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EVALUATING THE POTENTIAL ALKALI-SILICA REACTIVITY OF MINERAL FILLERS: A PRELIMINARY STUDY

Sinno, Noura[1,2] and Shehata, Medhat[1]
[1] Ryerson University, Canada
[2] noura.sinno@ryerson.ca

Abstract: Mineral fillers have been used in concrete to achieve certain properties such as self-consolidation. Different sources of fillers are present depending on the type of original rock from which the filler is produced. Their potential to cause alkali-silica reaction when used in concrete is a concern. This research focuses on adapting and adopting the current accelerated mortar bar test to evaluate reactivity of mineral fillers. The effect of different levels of fillers on mortars workability is investigated to evaluate the feasibility of introducing them to the mixture without sacrificing its workability. Three mineral fillers are tested: a calcium-carbonate filler, a carbonate filler with 27% SiO_2, and a siliceous filler produced from reactive aggregate. Two replacement methods of sand by filler are adopted where the filler is taken as a percent from the sand finest portion, or from the total graded sand. The results showed that workability of mortars decreases as the filler replacement level increases. In addition, a minor difference in workability was observed between samples obtained from the two replacement methods. As for the expansion due to alkali-silica reaction, when a percent of the total graded reactive sand was replaced by the carbonate filler containing silica, samples showed lower expansion than the control. However, this was not the case for samples where the sand finest portion was replaced. In this case, the expansion was the same as the control. Also, reactive fillers of 150 µm maximum size expanded more than fillers finer than 75 µm which had no expansion.

1 INTRODUCTION

Mineral fillers have been used frequently in concrete because of their beneficial effect specially in flowable mixtures such as self-consolidating concrete (SCC). SCC is one type of high performance concrete which is known for its highly flowable characteristics and its ability to be placed in formworks with minimal or no vibration (Khayat 1999). Although SCC is known for its high workability, it is necessary to balance between deformability and stability of the mix. To achieve this balance, powder materials are utilized in SCC to maintain stability and cohesiveness of the concrete mix (Uysal 2012). These additives can be either pozzolanic materials such as fly ash and slag or mineral fillers like limestone powder for example. The high amount of fillers added will decrease the water content in concrete leading to a denser material with less porosity (Uysal 2012). In addition, the amount of viscosity enhancing chemical admixtures required during the mix will be reduced or eliminated due to the addition of fillers instead. The use of mineral additives is cost effective and allows enhancement of the concrete's stability. Furthermore, it was found that mineral fillers have the potential to increase the rate of cement hydration due to their higher surface area promoting the reaction (Kjellsen and Lagerblad 1995). The incorporation of limestone filler enhanced the formation of calcium hydroxide at early ages probably due to the formation of nucleation sites allowing the growth of the hydration products (Pedersen 2004).

Each type of filler as well as its replacement level (RL) can affect the properties of the concrete differently. Due to their high surface area, the resistance to bleeding improves with the increase in mineral filler content. Elyamani et al. showed that the use of non-pozzolanic mineral fillers enhanced the resistance to bleeding and segregation in SCC compared to pozzolanic materials (Elyamani et al. 2014). This was more visible for higher replacement levels of fillers. Moreover, the average compressive strength of SCC samples with marble powder was found to be 25% higher than SCC samples without fillers (Alyamac and Ince 2009). Elyamani et al. pointed that the use of non-pozzolanic materials had similar effect on compressive strength compared to that of pozzolanic fillers. The observed performance of mineral fillers is due to the micro-filling ability, improving the microstructure of the matrix and the interfacial transition zone (Elyamani et al. 2014).

Many researches are ongoing in the study of mineral fillers and their effect on mechanical properties as well as durability. Some researchers replaced the cement by mineral fillers particularly in SCC applications (Craeye et al. 2010, Murthy et al. 2012, Turkel and Kademir 2010) as compared to others who considered the replacement should be taken from the aggregate portion (Pedersen 2004). The replacement method of fillers is still under debate in order to understand their effect on concrete's behavior. To study the effect of mineral fillers on alkali-silica reaction in concrete, Pedersen replaced a percent of the aggregates volume by mineral fillers (Pedersen 2004). In addition, he found that some types of mineral fillers can result in deleterious effect on concrete expansion due to the alkali-silica reaction. Some mineral fillers are still a concern due to their potential reactivity.

