

# **The Influence of Alternative Materials on Alkali Aggregate Reaction (AAR) Induced Development in Concrete**

Luan Regis Antunes

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**Department of Civil Engineering**  
**Faculty of Engineering**  
**University of Ottawa**

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## **Abstract**

Portland cement (PC) production is the main responsible for concrete's carbon footprint, accounting for about 7% of the annual man-made CO<sub>2</sub> emissions. Currently, concrete industry faces important challenges of finding cost-effective strategies to reduce the embodied energy associated with the production and use of PC in concrete. Therefore, the use of “conventional” by-products from other industries (i.e. supplementary cementing materials – SCMs) have been implemented with success in the concrete construction for many years. However, recent studies verified that the short and especially long-term availability of SCMs will not be enough to correspond to the rise in PC's demand, which reinforce the need for alternative solutions to partially replace PC. Amongst possible alternatives, biomass waste (i.e. wood-ash - WA) and mineral fillers (MF) are seen as viable products due to their large availability and interesting physicochemical features. Yet, the short and long-term behaviour of concrete incorporating WA or MF are still unclear, particularly their durability performance against important distress mechanisms such as alkali-aggregate reaction (AAR). In this context, this Thesis aims to assess the behaviour of concrete mixtures incorporating WA and MF against AAR-induced development. Concrete mixtures containing reactive and non-reactive coarse and fine aggregates were fabricated with a) WA at distinct replacement levels (i.e. 10, 20, 50 and 60%, by mass) and, b) four types of MF (i.e. reactive and non-reactive) replacing either PC or fine aggregates at a fixed replacement level of 15% (by mass). Accelerated mortar bar and concrete prism tests (i.e. AMBT and ACPT, respectively) along with the conventional concrete prism test (CPT) were performed. In summary, AMBT results indicate a potential benefit of using WA to mitigate AAR-induced expansion and deterioration whereas the ACTP clearly demonstrated that WA is not effective to suppress AAR. Otherwise, CPT results suggest a potential interest of using MFs to lower AAR-induced development, especially while replacing PC and depending upon the MF type. Furthermore, supplementary test procedures (i.e. chemical, mechanical and microscopic) were conducted to appraise the impact of these alternative materials on AAR-induced development. Finally, statistical analysis (i.e. analysis of variance – ANOVA) was conducted to prove the data and discussion made over this work.

**Keywords:** alkali-aggregate reaction, wood ash, mineral filler, reactivity, alternative materials

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## List of Symbols/Abbreviations

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AAR	Alkali-aggregate reaction
ACPT	Accelerated concrete prism test
AMBT	Accelerated mortar bar test
ASR	Alkali-silica reaction
ASTM	American Society for Testing and materials
CPT	Concrete prism test
CS	Compressive Strength
CSA	Canadian Standard Association
C-S-H	Calcium silicate hydrate
DRI	Damage Rate Index
F	Granite (quartz, feldspar, mica)
GU	General use Portland cement
GUL	General use Portland limestone cement
K	Dolomitic argillaceous limestone
LF	Limestone filler
LOI	Loss of ignition
ME	Modulus of elasticity
MF	Mineral filler
O	Limestone filler
PC	Portland cement
PDI	Plastic deformation index
PSD	Particle size Distribution
S	Crushed Greywacke
SCMs	Supplementary cementing materials
SDI	Stiffness Damage Index
SDT	Stiffness Damage Test
SPH	Spring hill aggregate
Tx	Texas sand aggregate
WA	Wood ash

WS	Wood ash with SPH
WT	Wood ash with Tx
XRD	X-Ray Diffraction
XRF	X-Ray Fluorescence

# Chapter One: Introduction

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## 1.1. Synopsis

Concrete is likely the most common construction material used in critical infrastructure worldwide. The life span of reinforced concrete structures varies according to the structure type and purpose; yet, 50 to 70 years are often targeted according to distinct standards. Portland cement (PC) is by far the most important ingredient of concrete; moreover, PC production is the main responsible for concrete carbon-footprint accounting for about 7% of the annual man-made CO<sub>2</sub> emission [1]. Yet, concrete industry currently faces crucial challenges of finding cost-effective strategies to reduce the carbon-footprint and embodied energy associated with the production of PC [2]. Moreover, one of the most adopted methods for reducing environmental impact of PC involve the use of supplementary cementing materials (SCMs) in concrete. Yet, recent studies show that their short and especially long-term availability will not be enough to correspond to the rise in PC's demand [3–6], which reinforce the need for new and renewable solutions to replace PC, at least partially.

Different alternatives such as biomass-waste (i.e. Wood-Ash – WA) and mineral fillers (MF) are seen as viable solutions due to their large availability towards a greener and more sustainable future of the civil industry [7–9]. Furthermore, the current amounts of generated WA and MF are raising concerns regarding their disposal. WA is frequently used as a soil supplement to improve its alkalinity for agriculture purposes [10,11]. However, a major portion (approximately 70%) is land-filled with a common method of disposal [12–14]. In addition, literature results suggested that WA may act as a pozzolanic material in concrete, consuming portlandite and forming additional C-S-H [15]. On the other hand, almost every inorganic mineral with no deleterious reaction with PC can be classified as an MF. Millions of tons of rock residues are produced worldwide every year, mostly improperly discarded in the environment. Overall, the use of MF in cement-based materials is a common practice; yet, limestone fillers (LF) are the most used materials due to their large availability and features (i.e. round shape and smooth texture) [16,17]. Different standards (i.e. CSA A3001 & ASTM C595, ASTM C1157, AASHTO M240) allow the use of up to 15% of LF pre-mixed with PC, the so-called

GUL cement (similar to ASTM type II). However, the current standards (i.e. CSA A23.1-14) do not deny the use of other MF types, but rather indicate them as a replacement for fine aggregates to optimize the materials gradation and thus enhance physical and mechanical properties of granular materials [18–23].

Although replacing PC by WA or MF has shown some promising preliminary results, the short and especially long-term behaviour of concrete incorporating WA or MF are still unclear, particularly their durability performance against critical damage mechanisms such as alkali-aggregate reaction (AAR), one of the most harmful deterioration mechanisms affecting the durability and long-term performance of concrete structures around the globe.

Over the past decades, several approaches and recommendations, including a variety of accelerated laboratory test procedures, have been developed to assess the potential reactivity of aggregates in the laboratory as well as the efficiency of preventive measures (i.e. SCMs such as fly ash, silica fume, blast furnace slag, natural pozzolans, etc.) to mitigate AAR in the field [3,4,18–25]. It has been found that AAR-induced expansion and distress may be prevented by the appropriate use of SCMs [3,25,26]. Nevertheless, there is currently a lack of literature attesting the efficiency of WA and the influence of MF (reactive and non-reactive) on AAR-induced expansion and deterioration, which makes room for major developments in the area.

## **1.2. Research Objectives**

The main goal of this research is to evaluate the impact of replacing PC by WA or a variety of MF on AAR-induced expansion and development. First of all, the finely ground materials (i.e. fillers and wood ash) are selected and physiochemically characterized through X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), particle size distribution (PSD) and specific gravity. Then, a wide range of concrete mixes are proportioned incorporating two highly reactive aggregates (i.e. greywacke coarse - SPH, and polymictic fine aggregate – Tx ), distinct WA replacement levels (i.e. 10%, 20%, 50% and 60% PC replacement, by mass) and four different types of MF (reactive and non-reactive) at a fixed replacement level of 15% (maximum allowed by CSA A3001 & ASTM C595, by mass). It is worth noting that WA was used to directly replace PC while MF has been used to replace either PC or fine aggregates. The manufactured concrete specimens were stored under controlled environmental

conditions enabling AAR-induced development. At selected exposure periods, physical (i.e. porosity), microscopic (i.e. Damage Rating Index) and mechanical (i.e. Stiffness Damage Test, modulus of elasticity and compressive strength) test procedures were conducted so that a comprehensive understanding of the effect of WA and MF on AAR-induced expansion and deterioration might be gathered; finally, considerations for future research in the area are proposed.

### 1.3. Thesis Organization

I hereby attest that all the activities conducted in this research (i.e. research design, experimental program, data analysis and interpretation, draft of papers and Thesis, etc.) has been performed by myself. The PhD candidate Diego Jesus de Souza and my MAsc supervisor Prof. Leandro Sanchez helped me with the *research methodology* besides *revising* the distinct chapters of the Thesis. The current document displays a paper-based format, comprised of five chapters (and an Appendix section) as follows:

**Chapter 1:** This chapter displays a brief synopsis, highlighting the environmental impact of concrete and its ingredients, particularly Portland cement (PC). Current challenges and future needs for finding alternative materials for the partial replacement of PC are then presented and possibilities of using wood-ash (WA) and mineral fillers (MF) are discussed. Finally, the thesis organization and structure are presented.

**Chapter 2:** This chapter displays a comprehensive literature review on concrete sustainability; furthermore, a detailed background on wood-ash (WA) production, including the physicochemical information of the material and its impact on concrete is presented. Likewise, chapter two covers the history of using mineral fillers (MF) in concrete, followed by the introduction of some distinct MF types and their influence on the fresh and hardened states of concrete. Finally, a summary section emphasizes the current research gaps in the field.

**Chapter 3:** Consists of a journal paper (submitted to Cement - Elsevier) which evaluates the influence of wood ash (WA) on alkali-aggregate reaction (AAR)-induced development. It is worth noting that a conference paper discussing the preliminary results of this research has been submitted and accepted for oral presentation in the “International Conference on Alkali-Aggregate Reaction” – ICAAR, Lisbon, 2020/2021.

**Chapter 4:** As for chapter 3, the current chapter consists of a journal paper (to be submitted to a high impact journal once the 365 days data are gathered and evaluated) which appraises the influence of different mineral fillers (MF) on AAR-induced expansion and deterioration.

**Chapter 5:** This chapter summarizes the main findings of the current research with recommendations and suggestions for future research in the area are presented.

**Appendix:** In this section, a series of raw data used in the project are displayed for the benefit of future students willing to conduct similar research.

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## Chapter Two: Background and Literature Review

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### 2.1. Environmental impact of concrete and Portland cement (PC) production

Concrete is likely the most common construction material used in critical infrastructure worldwide as a result of its availability, economic benefits, and outstanding mechanical and durability-related properties. Moreover, as the global demand for concrete increases progressively around the world, concrete industry currently faces crucial challenges of finding cost-effective strategies to reduce the carbon-footprint and embodied energy associated with the production of Portland cement (PC) [1]. Nowadays, concrete is heavily used with more than 10 billion tons produced annually [2]. Likewise, approximately 1.6 billion tons of PC and 10 billion tons of aggregates (fine and coarse) are produced annually [3,4]; consequently, non-negligible amounts of material residues produced are eventually discarded. Furthermore, the world population is expected to reach 9.7 billion in 2050 and could spike to nearly 11 billion around 2100; the latter would result in a huge increase in energy requirement. Hence, concrete demand is forecasted to grow approximately 18 billion tons annually by 2050 [5]. Similarly, an increase in the Portland cement (PC) demand is also expected.

The production of PC involves an intensive use of raw materials (i.e. limestone, clay, etc.) and energy (i.e. clinkerization); consequently, it releases high amounts of carbon dioxide into the atmosphere [6]. Moreover, limestone extraction for PC production requires high demands in fossil fuels burning resulting in significant environmental issues [7–9]. In practice, every ton of manufactured PC releases roughly 0.7 ton of CO<sub>2</sub> into the atmosphere, which emphasizes the need of alternatives to conventional PC [10].

Several studies [11–14] have been focusing on alternatives to partially replace PC in concrete [15,16]. One of the most adopted method for reducing environmental impact of PC involves the use of supplementary cementing materials (SCMs) in concrete. Yet, recent studies show that their short and especially long-term availability will not be enough to correspond to the rise in PC's demand [17–20], which reinforces the need for new and renewable solutions to replace PC. Different alternatives

such as biomass-waste (i.e. wood ash) and mineral fillers (MF) became viable solutions due to their large availability towards a greener and more durable future of the civil industry [15,16,21].

Wood is a potential source of energy as well as an environmentally friendly material; thus, it is expected that a significant increase in renewed wood usage for energy production will take place in the near future [22,23]. Therefore, an increment in the amount of wood ash (WA), a by-product of wood/biochar combustion is anticipated, raising concerns regarding its disposal. WA is frequently used as a soil supplement to improve its alkalinity for agriculture purposes [22,23]. Yet, a major portion (approximately 70%) of WA is land-filled as a common method of disposal [9,24,25]. Moreover, recent studies [11–14] suggest promising results in using WA to partially replace PC in concrete [15,16]. Although preliminary, these results are encouraging for improving WA waste management issues, contributing towards minimizing the energy consumption of high embodied energy PC mixtures.

On the other hand, the use of mineral fillers (MF) as cement replacement is a common practice worldwide. Apart from PC replacement, fillers (normally named “tailings” in this case) can also be used as an alternative to fine aggregates. Results suggest that the use of MF may significantly impact on the fresh and hardened state properties of concrete [26–31]. Moreover, even though fillers are considered to be “inert”, they may actually present small reactivity with PC.

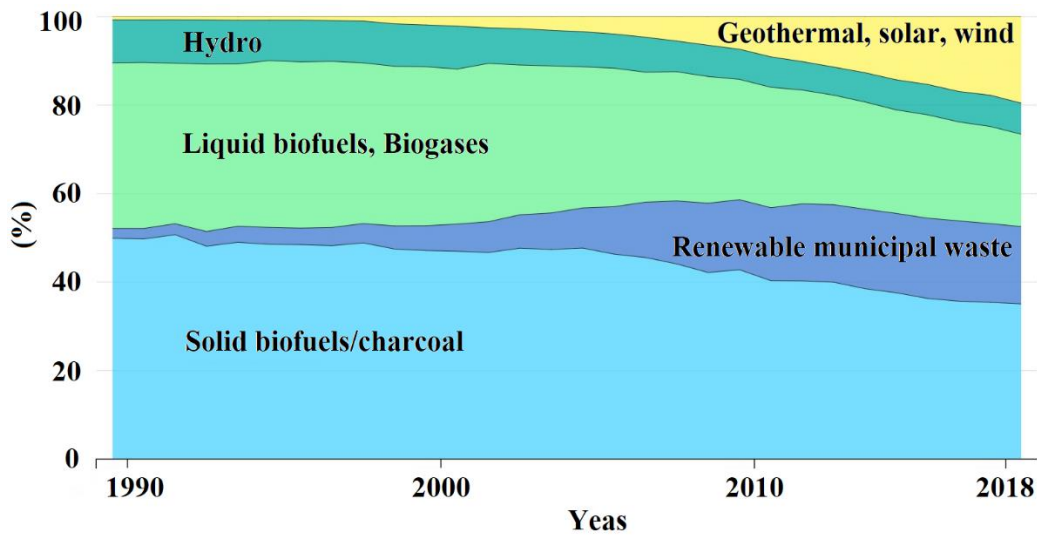
Despite the fact that the incorporation of WA and MF in concrete have shown quite promising results in the fresh and relatively short term (i.e. 28 days) hardened states, the long-term and durability-related properties of cementitious materials incorporating WA and MF are still not well understood, which prevent their widespread use in daily construction works, especially for structural applications.

## **2.2. Wood Ash (WA)**

### **2.2.1. Production of Wood Ash**

Solid biomass fuels, such as wood biomass, represent the most significant renewable energy source in Canada and worldwide according to the Organization for economic Co-operation and development – OECD, representing almost 40% of all energy sources nowadays (Figure 2.1) [32–34]. In general, biomass boilers are a widely known and efficient combusting method to generate energy [35]; yet, it

produces massive amounts of ash residues, raising concerns regarding its disposal and management. On average, burning of wood results in about 6–10% of ashes [15], which suggests a vast potential for developments in this area, aiming for a more efficient reuse of this material [22,23]. Moreover, the major portion (approximately 70%) is currently used as landfill disposal [33] and about 20% as soil supplement to improve its alkalinity for agriculture purposes [22,23]. The remaining 10% has been used for miscellaneous applications including construction materials, metal recovery, and pollution control [15].



**Figure 2.1: OECD renewable primary energy supply by-product [36]**

Although limited literature is available on the use of wood ash in construction, recent studies have investigated the use of WA as a supplementary cementing material (SCM) in concrete [8,15,21,24,37]. Preliminary results have showed suitable fresh and hardened state properties of concrete made of WA with replacement levels of up to 15% [7,14,37]; the latter highlights its promising character [6,21,38,39]. However, using WA in concrete bears important limitations, particularly related to the high variability of WA along with its unknown impact on the durability and long-term properties of concrete.

### 2.2.2. Physicochemical proprieties of WA

WA is the residue generated through the combustion of wood and wood products (i.e. chips, saw dust, bark, etc.). The physical and chemical properties of WA are important in determining potential

benefits in concrete. Although the main oxides present in industrial WA are SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and CaO, their amounts are governed by the tree's specie; thus, resulting in highly variable chemical compositions [15,21,39,40]. WA is chemically comprised of silica (4-40%), carbon (5-30%), calcium (7-33%), potassium (3-4%), magnesium (1-2%), manganese (0.3-1.3%), phosphorus (0.3-1.4%), sodium (0.2-0.5%) and loss on ignition (LOI) up to 60% [15,16,23,41]. For better visualization of the highly variability of WA, Table 2.1 provides the chemical composition of different biomass ashes [42]. It has been suggested that the combustion temperature significantly affects the total WA yield along with its chemical composition [43]. Carbonates compounds (i.e. CaCO<sub>3</sub>) are predominant in wood ash produced through incineration at temperatures lower than 500°C [6] Conversely, controlled combustion of wood/wood-waste at higher temperatures (i.e. about 1000°C) causes a substantial reduction in LOI due to the release of carbon oxides, enhancing thus the hydraulic or pozzolanic properties of the material [15,21,39,44,45].

**Table 2.1: Chemical composition of wood ash from several species of timber [42]**

<b>Wood - Biomass group and variety</b>	<b>SiO<sub>2</sub></b>	<b>CaO</b>	<b>K<sub>2</sub>O</b>	<b>P<sub>2</sub>O<sub>5</sub></b>	<b>Al<sub>2</sub>O<sub>3</sub></b>	<b>MgO</b>	<b>Fe<sub>2</sub>O<sub>3</sub></b>	<b>SO<sub>3</sub></b>	<b>Na<sub>2</sub>O</b>	<b>TiO<sub>2</sub></b>
<i>Alder-fir sawdust</i>	37.49	26.41	6.1	2.02	12.23	4.04	8.09	0.83	1.81	0.98
<i>Balsam bar</i>	26.06	45.76	10.7	4.87	1.91	2.33	2.65	2.86	2.65	0.21
<i>Beech bark</i>	12.4	68.2	2.6	2.3	0.12	11.5	1.1	0.8	0.9	0.1
<i>Birch bark</i>	4.38	69.06	8.99	4.13	0.55	5.92	2.24	2.75	1.85	0.13
<i>Elm bark</i>	4.48	83.46	5.47	1.62	0.12	2.49	0.37	1	0.87	0.12
<i>Eucalyptus bark</i>	10.04	57.74	9.29	2.35	3.1	10.91	1.12	3.47	1.86	0.12
<i>Forest residue</i>	20.65	47.55	10.23	5.05	2.99	7.2	1.42	2.91	1.6	0.4
<i>Hemlock bark</i>	2.34	59.62	5.12	11.12	2.34	14.57	1.45	2.11	1.22	0.11
<i>Land clearing wood</i>	65.82	5.79	2.19	0.66	14.85	1.81	1.81	0.36	2.7	0.55
<i>Maple bark</i>	8.95	67.36	7.03	0.79	3.98	6.59	1.43	1.99	1.76	0.12
<i>Oak wood</i>	48.95	17.48	9.49	1.8	9.49	1.1	8.49	2.6	0.5	0.1
<i>Olive wood</i>	10.24	41.47	25.16	10.75	2.02	3.03	0.88	2.65	3.67	0.13
<i>Pine bark</i>	9.2	56.83	7.78	5.02	7.2	6.19	2.79	2.83	1.97	0.19

It has been found that WA containing high LOI absorbs more water, which in turn increases the water or chemical admixtures demand in the concrete while negatively impacting on its fresh (i.e. workability) and hardened state (i.e. compressive strength) performance [21,46]. Furthermore, literature indicates that WA presents irregular morphology (i.e. shape and texture) and particle size distribution [15,21,39,44,45]. Values of specific surface area ranging from 5 m<sup>2</sup>/g to 35 m<sup>2</sup>/g and specific gravity varying from 2.2 to 2.8 are often found [41,47-49]. This particular and highly variable

character of WA emphasizes the importance of thoroughly characterizing the material prior to its use in concrete.

### **2.2.3. Effect of WA on concrete properties**

Very few researches have been conducted on the use of WA in construction. Nevertheless, WA is currently used in Europe as a raw material in the manufacturing of Portland cement [24]. Moreover, according to [50], wood ash has the potential to be used as a pozzolan in cement-based materials. On the other hand, some investigations have demonstrated the potential use of WA as road-base materials, whenever blended with cement [50]. Yet, there is still a needed of further developments in the field, from the selection and standardization of quality control protocols for biomass ash production (decreasing thus the variability of the final product), to the understanding of its impact on the short and long-term properties of concrete [51]. Some of the recent developments on the use of WA in concrete are presented hereafter:

#### **2.2.3.1. Fresh State**

It has been suggested that incorporating WA leads to an increase in the amount of water required to produce concrete with a desired consistency (i.e. slump value) [47,48]. Literature shows that replacing PC by WA in levels ranging from 0% to 30% (in mass), may significantly increase the material's consistency (i.e. slump decrease) without making use of chemical admixtures or changing the water-to-binder ratio [8]. Furthermore, although [48] observed that replacement levels of up to 10% only slightly modify the slump values of mixtures containing WA, important rheological changes are already verified with small replacement ratios (i.e. lower yield stress and viscosity) [52]. The authors explained that the high surface area, high organic content, LOI and irregular shape of WA allow water adsorption by its particles (hygroscopic by nature), leading to higher water demands and consistency [46–49,52]. In addition, it has been reported that replacing up to 30% of PC by different WA types might impact on the initial and final setting times of the mixture; the higher the replacement level, the longer the initial and final setting times observed [49].

