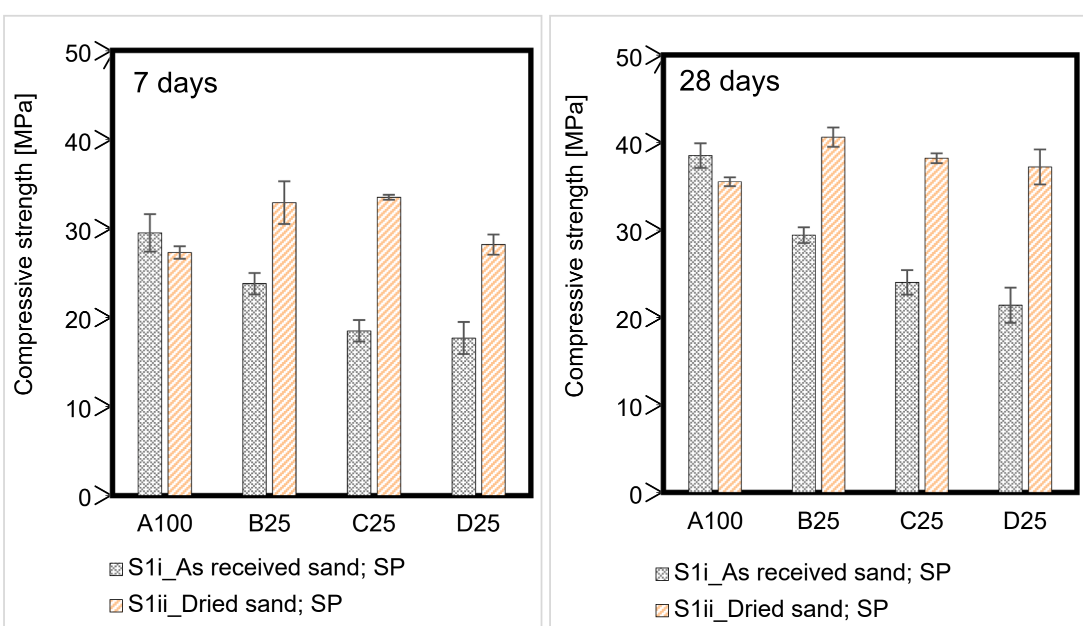


**Fig. 10.** Compressive strength of mortars at 7 and 28 days.

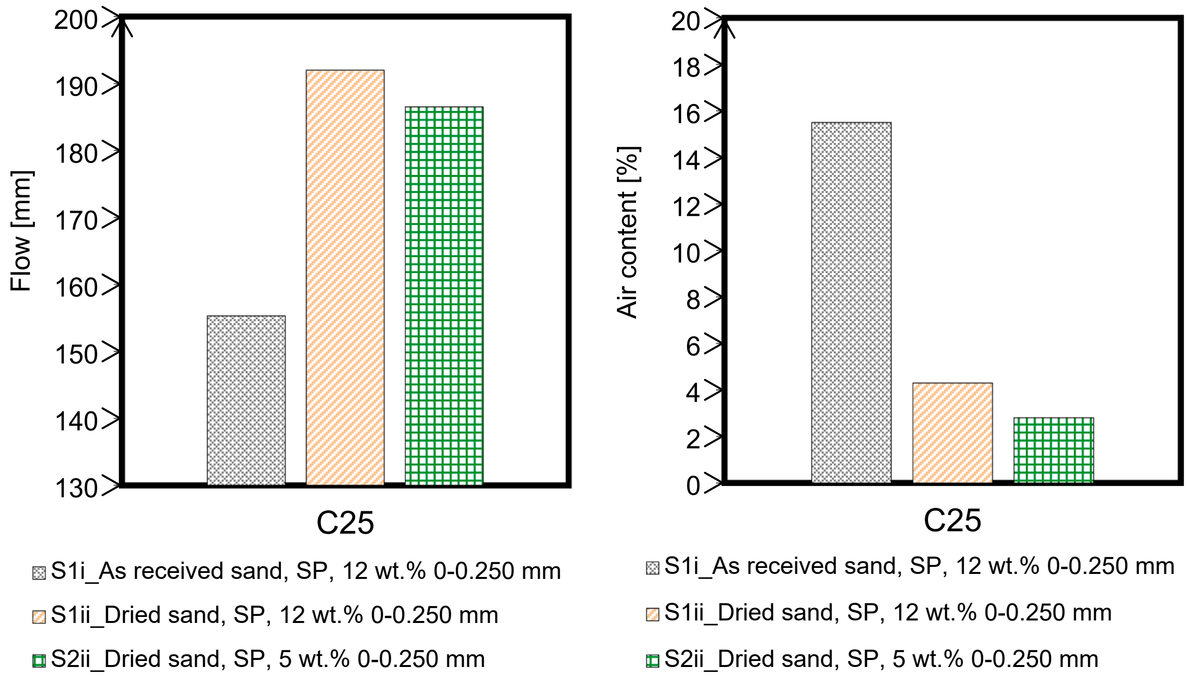


**Fig. 11.** Flow and air content of C25 mortars.

calculated for each set of data.

*2.3.4. Isothermal calorimetry*

The modified sequence was followed for the calorimetry tests. Isothermal calorimetry analysis was carried out to investigate possible differences in hydration kinetics of studied mortars with fRCA. The primary goal was to demonstrate whether the fRCA is reactive and whether it can cause certain delays in the hydration of cement due to its compositional heterogeneity compared to natural sand. The control sample was cement paste, CEM I 42.5 N with water-to-cement ratio 0.5 and SP. All the raw materials were conditioned at the measurement temperature. Samples were prepared outside the calorimeter. For a calorimetry test, the sample (mix of water, cement, river sand and fRCA) is stored in a glass ampoule (125 mL) and placed in the calorimeter (3-channel TAM Air, Thermometric). The content of fresh mortar was 28.8 g. The calorimeter consists of 3 parallel twin type measurement channels: one from the sample, the other for the reference. A glass ampoule with water was used as a reference. Once the thermal equilibrium is reached, the heat evolution is recorded at the constant temperature (25 $\pm$ 0.02 °C). The heat flow and the cumulative heat were normalized to the cement mass in the samples. Three replicates were measured simultaneously.