Alkali-silica reaction is one of the deleterious chemical reactions that can cause expansion and cracking in concrete. The reaction between siliceous aggregate and alkalis present in the cement will lead to the formation of a swelling gel. This gel, with the absorption of moisture, will cause pressure in the concrete. If the swelling pressures exceed that of the concrete tensile strength, cracks will form leading to expansion (Federal Highway Administration 2012, Sinno and Shehata 2016). Furthermore, the cracks will allow the mobility of the chloride ions as well as water which will cause later other durability issues such as reinforcement corrosion and additional cracks due to freezing and thawing. Hence, the importance of testing the alkali reactivity of mineral fillers. Different test methods are present to evaluate reactivity of aggregates which are the concrete prism test (CPT) and the accelerated mortar bar test (AMBT) described in the CSA A23.2-14A and CSA A23.2-25A respectively. Expansion limits of 0.040% and 0.10% are set to evaluate aggregates' reactivity in the CPT and AMBT, respectively. However, there are no current test methods for the evaluation of reactive fillers. Hence, the need for modified tests to avoid deleterious effects in concrete.

Supplementary cementing materials (SCM) are used as a preventive measure to reduce the reaction due to ASR. The use of SCM showed improved performance of concrete against ASR based on results obtained from CPT (Shehata and Thomas 2000, Thomas et al. 2006). Mineral fillers are being used instead of pozzolanic materials in SCC because they are less costly and show better performance. Hence, reactive mineral fillers need to be evaluated first before implementing it. When testing a Norwegian reactive filler, different results were obtained with CPT and AMBT. In CPT test, it showed higher expansion than the control which contradicted the results obtained in AMBT (Pedersen 2004). Also, the particle size distribution of the filler has a great importance on the expansion. A reactive filler of 0-20 µm size exhibited slightly higher expansion compared to fillers with 0-125 µm size with CPT. This might be due to the faster expansion of smaller particles. However, this was not the case with fillers, proven to be pozzolanic, which when tested, gave lower expansion with smaller size samples due to the higher pozzolanic activity (Pederson 2004).

2 RESEARCH OBJECTIVES

In order to get a better understanding of fillers behavior, the aim of this paper is to study the following:

1. Effect of replacement level on workability,
2. Effect of replacement method on expansion due to ASR,
3. Understanding the reactivity of mineral fillers in the AMBT.

Three different mineral fillers will be implemented in this study having different silica content: a calcium-carbonate filler, a carbonate filler with 27% SiO_2, and a siliceous filler produced from reactive aggregate. This will cover a wider range of fillers and will help in understanding the difference in their reactivity. Two

different replacement methods will be investigated which are replacement of mineral filler from total graded sand and from finest portion of the sand. In addition, the different mineral fillers will be tested with reactive and non-reactive aggregates using the accelerated mortar bar test to understand their ASR reactivity.

3 MATERIALS AND EXPERIMENTAL DETAILS

3.1 Materials

3.1.1 Aggregates and Cementing Materials

Spratt (SPR), a highly reactive coarse aggregate, is a siliceous limestone with 9% SiO_2 obtained from the city of Ottawa in Ontario. The bulk relative density of Spratt is 2692 kg/m^3 and its absorption is 0.53%.

Two different fine aggregates are tested during this research: sand with moderate reactivity in AMBT and non-reactive sand. Their fineness moduli are 2.64 and 2.43 respectively.

The cement used in this research is Type GU Portland cement with 0.99% Na_2O_e. Its chemical composition is presented in Table 1.

Table 1: Chemical composition of Type GU cement and carbonate silica filler

Oxide	GU Cement (Mass %)	Carbonate silica filler (Mass %)
SiO_2	19.54	27
Al_2O_3	5.21	1
Fe_2O_3	2.16	0.3
CaO	62.39	42
MgO	2.39	5
SO_3	4.03	-
Na_2O_e	0.99	-
Loss on Ignition	2.36	29

3.1.2 Mineral Fillers

The first mineral filler used, carbonate silica filler, consists of calcium carbonate (66%), quartz (11%) and silicate materials (23%). Its chemical composition is presented in Table 1 above. The median diameter is 40 µm and the particle size distribution is provided in Figure 1-a.

