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# Investigating the potential of electrostatic charging to separate cementitious binder and sand

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#### ABSTRACT

The environmental footprint of concrete is largely influenced by the binder. It is therefore of high interest to investigate the potential reuse of the binder retrieved by modern separation techniques. However, studies found that the recycled cement fraction (RCF) still contained a certain amount of siliceous concrete aggregates, which forms an obstacle in the upcycling of RCF. In this study, the potential of electrostatic separation as a method to separate cementitious binder (hydrated and unhydrated) and sand (silica) is evaluated. Different cementitious powders and silica (sand) were prepared, resulting in a total of 9 powders and 8 mixtures. The mixtures consisted of a combination of silica and one of the cementitious powders (50/50 wt%) with a particle size of the components <125 µm. The potential of the studied technique was evaluated through charging measurements and x-ray fluorescence (XRF). Silica was assumed to contain no CaO and the detected CaO was therefore assigned to the cementitious powders. Results showed that silica and silica-rich fly ash (FA) particles became negatively charged, blast furnace slag (BFS) particles remained largely charge neutral and all other cementitious particles obtained a positive charge. Through electrostatic separation an enrichment of the cementitious binder fraction for all mixtures was obtained at the negative electrode. FA-Silica achieved the highest enrichment (89.9%), CEM III/B-Silica the lowest (4.7%) and the hydrates were enriched ranging from 28.0 to 31.8%.

#### 1. Introduction

The reduction of greenhouse gas emissions is an important topic in the concrete industry. The clinker content of cement in concrete significantly determines the environmental (including  $CO_2$ ) footprint of concrete. Consequently, several studies [1–4] have explored the possibility of retrieving and reusing the cement fraction during the concrete recycling process with the potential to lower its environmental footprint. Although the results were promising, it was found that the presence of fine siliceous powder originating from sand reduced the quality of the cementitious powder fraction. Kwon et al. [2] found that a higher siliceous aggregate content in the fine fraction decreased the flowability of derived concrete mixtures. Moreover, Carriço et al. [5] mentioned that the presence of siliceous aggregates in thermally upcycled cementitious materials lowers the compressive strength of resulting concrete due to a decrease in rehydratable phases, which is also related to the lower effective binder content in the recycled cement fraction (RCF). Therefore,

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methods to separate siliceous aggregate-derived powder from cementitious powder are required to improve the quality of the RCF [2,5, 6].

Various separation methods have been discussed in literature, but they mostly focus on removing attached mortar from the aggregates [7–10]. Most methods, such as acid, and electrical treatment, are not preferable. Acid treatments completely dissolve the clinker, making it impossible to use in new concrete mixtures [7]. Methods using water as medium, such as electrical treatment [11,12] and liquid gravity separators [13], lead to hydration of any unreacted clinker particles present in the concrete. Preventing this is necessary to retain the possible cementing properties of unreacted particles [14]. Both mechanical [8] and thermal treatment combined with mechanical treatment [15] can remove attached mortar, but the resulting fines contain both binder related powders and siliceous aggregate. Therefore, additional treatment is required to further separate these powders. Magnetic separation [6] showed potential for particles larger than 125  $\mu$ m, but not for smaller particles, which are particularly of interest for reducing the environmental impact. Therefore, a method that is able to separate particles below 125  $\mu$ m is still required.

The method evaluated in this paper is based on the separation of particles by an electrostatic separator. This technique can be used for the separation of particles when the separation based on size and/or density alone is not effective [16]. Electrostatic separation relies on differences in triboelectric charging behaviour of particles. Triboelectric charging is highly surface sensitive, because charging of the particles occurs at its surface [17,18]. This mechanism works as follows: during triboelectric charging, particles acquire charge through frictional contact with the charging wall and may also charge upon collision with other particles. Electron transfer between the charging wall and the particles results in an equal but opposite charge (negative or positive). The polarity and charge value of a particle depend on factors such as the minimum amount of energy needed to remove an electron from the material (surface work function), the surface composition and defects, particle size and more. After charging, the particles enter an electric field generated by two electrostatic force, the gravitational force and the drag force induced by the air flow. The electrostatic force needs to be strong enough for a particle to move to one of the electrodes. The deflection changes, based on their polarity (negative or positive) and the magnitude of their charge and mass. Other factors, such as air flow, also affect particle deflection in the electric field [17–20]. A list of factors influencing the electrostatic separation process is mentioned in Ref. [17].

Electrostatic separation is used in various industries, such as the food (primarily in research), polymer recycling and the mineral processing industry. An example of a successful application of electrostatic separation is in fly ash treatment. In this application, carbon particles are separated from glassy aluminosilicate particles in fly ash, allowing the carbon depleted fly ash to be used as a partial clinker replacement in cement [17,21]. However, in the concrete recycling industry, electrostatic separation has not yet been applied as a method for improving the quality of the cement sized particle fraction of recycled concrete fines. Concrete fines obtained from concrete crushing and recycling techniques often contain silica particles stemming from the siliceous concrete aggregates. This lowers the cementitious content, which results in less reduction of the carbon footprint (less clinker replacement), a reduced flowability of the resulting concrete mix, a lower quantity of rehydratable phases, and the presence of silica may negatively affect the retrieval of cementitious properties during upcycling treatments [2,5,22]. Therefore, increasing the cementitious content, also referred to as enrichment, is required. To enhance the quality of this fine fraction towards an enriched cementitious fraction (hydrated and unhydrated), an effective separation of the respective siliceous and cementitious powders is therefore necessary [2,5,6]. The envisioned separation technique described in this research could potentially produce high quality material from the original fine fraction, resulting in clean sand that can directly replace primary sand. Additionally, it may allow the separation of hydrated from unhydrated cementitious material, in which the former can potentially be upcycled to new binder, while the latter can directly be used in the production of new concrete.