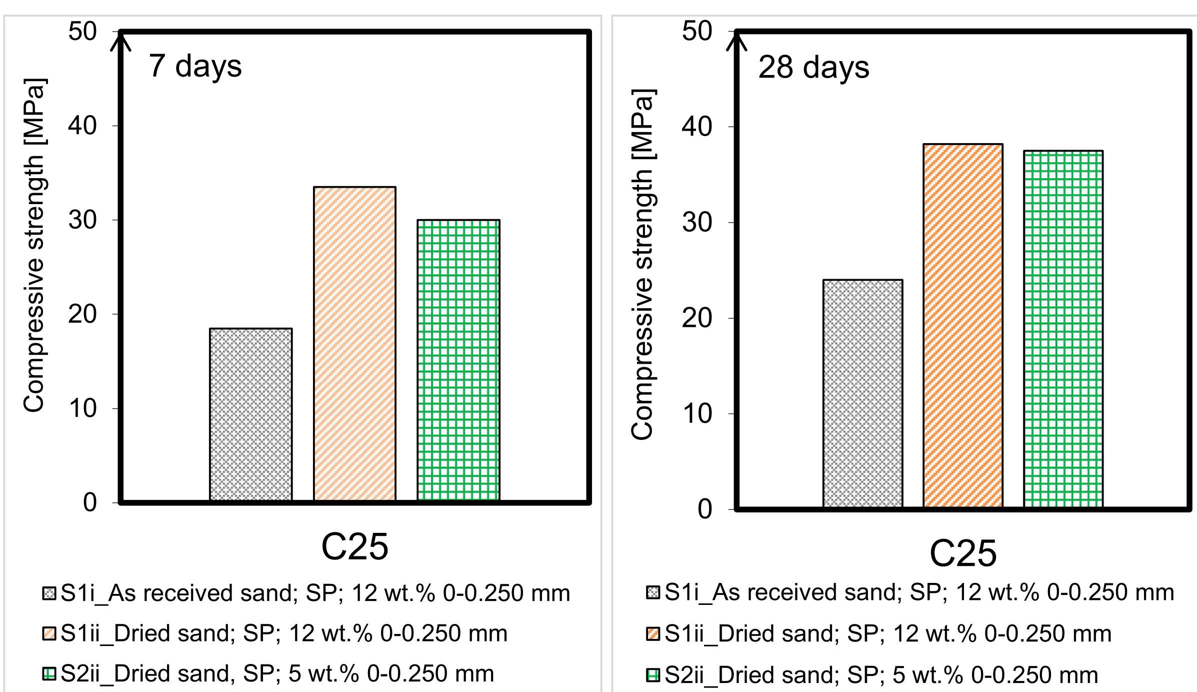


**Fig. 12.** Compressive strength of C25 mortars at 7 and 28 days.

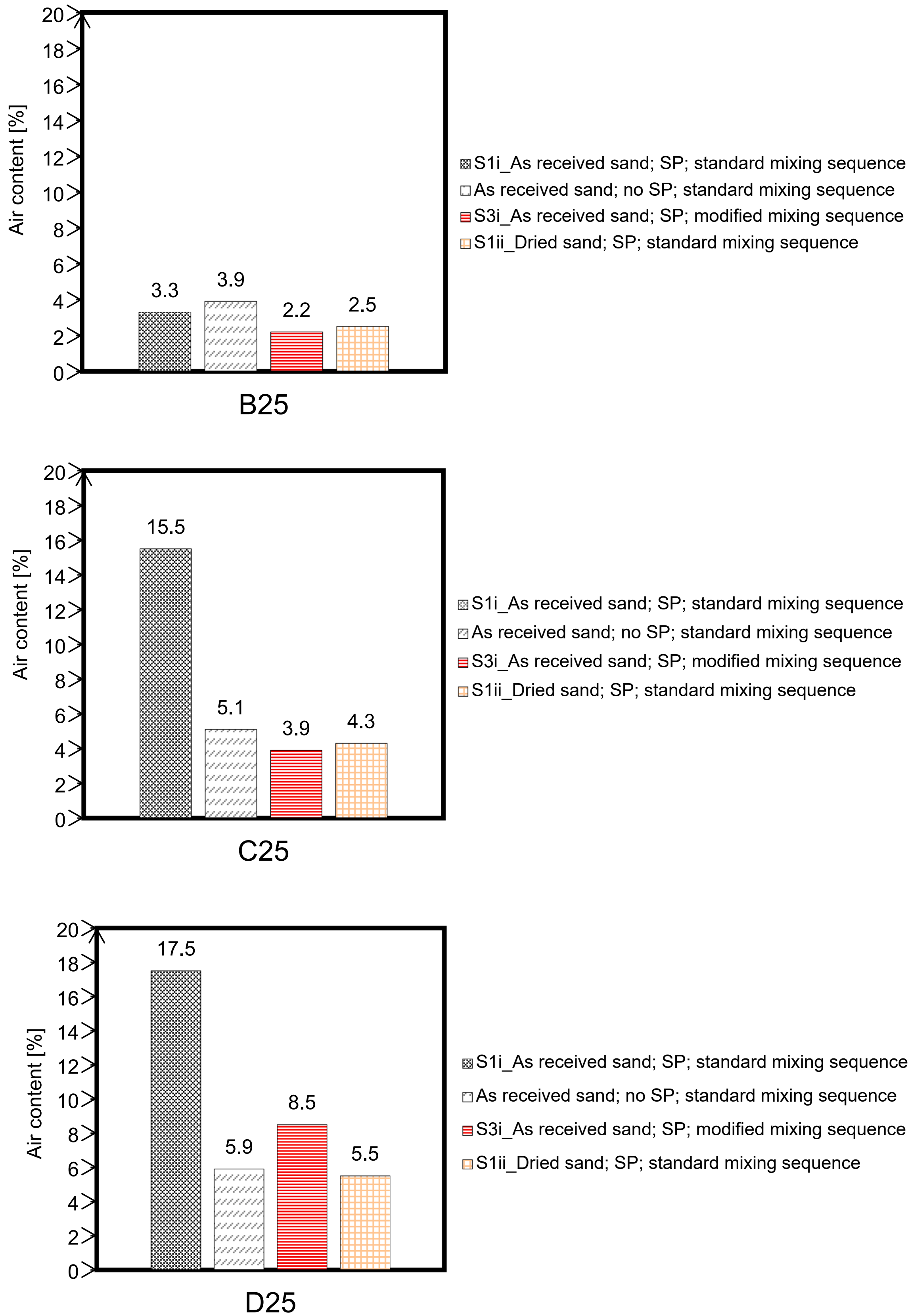


**Fig. 13.** Air content of mortars.

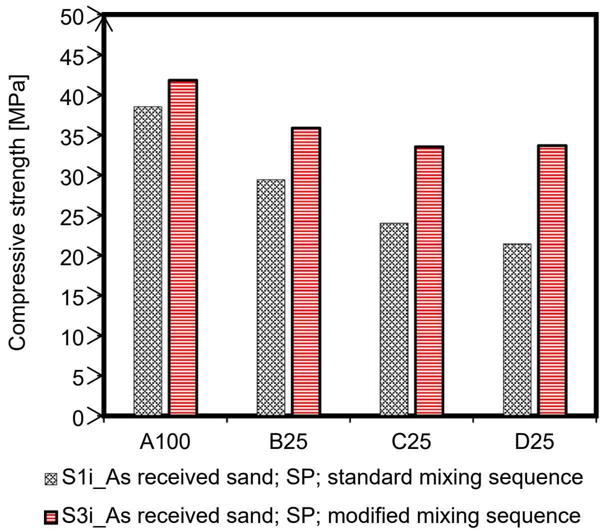


**Fig. 14.** Compressive strength of mortars at 28 days.

## 3. Results

### 3.1. *Effect of sand drying (Series 1)*

Fig. 9 shows flow and air content of mortars with as received and dried fRCA. It can be seen that the flow of the mortars increased when fRCA was used as dried in the mixing while the air content decreased. The air content was considerably higher in mixes C25 and D25 compared to reference mix A100 and mix B25. Fig. 10. shows effect of SP and sand drying on compressive strength of mortars at 7 and 28 days. It can be seen that the strength is positively influenced by the drying of the sand at both ages for all mortars. In case of two mortars C25 and D25 with as received sand, the air content is five to six times higher than of A100 and B25.