The carbonate filler is a dry ground calcium carbonate with a medium and closely sized particle distribution and excellent shrinkage resistance properties. The calcium carbonate content is 94% and magnesium carbonate content is 2.5%. The median diameter is 21 µm and its particle size distribution is shown in Figure 1-b.

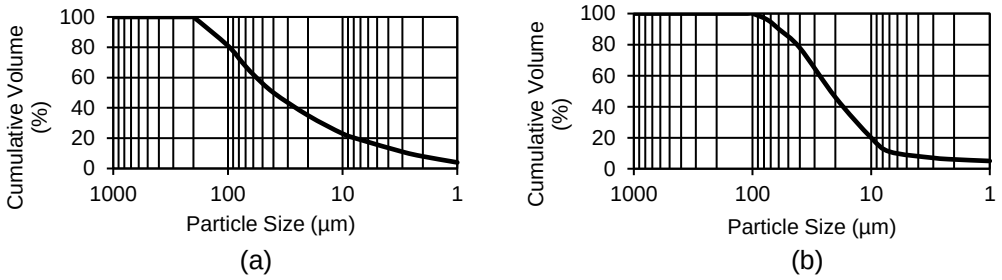

Figure 1: Particle size distribution of (a) the carbonate silica filler and (b) the carbonate filler

Springhill (SH) is an extremely reactive aggregate with an expansion of 0.22% at 1 year when tested using the concrete prism test and 0.46% at 14 days when tested using the accelerated mortar bar test. It is composed of coarse and fine materials. The fine portion was separated from the coarse aggregates. Crushing was completed on each separately to get two mineral fillers: one obtained from the coarse aggregate (SH-C) and the other from the fine portion (SH-F). SH fillers were crushed until all passed 75 µm sieve.

3.2 Experimental Procedure

3.2.1 Sample Preparation

Sieving of Fine Aggregates

The gradation of mortar samples is completed as specified in the accelerated mortar bar test described in ASTM C1260. The aggregates should have specific gradation expressed by mass of the total sample (990g) required to obtain 3 samples for each set as shown in Table 2.

Table 2: Grading Required as specified in ASTM C1260

Passing	Retained on	Mass (%)
4.75 mm	2.36 mm	10
2.36 mm	1.18 mm	25
1.18 mm	600 µm	25
600 µm	300 µm	25
300 µm	150 µm	15

Thus, the moderately reactive and non-reactive sands were sieved until the required gradations are obtained. Then, each portion was washed over its own sieve and dried in the oven.

Crushing of Spratt

For Spratt aggregate, a representative sample of 6 kgs was crushed using the crusher until all the aggregates passed 4.75 mm sieve. The crushing of the 6 kgs of Spratt was enough to obtain the required grading listed in Table 2. Once, the crushing is done, each portion is washed over its own sieve and dried.

Crushing of Springhill

Springhill was used as a mineral filler, obtained from reactive aggregate. Springhill aggregates were separated between fine and coarse to test the reactivity of each. The coarse aggregate was washed over 4.75 mm sieve to remove all the fines and the fine portion was washed over 75 µm to remove all the dust. They were allowed to dry. The coarse SH was crushed using the crusher first and then it was sieved. After sieving, each portion is crushed using the pulverizer until all passed 75 µm sieve to obtain SH-C filler. For the fine sample, the aggregate was sieved and larger size aggregates are crushed with the pulverizer until all passed the designated sieve. Then, the same procedure is applied for the subsequent aggregates retained on smaller size sieves until all passed 75 µm sieve obtaining SH-F filler.