Improving the quality of the cementitious fraction by separating the sand from the cementitious powders is important for maximizing environmental reduction. Thus, this research aims to evaluate the potential of electrostatic separation as a method for enriching the cementitious binder fraction and reducing the presence of siliceous aggregate fines. To do this, a set of defined powders, based on the raw materials that may be present in concrete, is used. First, the charging behaviour of different cementitious and siliceous (sand) powders is analysed. Then, the potential of these differences in charging behaviour to effectively separate cementitious from siliceous powders in powder mixtures is assessed.

#### 2. Materials and methods

#### 2.1. Powders

The materials analysed in this study are referred to as powders. Nine different powders (three hydrated and six unhydrated) were investigated. The powders were selected based on the composition of three commonly used cement types; Portland cement, Portland



Fig. 1. Schematic drawing of triboelectric charging via particle – wall interaction and subsequent particle separation in an electric field. Adapted from Ref. [17].

fly ash cement, and blast furnace slag cement. The nine powders investigated were: 1. Ordinary Portland Cement 52.5 R (CEM I), 2. Silica (derived from river sand), 3. blast furnace slag (BFS), 4. fly ash (FA), 5. Portland – Fly ash cement (CEM II/B-V), 6. Blast furnace slag cement (CEM III/B), 7. Ordinary Portland Cement 52.5 R hydrate (CEM I hydrate), 8. Portland – Fly ash cement hydrate (CEM II/B +V hydrate) and 9. Blast furnace slag cement hydrate (CEM III/B hydrate). For the preparation of CEM II/B-V and CEM III/B, CEM I was blended with FA or BFS in quantities of 30% FA and 70% BFS, respectively. To make the hydrated material, powder hydrates were made by mixing the binders with water, using a water-binder ratio of 0.43, which results in full hydration [23,24]. The powder hydrate pastes were cast (without sand) in prism moulds before being sealed in plastic and stored in an oven at 35 °C for three weeks to allow full hydrated powders were obtained by milling and grinding the samples with a Retsch ultra centrifugal mill ZM 200, and the silica powders were prepared by grinding sand separately in a Retsch mortar grinder RM200. Powders passing a 125 µm sieve (upper limit based on a grading curve of OPC) were collected and stored in closed jars until testing. Table 1 provides an overview of the powder combinations used in this study.

#### 2.2. Powder mixtures

The separation of the cementitious and siliceous (sand) powders in the recycled concrete fines is an important step for enhancing the quality of this recycled material [2,5,6]. Therefore, mixtures of powders were prepared to assess the separation efficiency of the cementitious and siliceous powders. These mixtures were made by taking the initial powders mentioned in Table 1 and mixing them in a ratio of 50% silica- and 50% cementitious powders. For this the assumption is made that, with the development of innovative concrete recycling techniques, the concrete components are fully separated from each other. An overview of the powder mixture combinations can be found in Table 2.

#### 2.3. Electrostatic separation

A custom bench-scale electrostatic separator [20] (Wageningen University & Research – Laboratory of Food Process Engineering) was used for the charging and separation experiments (see Fig. 2 for an image of the device). The separator consists of several parts: From top to bottom, these are the screw-feeding system, charging tube, chamber in which the separation occurs, and collecting bins. Two electrodes are installed in the separation chamber, with collectors attached to their lower parts. During the experiments, the collecting bins captured powders not collected by the electrode collectors. Nitrogen gas is used in the system as a carrier gas for the particles moving downwards through the charging tube. The charging tube consists of aluminium tubes with a length of 29.6 cm and an inner diameter of 0.8 cm. Particles were charged as they passed through it. After charging of the particles, the nitrogen carried the particles to the separation chamber. To create an electric field between the two electrodes, the voltage was set to 20 kV and the distance between the electrodes was 0.1 m. Depending on their charge, particles were attracted to either the left (ground in this study referred to as negative) or right (positive) electrode. For each test, 100 g of powder was loaded into the screw-feeding system at the top of the machine. The solids feeding rate was set to 0.5 kg/h and the nitrogen gas flow rate was 50 L/min. Both the voltage and feeding rate were based on the default values used at the testing location [20,25]. Powders collected by the two electrode collectors and the collecting bins were sealed and stored into separate bags until further analysis.

#### 2.4. Charging behaviour

In this study, the charging behaviour refers to the value and polarity of the charge of the different powders. An electrometer attached to the charging tube measured the impact charge obtained by the tube. When the particles collide with the charging tube, both the tube and the particles obtain the same charge but opposite polarities, meaning the measured charge is opposite to the actual charge of the particles. The cumulative charge of the powders is determined during the time needed to process 100 g of material. The specific particle charge, also called charge-to-mass ratio, is calculated by dividing the cumulative charge by the total processed weight of the powder [18].

#### 2.5. X-ray fluorescence

X-Ray Fluorescence (XRF) was used to quantitatively analyse the chemical composition of the powders before and after electrostatic separation. Other techniques were not considered as they provide only qualitative or semi quantitative analysis (XRD). Analysis of the chemical composition is performed to gain insight into the separation efficiency of the electrostatic separator for cementitious

 Table 1

 Overview of the composition of the 9 powder combinations

	· · Fourier communication			
Powders	Silica	CEM I	Blast furnace slag	Fly Ash
Silica	100%	-	_	-
CEM I	_	100%	-	-
CEM II/B-V	_	70%	-	30%
CEM III/B	_	30%	70%	-
CEM I hydrate	_	100%	-	-
CEM II/B-V hydrate	_	70%	-	30%
CEM III/B hydrate	_	30%	70%	-
Blast furnace slag	_	-	100%	-
Fly Ash	_	_	-	100%

#### A.T.M. Alberda van Ekenstein et al.

#### Table 2

Overview of 9 powder and 8	derived powder mixture	(50% silica- and 50%	cementitious powder) combin	nations.
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Powders		Powder mixtures						
	1	2	3	4	5	6	7	8
Silica	х	х	х	х	х	x	x	х
CEM I	х							
CEM II/B-V		x						
CEM III/B			х					
CEM I hydrate				х				
CEM II/B-V hydrate					х			
CEM III/B hydrate						х		
Blast furnace slag							х	
Fly Ash								x



Fig. 2. The electrostatic separator and its main parts as indicated in the figure [20].

powders containing siliceous aggregate (sand) particles. The XRF measurements were performed on the powder samples using a Panalytical Axios Max WD-XRF.