### **2.2.3.2. Cement Hydration**

Different studies have suggested that WA particles, independently of their pozzolanic activity, may enhance PC's hydration due to the formation of supplementary nucleation sites fastening PC's hydration kinetics [47,48,59]. Conversely, other studies have shown that WA may delay setting due to some of its features such as high surface area, high organic content, LOI and irregular shape [49]. The latter indicates quite contradictory results in the literature. Moreover, it has been reported that replacing up to 30% of PC by different types of WA, the initial setting time may range from 21% lower than pure PC to 113% higher; the same proportion has also been suggested for final setting time [49].

Results demonstrate that blended cement-based materials made of WA and PC have lower peaks of portlandite measured through X-ray diffractometry and thermal analysis [47]. The simple PC's replacement decreases the  $C_2S$  and  $C_3S$  contents, hence resulting in lower portlandite content, whereas the pozzolanic reactivity of WA also contributed to decrease portlandite peaks [47]. Moreover, the process and temperature of wood combustion, imply in variable carbon content on WA chemical composition, which results on variable peaks of the thermal decomposition of calcium carbonate found by different authors [47,49].

### **2.2.3.3. Hardened State**

Different authors have reported that the higher the WA replacement ratio, the higher the porosity and permeability of the hardened material and the lower the compressive strength at 28 days [8,15,21,37,38,41,48,49,62,63]. The splitting tensile and flexural strength of WA concrete generally follow a similar trend than the compressive strength [15,41]. Yet, results suggest that the inherent properties of the WA particles (i.e. specific surface area, texture, size and shape of particles and pozzolanic activity), which are strongly dependent on its production process, are the main cause of important variations on the hardened performance of WA concrete [21,25,47,48]. Moreover, since the pozzolanic activity of the WA is extremely variable, its reactivity with the portlandite from PC's hydration to form C-S-H pozzolanic, refining thus the microstructure of the concrete is also highly variable [15,49,63,64]. On the other hand, it has been reported that the higher porosity and absorption properties of the WA particles may indirectly enhance the mechanical properties of concrete by

improving the “internal” curing process of the material [8,15,65]; the latter might decrease the probability of cracks caused by drying shrinkage, self-desiccation, etc. Finally, the modulus of elasticity (ME) of WA concrete is quite comparable to conventional concrete up to 10% of replacement and for the same water-to-binder ratio. For replacement levels above and beyond 10%, significant ME reductions are observed [38].

#### **2.2.3.4. Durability of WA concrete**

The durability properties of concrete made of WA are still under investigation and contradictory results are reported in the literature. Research studies have been conducted on chloride penetration [66,67]; carbonation depth [60]; electrical resistivity [48,67] and resistance against freeze and thawing [62]. Regarding alkali-aggregate reaction (AAR), just a few data are available in the literature, mostly limited to mortar specimens [60,64,68]. The chloride permeability, directly linked to the permeability of concrete, has been reported to decrease [66]. However, contradictory results indicate that WA concrete may worsen microstructure quality and higher permeability [67]. Furthermore, different studies suggested that the electrical of the concrete decreases as highest the WA content in the blended matrix [48,67], which is explained by the formation of a more porous and less homogeneous matrix and a less compacted granular structure when using the WA. Yet, the resistance against freeze and thawing (FT) was improved, 15% of PC’s replacement by WA were able support similar total cycles of FT than conventional concrete (CC).

Few data are available in the literature regarding alkali-silica reaction, moreover, results are limited to the accelerated mortar bar test (AMBT) [60,64,68]. In general, the authors reported that mortars made of WA were able to modify AAR kinetics suggesting effectiveness in reducing AAR expansion; moreover, increasing WA content led to lower expansion. Esteves et al. [68] studied the incorporation of 20 and 30% and demonstrated reductions of 28% and 75% in the final expansion at 28 days of exposure to the AMBT, respectively. Similarly, AAR expansion was reduced between approximately 18% and 60% (replacement levels of 10% and 20%, respectively) when compared to neat PC mortar bars [60]. Although the mechanism of AAR mitigation was not well understood.

## **2.3. Fillers**

### **2.3.1. The use of mineral fillers (MF) in concrete production**

MF started to be used in the concrete industry due to economic reasons, where PC was initially replaced by different types of powder materials such as granite and sandstone [29]. Nowadays, limestone filler (LF) is the most widely used MF worldwide; in early 1980s, the cement replacement by LF was introduced into cement standards. The first standard developed on the use of LF was the Canadian CAN3-A5-M83, in 1983, which limited the use of LF to 5% [70]. Hereafter, by 1988 in Europe, those limits were raised to 20% in Portland-filler cements [70]. Currently, several national and international standards include MF for cement production [71]; hence, important developments on its usage may be currently found in the literature [72–75]. Nevertheless, most of the countries are restricted the PC replacement to the use of LF, as result some standard has been applied already allowing the use of general cement with limestone (GUL) as Portland-limestone cement with a limit up to 15% of LF presented in the cement, as per ASTM C595 / CSA A3000-18, even though there is still missing durability's (long-term) evaluation in the influence of MF. Yet, large amounts of different natures of fillers are also widely available and have been allowed the addition of up to 5% of minor mineral constituents, i.e. European standard EN 197-1, [26,27,29,76–78]. Moreover, additional literature provides detailed information of PC's replacement by different types of filler such as quartz [75,79–82], dolomite [82,83], granite [83–85], cristobalite [85,86], nepheline syenite [85,86], and wollastonite [86].

In summary, MF are utilized in order to enhance the granular filling factor and luck-up capillary pores, due to their low reactivity [75,87] enhancing the microstructure, reduce cement consumption, resulting in lower costs and decrease carbon footprint of the concrete [26,27,29,76–78]. Yet, concerns related to their structural performance and durability behaviour still prevent its widespread use for structural purposes.

### **2.3.2. General influence of MF on distinct methods of replacement on concrete properties**

Unlike SCMs, which take part in chemical reactions to produce gel and crystals for concrete strength development [88–94], MF displaying similar particle size distribution than cement and are not

considered to be an inert material [95,96]. In general, when properly selected and proportioned, MF may also increase the packing density of granular systems such as concrete, allowing thus PC reduction without compromising the mechanical properties of the material [17,97]. Apart from cement replacement, fillers (normally named “tailings” in this case) can also be used as an alternative to fine aggregates. Furthermore, although MF may derive from a wide range of rock types and there are some studies stated with detailed information of different types of MF such as quartz [75,79–82], dolomite [82,83], granite [83–85], cristobalite [85,86], nepheline syenite [85,86], and wollastonite [86], limestone fillers (LF) are the most used materials due to their large availability [75,87]. Different standards (i.e. CSA A3001 & ASTM C595, ASTM C1157, AASHTO M240) allow the use of up to 15% of LF pre-mixed with PC, the so-called GUL cement (similar to ASTM type II). Yet, the current standards (i.e. CSA A23.1-14) do not discard the use of other MF types, but rather indicate their use as a replacement for aggregates to optimize the materials gradation and thus enhance physical and mechanical properties [26–31]. Nevertheless, the long-term efficiency and durability behaviour of concrete incorporating distinct types of MF are still not fully understood, particularly when evaluating the performance of reactive aggregates in the laboratory

### **2.3.2.1. Cement Hydration**

It has been reported that three important physical effects may be observed while the use of MF regardless of their mineralogy [17,28,54,55,98]: (a) dilution enhancement of anhydrous PC particles; (b) enhancement of the particle size distribution (PSD) of the system (when properly selected and proportioned); and (c) formation of supplementary nucleation sites. Several authors investigated the use of basalt, granite, marble, dolomitic and limestone powders in cement pastes, mortars and concrete mixtures [97,99–107].

Carbonaceous fillers, such as limestone, magnesian limestone and dolomitic limestone can slightly react with aluminates phases of the clinker to form monocarboaluminate hydrates [97,99]. While MF containing siliceous phases (i.e. “pure” quartz, granite, etc.) may slightly contribute to form C-S-H along with delaying the formation of hydrogarnet and tobermorite [27,76]. Besides, results suggest that MF enhance the hydration process of cementitious materials [108], allowing thus PC reduction without compromising the mechanical properties gain at early ages [17,97]. MF with similar or lower

particle size distribution can enhance PC's hydration [108]; yet, MF with higher MgO content may slightly slow down PC's hydration kinetics [75].

### **2.3.2.2. Fresh State**

The fresh state behaviour of cementitious materials (the so-called workability) is defined as the easiness of the material to be mixed, handled, placed, and compacted with minimum homogeneity loss (i.e. segregation). Different authors have demonstrated that MF significantly improve the fresh state of the concrete [110–115], as well as reduce bleeding [116,117], settlement [41,118], and plastic shrinkage [119–121]. However, depending of the particle size distribution and specific surface area, the concrete might behave differently (positively or negatively in the fresh state) [122,123]. Moreover, the use of MF has show to increase the water demand to maintain normal consistency and setting time [124] while finer fillers induce higher plastic viscosity and yield stress than the coarser fillers [75,87,111,116,125,126]. In general, it has been suggested that increasing the volume of fillers in the paste matrix increased the maximum distances between the aggregates, leading to lower values of yield torque lowering consistency values [119]. On the other hand, the highest the volume of MF in the paste matrix, the highest the plastic viscosity of the concrete, once the distance between fine particles decreases [119,126,127].

### **2.3.2.3. Hardened State and Durability of MF in concrete**

When properly selected and proportioned, cementitious systems made of MF might display wider PSD and thus better packing density [128–130]. Yet, poor results were also observed, especially when conventional mix-proportioning approaches and simple PC's replacement (i.e. in %) were implemented [131]. Different authors have reported that the higher the MF replacement ratio, the higher the porosity and permeability of the hardened material and the lower the compressive strength at 28 days [132–134]. On the other hand, high-strength concrete mixtures were obtained through the use of MF [135,136]. Moreover, the splitting tensile, flexural strength and modulus of elasticity of concrete made of MF generally follow a similar trend than the compressive strength [135,136].

The durability of concrete made of MF and exposed to AAR development, very few data have been reported so far. Mostly, MF concrete have not shown significant mitigative behaviour against AAR

up to “safe” levels; yet, they may substantially change the AAR kinetics and expansion amplitudes [125,137–139]. However, the influence on AAR by adding different types of MF produced from alkali reactive rock types suggest promising results [125]; but, this influence either from alkali-reactive particles of “inert”, are still unclear [125,137–139]. It has been suggested that the particle size distribution of the MF is the main factor to govern AAR development [125]. In general, by decreasing the average particle size of reactive aggregate there is trend to accelerate AAR kinetics. Although, in the average size of the material (i.e. lower than 100 mm) the AAR kinetics is speed-down while the reactive particle starts to develop more pozzolanic reactivity than AAR [125,137–140]. However, [125] provides some opposite results while non-reactive filler was used, with finer fraction of filler (i.e. 0-20 mm) gives higher expansion than the coarser particles (i.e. 0-120 mm).

The general literature still needs further development in the area, specially investigating deeply the AAR-induced development on the mechanical and microscopic properties of concrete mixtures made with MF (either alkali-reactive or not) comparing with the most commonly used and standardized MF (i.e. limestone fillers). Which makes room for major developments in this area.

## **2.4. Alkali-Aggregate Reaction (AAR)**

AAR is one of the main processes affecting the durability and serviceability of concrete structures worldwide. It can be divided into two categories, the alkali-silica reaction (ASR) and the alkali-carbonate reaction (ACR). The most common, ASR, was firstly recognized by Thomas Stanton in the 1940s in California (USA) and its comprises of a chemical reaction between “unstable” silica mineral forms within fine and/or coarse aggregate materials and the alkali hydroxides (Na, K – OH) that are dissolved within the concrete pore solution [141–143]. It generates a secondary product, the so-called alkali-silica gel, that induces expansive pressure within the reacting aggregate material and adjacent cement paste upon moisture uptake, leading to microcracking, loss of material’s integrity and functionality of the affected structure [141–144]. On the other hand, ACR (first reported by Swenson in 1957 in Ontario, Canada) was found to be much faster and deleterious (although less frequent) than ASR [143,145]; yet, its mechanism is still unknown. However, several studies have verified that the dedolomitization process of the dolomite crystals inside carbonated rocks might be amongst the reasons of the chemical reaction and distress [143,145].

AAR distress degree and features depend upon the type (i.e. fine and coarse aggregate) and reactivity of the aggregates used, the amount of alkalis of the concrete, the temperature and relative humidity of the environment along with the exposure and confinement (e.g. reinforcement ratio, etc.) conditions of a given structure/structural member [144,146–151]. Yet, it has been found that AAR-induced expansion and distress may be prevented by the appropriate use of SCMs [142,152,153]. In this context, different authors have reported the efficiency of WA and specific types of MF may indicated a reduction induced expansion in due to AAR, even though most of these results were presented as per AMBT method; according to the studies, the higher their replacement ratio, the lower the AAR-induced expansion over time [60,68]. Nevertheless, the mechanism behind AAR mitigation while the use of WA or MF in concrete is still not fully understood nor discussed.

#### **2.4.1. The influence of mineral additions on AAR development**

Cement is the main source of alkalis (sodium  $Na^+$  and potassium  $K^+$ ) and also portlandite within the concrete. Therefore, it is convenient to use different binder composition able to reduce the final amount of at least one of these compounds. Industrial materials, such as, silica fume, fly ash and slag cements are probably the most widespread materials used to prevent AAR. Extensive research in Canada during the last years has confirmed that binary and ternary blends containing mixtures of Portland cement [154]. The use of mineral additions as a partial replacement of PC causes microstructural changes, modifying the pore size distribution [155]. In addition, the use of SCMs or MF decrease the alkali content available from the clinker and lower the rate of alkali release [155]. SCMs and MF are currently being used in concrete as they may reduce concrete costs and carbon footprint, improving thus its sustainability and durability. Regardless the type of the material (i.e. inert, cementitious or pozzolanic), change the hydration kinetics of PC [28,156] while the reactive materials (i.e. cementitious and pozzolanic) may also form supplementary C-S-H.

In the pozzolanic reaction, part of the alkali ions may be entrapped by the supplementary C-S-H particles [157]; furthermore, it consumes portlandite (CH) from the concrete pore solution. It has been suggested that  $Ca_2^+$  ions play a very important role in controlling the swelling properties of the ASR-gel [157]. Moreover, the CH may accelerate the diffusion of alkalis in the reactive particles [158]. Yet, high replacement levels high dosage of these materials (SCMs and MF) may negatively impact on other concrete properties such as setting, strength development, freeze–thawing and chlorides

permeability. Second, the availability of high quality materials has become uncertain and limited worldwide [159]; which raises the pressure of finding different sources of mineral additions, increasing the need for development and evaluation of non-conventional conventional sources such as biomass-waste (i.e. wood ash) and mineral fillers (MF).

#### **2.4.2. Influence of AAR on the mechanical proprieties of concrete**

Overall, the development of AAR induces a very significant and quick drop in modulus of elasticity and tensile strength of the affected concretes. These two properties are significantly more affected than the compressive strength, which begins to decrease significantly only at high levels of expansion [160–163]. This classic description of the ASR impact on the mechanical properties of concrete proposes that significant microcracking caused by this mechanism can be identified even before the material reaches significant expansion and mechanical distress, which might lead to noteworthy compressive strength loss [161,162].

It has been found that the reaction rate, the cracking patterns and also the mechanical properties reductions of ASR-affected concrete change as a function of the type/nature of coarse aggregate used in the material [164]. Based on the testing of slowly reactive aggregate types (i.e. greywacke, quartzite, granite, andesite, etc.) that had caused problems in concrete structures after 10 to 20 years in service, Reinhart & Mielich [165] showed that the dynamic modulus of elasticity, which can be measured through the ultrasonic pulse velocity procedure, is generally not a good indicator of concrete damaged due to AAR. On the other hand, the authors showed that the modulus of elasticity is the most sensitive parameter for assessing the degree of AAR damage of an affected material.

Moreover, Giaccio et al. [164], testing AAR-affected concrete, verified that the stress/strain behavior of the affected materials changed according to the coarse aggregate used. The authors concluded that those differences were related to the aggregates type (i.e. shape and etc.) , meaning the capability of the aggregates to provide a better bond with the cement paste (which depends on some aggregate's characteristics such as texture, toughness, hardness, soundness, etc.), as well as to the presence of pre-existing cracks within the particles generated over crushing/weathering processes. Finally, Alexander and Milne [166] showed that not only the aggregate types/natures and the concrete strength

(and maturity), but also the type of binder, plays a very important role on the stress/strain behavior of the concrete, again demonstrating the importance of the ITZ in the resulting behavior of concrete.

### **2.4.3. Tools for AAR assessment**

The distress caused by AAR-induced development may have harmful consequences for different types of properties (e.g. mechanical, durability and physical integrity) of concrete. Therefore, some tools were developed to measure the “damage” in its broader sense caused by AAR as defined hereafter: a) Different methods for measurements the expansion; 2) mechanical proprieties reduction of the material; 3) Microscopic tools to identify the durability loss related to the extension of cracks of the concrete. Moreover, damage can be defined as the harmful consequences of various types of mechanisms (e.g. loadings, shrinkage, creep, AAR, Delayed Ettringite Formation-DEF, freezing and thawing, etc.) on the mechanical properties, physical integrity and durability of a concrete element/material.

### **2.4.4. Assessing AAR-induced development in the laboratory**

Among the various test procedures currently used to identify the potential reactivity of aggregates in concrete, one may mention the accelerated mortar bar test - AMBT (ASTM C1260/CSA A23.2-25A) and the concrete prism test - CPT (ASTM C1293/ CSA A23.2-14A). The ABMT is the most widely used method for assessing AAR potential reactivity in the laboratory [54,55] due to its relatively quick response (i.e. 16 days). Mortar specimens are moulded and cured over 48 hours in water at 80°C; afterwards, the specimens are immersed in a highly concentrated alkaline solution (i.e. 1M NaOH) at 80°C for further 14 days. However, although the ABMT provides an interesting fast screening of ASR potential reactivity in the laboratory, this method has been criticized over the last decades due to two main reasons: a) first, it displays severe exposure conditions which can lead to false negatives and positives [56] and, b) the imposed accelerated procedure may not give enough time to evaluate binder compositions containing supplementary cementing materials [57,58]. On the other hand, the CPT (ASTM C1293/ CSA A23.2-14A) provides more reliable results which are much closer to what is experienced in the field; moreover, it is able to better classify the reactivity of aggregates along with more precisely appraise concrete mixtures incorporating a wide variety of binder compositions in the laboratory [57,59,60]. In the CPT method, concrete prisms are moulded

and cured in the moist-curing room over 24 hours. They are then demoulded and stored at 38°C and 100 of RH for either 1 year (in the case of evaluating the aggregates reactivity) or 2 years (while the assessment of preventive measures). The total content of binder materials and the water-to-cement (or binder) ratio are roughly fixed (i.e. narrow ranges are provided) in the mix. Furthermore, the amount of alkalis in the system is boosted through the addition of reagent NaOH grade to the mixing water so that the equivalent alkali content ( $\text{Na}_2\text{Oe}$ ) of the cement is raised to 1.25% (by mass).

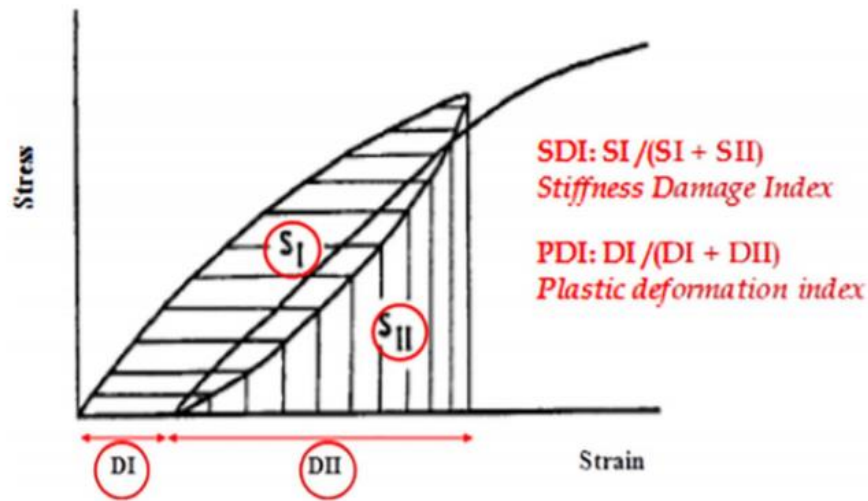
Even though the CPT is considered the most reliable existing test procedure, one important disadvantage of the method is its long testing period (1-2 years). Trying to solve this issue, accelerated CPT procedures were proposed in the 90's [173]. Among them, an accelerated CPT version (ACPT) conducted at 60°C stood out, showing promising results worldwide. It has been found that reliable results might be achieved within either 3-4 months or 6-8 months for evaluating the potential reactivity of aggregates or the efficiency of preventive measures, respectively. This method has been standardized in Europe [174], although in North America it is not currently a standard since some issues related to alkalis leaching and important variability in the test outcomes were found [175,176].

#### **2.4.5. Stiffness Damage Test (SDT)**

The SDT was initially developed by Walsh [177] who detected an interesting correlation between the inner crack density and the cycles of loading/unloading (stress/strain relationship) of rock specimens. Afterwards, Crouch proposed the use of the SDT to appraise concrete samples with diameters greater than 70mm. Moreover, in the early 1990's, Crisp and coworkers suggested using the SDT to quantify the distress degree of concrete affected by AAR, by applying five compression cycles (loading/unloading) with a fixed stress level of 5,5MPa [178]. Afterwards, Smaoui et al. [40] proposed the increase in stress level to 10 MPa so that the test could become more diagnostic. Nevertheless, it has been found that the SDT outcomes could significantly change as per the type of aggregates incorporated in the mix-design.

Sanchez et al. [147,151,179] continue the works from [40], by conducting the SDT on laboratory-made concrete specimens displaying various mix-designs (i.e. 25, 35 and 45 MPa), and incorporating a wide range of reactive aggregates types (i.e. coarse vs. fine) and natures (i.e. lithotypes), along with cores extracted from AAR-affected structures. After evaluating several parameters, it has been found

that the SDT should be performed with a percentage (i.e. ideally 40%) of the design (28-day) compressive strength instead of a fixed loading as per [40,178]. Moreover, the method was found very promising for assessing AAR-induced expansion and deterioration in concrete, especially when indices are used as the test outcomes, namely Stiffness Damage Index (SDI) and Plastic Deformation Index (PDI) illustrated on Figure 2.2, which represent respectively the ratio of dissipated energy/plastic deformation to the total energy/deformation implemented in the system [149]. Finally, the modulus of elasticity and the non-linearity index (NLI- the ratio between the slope of the stress-strain curve at the peak load and at half of the peak load) were also verified as suitable outcomes of the test to appraise AAR-induced progress.