### 3.2. *Effect of modified content of 0–0.250 mm (Series 2)*

The effect of reducing the content of subfraction 0–0.250 mm from 12.5 wt% to 5 wt% in mix C25 is shown in Fig. 11 and Fig. 12. Flow was slightly lower for mortar with 5 wt% 0–0.250 mm compared to mortar with 12.5 wt% 0–0.250 mm. Air content was also reduced with partial removal of subfraction 0–0.250 mm. The effect of 0–0.250 mm partial removal did have less influence on the compressive strength of the mortars (Fig. 12).

### 3.3. *Effect of modified mixing sequence (Series 3)*

Fig. 13 shows the effect of modified mixing sequence on the air content in different mortars, per fRCA-type. Mortar mixtures without SP were tested, and compared to the mixtures with SP, to isolate the effect of the SP addition on the air content. Other two mixtures were also added for comparison, where as received and dried sand with standard mixing sequence were used.

The air content of the reference mixture A100 was 2.1%. All B25 mortars exhibited air content values lower than 3.9 %, while mortar B25 with modified mixing sequence exhibited air content similar to reference mixture A100. In contrast, all C25 and D25 mortars exhibited air content values higher than 3.9 %, indicating the negative effect of fRCA source and quality on air content of mortars. As it can be seen the air content in all mortars with as received sand, SP and standard mixing sequence was very high in mortars C25 and D25. The air content of mortar without SP and standard mixing sequence was high but still lower than of mortars with SP and standard mixing sequence.

### 3.4. *Effect of cement increase (Series 4)*

With the increase of fRCA-fraction (from 25 wt% to 50 wt%), the mixes become ‘sandy’ and SP alone could not improve the flow of the mortars. The flow of mortars with 50 wt% fRCA was enhanced by increasing content of the cement paste and by that increasing Fig. 13cement content. Fig. 15 shows the flow and air content of mortars (50 wt% fRCA) with 0.3 wt% SP dosage and 10 wt% cement increase. The air content was higher in mixes C50 and D50 compared to reference mix A100 and B50, similar to what has been observed in mortars C25

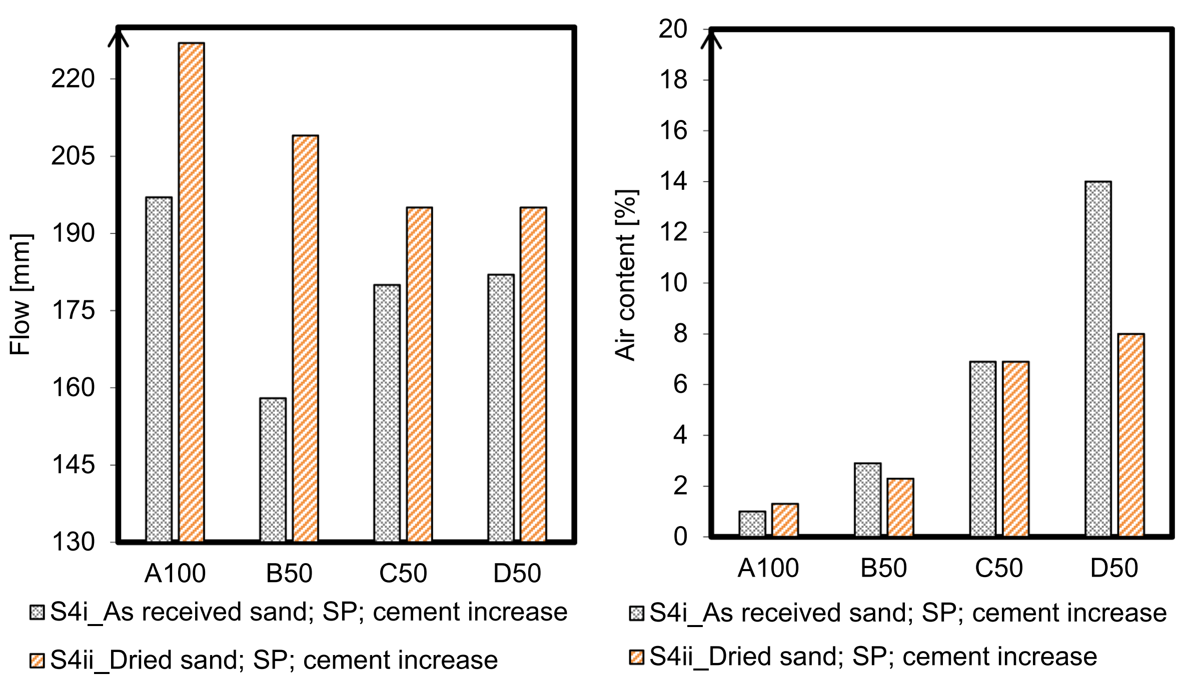


**Fig. 15.** Flow and air content of mortars.

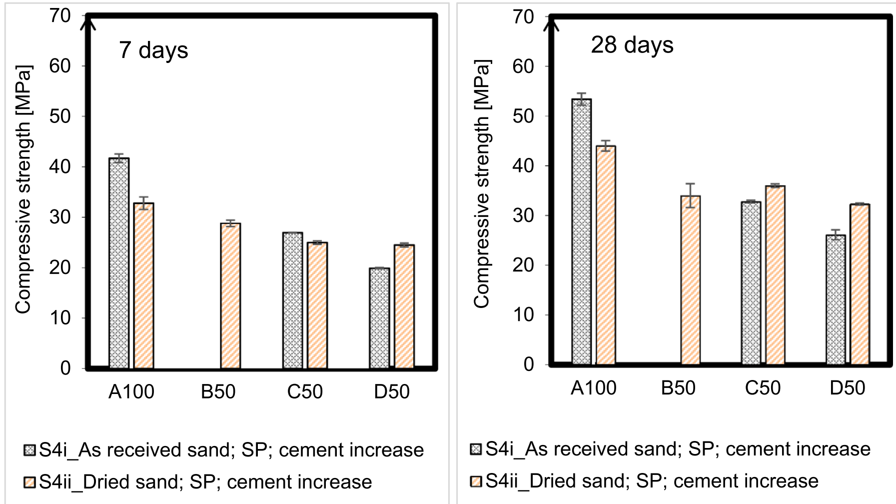


**Fig. 16.** Compressive strength of mortars at 7 and 28 days.

and D25.