#### 2.6. Separation efficiency

To get more insight in the separation efficiency, the XRF-determined percentages of the main compounds  $SiO_2$  and CaO in the powders are used. It was assumed that the siliceous aggregate powders used in concrete mixtures are composed of  $SiO_2$  and contain no significant amounts of CaO (Table 4). Detected CaO is therefore attributed to the cementitious compounds of the mixtures. Other oxides are not used as they are present in both the cementitious powders and the silica. The cementitious content of the samples in the electrode collectors after separation is determined by comparing the CaO content to the original powder. This is referred to as the cementitious recovery degree [25]. To illustrate: a value of 0% means no cementitious content was observed in the collector after separation, while a value of 100% indicates that the collector contains only cementitious material.

Cementitious recovery degree = 
$$\frac{Compound_{separation} [\%]}{Compound_{pure} [\%]} \times 100\%$$
 (1)

Where Compound<sub>separation</sub> is the CaO content after separation in % and Compound<sub>pure</sub> is the CaO content of the initial predefined powders in %. The yield was measured by taking the mass fractions of the materials present in the negative and positive electrode collectors and collecting bins. The amount of unrecovered material was determined by substracting the amount accumulated in the collectors and collecting bin from the total amount of material entering the machine. To compute the amount of material entering the machine the remaining mass of material in the feeder was subtracted from the starting mass (100 g) in the feeder. The yield is calculated by dividing the mass of a recovered fraction by the total mass of the initial mixture entering the machine before separation [25].

$$Yield = \frac{Mass_{fraction} [g]}{Mass_{mix entering machine} [g]} \times 100\%$$
<sup>(2)</sup>

Where Mass<sub>fraction</sub> is the mass fraction at a certain point in the separator in grams and Mass<sub>mix entering the machine</sub> is the total mass of the material entering the machine in grams. The enrichment of the cementitious binder fraction compared to the initial mixture is determined by the increase in the CaO compound after separation over the amount present in the initial mixture [25]. To illustrate: the initial mixtures contained 50% silica- and 50% cementitious powders. If the cementitious recovery degree shows a recovery of 60%, this indicates an increase in the cementitious content compared to the initial mixture of 20% ((60–50)/50)).

$$Cementitious enrichment = \frac{Compound_{separation} [\%] - Compound_{mix} [\%]}{Compound_{mix} [\%]} \times 100\%$$
(3)

Where Compound<sub>separation</sub> is the CaO content after separation in %, Compound<sub>mix</sub> is the CaO content of the powder mixture entering the separator in % and Compound<sub>pure</sub> is the CaO content of the initial predefined powders in %.

#### 3. Results

#### 3.1. Charging behaviour of powders

The triboelectric charging behaviour of the different powders was studied first. In total the charge of nine different powders (Table 1) was measured. Each powder was measured three times and the results were used to determine an average cumulative charging value per 100 g of powder, corresponding to a specific particle charge per gram of powder, also known as charge-to-mass ratio (Table 3).

Observed differences in the charge value and the polarity (Table 3) between the sand (silica) and cementitious powders appeared significant. This study aimed to establish whether a charge difference between virtually pure silica (SiO<sub>2</sub>) powder (sand) and cementitious powders with mixed CaO and SiO<sub>2</sub> ratios could be used to separate these from each other. The results show that silica and silica-rich FA particles acquire a negative charge, while BFS particles remained largely neutral, and all other cementitious particles obtained a positive charge. This indicates that charge differences can potentially be used to separate these particles in powder mixtures. Attention must be paid to CEM III/B and CEM II/B-V. Both materials are mixtures of two powders, namely CEM I and BFS or FA, respectively. The specific charge of these mixtures may therefore be an average of the charges measured for CEM I and BFS or FA particles. For the hydrates this is different, because blended mixtures of hydrated CEM I and FA or BFS are considered a single powder due to the hydration process.

Based on the difference between the specific particle charge of silica and the other materials, the electrostatic separation force can be determined, using the following equation:

Electrostatic separation force = 
$$\frac{|(SC_{mat1} - SC_{mat2})| \times EF}{D}$$
(4)

Where SC is the specific particle charge of the material in C/kg, EF is the electric field in volt and D is the distance between the two electrodes in meters. In the setup used, the distance between the electrode collectors was 0.1 m, and the value of the electrical field was 20 kV. Considering the specific particle charge difference between silica and the three hydrates, a difference ranging from 46.44 to 54.01 nC/g ( $46.44 \cdot 10^{-6} - 54.01 \cdot 10^{-6}$  C/kg) was found. Based on these values, the electrostatic separation force varies between 9.29 and 10.80 N/kg. As mentioned before, electrostatic separation is sometimes referred to when separation based on size and/or density alone is not possible [16]. For a separator based on density, a density separation force can be calculated as follows:

Density separation force 
$$=\frac{|(\rho_{mat1} - \rho_{mat2})| \times g}{\rho_{small}}$$
(5)

Where  $\rho$  is the density of the material in kg/m<sup>3</sup> and g is force of gravity in N/kg. Assuming the density of silica is 2600 kg/m<sup>3</sup> and that of the hydrates is 2000 kg/m<sup>3</sup>, the separation force is (2600 kg/m<sup>3</sup> – 2000 kg/m<sup>3</sup>) x 9.81 N/kg/2000 kg/m<sup>3</sup> = 2.94 N/kg. Comparing the range of the electrostatic separation force to the separation force of a density separator, the potential of electrostatic separation is clear. The different values indicate that the electrostatic separator generates a 3 times larger separation force than a density separator.