**Figure 2.2: Calculation of the Stiffness Damage Index (SDI) and Plastic Deformation Index (PDI) as by [179]**

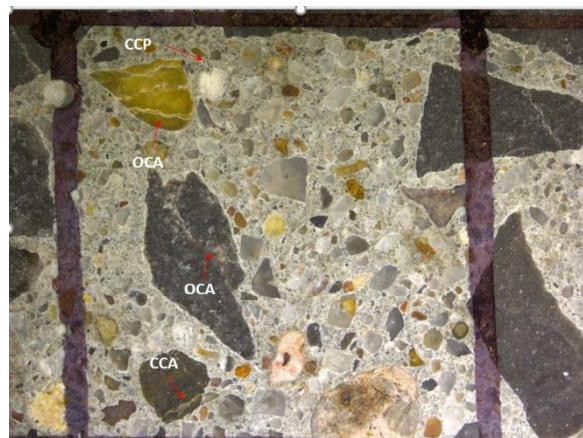
#### 2.4.6. Damage Rate Index (DRI)

The DRI is a petrographic analysis performed with the use of a stereomicroscope (15–16× magnification) where damage features generally associated with AAR are counted through a 1 cm<sup>2</sup> grid (i.e. 10 × 10 mm units) drawn on the surface of polished concrete sections [148]. The number of counts corresponding to each type of petrographic features is then multiplied by weighing factors (Table 2.2), whose purpose is to balance their relative importance towards the mechanism of distress, for instance AAR [148]. The factors used in the method were selected on a logical basis, but relatively arbitrarily; they were recently modified in order to reduce the variability between the petrographers

performing the test [180]. Ideally, a surface of at least 200 cm<sup>2</sup> should be used for DRI analysis, and it may be greater in the case of mass concrete incorporating larger aggregate particles. However, for comparative purposes, the final DRI value is normalized to a 100 cm<sup>2</sup> area [181]. The goal of DRI is to appraise the damage degree or extent of affected concrete, complementing conventional petrographic analysis whole main purpose is to detect the cause of the deterioration by observed the types of cracks (Figure 2.3). Moreover, presenting a direct correlation with expansion level, a damage scale to distinguish low, moderate and high damage levels caused by AAR were proposed by [148,179]. Yet, similar results were presented by [182,183] with concreted cores extracted from AAR-affected structures.

**Table 2.2: DRI weighing factors for petrographic features classification**

Petrographic features	Weighting factors	
Crack in coarse aggregate	CCA	0.25
Opened crack in coarse aggregate	OCA	2
Crack with reaction product in coarse aggregate	OCAG	2
Coarse aggregate debonded	CAD	3
Crack in cement paste	CCP	3
Crack with reaction product in cement paste	CCPG	3
Air void lined / filled with gel	AVG	--
Reaction rim around aggregate	RR	--
Disaggregate / corroded aggregate particle	DAP	2



**Figure 2.3: Indication of some crack types on the concrete sample**

## **2.5. Summary of the literature review**

As stated in the above sections, very few researches have been conducted on the use of wood ash in concrete, especially regarding durability parameters of cement-based materials such as the influence of concrete mixtures incorporating WA against AAR-induced development. To the best knowledge of the author, the only few works conducted in the past have been developed through the accelerated mortar bar test (AMBT), which is not a reliable test produced and could have generated false-positive and false-negative results. Furthermore, there is a lack of mechanical and microscopic assessment of AAR-induced deterioration in concrete bearing WA.

The situation above is quite similar for MF. Although the use of MF is much more disseminated than WA in concrete, and some research discussing the impact of MF on the fresh and hardened state properties of concrete are available, there is only few research studying the influence of distinct MF types (i.e. reactive vs non reactive, distinct mineralogy, etc.) on AAR-induced expansion and deterioration. Actually, the AAR-community is currently facing challenges regarding the assessment of concrete systems incorporating MF in the laboratory. First of all, the influence of MF fillers is not well understood and there are still doubts on the reliability and validity of using PC types containing MF such as GUL in Canada (or ASTM Type IL in the USA) to assess the reactivity of granular combinations (i.e. aggregates + MF) in the laboratory. Finally, questions regarding the way of introducing MF to the granular system either through PC or fine aggregates' replacement have been raised and are still open to debate.

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# Chapter Three: The use of Wood Ash as Partial Replacement of Cement to Investigate the Influence on ASR-induced Development in Concrete

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Antunes L.R.<sup>1</sup>, De Souza D.J.<sup>1</sup>, Sanchez L.F.M.<sup>1</sup>

<sup>1</sup>Department of Civil Engineering, University of Ottawa, Ottawa, Canada.

## Abstract

Over the past decades, several approaches and recommendations, including a variety of accelerated laboratory test procedures, have been developed to assess the potential reactivity of aggregates in the laboratory as well as the efficiency of preventive measures (e.g. supplementary cementitious materials-SCMs) to mitigate AAR in the field. It has been found that AAR-induced expansion and damage may be prevented by the appropriate use of SCMs. However, the anticipated depletion in the coming years of some of the most common SCMs used in concrete makes imperative the finding of new potential SCMs sources. This research aims to study the use of wood ash (WA), a by-product of biochar combustion, to analyze the influence on AAR physicochemical development. Two reactive aggregates and different WA replacement levels (i.e. 10%, 20%, 50% and 60%) are selected for this research and accelerated tests are performed in the laboratory (i.e. concrete prism test – CPT and the accelerated mortar bar test – AMBT). AMBT results indicated a potential benefit of using WA to mitigate ASR-development whereas the ACPT yielded completely opposite results. Therefore, the latter seems to indicate an example of a “false negative” result yielded by the accelerated mortar bar test. Furthermore, the mechanical properties losses (i.e. compressive strength, modulus of elasticity and SDI) are in agreement with the expansion levels obtained and microscopic analyses conducted through the DRI method. Although the addition of WA increased significantly the overall porosity of the material.

**Keywords:** alkali-aggregate reaction, durability of concrete, supplementary cementitious materials, wood ash.

### 3.1. Introduction

Concrete is likely the most common construction material used in critical infrastructure worldwide. The life span of reinforced concrete structures varies according to the structure type and purpose; yet, 50 to 70 years are often targeted according to distinct standards. Portland cement (PC) is by far the most important ingredient of concrete, being the main responsible for its carbon-footprint and accounting for about 7% of the annual man-made CO<sub>2</sub> emission [1]. However, the concrete industry currently faces crucial challenges of finding cost-effective strategies to reduce the carbon-footprint and embodied energy associated with the production of PC [2]. Moreover, one of the most adopted methods for reducing environmental impact of PC involves the use of supplementary cementing materials (SCMs) in concrete. Although the use of SCMs has progressively increased in the construction industry over the past decades, depletion in the coming years is expected to some of the most common SCMs (e.g. fly ash), which makes imperative the finding of alternative materials able to reduce the embodied energy of concrete [3,4]. Furthermore, recent findings suggest that the use of biomass-waste materials can be feasible to act as SCMs and thus partially replace PC in concrete [5–7].

Wood is a potential source of energy as well as an environmentally friendly material; thus, it is expected that a significant increase in renewed wood usage for energy production will take place in the near future [8,9]. Therefore, an increment in the amount of wood ash (WA), a by-product of wood/biochar combustion is anticipated, raising concerns regarding its disposal. WA is frequently used as a soil supplement to improve its alkalinity for agriculture purposes [8,9]. Yet, a major portion (approximately 70%) of WA is land-filled with a common method of disposal [10–12]. Moreover, due to its physicochemical properties, WA is being seen as a viable SCMs alternative towards a greener and more durable future of the civil industry [6,7]. Although past research has shown that the replacement of PC by WA may enhance the physical and mechanical properties of concrete [10,13–18], its impact on the long-term performance of the material is still not well understood, which prevent its widespread use in daily construction works, especially structural applications.

One of the main damage mechanisms affecting the durability and long-term performance of concrete structures worldwide is alkali-silica reaction (ASR). Over the past decades, several approaches and recommendations, including a variety of accelerated laboratory test procedures, have been developed

to assess the potential reactivity of aggregates in the laboratory as well as the efficiency of preventive measures (i.e. SCMs such as fly ash, silica fume, blast furnace slag, natural pozzolans, etc.) to mitigate ASR in the field [3,4,19–26]. It has been found that ASR-induced expansion and distress may be prevented by the appropriate use of SCMs [3,26,27]. Nevertheless, there is currently a lack of literature attesting the efficiency of WA to mitigate ASR-induced expansion and deterioration (to the best knowledge of the authors), which makes room for major developments in this area.

### **3.2. Background**

Concrete is a widely used material with over 10 billion tons produced annually in modern industrial society [28]. Moreover, the world population is expected to reach 9.7 billion in 2050 and could spike to nearly 11 billion around 2100, which would result in a huge increase in energy need. Thus, concrete demand is forecasted to grow approximately 18 billion tons annually by 2050 [29]. Similarly, an increase in the Portland cement (PC) demand is also expected.

The production of PC involves an intensive use of raw materials (i.e. limestone, clay, etc.) and energy (i.e. clinkerization); consequently, it releases high quantities of carbon dioxide into the atmosphere [13]. Moreover, the limestone extraction for PC production requires high demands in fossil fuels burning resulting in significant environmental issues [12,14,30]. In practice, every ton of manufactured PC releases roughly 0.7 ton of CO<sub>2</sub> into the atmosphere, which emphasizes the need of alternatives to conventional PC [31].

Recent studies [32–35] have been focusing on the use of by-products from distinct industries to partially replace PC in concrete [6,7]. One of the most promising materials studied is wood ash (WA), a by-product of biochar combustion, which may act as a pozzolan, consuming portlandite and forming additional C-S-H [36]. These findings displayed a potential solution for WA waste management issues, contributing towards minimizing the energy consumption of high embodied energy PC mixtures. Hence, the incorporation of WA in concrete would be beneficial not only for decreasing the carbon footprint of concrete but rather to reduce costs associated to clinker production [14,37].

### **3.2.1. Wood Ash (WA) as a supplementary cementing material (SCM)**

#### **3.2.1.1. Physicochemical properties of WA**

Wood ash (WA) is the residue generated due to combustion of wood and wood products (i.e. chips, saw dust, bark, etc.). The physical and chemical properties of WA are important in determining potential benefits in concrete. Although the main oxides present in industrial WA are  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{Fe}_2\text{O}_3$  and  $\text{CaO}$ , their quantities are governed by the tree specie; thus, resulting in highly variable chemical compositions [5,7,38,39]. WA is chemically comprised of silica (4-40%), carbon (5-30%), calcium (7-33%), potassium (3-4%), magnesium (1-2%), manganese (0.3-1.3%), phosphorus (0.3-1.4%), sodium (0.2-0.5%) and loss on ignition (LOI) up to 60% [6,7,9,17]. It has been suggested that the combustion temperature significantly affects the total WA yield along with its chemical composition [36]. Carbonates compounds (i.e.  $\text{CaCO}_3$ ) are predominant in wood ash produced through incineration at temperatures lower than  $500^\circ\text{C}$  [13]. Conversely, controlled combustion of wood/wood-waste at higher temperatures (i.e. about  $1000^\circ\text{C}$ ) cause a substantial reduction in LOI due to the release of carbon oxides, enhancing thus the hydraulic or pozzolanic properties of the material [5,7,18,38,40]. It has been found that WA containing high LOI absorbs more water, which in turn increases the water or chemical admixtures demand in the concrete while negatively impacting on its fresh (i.e. workability) and hardened state (i.e. compressive strength) performance [5,41]. Furthermore, literature indicates that WA presents irregular morphology (i.e. shape and texture) and particle size distribution [5,7,18,38,40]. Values of specific surface area ranging from  $5 \text{ m}^2/\text{g}$  to  $35 \text{ m}^2/\text{g}$  and specific gravity varying from 2.2 to 2.8 are often found [17,42-44]. This particular and highly variable character of WA emphasizes the importance of thoroughly characterizing the material prior to its use in concrete.

#### **3.2.1.2. Effect of WA on concrete properties**

##### **3.2.1.2.1. Fresh state**

It has been suggested that incorporating WA leads to an increase in the amount of water required to produce concrete with a desired consistency (i.e. slump value) [42,43]. Literature shows that replacing PC by WA in levels ranging from 0% to 30% (in mass), may significantly increase the material's

consistency (i.e. slump decrease) without making use of chemical admixtures or changing the water-to-binder ratio [14]. Furthermore, although [43] observed that replacement levels of up to 10% only slightly modify the slump values of mixtures containing WA, important rheological changes are already verified with small replacement ratios (i.e. lower yield stress and viscosity) [45]. The authors explained that the high surface area, high organic content, LOI and irregular shape of WA allow water adsorption by its particles (hygroscopic by nature), leading to higher water demands and consistency [41–45]. In addition, it has been reported that replacing up to 30% of PC by different WA types might impact on the initial and final setting times of the mixture; the higher the replacement level, the longer the initial and final setting times observed [44].

### **3.2.1.2.2. Hardened state**

The mechanical properties of concrete are directly related to the quality of its microstructure. Different authors have reported that the higher the WA replacement ratio, the higher the porosity and permeability of the hardened material and the lower the compressive strength at 28 days [5,7,14,17,43,44,46–49]. The splitting tensile and flexural strength of WA concrete generally follow a similar trend than the compressive strength [7,17]. Yet, results suggest that the inherent properties of the WA particles (i.e. specific surface area, texture, size and shape of particles and pozzolanic activity), which are strongly dependent on its production process, are the main cause of important variations on the hardened performance of WA concrete [5,11,42,43]. Moreover, since the pozzolanic activity of the WA is extremely variable, its reactivity with the portlandite from PC's hydration to form C-S-H pozzolanic, refining thus the microstructure of the concrete is also highly variable [7,44,49,50]. On the other hand, it has been reported that the higher porosity and absorption properties of the WA particles may indirectly enhance the mechanical properties of concrete by improving the “internal” curing process of the material [7,14,37]; the latter might decrease the probability of cracks caused by drying shrinkage, self-desiccation, etc. Finally, the modulus of elasticity (ME) of WA concrete is quite comparable to conventional concrete up to 10% of replacement and for the same water-to-binder ratio. For replacement levels above and beyond 10%, significant ME reductions are observed [47].

### **3.2.1.2.3. Durability of WA concrete**

The durability properties of concrete made of WA are still under investigation and contradictory results are reported in the literature. Research studies have been conducted on chloride penetration [51,52]; carbonation depth [15]; electrical resistivity [43,52] and resistance against freeze and thawing [46]. Conversely, very few researches has been performed on the behaviour of WA concrete incorporating reactive aggregates; the available results are limited to research conducted on mortar specimens [15,50,53].

Alkali-silica reaction (ASR) consists of a chemical reaction between “unstable” silica mineral forms within fine and/or coarse aggregate materials and the alkali hydroxides (Na, K – OH) that are dissolved within the concrete pore solution [4,27,54]. It generates a secondary product, the so-called alkali-silica gel, that induces expansive pressure within the reacting aggregate material and adjacent cement paste upon moisture uptake, leading to microcracking, loss of material’s integrity and functionality of the affected structure [4,27,54,55]. ASR distress degree and features depend upon the type (i.e. fine and coarse aggregate) and reactivity of the aggregates used, the amount of alkalis of the concrete, the temperature and relative humidity of the environment [55–61]. However, it has been found that ASR-induced expansion and distress may be prevented by the appropriate use of SCMs [3,26,27]. In this context, different authors have reported the efficiency of WA to mitigate induced expansion in mortar bar specimens; according to the studies, the higher the WA replacement ratio, the lower the ASR-induced expansion over time [15,53]. Nevertheless, the mechanism behind ASR mitigation while the use of WA in concrete is still not fully understood nor thoroughly discussed.

## **3.2.2. Tools for ASR assessment**

### **3.2.2.1. Assessing ASR-induced development in the laboratory**

Among the various test procedures currently used to identify the potential reactivity of aggregates in concrete, one may mention the accelerated mortar bar test - AMBT (ASTM C1260/CSA A23.2-25A) and the concrete prism test - CPT (ASTM C1293/ CSA A23.2-14A). The ABMT is the most widely used method for assessing ASR potential reactivity in the laboratory [62,63] due to its relatively quick response (i.e. 16 days). Mortar specimens are moulded and cured over 48 hours in water at 80°C; afterwards, the specimens are immersed in a highly concentrated alkaline solution (i.e. 1M NaOH) at

80°C for further 14 days. However, although the ABMT provides an interesting fast screening of ASR potential reactivity in the laboratory, this method has been criticized over the last decades due to two main reasons: a) first, it displays severe exposure conditions which can lead to false negatives and positives [64] and, b) the imposed accelerated procedure may not give enough time to evaluate binder compositions containing supplementary cementing materials [65,66]. On the other hand, the CPT (ASTM C1293/ CSA A23.2-14A) provides more reliable results which are much closer to what is experienced in the field; moreover, it is able to better classify the reactivity of aggregates along with more precisely appraise concrete mixtures incorporating a wide variety of binder compositions in the laboratory [65,67,68]. In the CPT method, concrete prisms are moulded and cured in the moist-curing room over 24 hours. They are then demoulded and stored at 38°C and 100% RH for either 1 year (in the case of evaluating the aggregates reactivity) or 2 years (while the assessment of preventive measures). The total content of binder materials and the water-to-cement (or binder) ratio are roughly fixed (i.e. narrow ranges are provided) in the mix. Furthermore, the amount of alkalis in the system is boosted through the addition of reagent NaOH grade to the mixing water so that the equivalent alkali content ( $\text{Na}_2\text{O}_e$ ) of the cement is raised to 1.25% (by mass).

Even though the CPT is considered the most reliable existing test procedure, one important disadvantage of the method is its long testing period (1-2 years). Trying to solve this issue, accelerated CPT procedures were proposed in the 90's [69]. Among them, an accelerated CPT version (i.e. ACPT) conducted at 60°C stood out, showing promising results worldwide. It has been found that reliable results might be achieved within either 3-4 months or 6-8 months for evaluating the potential reactivity of aggregates or the efficiency of preventive measures, respectively. This method has been standardized in Europe by RILEM [70], although in North America it is not currently a standard since some issues related to alkalis leaching and important variability in the test outcomes were found [71].

### **3.2.2.2. Stiffness Damage Test (SDT)**

The SDT was initially developed by Walsh [72] who detected an interesting correlation between the inner crack density and the cycles of loading/unloading (stress/strain relationship) of rock specimens. Afterwards, Crouch proposed the use of the SDT to appraise concrete samples with diameters greater than 70mm. Moreover, in the early 1990's, Crisp and coworkers proposed to use the SDT to quantify the distress degree of concrete affected by ASR, by applying five compression cycles

(loading/unloading) with a fixed stress level of 5,5MPa [73]. After several tests, Smaoui et al. [39] proposed some parameters for determining the extent of damage of ASR-affected concrete through the SDT, by increasing the fixed stress level up to 10MPa. It has been found later on that the SDT outcomes could significantly change as per the type of aggregates incorporated in the mix-design.

Sanchez et al. [57,61,74] continue the works from [39], by conducting the SDT on laboratory-made concrete specimens displaying various mix-designs (i.e. 25, 35 and 45MPa), and incorporating a wide range of reactive aggregates types (i.e. coarse vs. fine) and natures (i.e. lithotypes), along with cores extracted from ASR-affected structures. After evaluating several parameters, it has been found that the SDT needs to be performed with a percentage (i.e. ideally 40%) of the design (28-day) compressive strength instead of a fixed loading as per [39,73]. Moreover, the method was found very promising for assessing ASR-induced expansion and deterioration in concrete, especially when indices are used as the test outcomes, namely Stiffness Damage Index (SDI) and Plastic Deformation Index (PDI), which represent respectively the ratio of dissipated energy/plastic deformation to the total energy/deformation implemented in the system [59]. Finally, the modulus of elasticity and the non-linearity index (NLI) were also verified as suitable output parameters of the test to appraise ASR-induced progress.

### **3.2.2.3. Damage Rate Index (DRI)**

The DRI is a petrographic analysis performed with the use of a stereomicroscope (15–16× magnification) where damage features generally associated with ASR are counted through a 1 cm<sup>2</sup> grid (i.e. 10 × 10 mm units) drawn on the surface of polished concrete sections [58]. The number of counts corresponding to each type of petrographic features is then multiplied by weighing factors, whose purpose is to balance their relative importance towards the mechanism of distress, for instance ASR [58]. The factors used in the method were selected on a logical basis, but relatively arbitrarily; they were recently modified in order to reduce the variability between the petrographers performing the test [75]. Ideally, a surface of at least 200 cm<sup>2</sup> should be used for DRI analysis, and it may be greater in the case of mass concrete incorporating larger aggregate particles. However, for comparative purposes, the final DRI value is normalized to a 100 cm<sup>2</sup> area [76]. The goal of DRI is to appraise the damage degree or extent of affected concrete, complementing conventional petrographic analysis whole main purpose is to detect the cause of the deterioration. Moreover,

presenting a direct correlation with expansion level, a damage scale to distinguish low, moderate and high damage levels caused by ASR were proposed by [58,74]. Yet, similar results were presented by [77,78] with concreted cores extracted from ASR-affected structures.

### **3.3. Scope of the work**

As stated above, there is an urgent need to find alternatives to conventional SCMs towards a greener and more durable future in the concrete industry. Among possible alternatives, wood ash seems a viable solution; however, very few data have been reported on the long-term durability properties of concrete incorporating WA, particularly in concrete affected by alkali-silica reaction (ASR). This work focuses on the use of WA as a PC partial replacement to mitigate ASR-induced expansion in concrete. Mortar and concrete specimens incorporating two highly reactive aggregates (i.e. Springhill coarse aggregate and Texas sand), and distinct WA amounts (i.e. 10%, 20%, 50% and 60% PC replacement, by mass) are fabricated and stored under controlled environmental conditions enabling ASR development. At selected exposure periods, microscopic (i.e. Damage Rating Index), mechanical (i.e. Stiffness Damage Test, modulus of elasticity and compressive strength), and porosity analysis are conducted and a comprehensive evaluation of the influence of WA on ASR-induced expansion and damage is performed.