The mortars containing dried fRCA (B50, C50, D50) demonstrated similar strength results at both ages (7 and 28 days) as compared to corresponding mortars containing as received fRCA (Fig. 16). Mortars D50 with as received fRCA have lower strength results than mortar C50. This is in agreement with air content results for mortars C50 and D50. Although flow of mortars was satisfactory, a cement increase of 10 wt% was not sufficient to obtain comparable strength of mortars with fRCA to mortars with river sand.

### 3.5. Isothermal heat release

Isothermal calorimetry has been used in order to assess the effect of fRCA in mortars on the rate and heat development of the cement hydration at 25 °C. Fig. 17 shows the heat evolution of the three mixtures: cement paste, cement mortar with as received sand, modified mixing sequence and cement mortar with dried sand, standard mixing sequence for each type of fRCA. The SP was used for all mixes. All heat curves are normalised by the weight of cement.

The heat released from calorimetry curves shows that the overall pattern of reaction of the cement from mortars as compared to cement paste is similar. However, small changes are observed regarding influence of fRCA addition on cement hydration, depending on the moisture condition of the sand and on the source of the sand used in the mortar. During the first few hours exothermic heat events are observed for all mortars due to the dissolution of cement in water. Two calorimetric peaks were observed from the heat evolution rate curves. The first peak is comparable for all the mortars. The second peak is characteristic of mortar D25 (with both, as received and dried sand). To date, several studies have reported that the occurrence of this peak in the calorimetric curve corresponds to the sulphate depletion peak during hydration of Portland cement or cement with mineral additives [67-70]. Sulphate depletion peak can be observed when the proportions between the aluminate phase and gypsum decline from those enabling the covering of aluminate crystals surface with ettringite [68]. In general, upon depletion of the sulphates, the remaining tricalcium aluminate phase ($C_3A$) will react with the ettringite ($C_3(A,F){\cdot}CaSO_4{\cdot}32H_2O$) to form monosulphate ($C_3(A,F){\cdot}CaSO_4{\cdot}12H_2O$) or hydroxy-AFm solid solution [67]. Optical polarizing-and-fluorescence microscopy (PFM) study showed minor amounts of secondary ettringite in sand D in authors' previous study [16], which suggests that the sulfate attack was not the degradation mechanism acting on the parent concrete of the fRCA D. Since the same type of cement (CEM I 42.5 N) and the same type of SP (polycarboxylate-based) were applied for all investigated mortars (A100, B25, C25, D25), it is possible to hypothesise that the composition or amount of fine fraction (<0.250 mm) of fRCA D are likely a cause for sulphate depletion in mortar D25 rather than composition of cement. Hypothetically, since sand D has more fines or somehow more calcium silicate hydrate phases are produced during the acceleration period then part of sulphate would not be available to react with $C_3A$ but will be adsorbed on calcium silicate hydrate phases. Then at the decceleration it will be desorbed resulting in a fast dissolution of remaining $C_3A$ and fast formation of ettringite. Upon depletion of the sulphates, the remaining $C_3A$ will react with the ettringite to form monosulphate. This would not be or less the case with other sands (B, C). Other explanation is that the fRCA D might contain recycled gypsum plasters or gypsum boards and therefore it may affect the availability of soluble sulphates and the chemical composition of the pore solution in the mortar [71]. The high sulphate concentration in the pore solution is known to be stabilized by the solubility of gypsum. As gypsum stabilizes sulphate concentration in the pore solution, it might enable the transformation of ettringite to monosulphate [67] suggesting sulphate depletion. Third possible explanation for sulphate depletion could be that there is more unreacted $C_3A$ in sand D (compared to sands B and C), just because by coincidence the latter have more old cement paste with other cements than CEM I. To develop a full picture of sulphate depletion in mortars with recycled sand, additional studies will be needed that include pore solution analysis.

The first peak of mortars with as received sand are shifted to earlier hydration time (2 h difference) as compared to the corresponding mortars with dried sand. This can be attributed to the acceleration effect due to presence of impurities from unwashed fRCA. The release of hydration heat is reaching a plateau after 90 h for all mortars, and the main hydration reactions occur during first 48 h.

## 4. Discussion

### 4.1. Air content

Different methods have been tried in order to optimize and understand performance of mortars in Section 3. The tailor-made SP was used for all methods. The methods were: (1) drying of sand, (2) modified content of 0–0.250 mm, (3) modified mixing sequence, (4) cement increase. For all approaches, as received and dried sand were introduced as distinct materials. The fRCA dosage of 25 wt% and 50 wt% by mass replacement of river sand was considered. Based on the obtained results, mortars mixtures with fRCA resulted in an increased air content. Fig. 13 shows an overview of the effect of fRCA source and optimization

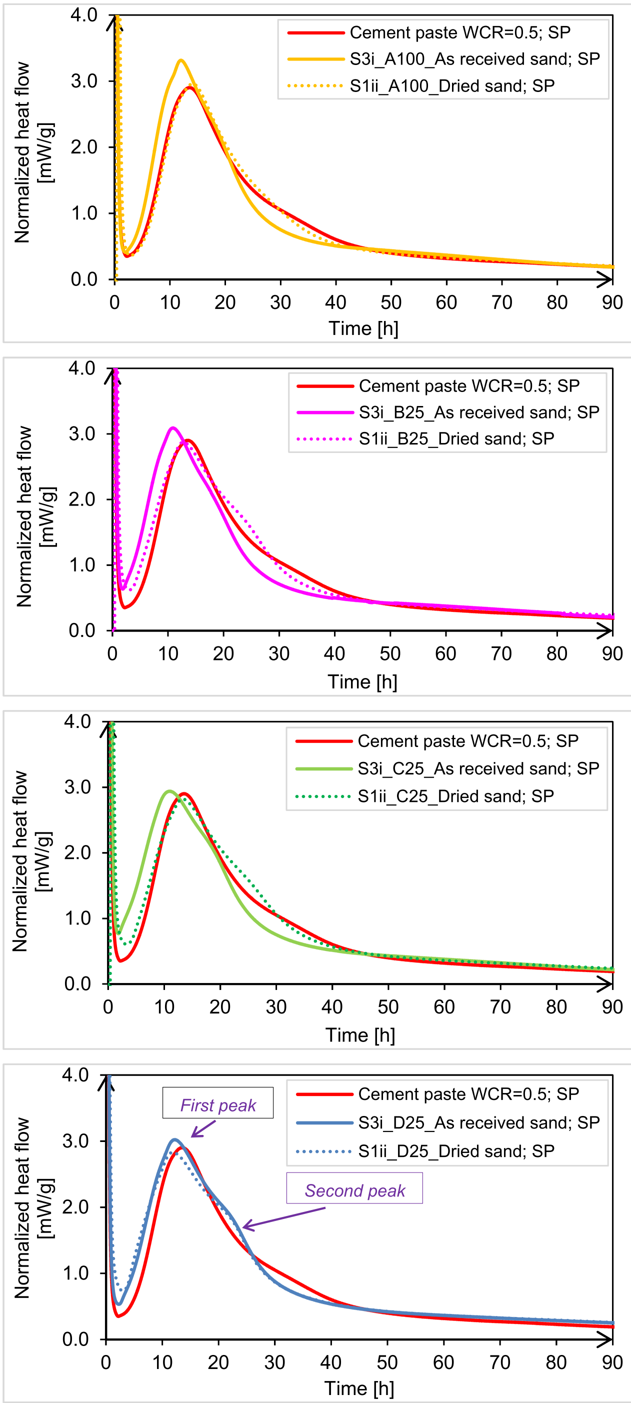


**Fig. 17.** Heat flow of cement paste and cement mortars.

approach on air content in different mortar series, per fRCA-type.