Based on the charging behaviour of the powders the deflection in an electric field was estimated. For this all factors such as particle diameter, distance to electrodes, horizontal and vertical flow velocity, were assumed to be identical, besides the density and the obtained charge of the powders. Additionally, a laminar flow (Reynolds number <2100) [26] was assumed. Fig. 3 shows the potential deflection of the different materials in the applied electric field. Powders with a higher charge value exhibit a larger horizontal travel distance and oppositely charged particles move in opposite directions. Fig. 3 also shows this opposite movement for positively and negatively charged particles.

Based on their polarity, the considered powders can be divided into five charge categories: high negative-, moderate negative-, neutral-, moderate positive- and high positive polarity. Fig. 4 provides an overview of the average charge of the considered powders

Table 3

Overview of the measured	l specific particl	e charges of the	e investigated powders
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Material	Specific particle charge [nC/g]
Silica	-28.43
CEM I	+3.427
CEM II/B-V	+2.290
CEM III/B	+1.480
CEM I hydrate	+25.58
CEM II/B-V hydrate	+18.01
CEM III/B hydrate	+20.35
Blast furnace slag	+0.294
Fly Ash	-14.70



Fig. 3. Schematic overview of the potential deflections of siliceous and cementitious particles in the applied electric field with a voltage of 20 kV and a distance between the electrodes of 100 mm.

and their relative position in these categories, corresponding with their horizontal travel distances in an electric field (Fig. 3). As mentioned before, a higher charge results in a larger travel distance. The powders in the high charge categories travel the furthest horizontally, while BFS, with its neutral charge, has the smallest travel distance. The moderate charge categories contain the powders with a travel distance that lies between the high and small travel distance.

The custom bench-scale electrostatic separator used in this study has two collectors for electrically charged particles. Based on the polarity, charge value and travel distance the unreacted and hydrated cementitious binder is expected to separate from the silica due to their opposite polarity and travel distance. This is also the case for BFS, but due to its almost neutral polarity the expectation is that most of the BFS falls into the collecting bins instead of the negative electrode collector. FA on the other hand, has the same polarity as silica and is therefore expected to move in the same direction, indicating that silica and FA are potentially difficult to separate in the current setup.

#### 3.2. Determination of efficiency of electrostatic separation of cementitious binder and siliceous powder mixtures through XRF analysis

To establish the baseline separation for the powder mixtures mentioned in Table 2, the elemental composition of the starting mixture and the powders accumulated in the two electrode collectors was determined. The change in  $SiO_2$  and CaO content, in which an increase in CaO content indicates an enrichment of the cementitious fraction, whereas an increase in  $SiO_2$  content refers to a fraction enriched with sand, were of particular interest. An overview of the obtained elemental compositions is shown in Table 4.

In total eight powder mixtures were analysed. The negative and positive electrode refer to the electrode collectors where powders accumulate after separation. From Table 4 it follows that powders retrieved from the negative electrode collectors show an increase in CaO and a decrease in SiO<sub>2</sub> content, while the opposite is true for the positive electrode collector. This means that enrichment of the cementitious fraction occurs at the negative electrode collector, whereas enrichment of the sand fraction occurs at the positive electrode collector. Fig. 5 shows a diagram of the CEM I hydrate and CEM I mixtures in which the change in CaO and SiO<sub>2</sub> is visualized.

Besides changes in the SiO<sub>2</sub> and CaO content, other elements also indicate an enrichment in the cementitious fractions at the negative electrode collector. Most of the elements shown in Table 4 are enriched in the cementitious fractions. For all the powders at the negative electrode collector an increase in  $Al_2O_3$  was observed and also MgO,  $Fe_2O_3$  and  $SO_3$  showed an increase in most of the cases. For the other elements, especially those present in smaller quantities, the deviations appeared insignificant. Specifically, in the FA samples,  $TiO_2$  and  $P_2O_5$  were present in larger quantities and enrichment of both compounds at the negative electrode collector was observed.

The increase in CaO content at the negative electrode collector as well as the increase of the amounts of other elements, show that electrostatic charging has potential to separate the cementitious binder and sand.

#### 3.3. Separation efficiency

Fig. 6 presents an overview of the cementitious content, yield and enrichment of the enriched fractions obtained after separation (negative electrode). The cementitious content is based on the presence of CaO in the material. The content in the mixtures after separation was estimated by comparing the CaO content to the CaO content of the considered powder combinations, which is seen as the value reached for full separation.



Fig. 4. Overview of the positioning of the charging behaviour of powders in different charge categories.

#### A.T.M. Alberda van Ekenstein et al.

#### Table 4

Overview of the elemental composition of the 9 powders and the different powder mixtures before (indicated mixture) and after (collected at negative and positive electrode) separation. The  $\Delta$  shows the difference between the initial mixture and the negative and positive collector, respectively (+increase; - decrease).