### **3.4. Materials and Methods**

#### **3.4.1. Materials**

Accelerated mortar bar (AMBT) and accelerated concrete prism (ACPT) tests were conducted incorporating two highly reactive coarse (Springhill) and fine (Texas sand) aggregates as described in Table 3.1. Table 3.2 provides information on the chemical composition of the different binder materials used in this research; i.e. PC-GU type (equivalent to ASTM type I) and WA. The available WA selected for this research (material produced close to Ottawa, Canada) displays a high amount of CaO, alkalis (K<sub>2</sub>O), and LOI, and a low presence of SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. The high CaO and LOI indicates a high amount of CaCO<sub>3</sub> (calcium carbonate) in the material, calculated as 52.7% (by stoichiometry). Furthermore, 48.97% of CaO and 23.18% of LOI were also obtained through XRF. Since the sample were fully dried, the LOI means CO<sub>2</sub> herein; therefore, using the LOI to subtract

the total amount of CaCO<sub>3</sub>, one notices that only 29.54% of CaO is combined as CaCO<sub>3</sub>, whereas 19.43% is likely “free” to be released to the concrete pore solution. Nevertheless, since this material is widely available within Ottawa, and the literature data was deemed promising, it has been decided (as a first trial) to assess its performance in concrete.

**Table 3.1: Reactive (R) and non-reactive (NR) aggregates used in the research.**

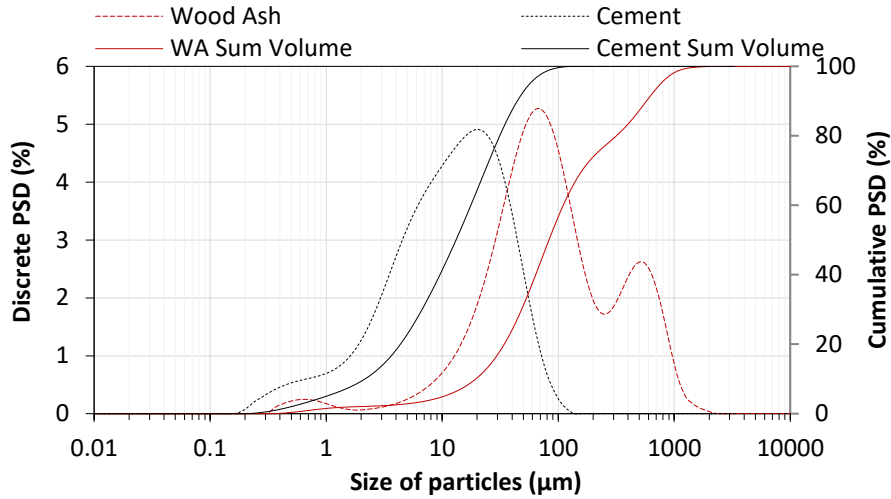
Aggregate	Reactivity	Rock Type	Specific gravity	Absorption (%)	AMBT <sup>a</sup> (%)	
Coarse	NC	NR	High-purity fine-grained limestone	2.79	0.42	0.01
	SPH	R	Crushed Greywacke	2.73	0.71	0.26
Fine	NF	NR	Natural derived from granite	2.67	0.82	0.09
	Tx	R	Polymictic sand (granitic, mixed volcanic, quartzite, chert, quartz)	2.63	0.91	0.85

<sup>a</sup> Results at 14 days of curing of the accelerated mortar bar testing (ASTM C 1260) performed on the aggregates selected for this research.

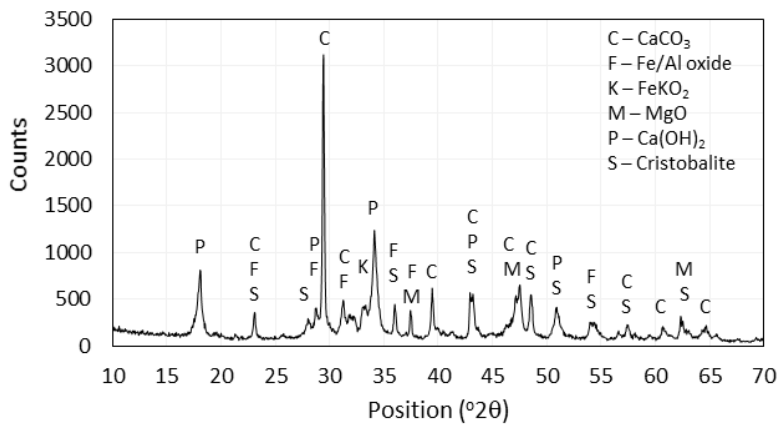
**Table 3.2: Chemical composition of the cement and wood ash.**

	Cement (%)	Wood Ash (%)
CaO	61.93	48.97
SiO <sub>2</sub>	20.1	6.51
Al <sub>2</sub> O <sub>3</sub>	5.02	0.92
Fe <sub>2</sub> O <sub>3</sub>	3.79	1.47
MgO	2.42	4.75
K <sub>2</sub> O	0.92	8.85
Na <sub>2</sub> O	0.3	0.25
P <sub>2</sub> O <sub>5</sub>	-	2.12
MnO	-	2.05
SO <sub>3</sub>	3.37	-
LOI	2.91	23.18
Na <sub>2</sub> O <sub>eq.</sub>	0.91	6.06

The particle size distribution (PSD) of the PC and WA were obtained through laser diffraction spectroscopy (Figure 3.1). WA displays a coarser PSD (D50 of 72.45 μm), which is around five times coarser than PC (D50 of 13.18 μm). The latter indicates that WA has the potential to enhance concrete packing, since it may fill the gaps in between the cement and fine aggregate particles. Moreover, X-Ray Diffraction-XRD has been conducted on the WA (Figure 3.2), where calcium carbonate (CaCO<sub>3</sub>) was identified as the predominant mineral phase of the material. The diffractogram of the material presents a slightly amorphous halo (i.e. amorphous fraction that can be reactive with the cement compounds – left portion); however, based on its chemical composition, WA is classified herein as an inert material.



**Figure 3.1: Particle size distribution (PSD) of the GU Portland cement and the Wood Ash: y1) discrete PSD and y2) Cumulative PSD.**



**Figure 3.2: X-ray diffractograms of the wood ash, with the predominant chemical phases identified with the ICDD database.**

### 3.4.2. Mixture proportions, curing and exposure conditions

The current research was divided into two phases. In the first phase, the expansion behaviour of eight different mortar bar mixtures, incorporating the two distinct reactive aggregate types (displayed in Table 3.3) and four different PC replacement levels by WA (i.e. 0%, 10%, 20% and 50%, by mass) was evaluated through the AMBT as per ASTM C 1260/1567 [79,80] (Table 3.3); the results obtained in this phase were taken as a first screening for the second research phase.

**Table 3.3: Mortar mixtures with the two different reactive aggregates, according to ASTM C 1260.**

Group ID	Fine materials (g)		Water (g)	Fine Aggregates
	Cement	Wood Ash		
Control	440	-	206.8	990g of Tx or SPH aggregates
WA10	396	44		
WA20	352	88		
WA50	220	220		

In the second phase, seventy-two 35 MPa concrete cylinders, 100 by 200 mm in size, were fabricated and appraised as per ASTM C1293 [81]; however, the specimens were stored under a higher temperature (i.e. 60°C instead of 38°C) to further accelerate ASR-induced development as per [70]. Six concrete mixtures, using the two reactive aggregates combined with non-reactive aggregates and different wood ash replacement levels (0%, 20% and 60%) were proportioned. It is worth noting that the water-to-binder ratio was kept constant for all mixes (Table 3.4).

The specimens were demoulded after 24 h of casting. Afterwards, small holes, 5mm in diameter by 15mm long, were drilled at both flat ends of the samples. Then, studs were glued in place with a fast-setting cement slurry in order to measure the longitudinal expansions. Next, the specimens were moist cured for over 24 h. After 48 h from casting, the “zero” reading was recorded, and the cylinders were placed in sealed buckets lined with a damp cloth and stored at 60°C and 100% RH. Every two weeks, the sealed buckets were removed from the chamber and stored at 23°C per  $16 \pm 4$  h prior to the sample’s measurements.

**Table 3.4: Concrete mixtures cast with different aggregates using the same volumetric amount of reactive aggregates.**

Mixture	Water-to-fines = 0.45			Aggregates (kg/m <sup>3</sup> )			
	Water (kg/m <sup>3</sup> )	Cement (kg/m <sup>3</sup> )	Wood Ash (kg/m <sup>3</sup> )	NF	Tx	NC	SPH
WS-C	189	420	-	836	-	-	938
WS-20		336	84	803	-	-	938
WS-60		168	252	738	-	-	938
WT-C		420	-	-	765	1020	-
WT-20		336	84	-	765	986	-
WT-60		168	252	-	765	917	-

### **3.4.3. Test methods**

#### **3.4.3.1. Stiffness Damage Test (SDT)**

Concrete specimens were subjected to five cycles of loading/unloading at a controlled loading rate of 0.10 MPa/s. The SDT was performed as per Sanchez et al.[57,59,61], i.e. using a loading level corresponding to 40% of the 28-day compressive strength. To characterize all mixtures at 28 days, samples were wrapped and placed at 12°C, since some of the specimens contained highly reactive aggregates and ASTM C 39 method could not be followed as they could develop some deterioration due to ASR. The cylinders were maintained at 12°C for a 47-day period, according to the maturity concept as per ASTM C 1074; they were then compressive strength tested and 40% of the obtained average value per concrete mixture was used to perform the SDT.

#### **3.4.3.2. Damage Rate Index (DRI)**

The DRI was conducted on one specimen from each concrete mixture as per Sanchez [55,74]. The samples were cut in half axially and polished using a standard polishing device which uses diamond-impregnated rubber disks (No 50 [coarse], 100, 400, 800, 1500, 3000 [very fine]); this device was found most suitable for the work, as it does not loose abrasive powders that can fill up cracks or voids in concrete, and high quality polishing is obtained with minimal water supply so that AAR-gel leaching is avoided. Afterwards, 1cm<sup>2</sup> grids were drawn on the surface of the polished sections and the DRI was performed as per [74]. The DRI final number presented hereafter is the normalized 100 cm<sup>2</sup> value.

#### **3.4.3.3. Compressive Strength (CS)**

Compressive strength was conducted on the samples with two different and specific goals. First, as previously mentioned, the 28 days compressive strength of each mixture was obtained considering the maturity concept as per ASTM C 107 to obtain the ultimate capacity of the mixtures designed. The second compressive strength measurements were performed on three cylinders used for stiffness damage testing, with the aim of verifying the compressive strength loss of the material as a function

of ASR development. This procedure was adopted and considered valid after Sanchez et al. [55,74] confirmed the non-destructive character of the SDT.

#### **3.4.3.4. Porosity**

The porosity of the concrete samples was evaluated according to the Archimedes immersion method. Every 30 days of exposure to 60°C and 100% RH, one sample per concrete mixture was taken and cut into three similar slices (i.e. about 3 cm high). Afterwards, the sliced samples were dried at 60°C over ten days and weighted to obtain their dry mass ( $M_D$ ). Next, the samples were fully immersed in water under a constant pressure of 28-30Hg for twenty-four hours, and then wiped to remove their surface moisture and weighted in a high precision scale to record their wet mass ( $M_{SSD}$ ). Finally, in a hydrostatic scale, the submerged mass ( $M_s$ ) was recorded. Thus, the apparent porosity can be calculated through the following equation.

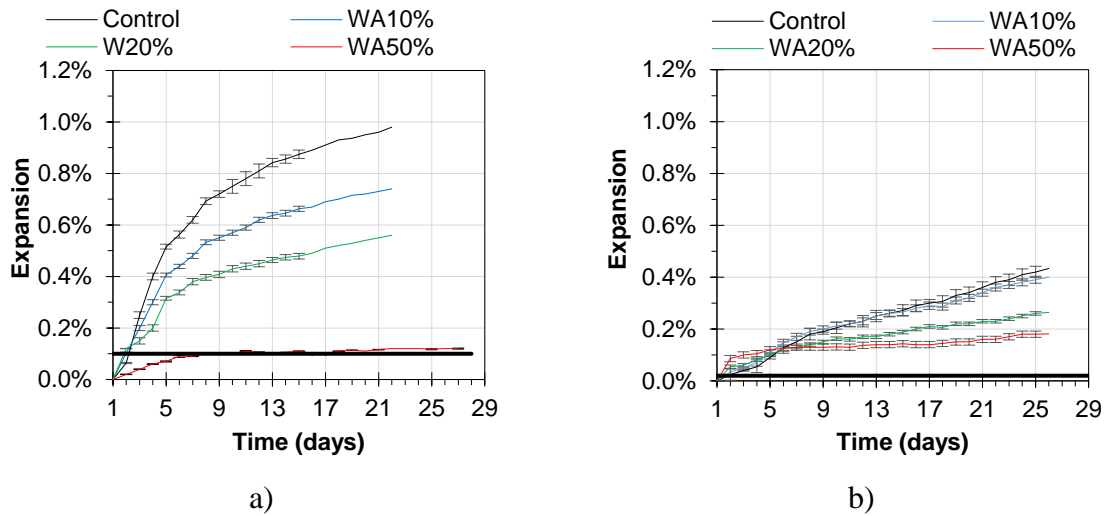
$$AP = \frac{M(ssd) - M(d)}{M(ssd) - M(s)} \quad \text{Equation 3.1}$$

### **3.5. Results**

#### **3.5.1. ASR-induced development**

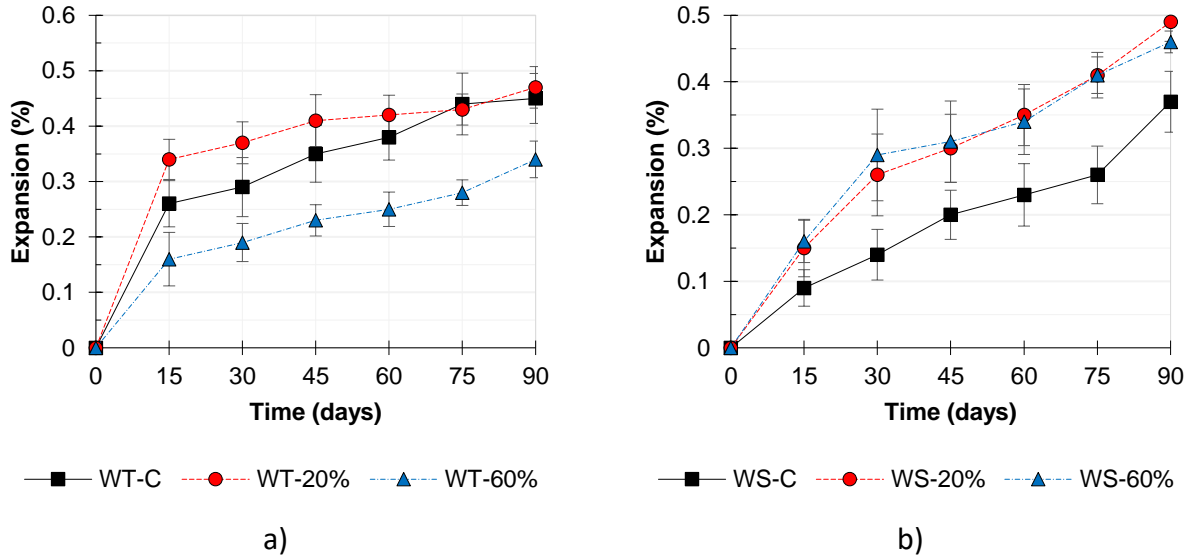
In this section, ASR-induced expansion is presented for all 8 mortar bar mixtures and 6 concrete mixes fabricated in the laboratory. Figure 3.3 displays the results obtained through the Accelerated Mortar Bar Test (AMBT). Overall, the variability of the analyzes was very low for all mixtures measurements. In general, the mixtures containing Tx presented faster kinetics and reactivity than those incorporating SPH. Figure 3.3a shows data from mixtures made of Tx sand with the control mixes displaying the highest expansion level followed by mixtures incorporating 10%, 20% and 60% of WA, respectively; in other words, the higher the amount of WA in the mix, the lower the induced expansion obtained. Figure 3.3b presents the results of mixtures incorporating the crushed coarse SPH aggregate. Analyzing the data, one notices that the same behaviour is found for SPH mixtures (i.e. lower induced expansion with higher incorporation of WA). Yet, for Tx sand mixtures, a PC replacement of 50% by WA was able to decrease the induced expansion down to levels below the limits proposed by ASTM C1260/C1567 (i.e. 0.1% at 16 days) and CSA A23.2-25A (0.15% at 16

days), while for SPH coarse mixture a PC replacement of 50% by WA was under the limits of CSA A23.2-25A only. Finally, these results are consistent with data found in the literature [15,50,53].



**Figure 3.3: Accelerate Mortar Bar Test (AMBT) expansion of ASR-affected mortars: a) Tx fine and b) SPH Coarse.**

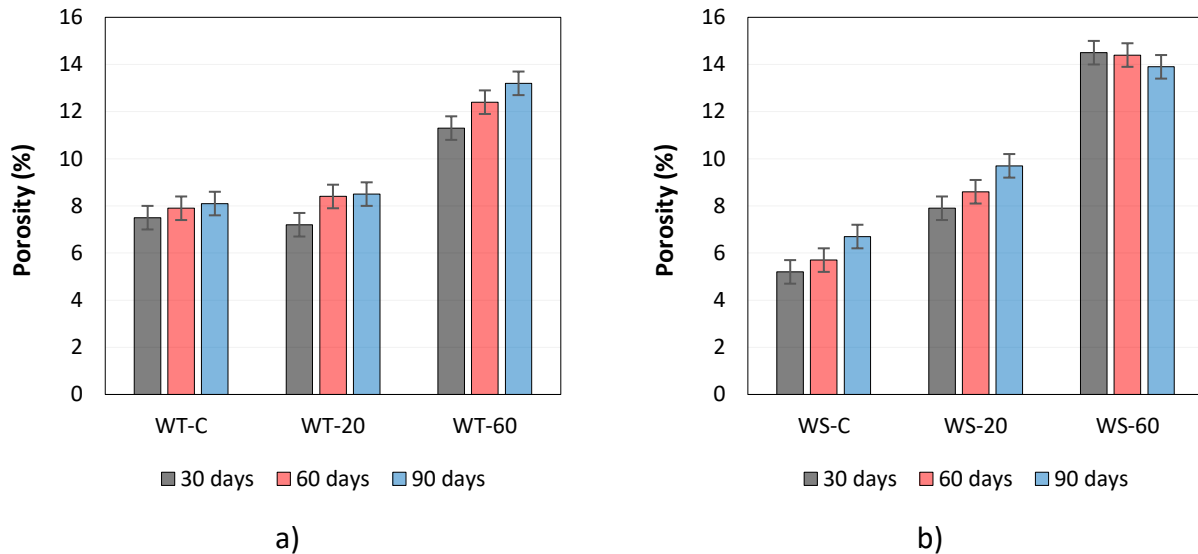
Figure 3.4 shows the specimens' expansions obtained through the ACPT as a function of time; Figure 3.4a illustrates Tx sand results while Figure 3.4b displays SPH data. Differently from the AMBT, the influence of WA on ASR-induced development was ambiguous. For concrete incorporating Tx sand, the use of 20% of WA was observed to yield a faster ASR-kinetics (i.e. induced development) but the same ultimate expansion was reached when compared to control samples. Conversely, at 60% replacement (i.e. WT-60), WA was able to decrease about 25% the expansion of the control mix. On the other hand, mixtures incorporating WA (i.e. both 20% and 60%) with SPH coarse aggregate displayed a much faster ASR kinetics and higher ultimate expansion than the control mix. Moreover, the replacement ratio did not seem to influence on ASR-induced development since mixtures presenting either 20% or 60% WA replacement yielded quite similar expansion results over time. The final expansion of WS-20 and WS-60 were between 0.45% and 0.5% whereas the control mix (i.e. WS-C) reached 0.37% at 90 days.