Regarding the source of fRCA, it is clear that the origin and the quality of fRCA play significant role. Considering the quality of fRCA, drying of the sand substantially decreased the air content. Mix B25 resulted in a slightly higher air content (2.5 %) when compared with reference mortar A100 (2.1 %). This value would be accepted for mortar mixtures in practice, as the limit of 3 % of air content is not surpassed. However, compared to reference mixture, the air content of mortars C25 and D25 with as received fRCA, showed substantially higher values than that of the reference by 86.5 % and 88 %, respectively. There are several possible explanations for this large difference in the air content.

1) fRCA consist of sand particles and residual cement mortar that introduce additional porosity.

As shown in Fig. 18, reference river sand has compact and round particles while fRCA have porous clusters of pure sand particles and residual cement mortar depending on the quality of parent concrete (e.g. presence of poorly compacted concrete, high water-to-cement ratio). Residual cement mortar can accommodate more air on its surface and inside [72] and it can have a large volume of voids as it can be seen in Fig. 18 (right photo). These empty spaces can potentially entrap air and give rise to air content of the mixture, such as in C25 and D25 mortars.

2) Agglomeration of fRCA due to outdoor storage.

As received fRCA C and D are considerably agglomerated (Fig. 1) due to outdoor storage conditions, compared to sand B which was not stored after production. Agglomeration is promoted by several factors:

- Cement binder. fRCA contain residual cement mortar of different compositions [16]. There is limited knowledge of the impact of residual cement mortar, and therefore, type of cement binder, on the agglomeration potential of stored fRCA. It is well known that Portland cement is often used as a binder to increase the strength of agglomerates for precious metal heap leaching because of the cementing effect that strengthens the agglomerates [73]. Similar to the heap leaching, mechanism of agglomeration in fRCA bulk storage may be explained by binding of fRCA fine particles together with coarser particles via the cement hydration products present in the residual cement mortar.
- Increased content of fraction 0–0.250 mm in fRCA compared to natural sand (the more crushing cycles, the more fines are being produced). This fine fraction has high surface area. Fine powder, or powder with high surface area, typically exhibits high cohesive forces [74]. The agglomerates in a cohesive powder are either due to attractive forces (e.g., electrostatic charging and/or van der Waals forces) between particles, or due to bonding between the particles due to moisture [75]. The fine particles are agglomerating to the surfaces of larger particles.
- As a material inherent property, fRCA have high ability to absorb water and water–vapor from the surrounding air producing agglomerated particles. High water absorption of fRCA (Table 1) causes more inter-particle forces (fRCA particles have more water bridges that are increased by wet conditions in the outdoor storage compared with reference river sand, Fig. 19).
- During the outdoor storage carbonation may take place via $CO_2$ physical and/or chemical uptake at the surface of fRCA particles in the presence of moisture. This can result in binding and agglomeration of fine particles in the C-S-H gel on the surface of fRCA. Similar agglomeration mechanism has been reported for quenched bottom ash consisted of a C-S-H phase [76]. Fig. 19. illustrates $CO_2$ uptake by the residual cement mortar and agglomeration of fRCA particles during the outdoor storage. Relative humidity ranges from 60% to 98% with 0.04% v/v $CO_2$ in Dutch outdoor conditions [77].

These types of agglomerates are most probably not falling apart during short mixing process. Consequently, additional air in as received fRCA C and D is expected due to empty pockets that entrap air (as illustrated in Fig. 20). There are no characterization tools for assessment of the strength of fRCA agglomerates. In case of a dried sand, the agglomeration effect could be evaluated through the particle size distribution testing of fRCA before and after mixing (before adding other ingredients to fRCA) in a mortar mixer for longer times (>5 min). Nevertheless, agglomerates from different production batches will behave differently in terms of entrapped air, size, strength, storage duration. To overcome agglomeration, the storage of fRCA should be avoided or fRCA should be stored in dried conditions.

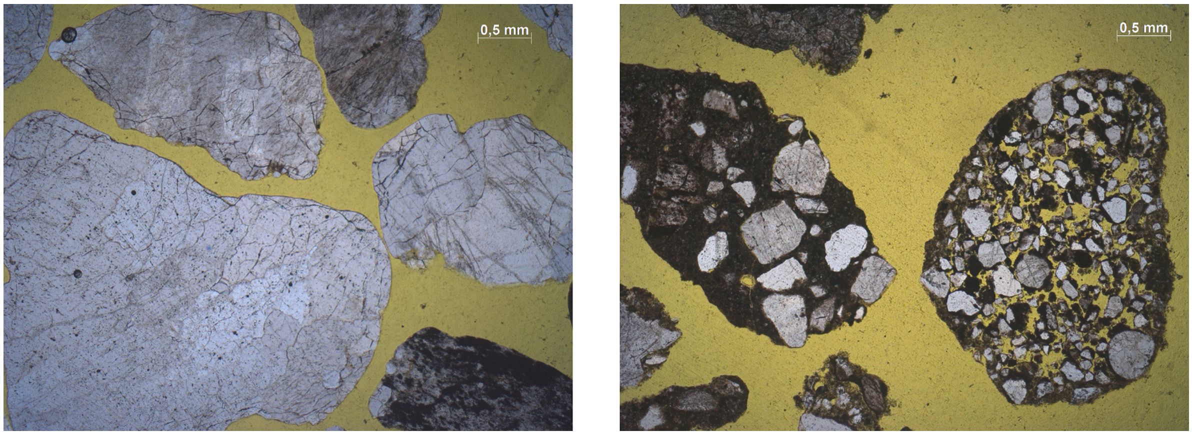


**Fig. 18.** River sand particles (left) and particles in fRCA D (right) [16].

3) Presence of impurities in as received fRCA causes air stabilization in mixing water.