	$SiO_2$	CaO	$Al_2O_3$	MgO	Fe <sub>2</sub> O <sub>3</sub>	$SO_3$	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	$P_2O_5$
Silica	94.9	0.2	2.6	0.3	0.9	0.0	0.5	0.5	0.1	0.0
CEM I hydrate	19.8	66.4	5.5	1.1	2.7	2.4	0.7	0.2	0.4	0.1
CEM I	19.8	67.1	4.3	1.2	2.6	3.1	0.8	0.0	0.3	0.1
CEM II/B-V	29.1	48.6	10.9	1.3	4.4	2.6	1.0	0.6	0.6	0.4
CEM II/B-V hydrate	28.0	51.4	9.8	1.4	4.5	2.0	0.9	0.5	0.7	0.3
CEM III/B	28.8	47.6	11.3	7.0	0.9	2.0	0.6	0.5	0.8	0.0
CEM III/B hydrate	27.0	51.6	9.8	5.4	1.3	1.9	0.6	0.5	1.1	0.1
BFS	32.6	40.6	13.5	9.0	0.3	1.6	0.5	0.6	0.9	0.0
FA	56.7	4.2	25.1	2.1	5.7	0.7	1.4	1.5	1.1	1.1
CEM I hydrate - Silica mixture	58.5	32.3	3.9	0.6	1.7	1.5	0.6	0.3	0.2	0.1
CEM I hydrate - Silica negative electrode	46.9	42.6	4.3	0.8	2.1	1.7	0.7	0.1	0.2	0.1
CEM I hydrate - Silica positive electrode	65.1	25.7	3.9	0.6	1.7	1.2	0.6	0.5	0.2	0.1
$\Delta$ Mixture and negative electrode	-11.6	10.3	0.5	0.2	0.5	0.2	0.0	-0.2	0.0	0.0
$\Delta$ Mixture and positive electrode	6.7	-6.6	0.1	0.0	0.0	-0.3	0.0	0.2	-0.1	0.0
CEM I - Silica mixture	51.7	37.5	4.6	0.8	1.9	2.2	0.7	0.0	0.2	0.1
CEM I - Silica negative electrode	40.9	46.9	5.0	0.9	2.2	2.1	0.7	0.5	0.3	0.1
CEM I - Silica positive electrode	55.3	33.8	4.4	0.7	1.8	2.1	0.7	0.4	0.3	0.1
$\Delta$ Mixture and negative electrode	-10.7	9.3	0.4	0.1	0.3	-0.1	0.0	0.5	0.1	0.0
$\Delta$ Mixture and positive electrode	3.7	-3.7	-0.2	0.0	-0.1	-0.1	0.0	0.4	0.1	0.0
CEM II /P. V. Silico minturo	EQ /	27.0	7.2	0.0	2.5	1.7	0.0	0.5	0.2	0.2
CEM II/B-V - Silica negative electrode	43.0	27.0	7.5	0.0	2.3	1./	0.8	0.5	0.5	0.2
CEM II/B V Silica negative electrode	43.0	11 1	4.4	0.7	1.0	1.0	0.5	0.0	0.5	0.2
A Mixture and possible electrode	15 4	12.1	1.7	0.7	1.0	0.0	0.5	0.4	0.2	0.1
A Mixture and positive electrode	-13.4	15.1	2.0	0.4	1.5	0.0	0.0	-0.5	0.2	0.0
	21.9	-13.9	-2.9	-0.2	-1.5	-0.0	-0.2	-0.1	-0.1	-0.1
CEM II/B-V hydrate - Silica mixture	62.9	24.4	6.3	0.7	2.4	1.1	0.7	0.4	0.4	0.2
CEM II/B-V hydrate - Silica negative electrode	53.8	31.3	7.5	1.0	2.9	1.4	0.7	0.4	0.4	0.2
CEM II/B-V hydrate - Silica positive electrode	69.2	19.2	5.8	0.8	2.0	1.0	0.7	0.3	0.3	0.2
$\Delta$ Mixture and negative electrode	-9.1	6.8	1.2	0.2	0.6	0.2	0.0	0.0	0.1	0.0
$\Delta$ Mixture and positive electrode	6.3	-5.2	-0.5	0.0	-0.4	-0.1	0.0	-0.1	0.0	0.0
CEM III/B - Silica mixture	54.7	29.4	7.4	4.1	1.0	1.4	0.6	0.4	0.6	0.0
CEM III/B - Silica negative electrode	53.0	30.8	7.8	4.0	1.1	1.5	0.6	0.3	0.5	0.0
CEM III/B - Silica positive electrode	55.7	28.1	7.7	4.4	1.0	1.4	0.5	0.4	0.4	0.0
$\Delta$ Mixture and negative electrode	-1.7	1.4	0.4	0.0	0.1	0.0	0.0	-0.1	-0.1	0.0
$\Delta$ Mixture and positive electrode	1.0	-1.2	0.2	0.3	0.0	0.0	-0.1	0.0	-0.2	0.0
CEM III/B hydrate - Silica mixture	60.9	25.9	6.2	2.8	1.1	1.2	0.6	0.5	0.5	0.1
CEM III/B hydrate - Silica negative electrode	50.8	33.7	7.2	3.6	1.3	1.3	0.6	0.3	0.7	0.1
CEM III/B hydrate - Silica positive electrode	68.4	19.9	5.5	2.4	1.0	1.0	0.5	0.5	0.4	0.1
$\Delta$ Mixture and negative electrode	-10.1	7.8	1.0	0.8	0.2	0.2	0.0	-0.2	0.2	0.0
$\Delta$ Mixture and positive electrode	7.5	-6.1	-0.6	-0.4	-0.1	-0.2	0.0	0.0	-0.1	0.0
BFS - Silica mixture	55.9	25.3	9.2	5.5	0.7	1.1	0.6	0.6	0.8	0.0
BFS - Silica negative electrode	44.8	32.3	11.4	7.1	0.6	1.3	0.5	0.6	1.0	0.0
BFS - Silica positive electrode	75.8	12.7	5.8	3.1	0.5	0.7	0.5	0.3	0.3	0.0
$\Delta$ Mixture and negative electrode	-11.1	7.0	2.2	1.6	-0.1	0.2	0.0	0.0	0.2	0.0
$\Delta$ Mixture and positive electrode	19.9	-12.6	-3.4	-2.3	-0.2	-0.4	-0.1	-0.2	-0.5	0.0
FA - Silica mixture	74.0	2.5	14.4	1.3	3.6	0.4	1.0	0.9	0.7	0.6
FA - Silica negative electrode	62.5	4.8	20.3	1.8	5.5	0.6	1.3	1.0	0.8	0.9
FA - Silica positive electrode	86.1	1.6	7.9	0.8	1.4	0.3	0.7	0.5	0.3	0.4
$\Delta$ Mixture and negative electrode	-11.5	2.3	6.0	0.5	1.8	0.2	0.2	0.1	0.1	0.2
$\Delta$ Mixture and positive electrode	12.1	-0.9	-6.4	-0.5	-2.3	-0.2	-0.4	-0.4	-0.5	-0.3