Samples used for the tests

Two different replacement methods are tested in this paper:

1. Replacement of the sand finest portion by filler: for example, for a 10% replacement level, all will be replaced from the smallest size aggregate (passing 300 µm and retained on 150 µm) and only 5% will be remained from this portion of the sand. As for 20% replacement level, all the required gradation for the particles passing 300 µm and retained on 150 µm will be eliminated and 5% extra will be removed from the sand passing 600 µm sieve and retained on 300 µm sieve (Table 2).

2. Replacement of the total graded aggregate by filler: for example, a 10% replacement level will be taken from the total weight of the aggregate sample of 990 g. The rest 891 g will be divided based on the gradation requirements of ASTM C1260 listed in Table 2.

3.2.2 Mixing of Mortars

The mixing procedure followed was based on the requirements described in ASTM C305. The water to cement ratio used is 0.47 for all the samples as specified in ASTM C1260 except for the Spratt control which was 0.5. This sample was tested in a previous research work and results are reported in this paper for comparison. The water is put first in the mixer and then the cement. They were allowed to mix for 30 seconds at low speed. Following this, the aggregate obtained from the gradation requirements explained previously and the mineral filler are added to the paste over a period of 30 seconds while mixing. After the 30 seconds had elapsed, the mixer speed was changed to medium and allowed to mix for 30 seconds. This is followed by a 90 seconds of rest period and then mixing for another 90 seconds at medium speed.

3.2.3 Flow Test for Mortars

The workability of mortars was tested according to the procedure described in ASTM C1437. After finishing the mixing, a first layer of about 25 mm thickness was placed in the mold located on the flow test table and tamped 20 times. A second layer was added until the top of the mold and the same tamping was required. Once the tamping is done, the mold was removed and immediately the flow table was allowed to drop for 25 times. The diameter of the sample was measured from 4 different angles. The flow is the change in the average diameter and is expressed as a percent from the original inner diameter of the mold's base.

3.2.4 Accelerated Mortar Bar Test

The procedure followed is explained in ASTM C1260. After mixing, the samples were casted in the specified 25 by 25 by 285 mm molds and were placed in the curing room for 24 hours. Then, they were demolded and put in water at 80ºC for another 24 hours. Following this, the samples were taken out from water and zero measurements were obtained. They were soaked after in 1M NaOH solution and placed in an oven at 80ºC. Subsequent measurements were taken at 1, 3, 7 days and then once per week until 56 days.

4 RESULTS AND DISCUSSIONS

4.1 Workability of Mortars

4.1.1 Effect of Replacement Level

Samples with carbonate filler of 10, 15, 20, 30 and 100% RLs from the finest portion of the moderately reactive sand were tested to check the effect of mineral fillers addition on workability. The results obtained are presented in Figure 2.

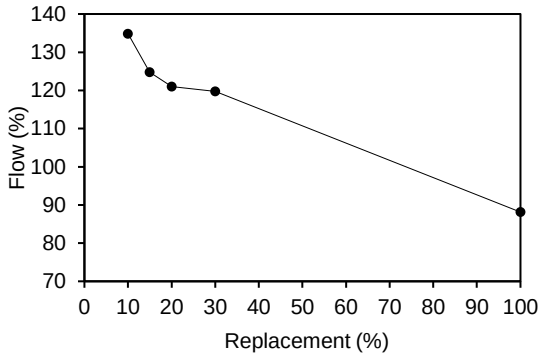

Figure 2: Flow data of moderately reactive sand with different RLs of carbonate filler taken from sand finest portion

As shown in the figure, the flow is 135% at 10% RL and 121% at 20% RL. There is a decrease in the flow with the increase in the replacement level reaching a flow of 80% at 100% RL. It should be noted that with 30% replacement level, 2 mL of superplasticizer was added to the mix. Hence, the decrease was not clearly shown from 20 to 30%. The workability of mortars decreases with the increase of the mineral filler replacement level. Similar results were observed with the other mineral fillers tested.

4.1.2 Effect of Replacement Method

Non-reactive sand samples with carbonate, carbonate silica and SH fillers at 20% RL from the sand finest portion and from the total graded sand were casted and flow was found to understand the effect of different replacement methods on workability. Figure 3 shows the flow results obtained from the two different replacement methods.