As received fRCA is not washed and, therefore, fRCA may contain different impurities. For instance, fRCA obtained from old buildings or old pavements may contain small amounts of soft soils, natural stones, clay bricks, and other impurities like paper, wood, glass, ceramics and metals (aluminum and steel) [72,78]. This means that some of surface active agents (as illustrated in Fig. 20 in red colour) and impurities from fRCA (contamination from enviroment, contamination from demolition works, contamination from soil, coatings and hydrophobic impregnation) can be water soluble. Once that fRCA is mixed with water designed for the mortar, the water might be polluted by multiple impurities. These water-soluble impurities can be detrimental for concrete properties [79], as shown even for concrete with natural aggregates which might have coatings including dust, silt, clay, iron oxide and calcium carbonates from the processing (quarried deposits, ground water). The quality of mixing water plays key role in stabilizing the air bubbles in fresh mortar/concrete mixture [45]. Water with significant impurities present in it may have an effect (positive or negative) on air entrainment. Similarly, unwashed fRCA containing various impurities can increase air entrainment when mixed with tap water and cement.

4) Presence of impurities in as received fRCA reduces effectiveness of SP.

It is reported that the foaming property of surfactants from SP can be affected by the impurities present in the host solution [45]. Inorganic electrolytes are most effective with ionic surfactants and polar organic additives can affect all types of surfactant [80]. Macromolecular materials can also influence the formation and stability of air bubbles [45]. SP could not be hardly adsorbed on the cement particles due to presence of unknown impurities on the cement particles or in the mixing water, as shown in Fig. 20, affecting the working mechanism of SP. This consequently could lead to air bubbles to be kept by the SP chains in the fresh mortar mix. In addition, some chemical admixtures reduce the water absorption by capillarity, creating small air bubbles in mortar or even stabilizing them [81,82].

To mitigate the (possible influence) of the last two effects (3, 4), a modified mixing sequence on the air content of mortars was tested. First reference sand, cement, water and SP were added to the mixing bowl and premixed without fRCA in order to avoid initial pollution of the mixing water with unwashed fRCA. Afterwards the fRCA was added. It was assumed that by changing the mixing sequence, SP can be adsorbed by cement particles effectively. It is found that this approach provides significant reduction of air content in all mixes, indicating that fRCA contain some matters that are reducing effectiveness of SP when the standard mixing sequence is applied. Using modified mixing sequence (when fRCA was added the last in the fresh mortar mix) in addition to drying, can effectively reduce the air content of mortars as shown in Fig. 13, independent of fRCA quality and source.

The air content was reduced largely in mortars with dried sand and SP. For measuring particle size distribution in Fig. 2 and Fig. 3, sand was first dried (by which the agglomeration of particles has partially disappeared).When moisture disappears from the surface of particles, i.e. by drying, then any charge induced by the presence of surface active agents and impurities on the cement particles is removed or immobilized (Fig. 20). An example of such agents is given in [83]. This makes the SP easier to redistribute and adsorb. It should be noted that besides the drying theory, other explanations may also exist for the air content increase in the presence of SP. In reviewing the literature, no data was found on the association between the air content and SP in presence of fRCA. For a given air content in concrete, the larger the air-void diameter, the larger the spacing factor and specific surface area, and the lower the freeze–thaw resistance [45]. A further study with more focus on air content in mortars and concretes with fRCA is therefore suggested.

### 4.2. Compressive strength and reaction kinetics

The increased air content in the mortar influences its strength. This behavior is observed in Figs. 10, 14 and 16. The compressive strength for mortars with as received sand varies with the source of fRCA. This is due to the variation in quality of fRCA which originates from an unknown quality of parent concrete. fRCA originate from various sources and therefore, chemical, physical and mechanical properties of such material, vary largely.

The strength of mortars B50, C50, D50 (Fig. 16) is lower than that of mortars B25, C25, D25 (Fig. 10). The replacement of river sand with 50 wt% fRCA reduces the content of hard aggregate material in mortar. As shown in Table 1, on average, fRCA consist of 71 wt% fraction of hard aggregates and the remaining fraction is occupied by the old cement paste. This negatively affects compressive strength of investigated mortars in agreement with previous studies [84,85]. This effect can be explained through the quality of parent concrete, i.e. porosity of old cement paste and ITZ in fRCA particles: on one hand, particles from good quality parent concrete have more reaction products and denser microstructure, but on the other hand the particles from less good quality parent concrete have detrimental effect on the strength due to their large voids and porosity, as shown in Fig. 18 (right). As a result, the strength of mortar will be influenced by the proportion of such particles. There are, however, other possible explanations. It is found that mortar with 100 wt% fRCA had a higher fraction of loosely packed low-density calcium silicate hydrate phases than mortar with natural sand [86].