**Figure 3.4: Accelerated Concrete Prism Test (ACPT) expansion of ASR-affected samples cured at 60°C and 100% of R.H.: a) Tx fine and b) SPH Coarse.**

### 3.5.2. Porosity

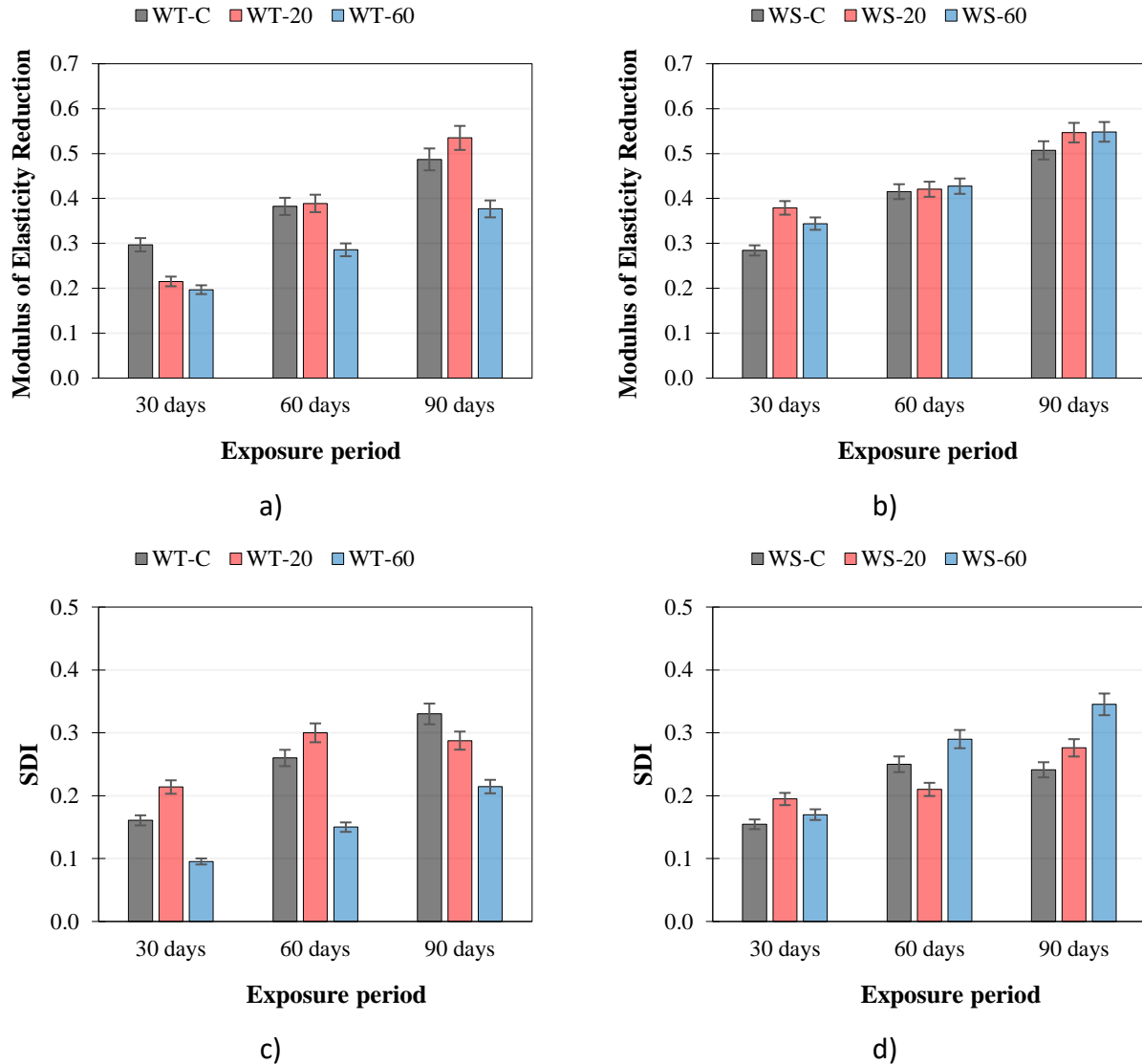
Figure 3.5 displays the porosity obtained from the ASR-affected concrete specimens at 30, 60 and 90 days of expansion test for all mixes containing the two different reactive aggregates. The results indicate that the higher the WA replacement level, the higher the porosity obtained (regardless of the age of test), which is quite expected since PC's replacement was made directly (by mass) without accounting for the distinct PSD of the WA material. The porosity of mixtures incorporating 60% of WA increased 64% and 174% (on average) for concrete mixtures incorporating Texas sand and SPH coarse aggregate, respectively. Furthermore, in general, the porosity of the samples exposed to ASR development increase over time, with the exception of the mixes WS-60 and WT-C.



**Figure 3.5: Porosity of the ASR-affected concrete specimens: a) Polymictic Fine (Tx) and b) Greywacke Coarse (SPH).**

### 3.5.3. Mechanical Properties Assessment

This section evaluates the stiffness damage index (SDI) and the losses in modulus of elasticity (ME) and compressive strength (CS) of the various concrete mixtures investigated. The mechanical data presented hereafter displays the ratio of values obtained from an ASR-induced expansion at 180 days over the values gathered on sound concrete specimens presenting “equivalent maturity” than the damaged samples as previous discussed in 3.2.2.1. Figure 3.6a displays the SDI results for Tx mixtures whereas Figure 3.6b illustrates the results for SPH mixtures; moreover, Figure 3.6c and d (for Tx and SHP aggregates, respectively) show the ME reduction of all ASR-affected concrete specimens.

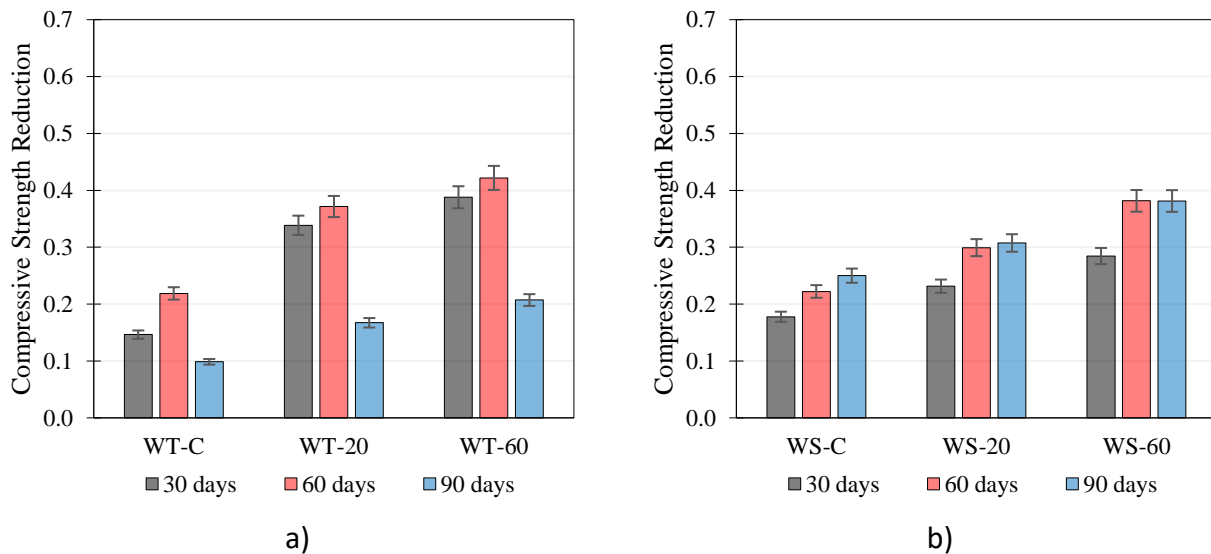


**Figure 3.6: Assessment of the mechanical properties of the concrete samples: Variation ratio in Modulus of Elasticity for a) Tx mixtures and b) SPH mixtures; and Stiffness Damage Index c) Tx mixtures and d) SPH mixtures**

Analyzing Figure 3.6a and b, there is an almost linear increase in the modulus of elasticity loss as a function of time for all mixtures. Furthermore, regarding the Tx sand mixtures, the control and 20% of WA displayed the highest ME loss (i.e. 49% and 53%) respectively, while PC replacement by 60% of WA presents 38% of ME loss at 90 days. On the other hand, the SPH mixtures provide similar ME loss at 90 days for all mixtures: control, 20% and 60% of PC replacement by WA (i.e. 51%, 55% and 55%, respectively).

The SDI results (Figure 3.6c and d) also showed an increase as a function of time for almost all mixtures. Yet, WT mixes presented a decrease in the SDI for higher WA replacement ratios. The WT control mix displayed the highest value (i.e. 0.33) at 90 days, followed by WT-20 and WT-60 (i.e. 0.29 and 0.21), respectively. Conversely, SPH mixes showed a distinct behaviour: an increase in SDI for higher WA replacement levels. The WS control mix presented the lowest SDI value (i.e. 0.24) at 90 days, while WS-60 displayed the highest value (i.e. 0.35).

Figure 3.7a and b give a plot of the CS variation of all ASR-affected concrete specimens. The results demonstrate a clear increase in compressive strength reduction (i.e. decrease in compressive strength) as a function of time for all mixtures. Moreover, WT-C and WT-20 yielded the highest CS losses (i.e. 39% and 42%), while WT-60 presented the lowest CS reduction (i.e. 21%). Furthermore, WS-C yielded the lowest CS loss (i.e. 28%), while WS-20 and WS-60 displayed a similar reduction of 38%.

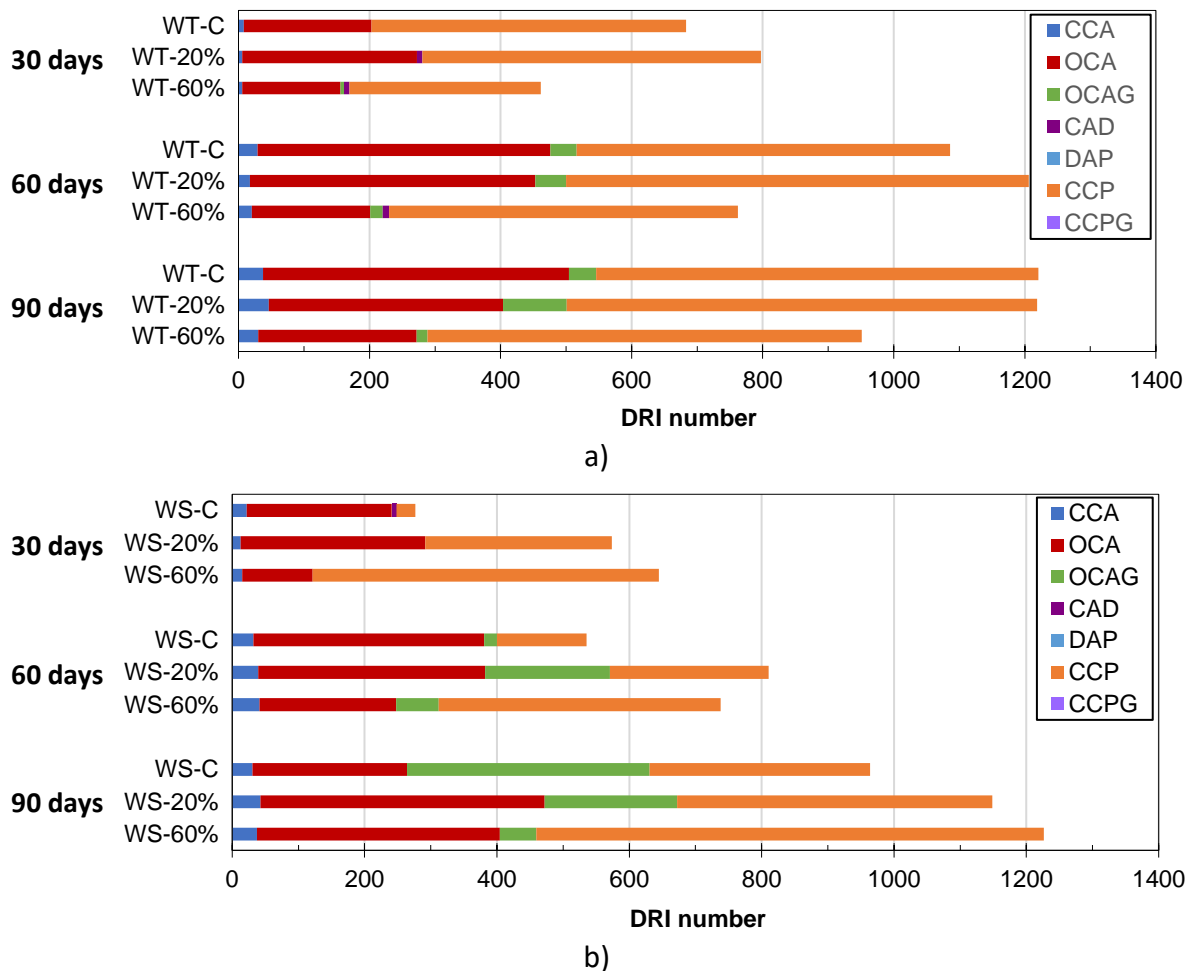


**Figure 3.7: Compressive strength reduction ratio of the ASR-affected concrete specimens: a) Tx mixtures and b) SPH mixtures.**

### 3.5.4. Microscopy Assessment

Figure 3.8 provides the microscopic damage features and DRI numbers obtained from the ASR-affected concrete specimens at 30, 60 and 90 days of test for all mixes containing the two different reactive aggregates a) Tx fine and b) SHP coarse. As expected, the DRI number increased as a function of the exposure period; moreover, a quite high DRI number was observed for mixtures incorporating Texas sand in both control and WT-20% specimens (i.e. 1221 and 1219, respectively).

However, specimens made of PC and 60% of WA yielded lower DRI numbers (i.e. 951) at 90 days. It worth nothing that mixtures incorporating Tx sand showed significant crack numbers within the aggregate particles (OCA, OCAG); yet, most of the cracks were found in the cement paste (CCP). The presence ASR-gel was mostly found within the aggregate particles but increasing WA replacement level, a higher amount of gel was observed within concrete pores. Furthermore, mixtures made of SPH coarse aggregate demonstrated a higher DRI number at 90 days. In other words, the highest the amount of WA for SPH concrete, the highest the DRI value. Therefore, the control mix displayed the lowest result (i.e. 964), while WS-20 and WS-60 yielded higher DRI numbers (i.e. 1149 and 1226, respectively). Moreover, the number of open cracks in aggregates (OCA) was observed to be larger for higher WA replacement levels although the amount of OCAG decreased. Finally, higher number of CCP were displayed for 60% of PC's replacement at 90 days of exposure.



**Figure 3.8: Damage Rating Index of the ASR-affected concrete specimens: a) Tx mixtures and b) SPH mixtures.**

## 3.6. Discussion

### 3.6.1. ASR kinetics and development

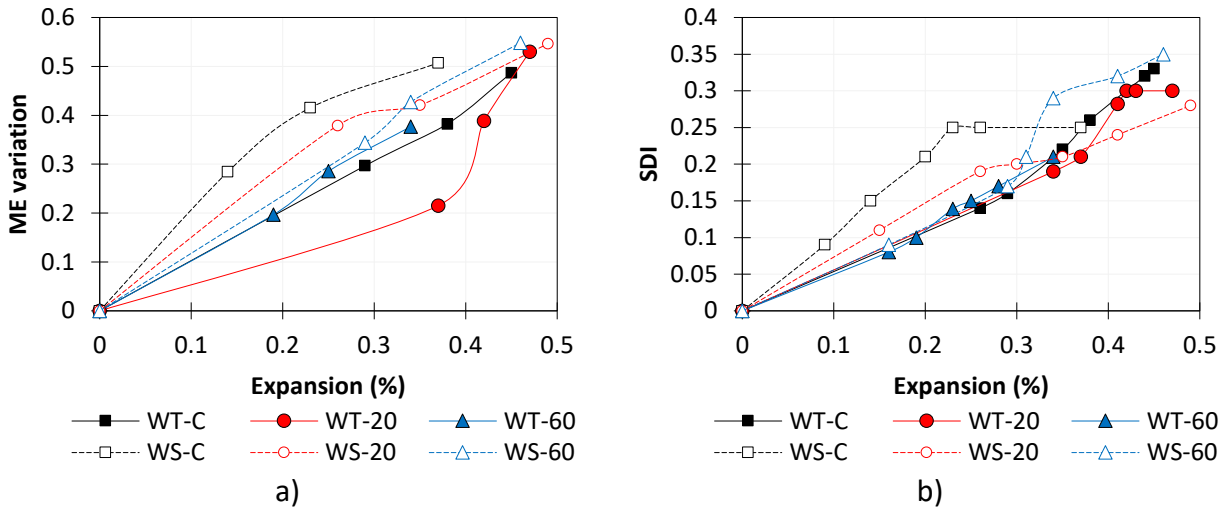
According to the Accelerated Mortar Bar Test – (AMBT), it is clear that replacing PC by wood ash, ASR-induced expansion is lowered; i.e. the higher the replacement level, the lower the ASR-induced expansion. Additionally, replacing 50% of PC by WA, one may notice that Tx reactivity is mitigated down to a non-reactive behaviour as per ASTM C 1260 (and CSA.A23-25A), whereas the mixture proportioned with SPH showed expansion levels slightly above the limit (0.10% at 14 days in solution) but also presented mitigation behaviour as per CSA.A23-25A. These results are consistent with the results found in the literature [15,50,53]. Conversely, specimens tested in the ACPT did not display the same ASR-induced development. It is worth noting that the selection of 60% of PC's replacement by WA in the ACPT was made due to the positive behaviour obtained with 50% replacement in the AMBT (i.e. 50% replacement generated expansion levels slightly above the AMBT reactivity limit for mixtures made of fine and coarse reactive aggregates). Therefore, it has been decided that an increase in the amount of WA in the system might be beneficial to completely suppress ASR-induced development. However, in the case of concrete mixtures made of SPH coarse aggregate, ACPT results indicated a completely opposite behaviour when compared to AMBT; i.e. mixtures incorporating WA presented a higher ASR-induced development than the control mix. However, the amount of WA in the system seemed to not interfere on the induced expansion since quite close results (i.e.  $\approx 0.45\%$ ) were obtained at 90 days for mixtures incorporating 20% and 60% of WA. On the other hand, mixtures made of Tx (known to be more reactive than SPH as per the CPT test) did not display the same behaviour, since mixtures incorporating 20% of WA yielded faster ASR kinetics and similar induced expansion than the control mix (i.e. 0.45%) at 90 days, while the mixtures presenting 60% WA yielded about 25% less expansion (i.e. 0.34) at 90 days. Nevertheless, although WT-60 specimens reached a significantly lower induced-expansion, the results still demonstrate a quite reactive behaviour as per ACPT test, once the expansion threshold is 0.04% at 90 days [82].

The results above emphasize that the reactive behaviour of mixtures made of WA depend on the aggregate type and nature (i.e. fine vs coarse, lithotype, reactivity degree, etc.), since very distinct performance was obtained for the same replacement level while the use of distinct aggregates. Moreover, the results obtained in this work clearly indicate the incompatibility between the AMBT

and ACPT tests for assessing the efficiency of preventive measures to mitigate ASR-induced expansion. Hence, the promising results gathered through the AMBT could simply be a “false negative” behaviour as previously reported in the literature [83,84], Although the AMBT is a very fast, non-expansive and straightforward method, the environment provided by the test is extremely aggressive (i.e. temperature and solution) when compared to other test procedures such as the CPT, ACPT or even field conditions; moreover, while the crushing process, some reactive mineral phases may be lost, which could impact quite significantly on the reactivity of the material assessed. Therefore, the AMBT has been observed to display very often “false-negative and “false-positive” results. In this case, a false-negative result has been gathered very likely due to one of the above conditions. Nevertheless, further research is still required to fully understand the behaviour obtained in this work.

### **3.6.2. Mechanical and microscopic evaluation**

Mechanical and microscopic analyses were performed to assess the extension of damage of the ASR affected mixtures. In general, the results agreed with the expansion levels obtained (i.e. the higher the expansion level, the higher the losses in mechanical properties, displayed in Figure 3.9a and b. Yet, the type and nature of the reactive aggregate showed to play an important role on the reaction kinetics and distress development. Even though the mixtures made of Tx sand developed higher expansion levels than the ones made of SPH coarse aggregate for the control group, the losses in the mechanical properties did not follow the same trend. However, this behaviour was quite expected, since coarse aggregates have more influence on the concrete mechanical properties, particularly on the stiffness or modulus of elasticity. Furthermore, the DRI results presented in Figure 3.8 showed evidences that more open cracks in the aggregates were found in SPH coarse aggregate specimens while the use of WA, which may explain the higher loss of stiffness obtained.



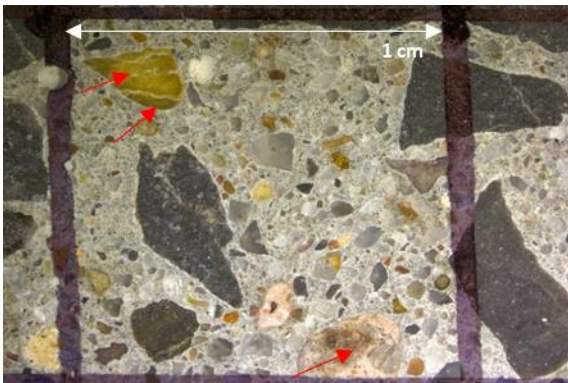
**Figure 3.9: Correlation between the ACPT expansion amplitudes and a) modulus of elasticity and b) stiffness damage index.**

Analyzing Figure 3.9a and b, it is clear that ME losses displayed a “concave” trend of reduction towards ASR development; moreover, even though the coarse SPH demonstrate higher ME reduction over time, similar “reduction trends” were generally observed as function of the expansion level for both reactive aggregates, comparable with results in the literature [59,61]. The only exception is WT-20 which was found to have one of the lowest ME losses at 30 days of exposure; yet, it yielded the highest at 90 days. Furthermore, Figure 3.9b displays a somewhat linear correlation between SDI and expansion.

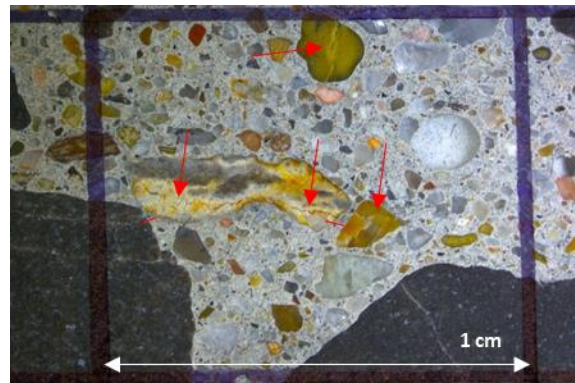
ASR distress development has been widely studied and described by [58,74]. At the beginning of the physicochemical reaction, cracks are formed within the aggregate particles; as the ASR-induced damage raises, new cracks are still developed within the aggregate particles yet, the pre-existing cracks keep increasing in width and length reaching the cement paste. At higher levels of expansion, the cracks keep propagating through the aggregate particles and cement paste and due to the “minimum energy law”, they start to connect to each other forming a high crack networking compromising the mechanical properties of the affected concrete [58,74]. The crack development following the “minimum energy law” is not only restricted to cracks formed by ASR, but also through pre-existing cracks within the particles generated over crushing/weathering processes. For instance, Figure 3.10a (WT-C specimen) reveals several OCA features in the non-reactive coarse aggregate besides the significant increase in the number of cracks generated in the reactive Tx sand. Yet, WT-20 specimen (Figure 3.10b) and WT-60 (Figure 3.10c) indicates a notable decrease in the quality of

the microstructure since more pores were detected and thus thin cracks may have been originated through a different mechanism than ASR (e.g. shrinkage).

Following the minimum energy law, WA particles display low mechanical properties and high porosity which may explain the fact that several cracks propagated through the coarser WA particles. Moreover, it has been observed for both WT-20 and WT-60 specimens a notable number of pores filled by ASR-gel; likewise, most of the cracks were already connected to each other, which indicates that the samples containing WA were further damaged. WS-C (Figure 3.10a), WS-20 (Figure 3.10b) and WS-60 (Figure 3.10c) displayed similar evidences as aforementioned for WT mixtures, yet the reactive coarse aggregate contributed more towards the cracks propagation and distress, as expected.