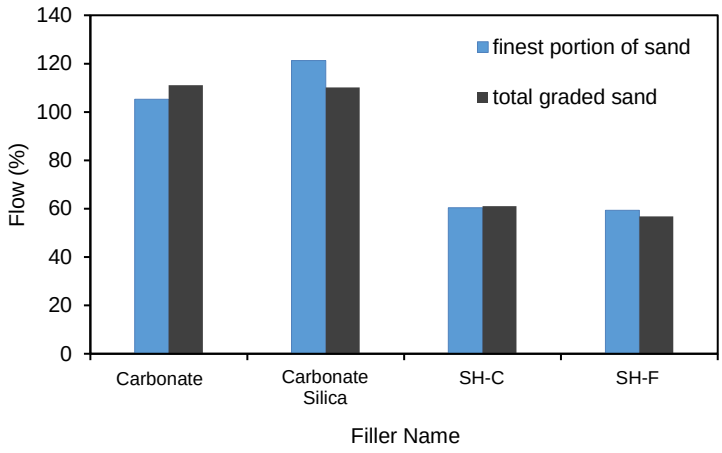

Figure 3: Comparison of flow data between different methods of replacement

As shown in the figure, the difference in workability is very minimal between the two methods. For example, with the carbonate filler, the flow values are 105% and 110% for the samples replaced from the finest portion and from the total graded sand respectively. A maximum difference of 11% between the two methods is observed for samples with carbonate silica filler. It can be concluded that both methods have the same effect on the flow of mortars.

From another side, as Figure 3 indicates, there is a difference between workability of samples with carbonate, carbonate silica and SH fillers. The flow of samples with carbonate and carbonate silica fillers are between 100% and 120%. However, for the SH filler samples, it was much lower with around 60% for both SH-C and SH-F fillers. This might be due to the difference in fillers' sizes. The maximum size of SH filler was smaller than 75 µm. However, carbonate silica filler had 30% of its sample with material coarser than 75 µm as shown in the gradation curve in Figure 1-a. Hence, with smaller size mineral fillers, the decrease in workability is more evident. It is recommended to use superplasticizer with samples containing SH fillers at RLs of more than 10%. At 10% RL, the flow of the SH sample with non-reactive sand was measured and it was still acceptable with a value of 103%. Although the workability of SH filler samples was low, it was still possible to place the samples in their molds and compact them in a similar way to other samples.

4.1.3 Effect of Replacement Method on Expansion

The replacement methods of mineral fillers might have different impact on expansion. 10% and 20% replacement levels from both sands by carbonate silica filler were tested using the AMBT. The data are obtained by taking the average of 3 samples and presented in Figures 4 and 5.

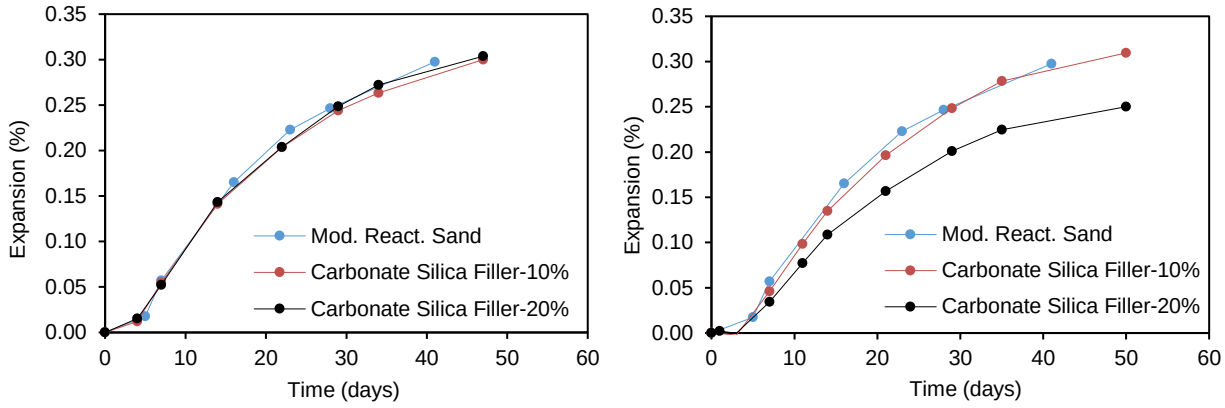

Figure 4: Expansion data of moderately reactive sand with carbonate silica filler taken as (a) percent from finest portion (b) percent from total graded sand