For the negative electrode collector an increase in the cementitious content was observed for all the mixtures. Enrichment of the cementitious materials therefore occurs at the negative electrode. The hydrates showed an enrichment in a relatively narrow range of 28.0–31.8%, while the unhydrated binders showed a wider range of 4.7–48.4%. The CEM III/B – Silica fraction had the lowest (4.7%) enrichment in cementitious content, whereas CEM II/B-V – Silica showed one of the highest enrichments (48.4%). Only FA – Silica obtained a higher enrichment (89.9%) at the negative electrode. CEM II/B-V hydrate – Silica, on the other hand, only showed an enrichment of 28.0%.

The enriched cementitious fractions reached a cementitious content higher than 60%. However, differences in the content of the recovered material were observed. The enriched fractions containing the hydrates varied in cementitious content between 60 and 66%, while the unhydrated cementitious material mixtures obtained a higher cementitious content, with the exception of the CEM III/B – Silica mixture. Based on the charging behaviour of the powders, FA was expected to move in the same direction as silica, as both obtained the same polarity (i.e. negative charge). BFS was expected to fall straight down and not in one of the electrode collectors due







Fig. 6. Overview of the cementitious content in the enriched cementitious fraction (negative electrode) after separation. The yield of the enriched fraction and the enrichment are also shown.

to its neutral polarity. However, both the FA – Silica and BFS – Silica mixtures show high cementitious content after separation, which was unexpected based on the charge values of the individual powders.

Fig. 7 presents the yield of the mixtures during the electrostatic separation process. The CEM I hydrate, CEM III/B hydrate and CEM II/B-V hydrate – Silica mixtures showed a similar yield in the negative electrode collector (8.42–8.96%). The mixtures based on the unhydrated binders, specifically CEM I, CEM III/B and CEM II/B-V – Silica, achieved a slightly higher yield in the negative electrode collector (9.53–11.48%). Based on the charge value, however, the opposite was expected, because the hydrates obtained a higher specific particle charge.

The cementitious content after separation (negative electrode) and the enrichment demonstrate that separation of cementitious



Fig. 7. Yield for the different mixtures after separation and the amount of unrecovered material. The yield is based on the amount of material entering the machine. Material remaining in the feeder is not taken into account.

binder and sand occurs. In this first separation step 11–20% of the total initial cementitious amount entering the machine was recovered at the negative electrode for the different fractions. To increase this amount the yield, cementitious content and enrichment of the cementitious fraction should be enhanced. Additionally, the powder collected in the collecting bins was not taken into account during this study. In the current experimental setup, part of the powders flows past the electrodes, because they do not span the entire width of the equipment, ending up in the collecting bins together with the non-separated and neutrally charged material. The powder collected from the electrodes are therefore more enriched or depleted in specific components than those collected from the collecting bins. For future improved and scaled-up equipment designs, a similiar enrichment to that of the powders at the electrodes is expected, hence only the powders collected from the electrodes were analysed. Moreover, it has been demonstrated for different materials that higher yields for the electrodes can be achieved by recirculating the materials from the collecting bins [27].

#### 4. Discussion

#### 4.1. Charge behaviour

The charging behaviour of the powders provides an initial indication of the possibility of using electrostatic charging for the separation of cementitious binder and sand. Materials can be ranked according to the polarity they obtain during friction, i.e. a triboelectric series [28]. In such a series, quartz is often at the very negative end, generally resulting in a negative charge. Since most minerals are more positively charged, separation of quartz and other minerals can potentially be achieved [28]. Silica consists mainly of quartz, leading to its negative polarity. Quartz is also present in fly ash, but in lower amounts than in silica, resulting in a lower negative polarity. Tanaka et al. [29] measured the electric charge of Portland cement and its main minerals (tricalcium silicate, dicalcium silicate, tricalcium aluminate, tetracalcium aluminoferrite) and found that they had a positive charge. Similar polarity (positive charge for cement) was also observed in this study.

Currently, the custom bench-scale electrostatic separator has only two collectors for electrically charged particles. With further development by specifically considering the powder charge values, it should be possible to obtain at least five fractions related to the five indicated categories. The five collectors would specifically, and respectively going from negative to positively charged particles, collect enriched sand, FA, BFS, unreacted cementitious binder and hydrated cementitious binder. However, the separation experiments showed that FA and BFS were also present at the negative electrode. Therefore, other factors besides the polarity and charge value must be considered. Further research is therefore needed to determine the number of collectors. Additionally, changes in parameters such as the distance between the electrodes and the strength of the electric field [17], may further increase the horizontal separation distance and thus potentially improve the separation process.

#### 4.2. Separation optimization of sand and cementitious binder

Based on the charging behaviour of the 9 powder combinations, FA and silica were expected to be difficult to separate. However, the results showed that FA - Silica obtained the highest enrichment at the negative electrode. This indicates that not only is the polarity important for the separation, but other factors also have to be taken into account. This also applies to BFS-silica. Because BFS obtained a largely neutral charge, most of the BFS powder was expected to fall in the collecting bins instead of the electrode collectors, which would result in a lower enrichment. Nevertheless, after the separation experiments the BFS-silica mixture obtained a higher enrichment than the CEM I – Silica mixture, despite CEM I obtaining a positive charge. As mentioned before, CEM II/B-V – Silica and CEM III/ B - Silica mixtures are powder mixtures containing CEM I, sand and either FA or BFS. CEM III/B - Silica obtained the lowest enrichment, whereas CEM II/B-V - Silica obtained the second highest enrichment. For unreacted powder mixtures, an increase in the FA content seems to result in a larger enrichment for the current equipment design. For the hydrated powders, the hydration reaction leads to the formation of hydration products, which physically connects the CEM I with FA or BFS and alters the particles. The hydrates are therefore seen as one powder or conglomerate instead of a mixture of multiple separate powders. A difference in the enrichment between the unreacted and hydrated mixtures is observed. The unreacted mixtures have a wider range and the hydrated mixtures are comparable. This again indicates that other factors besides the polarity influence the separation process. To further improve the separation of cementitious binder and sand more research is therefore needed. A number of factors influence the separation process. Mirkowska et al. [17] listed several factors to take into account with regard to electrostatic separation. They state that the factors can be divided into four categories.