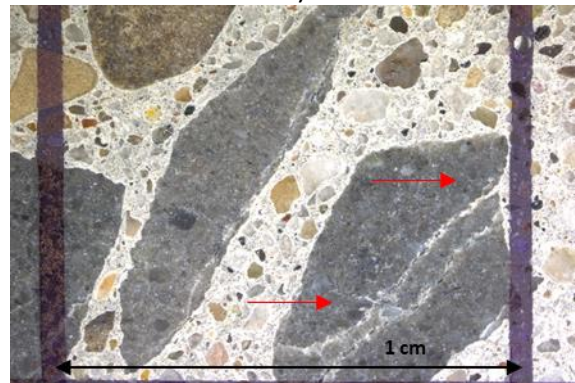
a)



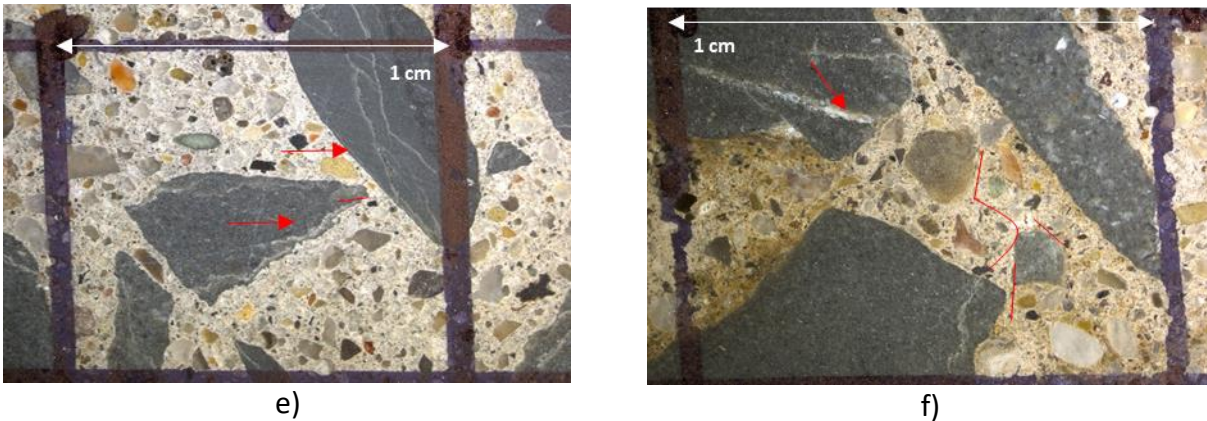
b)



c)



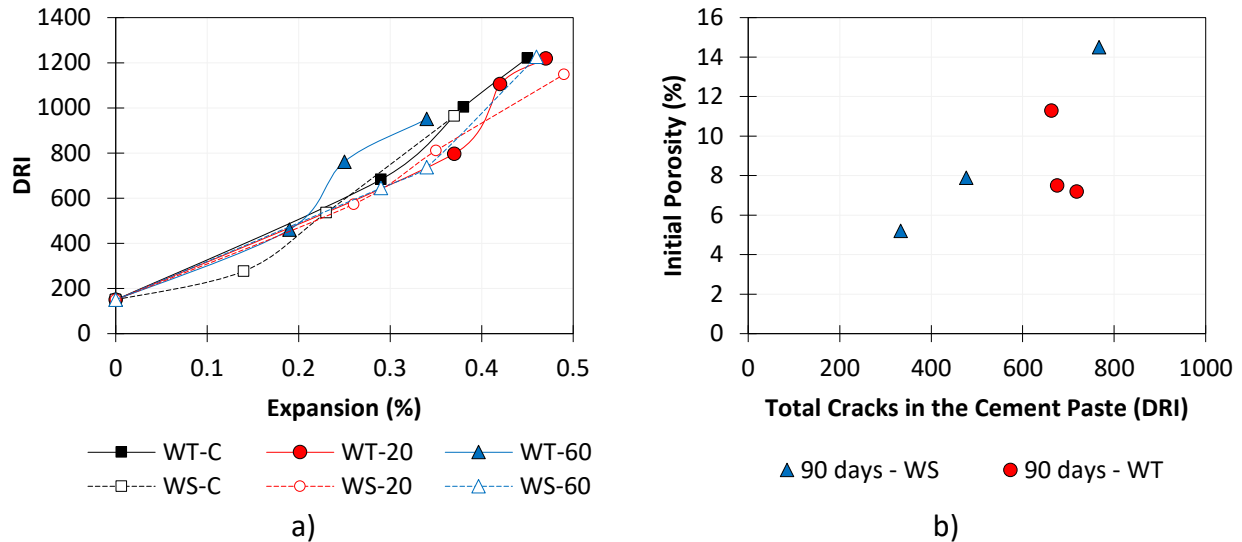
d)



**Figure 3.10: Pictures captured during the DRI test for mixtures incorporating Tx sand: a) WT-C; b) WT-20% and c) WT-60% and SPH coarse: d) WS-C; e) WS-20% and f) WS-60%**

The total number of cracks obtained through DRI was found to increase almost linearly with the measured induced expansions (Figure 3.11a); likewise, it was observed that the DRI also increase proportionally with the increase in PC's replacement, except for WT-60 mix. The latter may be explained by the higher porosity of the material (Figure 3.5), since these empty spaces in the matrix can be filled by gel (Figure 3.10c) decelerating thus the induced expansion process. Moreover, it has been verified an important number of CCP (i.e. cracks in the cement paste) nearby pores filled by gel.

It worth noting that in this study, the specimens were maintained at 60°C over 90 days, which further accelerates ASR kinetics; therefore, it was clear that ASR-generated cracks extended much faster through the cement paste, even at moderate expansion levels. These cracks started building an important crack network at lower expansion levels, which seems to be affected by the WA particle features (i.e. high porosity, angular shape and texture) and the likely lower microstructure quality of the WA made mixtures. The latter suggests that the use of WA slightly changed ASR-induced damage mechanism by fastening the crack propagation through the cement paste (Figure 3.11b). Moreover, it highlights that the mechanical and microscopic behaviours of WA made concrete mixtures are associated to the induced expansion measured in the ACPT; this indicates that further research is still required to better understand the durability behaviour of WA concrete affected by ASR, since the AMBT has showed promising and similar results to the ones currently found in the literature [15,50,53]. Nevertheless, this could be simply another example of a “false negative” behaviour observed through the AMBT.



**Figure 3.11: Correlation between a) ACPT expansion amplitudes and damage rating index values; and b) porosity vs DRI-CCP.**

### 3.7. Conclusions

The main objective of this research was to determine the influence and efficiency of different amounts of WA to mitigate ASR-induced expansion and damage. The main findings of the current research are presented hereafter:

- The accelerated mortar bar test (AMBT) showed that the higher the replacement level of PC by WA, the lower the expansion levels reached by mixtures incorporating highly reactive Tx sand and SPH coarse aggregates. Both Tx and SPH mixtures made with 50% WA replacement were considered either non-reactive as per CSA A23.2-25A. Yet, Tx sand mixture with 50% of WA also presented under the reactivity limit as per ASTM C 1260-1567.
- The results obtained through the ACPT are not in agreement with the results obtained through the AMBT for both aggregates used. The latter seems to indicate an example of a “false negative” result yielded by the accelerated mortar bar test. The ACPT results found for the SPH aggregate seem to be counterintuitive since the higher the amount of WA replacement, the higher the distress obtained. Further analyses are still required to fully understand the results gathered in this work.
- The mechanical properties losses (i.e. compressive strength, modulus of elasticity and SDI) are in agreement with the expansion levels obtained and microscopic analyses conducted

through the DRI method. Although the mechanisms observed while the use of WA seem to change as a function of the aggregate type and may be somewhat counterintuitive, the use of the multi-level assessment clearly indicated that the induced expansion results obtained were correct.

- The preliminary results obtained in this research seem to indicate that WA is not a suitable material to be used as an SCM to mitigate ASR-induced development in concrete. Yet, trials should still be conducted to use such a material as a filler towards a greener future of the construction industry.
- A good linear correlation between porosity and compressive strength was verified in this research confirming that the addition of WA increased significantly the overall porosity of the material; however, no correlation between porosity and expansion was observed.

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## Chapter Four: Influence of Mineral Fillers (MF) on AAR-induced Expansion and Deterioration

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Antunes L.R. 1, Sanchez L.F.M. 1, De Souza D.J. 1

<sup>1</sup>Department of Civil Engineering, University of Ottawa, Ottawa, Canada.

### Abstract

Alkali-aggregate reaction (AAR) is one of the most harmful distress mechanisms affecting concrete infrastructure worldwide. Over the past decades, several approaches, including a variety of accelerated test procedures, have been developed to assess the potential reactivity of aggregates in the laboratory. If on the one hand, these methods are quite established for assessing the reactivity of fine and coarse aggregates, it is still very unclear how to appraise the reactivity and impact of using mineral fillers in the laboratory. This project aims to understand the effect of MF on AAR-induced expansion and deterioration. Concrete specimens containing reactive and non-reactive coarse and fine aggregates were fabricated with four types of fillers (reactive and non-reactive) at a replacement level of 15% using two distinct design approaches (i.e. replacement of PC or fine aggregates). These specimens were stored in conditions enabling AAR-induced development over 180 days and their expansion over time was monitored throughout. At 180, physical (i.e. porosity), mechanical (stiffness damage test, modulus of elasticity and compressive strength) and microscopic (damage rating index) were conducted. Results indicate that mixtures incorporating MF and replacing PC display lower expansion and damage over time. Moreover, the MF type was also deemed to significantly change AAR-induced expansion and damage; granite, greywacke and dolomite fillers showed no or positive impact on AAR-development while limestone fillers worsen the expansion and deterioration process.

**Keywords:** alkali-aggregate reaction, mineral filler, CO<sub>2</sub> mitigation, reactivity.

## 4.1. Introduction

Concrete is likely the most common construction material used in critical infrastructure worldwide as a result of its availability, economic benefits, and outstanding mechanical and durability-related properties. As the global demand for concrete increases progressively around the world, concrete industry currently faces crucial challenges of finding cost-effective strategies to reduce the carbon-footprint and embodied energy associated with the production of Portland cement (PC) [1]. PC is by far the most important ingredient of concrete; moreover, PC production along with the extraction and manufacturing of natural aggregates is the main responsible for the overall concrete carbon-footprint, accounting for about 7% of the annual man-made CO<sub>2</sub> emission [2]. In this context, one of the most commonly adopted methods for reducing the environmental impact of PC involves the use of supplementary cementing materials (SCMs) in concrete. Yet, recent studies demonstrated that their short and especially long-term availability will not be enough to correspond to the rise in PC's demand [3–6]. However, mineral fillers (MF) might become one of the viable solutions due to their large availability [7]. Almost every inorganic mineral with no deleterious reaction with PC can be classified as a filler. Moreover, millions of tons of rock residues are produced throughout the world every year, mostly improperly discarded in the environment. Literature suggests that the incorporation of these residues into cementitious materials may keep (or even improve) their fresh and hardened state performance while increasing sustainability [8–13].

In general, the use of fillers in cement-based materials is a common practice worldwide. Results suggest that MF may enhance hydration due to the formation of supplementary nucleation sites and shearing conditions [14]. Moreover, MF may also increase the packing density of granular systems such as concrete, allowing thus PC reduction without compromising the mechanical properties of the material [3,15]. Apart from cement replacement, fillers can also be used as an alternative to fine aggregates. Furthermore, although MF may derive from a wide range of rock types such as limestone, granite, sandstone, quartz and dolomite, limestone fillers (LF) are the most used materials due to their large availability [7,16].

Different standards (i.e. CSA A3001 & ASTM C595, ASTM C1157, AASHTO M240) allow the use of up to 15% of LF pre-mixed with PC, the so-called GUL cement in Canada (i.e. similar to ASTM type IL). Yet, current standards (i.e. CSA A23.1-14) do not deny the use of other MF types in concrete, but rather indicate their use as a replacement for aggregates to optimize the materials gradation and thus enhance physical and mechanical properties [8–13]. Nevertheless, the long-term

efficiency and durability behaviour of concrete incorporating distinct types of MF are still not fully understood, particularly when evaluating the performance of reactive aggregates in the laboratory.

## **4.2. Background**

### **4.2.1. The use of fillers in concrete production**

MF started to be used in the concrete industry due to economic reasons, where PC was initially replaced by different types of powder materials such as granite and sandstone [29]; nowadays, limestone filler (LF) is the most widely used MF worldwide. In early 1980s, PC replacement by LF was introduced into cement standards. The first standard to adopt the use of LF was the Canadian CAN3-A5-M83 in 1983, which limited its use to 5% [30]. Hereafter, by 1988 in Europe, this limit has been raised to 20% in Portland-filler cements [30]. Currently, several national and international standards include fillers for cement production [31]; hence, important developments on its usage may be currently found in the literature [16,32–34]. Nevertheless, most of the countries are restricted to the use of LF; some current standards allow the use of general cement with limestone (GUL) as Portland-limestone cement with a limit of up to 15% by mass (e.g. ASTM C595 / CSA A3000-18), while others enable the use of MF other than LF in smaller replacement ratios (i.e. 5% in the European standard EN 197-1) [8,9,11,35–37]. Although it has been demonstrated that concrete mixtures incorporating the above MF limits may display suitable fresh [26–31] and hardened state [32–34] properties, much less has been discussed in the literature on the long-term performance of systems containing MF.

### **4.2.2. Alkali Aggregate Reaction (AAR)**

Alkali–aggregate reaction (AAR), a harmful chemical reaction between certain mineral phases of the aggregates and the alkali hydroxides of the concrete pore solution [38]. Overall, AAR can be divided in two main reaction types: alkali–silica reaction (ASR) and alkali-carbonate reaction (ACR). ASR is by far the most common reaction type found worldwide, and its distress mechanism is fairly well understood, at least in its major steps. Alkali-silica reaction (ASR) consists of a chemical reaction between “unstable” silica mineral forms within fine and/or coarse aggregate materials and the alkali hydroxides (Na, K – OH) that are dissolved within the concrete pore solution [26,28,39]. It generates a secondary product, the so-called alkali-silica gel, that induces expansive pressure within the reacting aggregate material and adjacent cement paste upon moisture uptake, leading to microcracking, loss

of material's integrity and functionality of the affected structure [26,28,39,40]. ASR distress degree and features depend upon the type (i.e. fine and coarse aggregate) and reactivity of the aggregates used, the amount of alkalis of the concrete, the temperature and relative humidity of the environment [40–46]. On the other hand, ACR is a much less common concrete distress whose mechanism is still under debate, being considered as a form of ASR by some authors [47,48], while other researchers believe that ACR follows a “different” distress mechanism [49,50]

Over the past decades, several approaches and recommendations, including a variety of accelerated laboratory test procedures (especially the accelerated mortar bar test - AMBT as per ASTM C1260/CSA A23.2-25A and the concrete prism test - CPT as per ASTM C1293/ CSA A23.2-14A), have been developed to assess the potential reactivity of aggregates in the laboratory as well as the efficiency of preventive measures (i.e. the use of supplementary cementing materials- SCMs) to mitigate ASR in the field [17–26]. However, there is currently a lack of literature data on the influence of MF on AAR-induced expansion and deterioration. Few studies have suggested that some types of -reactive mineral fillers (i.e. rhyolite) may be beneficial to decrease ASR-induced development (reducing both the expansion rate and level), particularly when MF is used to replace PC [27]. Yet, depending on the physicochemical proprieties (i.e. shape, texture and particle size distribution), mineralogy and chemical composition (e.g. presence of alkalis), and reactivity, the influence of MF on AAR-induced development might be completely different and could increase the potential of AAR-induced deterioration [17,25,28]. Moreover, the likely distinct impact of replacing PC or fine aggregates by MF on AAR-induced progress is still not completely understood. All the above attests the need of further research in the field.

### **4.3. Tools for ASR assessment**

#### **4.3.1. Stiffness Damage Test (SDT)**

A full review on the SDT development as a diagnostic tool for assessing ASR affected concrete is presented in [101]. Sanchez et al. [89,94,102] verified that the SDT should be performed through five loading/unloading cycles using a percentage (i.e. ideally 40%) of the design (28-day) compressive strength of the specimen under analysis, instead of using a fixed load as per [101,103]. Moreover, the method was deemed very promising for assessing AAR-induced damage and progress in concrete, especially when indices are used as output parameters of the test, namely Stiffness Damage Index (SDI) and Plastic Deformation Index (PDI); the latter represents respectively the ratio of dissipated

energy/plastic deformation to the total energy/deformation implemented in the system [91]. Moreover, the modulus of elasticity and the non-linearity index (NLI) were also verified as suitable output parameters of the test to appraise ASR-induced progress.

### **4.3.2. Damage Rate Index (DRI)**

The DRI is a petrographic analysis performed with the use of a stereomicroscope (15–16× magnification) where damage features generally associated with ASR are counted through a 1 cm<sup>2</sup> grid (i.e. 10 × 10 mm units) drawn on the surface of polished concrete sections [90]. The number of counts corresponding to each type of petrographic features is then multiplied by weighing factors, whose purpose is to balance their relative importance towards the mechanism of distress, for instance ASR [90]. The factors used in the method were selected on a logical basis, but relatively arbitrarily; they were recently modified in order to reduce the variability between the petrographers performing the test [104]. Ideally, a surface of at least 200 cm<sup>2</sup> should be used for DRI analysis, and it may be greater in the case of mass concrete incorporating larger aggregate particles. However, for comparative purposes, the final DRI value is normalized to a 100 cm<sup>2</sup> area [105].

The goal of DRI is to appraise the damage “degree or extent” of affected concrete, complementing conventional petrographic analysis whose main purpose is to detect the “cause” of deterioration. Moreover, presenting a direct correlation with the induced expansion level, a damage scale to distinguish low, moderate and high damage levels caused by ASR was proposed by [90,102]. Likewise, similar results were presented by [106,107] assessing cores extracted from ASR-affected structures.

## **4.4. Scope of the work**

As stated above, several techniques and methods were well established over the years to understand and characterize ASR-induced expansion and damage in the laboratory. However, the influence of fillers (i.e. reactive or not) on the expansion behaviour of concrete is not fully understood nor well studied. Furthermore, there is currently a lack in the literature on protocols (e.g. replacing fine aggregates or PC) to appraise the impact of fillers on reactive systems, which prevents a proper evaluation of the reactivity of concrete mixtures containing MF. The proposed research aims to understand the effect of MF on ASR-induced expansion and development. Concrete mixtures containing reactive and non-reactive coarse and fine aggregates will be fabricated with four types of

MF (from alkali-reactive and non-reactive rocks) at a replacement level of 15% (maximum allowed by CSA A3001 & ASTM C595) of either fine aggregates or PC. Concrete prism tests (CPT) will be performed over one year and chemical (i.e. X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD)), mechanical (i.e. Stiffness Damage Test, modulus of elasticity and compressive strength), microscopic (i.e. Damage Rating Index (DRI)) and porosity evaluations will be conducted at selected time periods to understand the impact of distinct fillers on ASR-overall development.

## **4.5. Materials and Methods**

### **4.5.1. Raw materials**

A conventional Portland cement (GU type, equivalent to ASTM Type I), four different types of MF (crushed greywacke – (SH), limestone – (O), granite – (F) and dolomitic argillaceous limestone – (K)) and two highly reactive coarse (Springhill) and fine (Texas) aggregates were selected for this study to manufacture sixteen concrete mixture with a 0.45 water-to-fines ratio (i.e. amount of water divided by the amount of PC + fillers, in mass). The coarse aggregates ranged from 5 to 20 mm in size. Non-reactive fine (NF) and coarse (NC) aggregates were used in combination with the reactive aggregate materials for concrete manufacturing. Table 4.5 provides information on the different aggregates and MF used in this study. It is worth noting that the fillers SH and K were manufactured by crushing, sieving and selecting aggregate particles below 150 $\mu$ m, while the limestone filler (O) and granite filler (F) were acquired directly from suppliers. Moreover, the greywacke and dolomitic argillaceous limestone aggregates used to produce SH and K fillers, respectively, are known as highly reactive aggregates to the development of alkali-silica reaction (ASR) and alkali-carbonate reaction (ACR) in concrete. Table 4.6 displays the chemical composition of the PC and filler materials used in this research.

**Table 4.5: Reactive (R) and non-reactive (NR) materials used in the research**

Aggregate	Reactivity		Rock Type	Specific gravity	Absorption (%)	AMBT <sup>a</sup> (%)
Coarse	NC	NR	High-purity fine-grained limestone	2.79	0.70	0.01
	SPH	R	Crushed Greywacke	2.73	0.71	0.26
Fine	NF	NR	Natural derived from granite	2.67	0.75	0.09
	Tx	R	Polymictic sand (granitic, mixed volcanic, quartzite, chert, quartz)	2.63	0.79	0.85
Filler	SH <sup>b</sup>	-	Crushed Greywacke	2.73	-	-
	O	-	Calcium carbonate (limestone)	2.66	-	-
	F	-	Granite (quartz, feldspar, mica)	2.70	-	-
	K <sup>c</sup>	-	Dolomitic argillaceous limestone	2.69	-	-

<sup>a</sup> Results at 14 days of curing of the accelerated mortar bar testing (ASTM C 1260) performed on the aggregates selected for this research.

<sup>b</sup> Manufactured by crushing SPH aggregate;

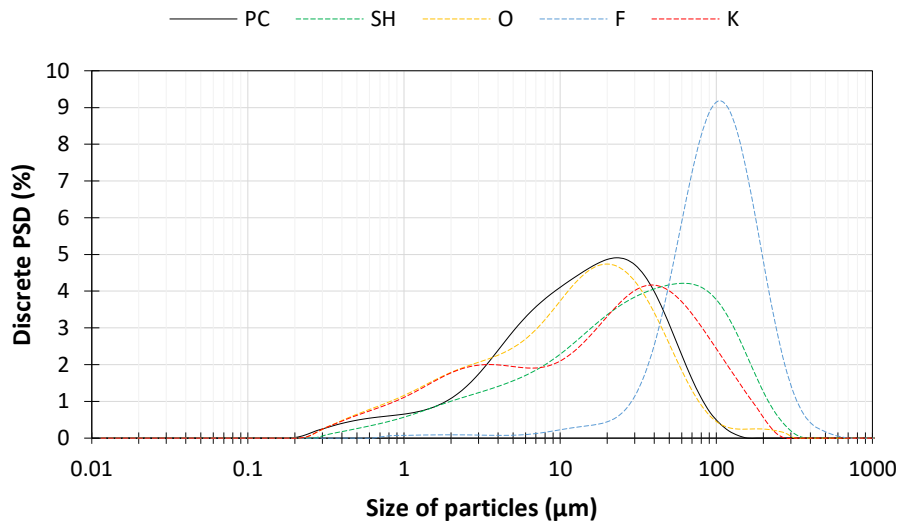
<sup>c</sup> Manufactured by crushing a known alkali-carbonate reaction aggregate [46].