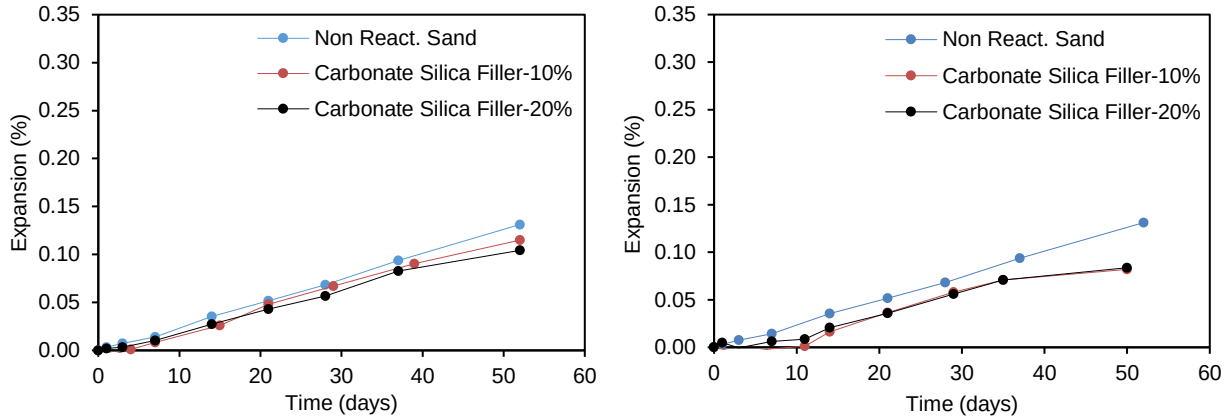

Figure 5: Expansion data of non-reactive sand with carbonate silica filler taken as (a) percent from finest portion (b) percent from total graded sand

As shown in Figures 4 and 5, at 80ºC, the moderately reactive sand has an expansion of 0.17% at 14 days as opposed to the non-reactive sand, which has an expansion of 0.03%. From Figure 4-a, it can be observed that the expansion of the samples with carbonate silica filler at 10% and 20% are the same as the control sample. They have an expansion of 0.27% at 35 days. However, in Figure 4-b, when replacing the total graded sand with 20% carbonate silica filler, the expansion was lower than the control with a value of 0.23% for the carbonate silica filler sample and 0.28% for the control at 35 days. The difference obtained between the two samples (0.05%) is outside the acceptable range of the within-laboratory precision stated in ASTM C1260 which specifies that it should be lower than 8.3% of the mean expansion (8.3%*0.26% = 0.02%). This difference in expansion between the two methods might be due to the fact that the finest portion of the sand has lost reactivity as compared to the particles of larger sizes. Thus, when replacing from the finest portion, the reactivity of the sample is probably not affected as compared to the replacement from the total graded sand. In this method, sand is being removed from the larger size aggregates as well, which perhaps are more responsible for the expansion. The same behavior was observed with non-reactive sand samples (Figure 5). However, the difference was not that clear since the sand is non-reactive. The control has an expansion of 0.13% at 56 days. However, the samples with 10% and 20% RLs have lower expansion of 0.08%. The same behavior was observed with samples containing SH filler when tested at 20% RL.

4.2 Reactivity of Mineral Fillers

4.2.1 Non-Reactive Mineral Fillers

The minerals fillers studied are obtained from different sources, i.e. they might have different reactivity and thus expansion. The carbonate filler is known to be non-reactive since it doesn't contain silica. Figure 6 presents the expansion data of the carbonate filler with non-reactive sand.