- Design of the equipment
- Environmental influences
- Material characteristics
- Powder particle characteristics

#### 4.2.1. Design of the equipment and environmental influences

As stated in section 4.1, development of the equipment might make it possible to obtain multiple separated fractions, but to optimize the current separation process a closer look at the design is of interest. Manouchehri et al. [30] found that changing the charging wall materials influenced the charging behaviour of the materials. PVC resulted in a positive charge of quartz, while other charge wall materials, such as aluminium, steel, nickel and copper, led to a negative polarity. Besides charging wall material, other parameters of interest include the feeding rate, type of electrode and charging unit, time between charging and separation, distance between electrodes, strength of the electric field, etc [17,31]. Decreasing the distance between the electrodes and increasing the strength of the electric field results in higher electrostatic separation forces and therefore might increase the separation. In the current

setup, nitrogen gas is used as a carrier gas, but air could be used instead. The flow rate of the gas influences the velocity in the charging tube. Wang et al. [26] found that an increase in the gas flow rate positively contributes to the charging process. However, the velocity between the electrodes should not be too high or too low, since the particles should be able to deposit on the electrodes. This is also related to the distance between the electrodes. Research [26] found that a larger distance had a negative effect on the separation efficiency due to a reduced gas flow and increased residence time of the particles. Sufficient cleaning of the electrodes to avoid an accumulation of charge that might result in a shielding effect is of interest, as this can prevent particles from depositing on the electrode. Changes in the design of the equipment may therefore positively influence the separation process.

The humidity of the air flowing within the equipment influences the conductivity of the particles. Moisture presence is said to decrease the yield, but on the other hand is important for triboelectrification [17,28,31]. The influence of moisture on the separation process is complex. For moisture, the temperature is also of importance and related to the humidity. An increase in temperature results in a decrease of relative humidity, which could improve the charging behaviour. The origin and history of the material is of importance as materials in high humidity environments might require pre-drying to improve the separation process [28]. This is particularly relevant for the cementitious hydrates. The lower yield of the hydrated mixtures containing the hydrated powders may be related to the presence of water in the hydrates. In industrial operations, pre-drying of the materials is not preferred due to increased energy use and environmental impact. Therefore, the choice was made in this research not to dry the hydrated materials before electrostatic separation. Moisture can influence the surface conductivity of the particles and the distribution of the charge on their surface [17,32]. Wang et al. [18] studied the influence of the relative humidity on the charging of spherical polystyrene particles and wheat gluten. No effect of the humidity on the charging was detected. Rowley and Mackin [33] on the other hand, found a decrease in particle charge for sodium starch glycolate particles with increased moisture content. Differences between the observed effect of the humidity on the charging of the surface properties of the materials [18]. Further research should therefore include the effect of humidity on the electrostatic separation potential related to the surface properties of the materials.

#### 4.2.2. Material and powder particle characteristics

Electrostatic separation is a surface sensitive technique. Differences in preparation of the materials can result in charge variations. Surface roughness, contamination, orientation of surface planes and impurities influence the type of contact and charge transport [17]. Material treatment before separation might therefore be of importance. The cementitious hydrates in this study were milled to the preferred particle size. Other size reduction techniques or treatments may increase the separation efficiency. Heating or drying of the materials to remove the water present may also be of importance. Other treatments, such as surface doping, cleaning or the addition of chemicals could influence the charging behaviour [17,31,34]. Wen and Chung [35] found that the addition of different admixtures can either increase or decrease the dielectric constant of cement paste. Particle size also affects the process. Smaller particles have a relatively larger surface area, leading to more particle-wall contact and resulting in a higher cumulative charge [17,18,34]. When reducing the particle size from  $<125 \,\mu$ m to  $<63 \,\mu$ m, the separation may be optimized as the cumulative charge is expected to increase. However, influences from for example air flow may have a negative effect on the separation as the particle size decreases. In the case of powder mixtures, particle – particle interactions may also affect the charging, leading to different charges than those observed when only considering particle - wall contact [20]. This can influence the observed separation efficiencies and predictability based on charging behaviour, especially for composite materials and mixtures. Therefore, it is advised to perform both charging behaviour and separation experiments. However, results obtained through the separation experiments are assumed to give a good indication of the separation potential. Another important aspect is that individual particles should be freely dispersed in the gas phase, as agglomerated particles cannot be efficiently separated [17,28]. Other factors influencing the separation efficiency include e.g. travel distance, the chemical composition, crystallographic structure, particle shape, and charge distribution on the surface [17]. Regarding the crystallographic structure, both the unhydrated CEM II/B-V - Silica and CEM III/B - Silica mixtures can be seen as powder mixtures composed of three major components. This is different for the hydrated versions of these specific mixtures. Although the elemental composition of these mixtures is comparable, the hydration reactions result in differences between the crystal structures of the hydrated and non-hydrated mixtures. Comparing the enrichment of the CEM II/B-V - Silica, FA - Silica and CEM II/B-V hydrate - Silica mixtures this is also of interest. The difference between these three mixtures might be due to the presence of unreacted FA in the first two. FA and sand in a mixture may interact in such a way that it increases the separation efficiency. Adding CEM I to the mixture (CEM II/B-V – Silica) appears to maintain this effect. In the hydrated version, CEM I and FA are no longer separate powders within the mixture. The hydration reaction changed the crystal structure and combined the CEM I and FA in new reaction products, which may influence the separation process. Additionally, the approach used to produce the hydrated fractions (in this study milling of the samples) influences characteristics such as particle size distribution and surface roughness [17]. The lower enrichment for CEM III/B -Silica might also be related to it being a mixture of three unreacted materials and the interaction of these materials in the separation process (particle – particle interaction). The unexpected enrichment of BFS – Silica may also be due to the interaction between the particles, which possibly increase the separability of both materials. The differences in the results between CEM I and the CEM I hydrates may be related to the presence of water, the pretreatment method, changes in the crystal structure due to the hydration reaction or other previously mentioned factors.