**Table 4.6: Chemical composition of the cement and fillers by XRF analyse**

Chemical Composition	Cement	SH	O	F	K
<i>CaO</i>	61.93	5.21	52.28	3.09	41.51
<i>SiO<sub>2</sub></i>	20.1	60.45	4.09	65.93	9.47
<i>Al<sub>2</sub>O<sub>3</sub></i>	5.03	12.17	0.23	15.17	2.66
<i>Fe<sub>2</sub>O<sub>3</sub></i>	3.80	5.21	0.15	4.62	0.9
<i>MgO</i>	2.42	3.50	1.69	1.70	5.48
<i>K<sub>2</sub>O</i>	0.92	2.67	0.07	3.8	0.82
<i>Na<sub>2</sub>O</i>	0.3	1.41	0.04	3.69	0.17
<i>SO<sub>3</sub></i>	3.37	0.30	-	-	0.33
<i>C<sub>3</sub>S</i>	43.24	-	-	-	-
<i>C<sub>2</sub>S</i>	25.01	-	-	-	-
<i>C<sub>3</sub>A</i>	6.90	-	-	-	-
<i>C<sub>4</sub>AF</i>	11.56	-	-	-	-
<i>Na<sub>2</sub>O<sub>eq.</sub></i>	0.95	3.17	0.09	6.1	0.71
<i>LOI</i>	2.91	8.25	41.34	0.81	38.58

The particle size distribution (PSD) of the PC and fillers were obtained through laser diffraction spectroscopy (Figure 4.12). The limestone filler (O) shows the smallest PSD (D50 of 12 mm), yet, quite similar to PC (D50 of 13 mm). The fillers K, SH and F displayed average PSD (i.e. D50) of 19 mm, 30 mm and 95 mm, respectively, all coarser than PC. The latter indicates that the use of these materials (i.e. SH, F and K) have the potential to enhance concrete packing, since they may fill the gaps in between the cement particles and the fine aggregate particles. Moreover, X-Ray Diffraction - XRD has been conducted on the filler materials (Figure 4.13). The predominant minerals phases of SH (Figure 4.13a) were identified as quartz, orthoclase, illite, albite and kaolinite. Similar

predominant minerals phases were also observed on F samples (Figure 4.13c), the exception was kaolinite; yet, bytownite and muscovite were identified. The diffractograms of O and K (Figure 4.13b and Figure 4.13d, respectively) display major peaks of calcite, which was expected; furthermore, due to the higher MgO content seen in Table 4.13, it was identified pronounced peaks of dolomite. Besides the predominant minerals phases, it was calculated the relative crystallinity/amorphous portion through the XRD analysis using the Frost et al. [108] method through baseline correction and subtraction of amorphous background scattering from the coherent crystalline scattering. The data obtained indicates that the fillers SH, F and K have relative crystallinity of 78.12%, 81.35% and 84.93%, respectively, while O displays 91.64%. It is worth noting that this information may indicate that materials containing quartz, cristobalite, kaolinite and illite may present a slightly pozzolanic reactivity, although not enough to be classified as supplementary cementing materials.



**Figure 4.12: Particle size distribution of the PC and filler materials**



**Table 4.7: Concrete mix proportions.**

Group ID <sup>a</sup>	Fillers	Replacement (%)	kg/m <sup>3</sup>				
			Filler	FA	CA	PC	Water
Control - SPH	-	-	-	781	993	420	189
Control - Tx	-	-	-	781	993	420	189
15 - S	SH	15	63	719	993	420	189
15C - S <sup>b</sup>	SH	15	63	773	993	357	189
15 - O	O	15	63	719	993	420	189
15C - O <sup>b</sup>	O	15	63	773	993	357	189
15 - F	F	15	63	719	993	420	189
15C - F <sup>b</sup>	F	15	63	773	993	357	189
15 - K	K	15	63	719	993	420	189

<sup>a</sup> Besides control mixes, all other mixtures should be considered twice, either with Tx or SPH as reactive aggregates.

<sup>b</sup> C – Indicated cement replacement, otherwise is consider sand replacement.

The specimens were demoulded after 24 h of casting. Small holes, 5mm in diameter by 15mm long, were drilled at both flat ends of the samples. Then, studs were glued in place, with a fast-setting cement slurry in order to measure the longitudinal expansions. Next, the specimens were moist cured for over 24 h. After 48h from casting, the “zero” reading was recorded, and the cylinders were placed in sealed buckets (4 cylinders per bucket) and then stored at 38°C and 100% RH. The cylinders were monitored for length changes over time (i.e. 30, 90, and 180 days); however, ongoing analyzes will be performed up to 360 days. As per ASTM C1293, the buckets were cooled at 23°C for 16 ± 4h prior to the axial measurements. When the above selected periods were reached, the specimens were wrapped in plastic films and stored at 12°C until testing (because of testing capacity limitations). In order to perform each test selected (i.e. mechanical, microscopy and porosity), the samples were conditioned/prepared as per following section.

### 4.5.3. Test methods

#### 4.5.3.1. Porosity

According to the Archimedes immersion method, the porosity of the concrete samples was evaluated. Every 30 days of exposure to 60°C and 100% RH, one sample per concrete mixture was taken and cut into three similar slices (i.e. about 3 cm high). Afterwards, the sliced samples were dried at 60°C during a 10-day period and weighted to obtain their dry mass ( $M_D$ ). Next, the samples were fully immersed in water under a constant pressure of 28-30Hg over twenty-four hours, and then wiped to

remove their surface moisture and weighted in a high precision scale to record their wet mass ( $M_{SSD}$ ). Finally, in a hydrostatic scale, the submerged mass ( $M_s$ ) was recorded. Thus, the apparent porosity could be calculated through the following equation 1. The results presented in this work are the average of three concrete slices.

$$AP = \frac{M(ssd) - M(d)}{M(ssd) - M(s)} \quad \text{Equation 4.2}$$

#### **4.5.3.2. Compressive Strength Test**

Compressive strength was conducted on the samples with two different and specific goals. First, the 28 days compressive strength of each mixture was obtained considering the maturity concept as per ASTM C 107; i.e. three samples per mixture were placed at 12°C for 47 days according to the maturity concept to obtain the ultimate capacity of the mixtures designed. Second, the compressive strength was determined in two out of three specimens from each concrete mixture after being subjected to SDT (5.3.3), with the aim of verifying the compressive strength reductions as a function of ASR development. This procedure was adopted and considered valid after Sanchez et al. [40,66] confirmed the non-destructive character of the SDT.

#### **4.5.3.3. Stiffness Damage Test (SDT)**

The SDT procedure was performed as per Sanchez et al. [42,44,46], i.e. using five loading/unloading cycles at a controlled loading rate of 0.10MPa/s with a maximum load corresponding to 40% of the 28-day compressive strength (i.e. obtained for the distinct mixtures following ASTM C 107 – see 5.3.2). Moreover, before the SDT was conducted, the samples were unwrapped, and expansion readings were taken to confirm that no outstanding expansions took place during the storage period. Afterwards, the specimens were re-conditioned under 100% R.H. for 48 hours as per CSA A23.2-14A and ASTM C1293 according to [74]. Finally, the samples were stiffness damage tested and the results presented hereafter are the average values corresponding to two specimens from a given mix at a specific time period.

#### **4.5.3.4. Damage Rating Index (DRI)**

After being removed from the storage place at 12°C, the samples were conditioned 23°C over 48 hours, therefore, the Damage Rate Index was performed on one specimen from each concrete mixture

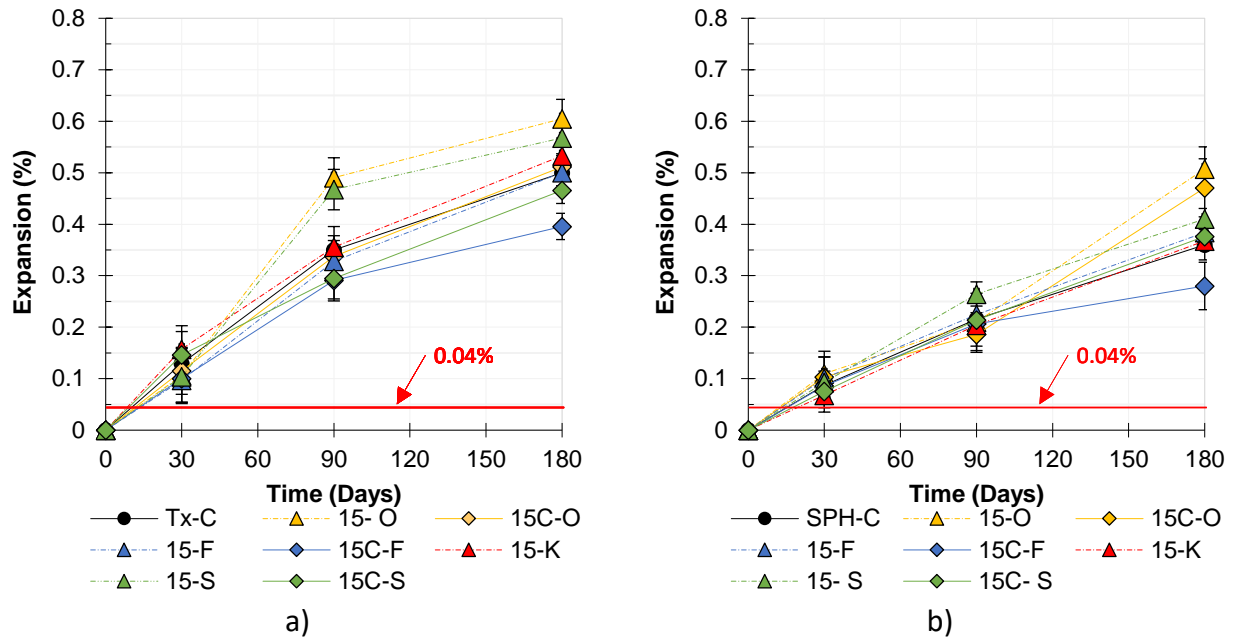
as per Sanchez [90,105]. The samples were cut in half axially, polished by a hand polishing device, which uses diamond-impregnated rubber disks (nº. 50 (coarse), 100, 400, 800, 1500 to 3000 (very fine)) and 1cm<sup>2</sup> grids were drawn on the surface of the polished sections. Yet, was performed the counting cracks by a stereomicroscope (16× magnification) [107]. Finally, DRI final number presented hereafter is the normalized 100 cm<sup>2</sup> value.

## **4.6. Results**

### **4.6.1. ASR-induced development**

In this section, ASR-induced expansion over time is presented for all mixtures developed in the laboratory with different amounts of MF and replacement methods (i.e. replacement of aggregates or PC, in mass) as presented in section 4.2. Figure 4.14 displays the results obtained through the concrete prisms test; Figure 4.14a presents the results of mixtures incorporating the natural Tx fine aggregate while Figure 4.14b shows data from mixtures made of crushed SPH coarse.

In general, Tx mixtures show a faster expansion rate than those containing SPH aggregates. The highest expansion levels obtained over 180 days were 0.61% for Tx mixtures incorporating limestone filler (15-O, replacing fine aggregates) and 0.52% for SPH mixes incorporating the same replacement level, filler type and replacement method (i.e. 15-O replacing fine aggregates, respectively). Furthermore, all mixes displayed higher induced expansion than 0.04% (threshold to classify reactive and non reactive behaviour as per ASTM C1293) regardless of the reactive aggregate used; yet, it is clear that the use of MF have changed ASR-induced kinetics and final expansion of the distinct mixtures. Likewise, the replacement method (i.e. fine aggregates or the cement) was also relevant, since mixtures presenting 15% replacement of PC by MF yielded lower expansion levels. Otherwise, replacing the fine aggregates seems to accelerate ASR-induced development, even for mixtures where the reactive fine aggregate was replaced (i.e. Tx).



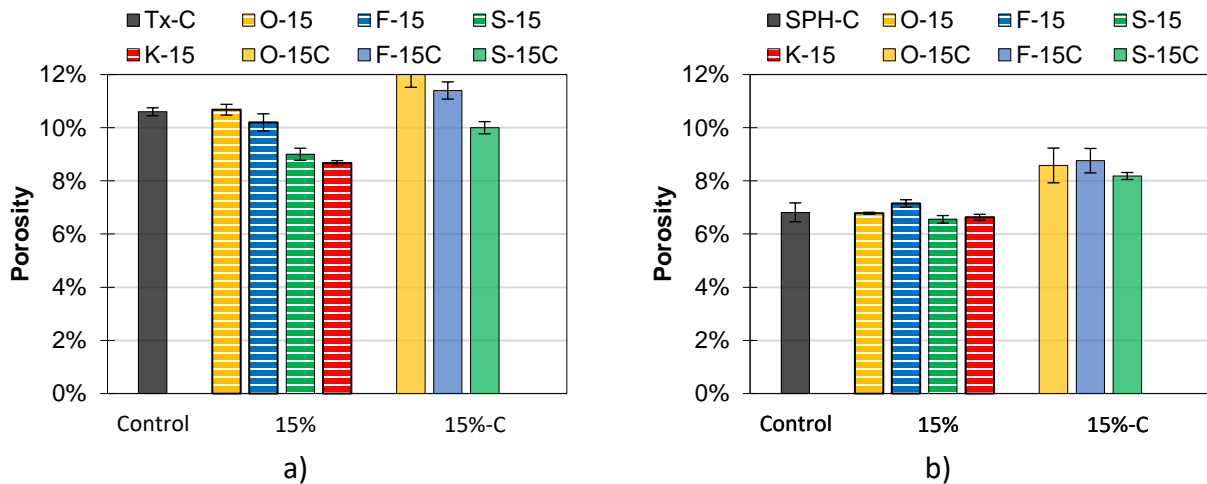
**Figure 4.14: Accelerated Concrete Prism Test (ACPT) expansion of ASR-affected samples: a) Tx fine and b) SPH coarse.**

Regarding the different MF types used, the mixtures containing limestone filler (O) displayed higher expansion levels regardless of the replacement procedure adopted for mixtures incorporating SPH (i.e. 0.52% and 0.47% for 15-O and 15C-O, respectively). Likewise, 15-O Tx mixtures developed faster ASR kinetics than the other mixtures, while 15C-O yielded similar expansion than the control mix. On the other hand, the samples containing crushed granite filler (F) incorporating both reactive aggregates (i.e. SPH and Tx) presented lower expansion levels than the control groups (i.e. SPH-C and Tx-C). Specimens incorporating SPH and having 15% of PC replacement by granite fillers presented 0.28% of expansion (28% lower than SPH-C), while Tx samples bearing the same fillers yielded 0.40% of expansion (20% lower than Texas sand control). However, it was not possible to notice important expansion differences between mixtures incorporating granite fillers replacing fine aggregates and their respective control mixes for both SPH and Tx aggregates. Likewise, 15-K, 15-S and 15C-S mixtures developed similar expansion levels than their respective controls made of SPH and Tx control (although both MFs (i.e. K and S) were produced by crushing two highly reactive aggregates).

#### 4.6.2. Porosity

Porosity is closely linked to the quality of concrete microstructure. Figure 4.15 provides the porosity results of the ASR-affected concrete specimens after 180 days of exposure for all mixes (Figure 4.15a

- Tx mixtures and Figure 4.15b - SPH mixtures). In general, concrete manufactured with Tx presented higher porosity than SPH (on average). The highest values measured after 180 days of exposure were 12% and 11.4%, achieved by the mixtures O-15C (PC's replacement by limestone filler) and F-15C (PC's replacement by granite filler), respectively. It is important to highlight, that the water-to-cement ratio was slightly different for mixtures displaying distinct replacement approaches (i.e. PC vs fine aggregates) since the total amount of water per m<sup>3</sup> was kept constant to simulate what would likely be done in practice while the use of limestone cements (i.e. GUL). Yet, S-15C mixtures (using an alkali-reactive filler) displayed porosity of 10%, which is 6% lower than the control Tx-C (10.6%). On the other hand, replacing the fine aggregates by the distinct MFs kept the microstructure similar to the control groups regardless of the reactive aggregate used (i.e. Tx or SPH). This behaviour was quite expected, since the water-to-cement ratio was kept constant for all sand replacement mixes and control groups, following the recommendation of CSA A3001 and ASTM C595. The concrete mixtures made of SPH displayed generally lower porosity than Tx mixtures; however, the influence of the MFs and different replacement methods on the porosity results were similar to Tx mixtures. Finally, the mixtures O-15C and F-15C showed higher porosity than the controls, whereas the mixtures made of reactive fillers (i.e. K and S) displayed lower porosity values (both with 6.6%).



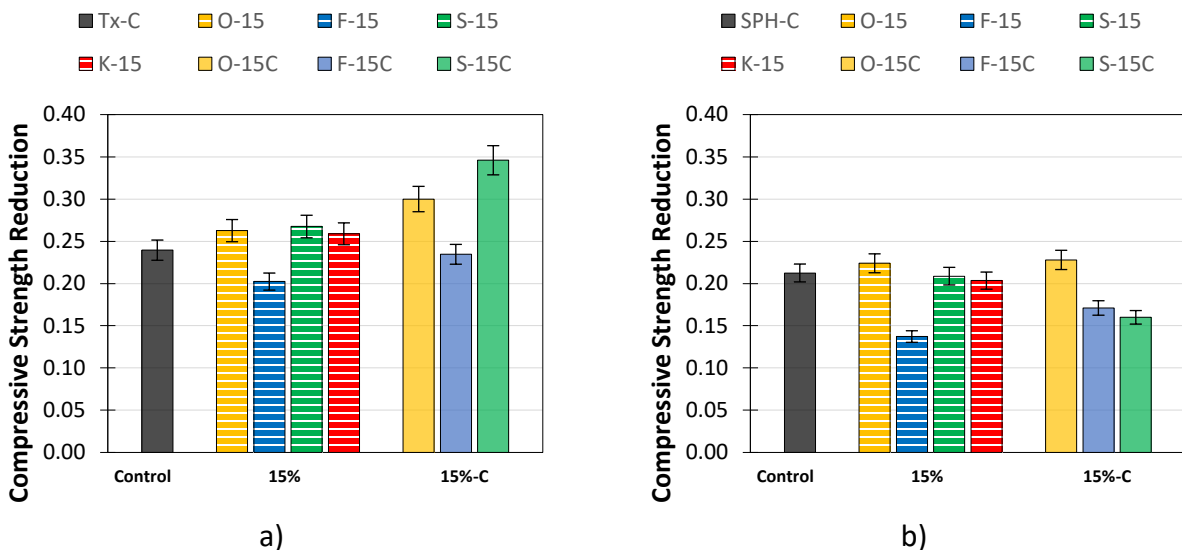
**Figure 4.15: Porosity results – a) Tx mixtures and b) SPH mixtures.**

### 4.6.3. Mechanical Properties Assessment

This section evaluates the compressive strength (CS) reduction, stiffness damage index (SDI) and the losses in modulus of elasticity (ME) of the various concrete mixtures investigated. The SDI values presented herein are the average of three specimens at 180 days for the distinct concrete mixtures while the CS and ME results are represented by the average loss of three specimens (i.e. ratio between

the values gathered at 180 days and the 28-day values) of the different mixtures. It is worth noting that the compressive strength and modulus of elasticity values were obtained at 28 days as per the maturity concept discussed in 2.4.4.2.

Figure 4.16 provides the CS losses obtained for all mixtures. Overall, all mixtures containing Tx sand (Figure 4.16a) obtained higher CS losses than the SPH coarse aggregate mixes (Figure 4.16b). The highest CS loss obtained over 180 days were 35% for Tx mixtures incorporating greywacke filler (15C-S, replacing cement) and 23% for SPH mixes incorporating 15C-O (replacing the cement). Furthermore, Tx-C presented similar losses (i.e. about 25%) than the mixes O-15, S-15 and K-15 (sand replacement mixtures) while the use of granite filler as a sand replacement (F-15) yielded slightly lower losses (i.e. 20%). Nevertheless, for mixtures incorporating Tx sand and evaluating both replacement method (i.e. fine aggregates or cement) the compressive strength losses were more significant (on average) for mixtures that PC was replaced by MF. On the other hand, SPH mixtures yielded a quite different behaviour when compared to Tx mixtures since mixtures designed with the sand replacement approach displayed similar CS losses than the ones proportioned with the cement replacement method. Moreover, while the CS losses for O-15 (22%), S-15 (21%) and K-15 (20%) were close to what was achieved by SPH-C (21%), the reduction of F-15 was lowest among all mixtures, with only 14% of loss.