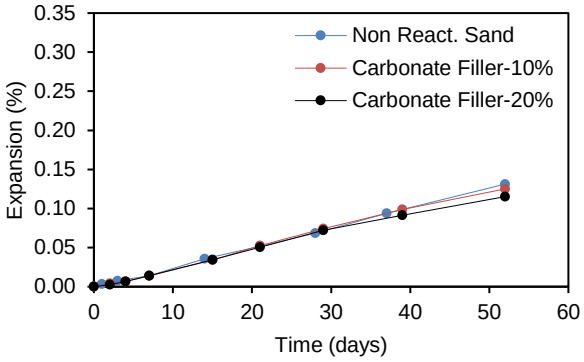

Figure 6: Expansion data of non-reactive sand with carbonate filler taken as a percent of finest portion

The expansion values confirm the non-reactivity of the carbonate filler. The expansion of the samples with 10% and 20% carbonate filler is 0.12% at 52 days which is very close to the control sample expansion of 0.13%. Comparing the data of the carbonate filler samples with carbonate silica filler samples (shown in Figure 5-a), both samples have similar expansions. Although carbonate silica filler has 23% silicate materials, however it didn't show any expansion higher than the control samples even with the moderately reactive sand. This suggests that maybe the silica content might not be enough to cause expansion or the mineral filler has lost reactivity when grinded to its small size.

4.2.2 Springhill Reactivity

Since coarse and fine aggregates from the same source might have different effect on ASR expansion, SH, from coarse and fine aggregates, were crushed separately to test the effect of mineral fillers from each fraction (coarse or fine) on expansion. The expansion data of these two mineral fillers at 20% replacement level are reported in Figure 7.

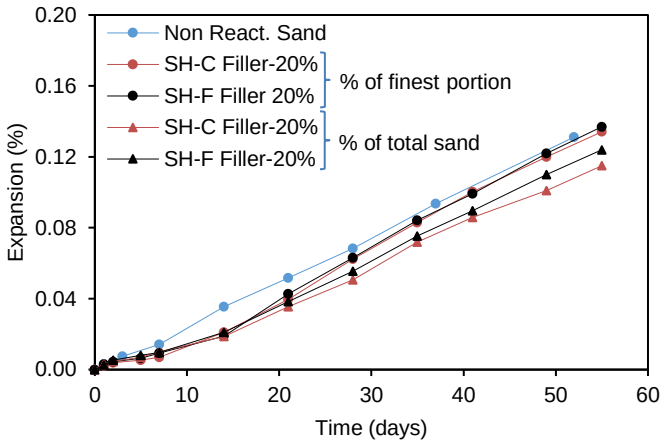

Figure 7: Expansion data of non-reactive sand with 20% SH filler

At 55 days, the expansion of SH-C and SH-F fillers is 0.14% for the samples with a replacement from the finest portion of the sand. As for the ones with replacement from the total graded sand, the expansion is

0.12% for both SH-C and SH-F fillers. Thus, the expansions of SH-C and SH-F fillers with the same replacement method are similar which suggests that they have same reactivity. When compared to the non-reactive sand sample, the decrease in expansion was higher with samples of 20% replacement from total graded sand compared to the other method as expected (Section 4.1.3). In addition, the expansion of SH samples with 20% RL from both methods showed slightly lower expansion that the non-reactive sand. This finding was not expected since the SH filler was obtained from an extremely reactive aggregate. The reactivity of SH filler is still not clear whether the filler has lost reactivity when ground to passing 75 µm.

To investigate whether or not the materials passing 75 µm of Springhill has a pozzolanic reactivity, a sample of Spratt aggregate with 20% SH-C filler was tested. The expansion data are presented in Figure 8-a.