To further optimize the separation of sand and cementitious binder, all the above considered factors should be investigated in for example a parametric study. This research focused on siliceous aggregates, but other aggregate types, such as limestone aggregates, may also be of interest. Non-siliceous aggregates may lead to different results. In the case of limestone the cementitious content after separation may be lower due to its high CaO content. However, it remains uncertain if this results in a lower quality recycled concrete fines. Depending on the chosen upcycling method, separation may not be necessary for high calcium aggregates such as limestone. Therefore, when the recycled fines are used as raw material in the kiln, a separation for this type of aggregate may not be required.

Additionally, materials accumulated in the collecting bins might be analysed to determine if enrichment of cementitious binder occurred in one of the bins. For this study, it was assumed that the innovative recycling techniques are able to fully separate the concrete components (gravel, sand, cementitious binder). However, in practice, some of the fine sand and cementitious particles may still be attached and additional pretreatment might be required. This research studies the potential after one separation cycle, which is considered the base line of the separation. Further research might also include multiple electrostatic separation steps, as these are expected to improve the efficiency [25], and key parameters for the separation optimization.

#### 5. Conclusion and future research

This study investigated the fundamental question of whether it is possible to separate different cementitious components that can be used in concrete from sand using electrostatic charging with the goal to reduce the environmental impact of concrete. The charging behaviour of the individual components was determined and differences in polarity and charge magnitude theoretically suggest that the cementitious powders can be separated from sand. Several separation experiments were conducted to test this hypothesis. The main findings of this research are.

- Sand (silica) obtained a negative polarity, while the cementitious powders obtained a positive polarity, except for BFS (neutral) and FA (negative). The negative charge value of sand was twice as high as that of FA.
- Mixtures of cementitious material and sand showed an enrichment of the cementitious content in the negative electrode collector. FA – Silica achieved the highest enrichment (89.9%) and CEM III/B – Silica the lowest enrichment (4.7%). An enrichment in the range of 28.0–31.8% was achieved for the hydrates.
- Among the hydrated cementitious powders, CEM III/B hydrate yielded the highest separation efficiency, followed by CEM I hydrate and the lowest efficiency was observed for CEM II/B-V hydrate. For the unhydrated powders, the opposite was observed: CEM II/B-V had the highest separation efficiency, followed by CEM I and CEM III/B. Of all the eight mixtures the FA-mixture obtained the highest separation efficiency.

Based on the results, electrostatic charging shows potential for the separation of sand from cementitious binder. However, more research is required to improve the separation process and efficiency. Future research should include topics related to the equipment design, environmental influences, material and powder particle characteristics. Potential recommendations for future research include:

- The enriched cementitious fractions contained less than 40% silica. To further increase the quality of the enriched cementitious fractions that still contain silica, further improvement of the electrostatic separation system used in this research is needed. Further development of the design could allow separation into different collectors, potentially resulting in five fractions, namely sand, FA, BFS, unreacted cementitious binder and hydrated cementitious binder
- In total 11–20% of the initial amount of the cementitious materials entering the separator was retrieved. Multiple separation steps may increase the recovered amount and the cementitious content of the enriched cementitious fractions.
- Materials accumulated in the collectors bins (not effectively separated) were not characterized in this study. Follow up research should characterize these materials to determine the level of enrichment in the bins and the potential for further process steps.
- Numerous factors influence the electrostatic separation process. More research is needed to investigate how variations in these factors could lead to further optimization of the separation process and to what extent the parameters influence the separation efficiency. Expected parameters of interest are:
  - o Variations in chemical and mineralogical composition, potentially including other cementitious materials and different substitution levels for BFS and FA.
  - o Interaction between powders in composite materials within the separation setup. The triboelectric charging phenomena during electrostatic separation are not yet fully understood for composite materials and mixtures, which requires further research. Combination of different powder materials may influence the separation positively or negatively. This is of interest especially in the case of residual cementitious powders from the concrete recycling.
  - o Moisture content, which may influence the separation efficiency. Since the temperature may directly affect the presence of moisture, it should be included in further studies.
- o Particle size, as smaller particles may result in an improved charging behaviour and increased separation efficiency.
- o Surface treatments, such as doping, cleaning, or the addition of chemicals to improve the charging behaviour. Additionally, insight should be gained into the extent to which the mechanical treatment processes influence the separation efficiency over different production times, as this may affect the surface of the particles.
- To implement this method within industry, a cost analysis is of interest. This should include not only the economic costs, but also the societal and primary material costs, as implementing this innovative separation technique could reduce the need for primary materials and the environmental impact of concrete.

#### CRediT authorship contribution statement

A.T.M. Alberda van Ekenstein: Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Conceptualization. M.A.I. Schutyser: Writing – review & editing, Resources, Conceptualization. M. de Wit: Writing – review & editing, Resources, Investigation. H.M. Jonkers: Writing – review & editing, Supervision, Funding acquisition, Conceptualization. M. Ottelé: Writing – review & editing, Supervision, Funding acquisition, Conceptualization.

#### 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.

#### Data availability

Data will be made available on request.

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