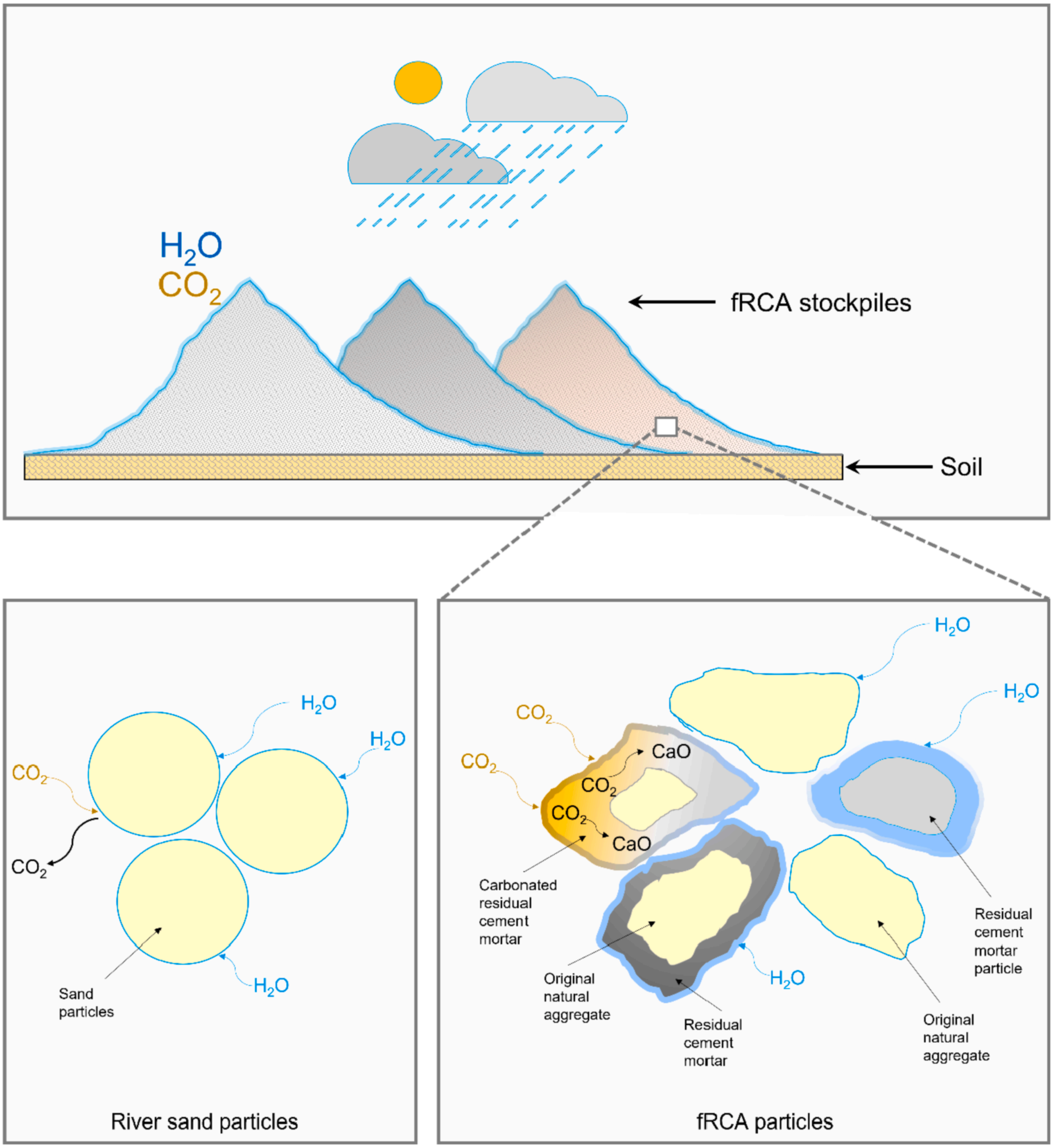


**Fig. 19.** Schematic representation of the agglomeration of fRCA particles during the outdoor storage in Dutch wheather conditions in comparison to river sand particles.

Porosity of calcium silicate hydrates account for more than a half of the total porosity. Therefore, porosity reduction of calcium silicate hydrates and increase of the fraction of optimally-packed high-density calcium silicate hydrates is required to improve the mechanical properties of mortars with 100 wt% fRCA [86]. This might be beneficial also for replacement levels such as in this study, for 50 wt% fRCA. Several studies have shown that carbonation and pre-treatment of fRCA using pozzolanic materials are the most efficient strategies to improve physical and chemical properties of fRCA and therefore, properties of mortars and concretes with fRCA [40,50,59,60,62,63].

The effect of the mixing sequence (standard versus modified) on the compressive strength of mortars with as received fRCA is shown in Fig. 14. According to Fig. 14, the compressive strength is lower for all mortars with as received sand when the standard mixing sequence is applied. In contrast, the strength of the mortars is increased when modified mixing sequence (Fig. 8) is applied. This is due to the respectively lower air content in these mortars, as shown in the previous subsection.

The adjustment of the content of the 0–0.250 mm fraction (Fig. 7) did not improve the properties of mortars significantly compared to other methods (drying of the sand, modified mixing sequence). Using adjustment of the whole particle size range (0–4 mm) to identify how this would affect the mortars' design (e.g. use of SP) and performance is recommended for the future studies. For this purpose, particle size distribution of a reference natural sand should be taken into account. In addition, the particle size distribution can only be adjusted if the sand is dried. It is not possible to obtain particle size distribution for the as received fRCA considering their moist state.

Considering the effect of origin, moisture condition of fRCA (as received or dried) and mixing sequence on the air content and compressive strength of mortars, as received sand with modified mixing sequence and SP and also dried sand with standard mixing sequence and SP show the best performances. These mixes have similar reaction kinetics (Fig. 21).

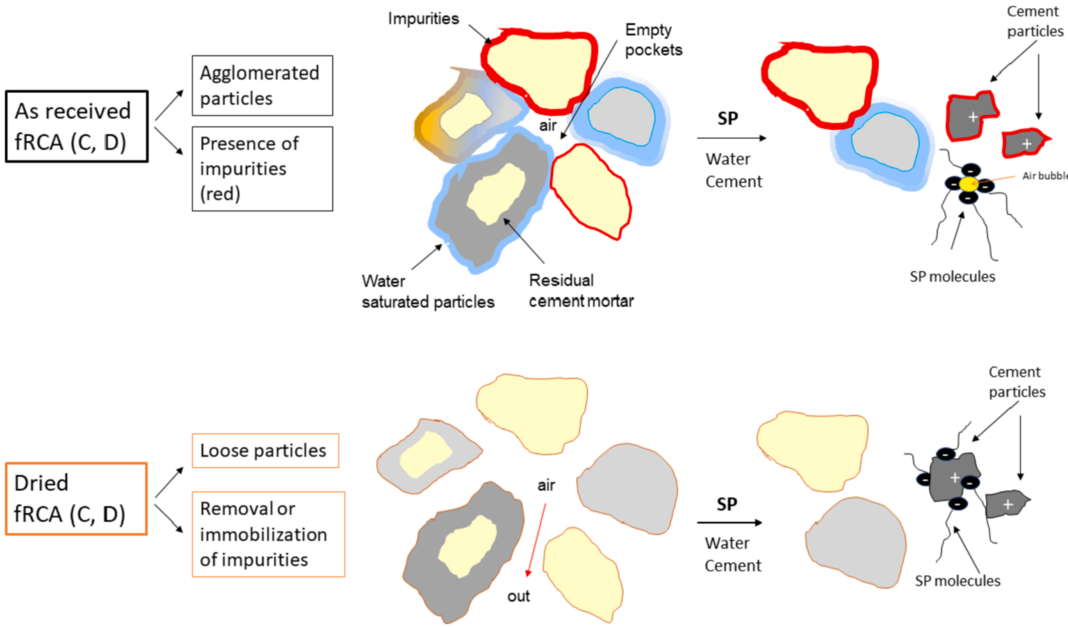


**Fig. 20.** Effect of fRCA drying on working mechanism of SP. Distribution of surfactant molecules from SP and the interaction with cement particles for two types of fRCA: as received and dried. Red colour highlights presence of impurities from unwashed fRCA materials. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