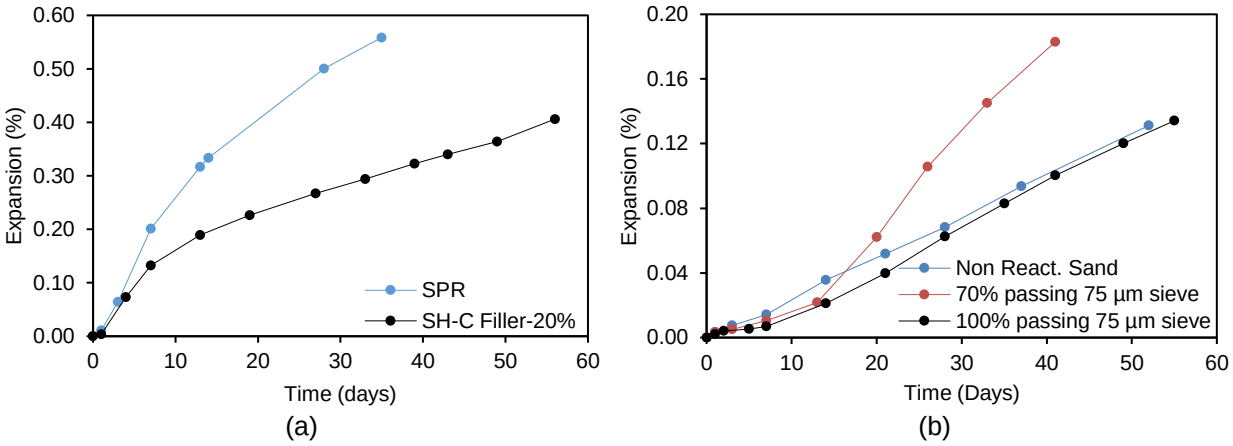

Figure 8: Expansion data of (a) SPR with 20% SH-C filler taken from total graded sand (b) non-reactive sand with 20% SH-C of different gradations taken from finest portion of the sand

The results show that the sample with SH-C filler has lower expansion than the control. At 39 days, the Spratt samples with 20% SH-C filler have an expansion of 0.32% as compared to 0.56% for the SPR sample. This decrease in expansion might be due to the fact that the sample with SH-C has 20% less of Spratt reactive aggregate.

SH samples with different gradation, 70% finer than 75 µm and 30% passing 150 µm sieve and retained on 75 µm sieve, were tested. This size is selected to have a gradation similar to commercial mineral fillers available in the market. This will enable a better prediction of reactivity of mineral fillers produced from reactive stones. Samples with this gradation were casted at 20% RL and results are shown in Figure 8-b. At 20% RL, SH-C samples with 100% finer than 75 µm have same expansion as the non-reactive sand. However, with the same replacement level, the SH-C filler with 70% passing 75 µm sieve showed expansion higher than that of the non-reactive sand. At 41 days, an expansion of 0.18% was observed as compared to 0.10% for the non-reactive sand as well as the SH-C samples finer than 75 µm. This suggests that maybe with finer gradation, the sample loses reactivity or the permeability is reduced hindering the alkalis to reach the core. The behavior of reactive fillers is not clearly understood yet. Thus, more testing should be done to evaluate the reactivity of SH filler using the concrete prism test which is a more reliable test method. It should also be stated that testing beyond 14 days was required to see expansion in mortar bar samples with mineral filler.

5 CONCLUSIONS

Based on the results, the following conclusions are drawn:

1. The higher the replacement level of the mineral filler, the less the workability of the sample. Depending on the gradation of the mineral fillers, the workability will decrease with reduced size of filler. For samples of 0-150 µm size, it is recommended to use superplasticizer for a replacement level above

20%. For samples with particles smaller than 75 µm, the use of superplasticizer is recommended at levels above 10%.

2. The replacement method seems not to have remarkable difference on workability of samples. Whether the replacement is taken from the total graded sand or from the sand finest portion, the workability is almost the same at 20% replacement levels.

3. As for the ASR expansion, replacing the finest portion of the sand by fillers gives higher expansion than replacing a percent of the total graded sand. This might be due to the fact that when using the first replacement method, the finest portion is being removed which might have already lost reactivity compared to coarser particles.

4. Mineral fillers passing 75 µm sieve, obtained from reactive aggregate, showed lower expansion when tested with Spratt reactive aggregate compared to samples with only Spratt. This is likely to be the effect of diluting the reactive aggregate, Spratt, content in the sample assuming that mineral filler of this size is not reactive.

5. Mineral fillers with 70% materials passing 75 µm showed higher expansion than samples with maximum size of 75 µm which showed same expansion as that of the control samples. To understand whether finer materials lose reactivity or reduce permeability, more testing should be done using the CPT.

Acknowledgements

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