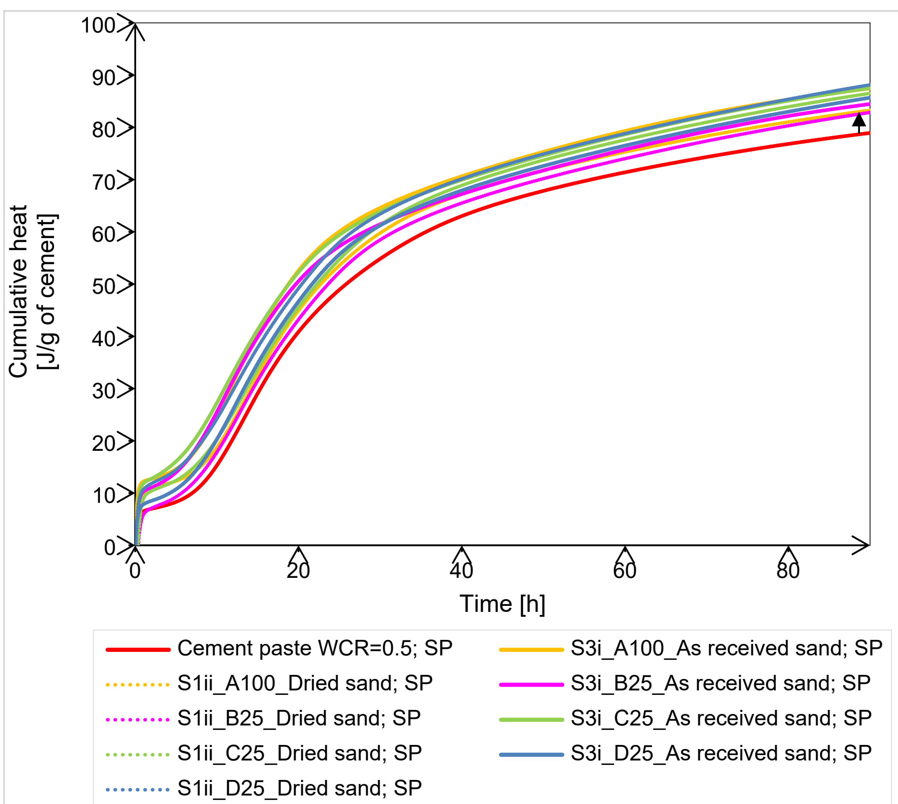


**Fig. 21.** Cumulative heat of cement paste (WCR = 0.5, SP) and cement mortars with river and fRCA sands after 90 h.

Mortars have slightly higher cumulative heat compared to cement paste as shown in Table 3. The higher cumulative heat per g cement of the reference mortar with quartz (A100) and of the mortars with fRCA (B25, C25, D25) compared to cement paste, indicates that the nucleation sites on the inert quartz sand particles served as favourable sites for nucleation and growth of cement hydration products which agrees with previous observations [87,88]. Finally, values for cumulative heat reported in Table 3, indicate that in the first 90 h of reaction the effective water for cement hydration was the same in both mortars with as received and dried sand. The reaction kinetics of two systems of mortars is similar and reveals a very low error, which could have been induced by sample preparation and placing in the calorimeter. Furthermore,

**Table 3**
Cumulative heat of cement paste and cement mortar samples after 90 h.

| | A100 | B25 | C25 | D25 | Cement paste |
|---|---|---|---|---|---|
| | Cumulative heat [J/g of cement] after 90 h | | | | |
| S3i_As received sand; SP; modified mixing sequence | 87.5 | 84.5 | 87.5 | 88.1 | 78.9 |
| S1ii_Dried sand; SP; standard mixing sequence | 83.3 | 82.9 | 86.5 | 85.7 | |

based on calorimetry results, the substitution of 25 wt% river sand by fRCA does not change the degree of cement hydration in agreement with study of Delsaute and Staquet [89]. The positive side of this is that the fRCA can be considered as non-reactive.

Considering the overall influences of fRCA on flow, air content, strength of mortars, it seems that 25 wt% substitution of river sand by fRCA shows the optimal performances. Nonetheless, it should be noted that even the compressive strength of mortars with 50 wt% fRCA, 32 MPa at 28 days, is already adequate for some concrete applications in practice.

## 5. Conclusions

In this investigation, the aim was to assess the influence of different optimization methods on the performance of mortars with fRCA. The following conclusions can be drawn based on the experimental findings:

(1) The flow, air content and strength of mortars largely depends on the origin, recycling process and storage of fRCA and therefore, on the key properties of the fRCA, such as surface state (presence of impurities) and degree of agglomeration. fRCA B was the least agglomerated and mortars with fRCA B performed better than with fRCA C and D.
(2) The replacement of river sand by 25 wt% of fRCA needs addition of SP. The use of SP at a dosage of 0.3 % by cement weight largely helped to achieve consistent mixtures and adequate workability. This could be also achieved with additional cement content, however, this was not done for sustainability and costs reasons. The use of 50 wt% fRCA in mortar tends to make the mixture stiff and SP cannot improve alone the flow. Therefore, increase of cement content was necessary in mortars with 50 wt% fRCA.
(3) This study has shown that the air content is the key quality indicator for performance of mortars with fRCA. Compared to river sand, as received, uwashed fRCA increase the air content of cement-based mortar mixture, except for fRCA B. The partial removal of 0–0.250 mm subfraction did not significantly reduce the air content in mortars. It is believed that the air content increased due to agglomeration of fine particles (in as received sand) and due to quality of the mixing water (due to presence of impurities from the unwashed fRCA). It should be noted here that the quality of the water is good; only when fRCA are mixed with water, pollutants will dissolve in the mixing water.
(4) The research has also shown that the large air content can be reduced by drying of fRCA or by modified mixing sequence (in this case fRCA would be added last to the fresh mortar mix). It is assumed that drying reduces agglomeration effect and removes partially surface active agents allowing SP to be adsorbed on cement particles. The modified mixing sequence also allowed SP to be adsorbed on the cement particles by adding fRCA in the end of the mixing procedure.
(5) Compressive strength was maintained or even higher compared to the reference mortar, when SP was used in the mixes with 25 wt% fRCA. The strength was reduced with increasing percentage replacement (50 wt%) of river sand with fRCA regardless of their type (B, C, D). The strength of mortar is influenced by the proportion of particles originated from good concrete quality and less good concrete quality (e.g. particles from poorly compacted concrete, high water-to-cement ratio). Increasing the replacement percentage of natural sand by fRCA, the possibility of introducing more 'weaker' particles from fRCA increases.
(6) The presence of fRCA and the old adhered mortar did not alter the reaction of the cement in mortars. The cumulative heat of mortars with fRCA was similar to reference mortar mix. The positive side of this is that the fRCA can be truly considered as non-reactive.
(7) Not only advanced solutions for recycling technology are required, but much more is needed such as improvements within concrete recycling facilities to advance quality of fRCA. An important practical implication of this study is that no storage or storage of fRCA in dried condition after production may substantially reduce their tendency to agglomerate. The longer the storage of fRCA is, the larger the agglomeration impact would be.
(8) Procedures for ensuring that the fRCA taken from the stockpile have not severely agglomerated and deteriorated in such a way that its conformity is compromised, should be specified in the future. In addition, knowing the origin of fRCA should help to improve predictions of the impact of their use on the properties of new concrete by separating stockpiles of fRCA based on the properties of parent concrete. This would be a fruitful area for further work.
(9) Follow up study is designed to scale up the mortar mix design to concrete using the same amount of SP and modified mixing sequence and design concrete mixtures with both coarse and fine recycled concrete aggregates.

## CRediT authorship contribution statement

**Marija Nedeljković:** Conceptualization, Investigation, Methodology, Visualization, Writing – original draft. **Akis Mylonas:** Investigation, Methodology, Writing – review & editing. **Virginie Wiktor:** Conceptualization, Investigation, Methodology, Supervision, Writing – review & editing. **Erik Schlangen:** Funding acquisition, Writing – review & editing. **Jeanette Visser:** Conceptualization, Methodology, Supervision, Writing – review & editing.

## Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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