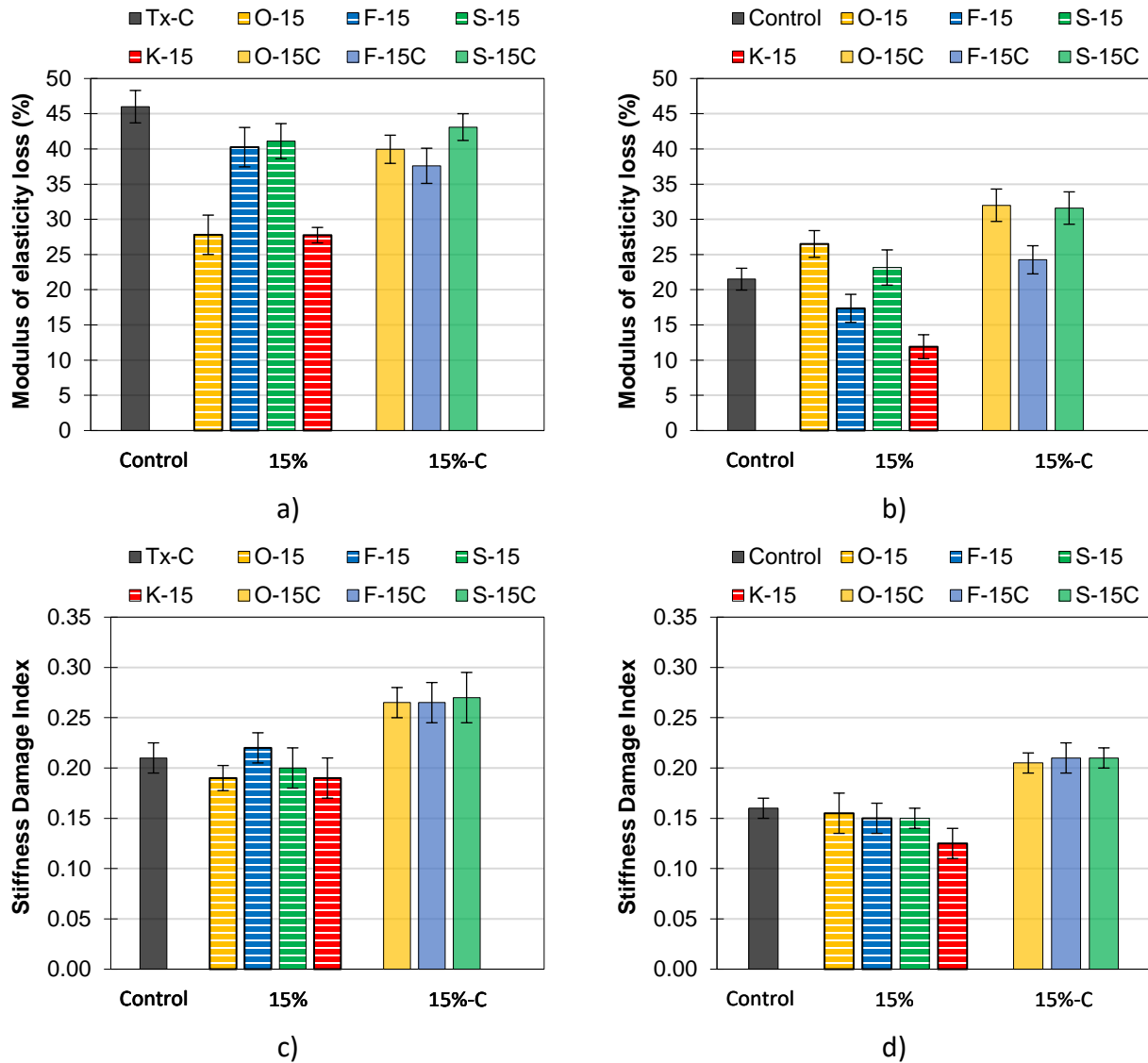


**Figure 4.16: Compressive strength reduction – a) Tx mixtures and b) SPH mixtures**

Figure 4.17a) and b) display the modulus of elasticity (ME) loss for Tx sand and SPH respectively. Moreover, Figure 4.17c) and d) (Tx and SPH aggregates) show the stiffness damage index (SDI) of

the AAR-affected concrete specimens after 180 days of ASR-induced exposure. Verifying the data, one observes that the damage displayed by the Tx mixtures was more pronounced than for SPH mixes, regardless of the test procedure used to measure damage (i.e. ME or SDI). Moreover, in general, the mechanical properties measured through the ME and SDI indicate that the PC replacement results in higher overall values (i.e. higher ME losses and SDI values), compared to the sand replacement. For concrete made of Tx sand, the highest ME losses obtained for the sand replacement approach were for mixtures F-15 and S-15 (41%), followed by O-15 and K-15 mixtures (i.e. about 28%). Yet, S-15C (cement replacement method) exhibited the higher ME loss among all mixtures containing MF with a 43% loss. For the mixtures incorporating limestone and granite fillers the obtained ME losses were 40% and 37%, respectively. Furthermore, for SPH mixtures, the sand replacement method resulted in a quite similar or lower results than SPH-C. While O-15 and S-15 presented 26% and 23% of ME losses (23% and 8% higher than SPH-C), F-15 and K-15 displayed 17 and 15%, respectively (26% lower than the SPH-C). Comparable behaviour was observed in cement replacement, while O-15C and S-15C showed higher results (both with 32% of ME losses, 49% higher than SPH-C), while granite filler (F-15) samples yielded 23% of ME loss.

Overall, the SDI results obtained a similar trend for both reactive aggregates mixture (Tx sand and SPH coarse). For Tx mixes designed with the fine aggregate replacement method, the SDI results varied from 0.19 to 0.21. Furthermore, all Tx mixtures in which the PC was replaced by the MF reached similar SDI results of around 0.27. Overall, the SDI results obtained for SPH mixtures were quite similar to the ME outcomes. Therefore, while sand was replaced by MF, O-15, F-15 and S-15 mixtures yielded SDI results of around 0.15, similar to the SPH control, and K-15 mixtures displayed a slightly lower SDI value of 0.12, indicating a lower induced damage. The cement replacement by the fillers provided higher SDI values; however, the results were similar amongst each other (around 0.21).

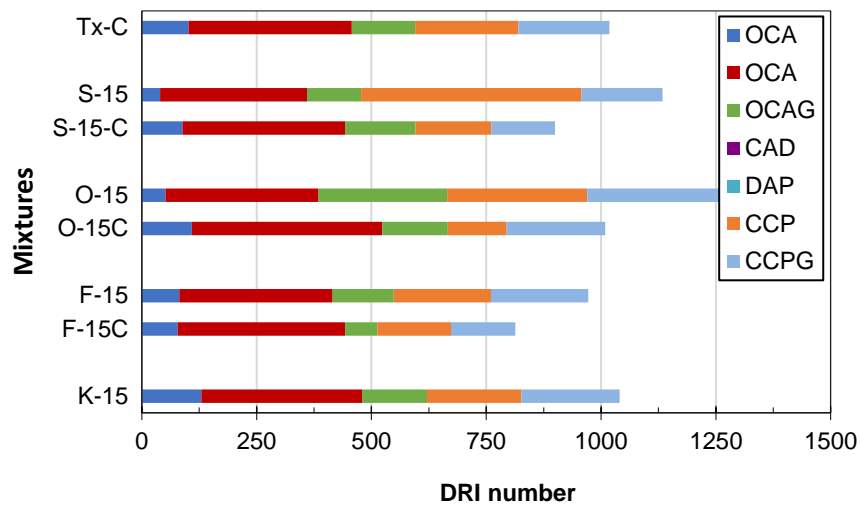


**Figure 4.17: Assessment of the mechanical properties of the concrete samples: Variation percentage in Modulus of Elasticity for a) Tx sand mixtures and b) SPH mixtures; Stiffness Damage Index c) Tx mixtures and d) SPH mixtures.**

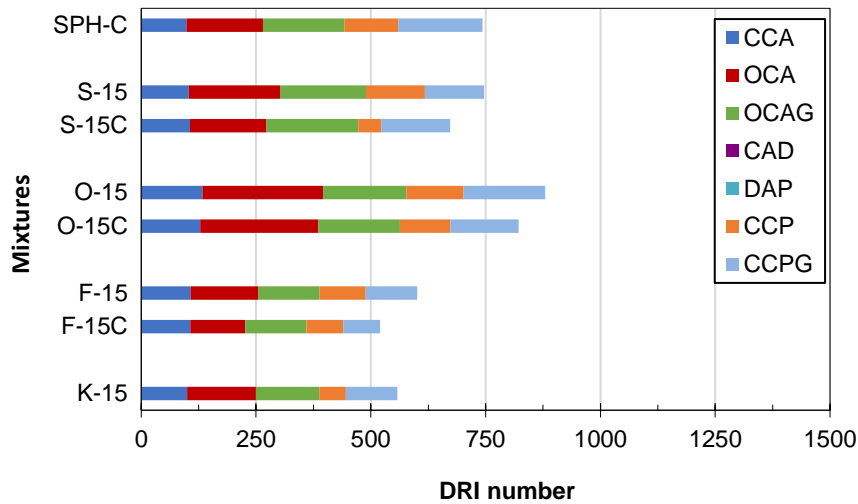
#### 4.6.4. Microscopy Assessment

Figure 4.18 illustrates the DRI numbers after 180 days of exposure to AAR development for both reactive aggregates. As expected, the DRI numbers for SPH mixtures are lower than for Tx mixes, once the SPH mixes reached lower expansion levels at 180 days. Globally, it was found that specimens design with the cement replacement procedure achieved lower DRI results than the ones made by sand replacement. Yet, the O-15 mix was the worsen result amongst Tx sand samples (i.e. 1268), which means 25% more cracks than the control Tx-C. Moreover, the mixtures which

incorporated reactive fillers as sand replacements (i.e. S and K) displayed similar DRI results than the control Tx-C (i.e. 1000 to 1125, respectively), followed by the F-15 mix (i.e. 970). Regarding the cement replacement mixtures, O-15C yielded the same DRI value than the Tx-control, while S-15C and F-15C were observed to have lower DRI numbers (i.e. 900 and 813, respectively). On the other hand for SPH mixtures, 15-O presented the highest DRI value (880) followed by 15C-O, 15-S, SPH-C, 15C-S, 15-F, 15-K and 15C-F (DRI of 822, 746, 743, 673, 601, 558 and 520, respectively). In general, it was observed that the cracks in cement paste (CCP) were lower for mixtures replacing PC when compared to the sand replacement; in addition, the presence of ASR-gel was mostly found in the cement paste for mixtures incorporating Tx reactive sand.



a)



b)

**Figure 4.18: Damage Rating Index of the ASR-affected concrete specimens at 180 days of exposure: a) Tx mixtures and b) SPH mixtures.**

## 4.7. Discussion and overall assessment

### 4.7.1. ASR kinetics and development

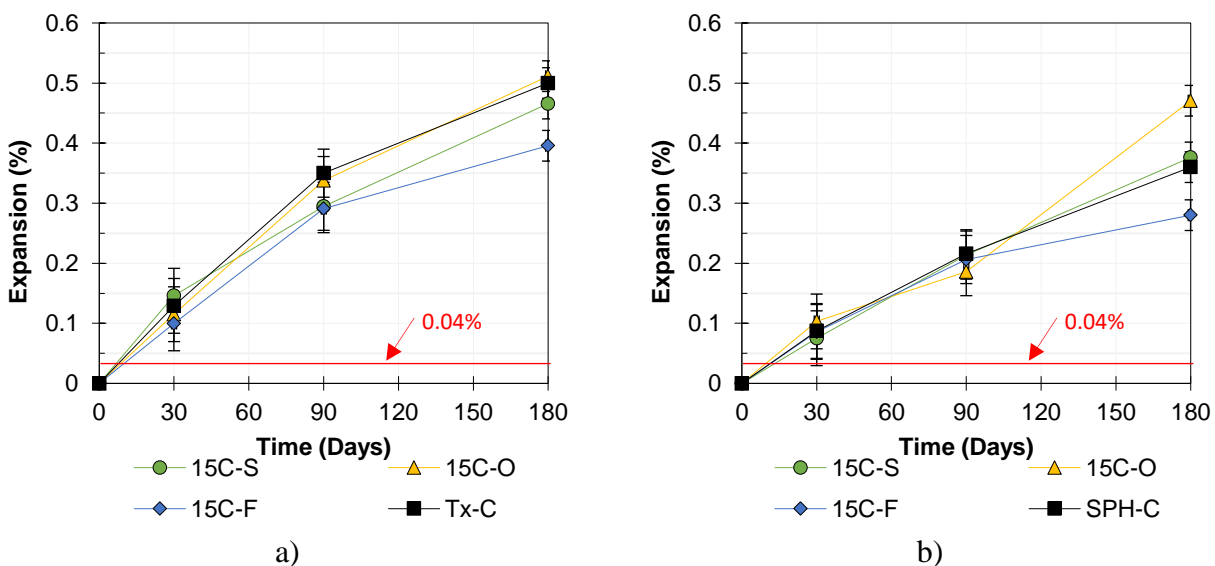
According to the concrete prism test (CPT), the mixtures containing Tx sand yielded faster ASR kinetics than the specimens made of SPH coarse. This was quite expected since Tx sand bears important amounts of chert in its coarse particles and thus is a more reactive material than SPH. Moreover, it was clear that depending upon the design approach (i.e. PC or sand replacement) the incorporation of MF influence different on ASR-induced expansion, as indicated by the statistical analysis displayed in Table 4.8. Overall, the sand replacement suggests faster ASR kinetics and higher expansion levels (up to 180 days) than the results obtained for concrete proportioned by cement replacement, even for mixtures where the reactive fine aggregate was replaced (i.e. Tx). The only exceptions were the mixtures proportioned with limestone filler (O) and SPH aggregate, which both replacement methods resulted in statistically similar expansion levels, indicating lower F-values than the F-critic (also P-value higher than significance level 0.05) by the ANOVA analysis (Table 4.8). Furthermore, all other combination tested indicate statistically significant impact in the results (comparing sand and cement replacement). Interestingly, the use of the dolomitic argillaceous limestone filler (K), crushed from a widely known ACR reactive aggregate has, somehow, demonstrated no significant influence on the expansion results (Table 4.8).

**Table 4.8: Two-variable ANOVA (significance level of 0.05) on the induced-expansion results for the concrete mixtures incorporating different aggregate types/natures, different types of filler and different replacement procedures after 180 days of exposure to ASR development.**

Aggregate	Source of Variation	F	P-value	F crit	
SPH vs Tx	Control SPH vs Control Tx	44.64	2.17 E <sup>-06</sup>	4.38	
	Control vs Fillers	36.03	1.65 E <sup>-25</sup>	2.10	
	Control vs FA Replacement	29.33	6.06 E <sup>-14</sup>	2.51	
	Control vs Cem. Replacement	41.48	6.16 E <sup>-14</sup>	2.78	
	SPH	Control vs 15-K	1.59	0.22	4.26
		O - FA vs Cem. Replacement	3.82	0.061	4.20
		F - FA vs Cem. Replacement	26.85	1.69 E <sup>-05</sup>	4.20
		S - FA vs Cem. Replacement	11.07	0.002	4.20
Tx	Control vs Fillers	32.37	7.79 E <sup>-20</sup>	2.14	
	Control vs FA Replacement	10.31	2.49 E <sup>-06</sup>	2.54	
	Control vs Cem. Replacement	19.83	3.46 E <sup>-07</sup>	2.93	
	Control vs 15-K	0.51	0.48	4.38	
	O - FA vs Cem. Replacement	33.63	9.33 E <sup>-06</sup>	4.32	
	F - FA vs Cem. Replacement	33.91	4.42 E <sup>-05</sup>	4.60	
	S - FA vs Cem. Replacement	28.94	3.43 E <sup>-05</sup>	4.38	

Regarding the cement replacement method, the use of limestone filler (15C-O) made mixtures somewhat more susceptible to ASR development; with kinetics and expansion amplitudes similar (for Tx mixture as indicated in

Table 4.9 and higher (for SPH mix) than the control groups. This behaviour is quite similar to what was found in the literature [49]. Figure 4.19 presents the relationship and influence of PC replacement by MF when compared to the control groups. It worth noting that the 15C-O mixtures may be directly correlated to the potential behaviour of using limestone cement (GUL/Type IL), since the 15% of PC's replacement is the maximum allowed for GUL production. The latter suggests that the use of this type of cement to assess the potential reactivity of aggregates in the laboratory may result in higher induced expansion and thus should be treated carefully. On the other hand, the use of granite filler (F) for partially replacing PC yielded the best results so far, somewhat mitigating AAR-induced expansion (i.e. 25% lower than control mixes, on average). Furthermore, the PSD of F suggests that this material may enhance the packing density of the mixtures since most of the F particles display particle sizes between the gap from PC particles and fine aggregate. Therefore, the F filler might be considered to be further combined with other preventive measures such as SCMs to both enhance the capacity for mitigating AAR and lower the carbon footprint of concrete mixtures.



**Figure 4.19: ASR kinetics with cement replacement by filler – a) Tx mixtures and b) SPH mixtures**

**Table 4.9: One-variable ANOVA (significance level of 0.05) on the induced-expansion results for the concrete mixtures incorporating different aggregate types/natures and different types of fillers replacing 15% of the total mass of cement after 180 days of exposure to ASR development.**

Source of Variation	F	P-value	F crit
Control SPH vs 15C-O	20.06	1.4 E <sup>-04</sup>	4.24
Control SPH vs 15C-F	31.62	8.66 E <sup>-06</sup>	4.26
Control SPH vs 15C-S	0.48	0.50	4.26
Control Tx vs 15C-O	0.27	0.61	4.49
Control Tx vs 15C-F	83.11	9.79 E <sup>-08</sup>	4.49
Control Tx vs 15C-S	4.38	0.054	4.54

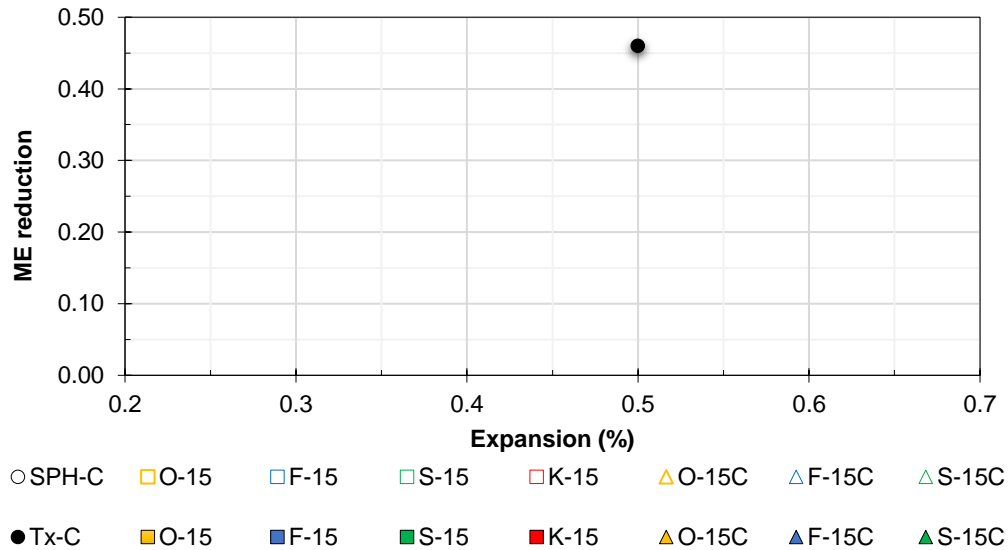
Finally, the present study has also considered the practical implications of using distinct granular systems incorporating reactive and non-reactive MF as either PC or fine aggregates replacement. The results clearly evidenced that the use of reactive MF (i.e. K and S) do not impact on ASR kinetics and final expansion. Yet, it should be noticed that these results were obtained in concrete systems incorporating highly reactive aggregates (i.e. SPH and Tx) and should be confirmed testing mixtures containing marginally to moderately reactive aggregates.

#### **4.7.2. Mechanical and microscope evaluation**

Mechanical and microscopic tests were performed to evaluate the influence of a variety of MF on ASR-induced deterioration. First of all, the type and nature of the reactive aggregate showed to play an important role in the reaction kinetics and distress development. Furthermore, different MF types and replacement methods (replacing PC or sand) were also deemed to impact the overall distress mechanism. The influence of the MF type was more pronounced than the replacement method on the compressive strength reductions of AAR-affected concrete. However, it is known that the compressive strength is not the best indicator for assessing AAR-induced deterioration and thus a better understanding of the influence of MF on the mechanical properties of affected concrete might be easier through the ME results.

The reductions in ME results agreed with the expansion levels obtained by the distinct mixtures (i.e. the higher the expansion level, the higher the losses in modulus of elasticity) as indicated in Figure 4.20. Furthermore, the DRI results presented in Figure 4.18 show evidences that a higher number of

cracks in the aggregates were found for Tx specimens and cement replacement, which may explain the higher loss of stiffness obtained.

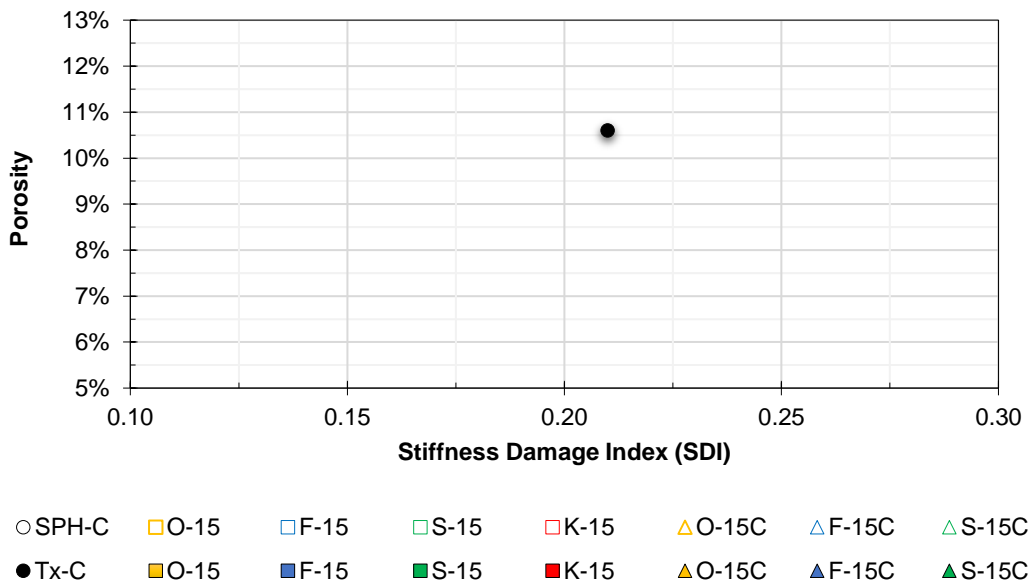


**Figure 4.20: Modulus of elasticity reduction vs the induced expansion for all concrete mixtures after 180 days of exposure to ASR development.**

AAR distress development has been widely studied and described by [33,51]. At the beginning of the physicochemical reaction, cracks are mainly generated within the aggregate particles; as the ASR-induced damage raises, new cracks are still developed within the aggregate particles yet, the pre-existing cracks keep increasing in width and length reaching the cement paste and thus forming an important network of cracks. The development of cracks within the aggregate particles and the propagation through the cement paste leads to an extension of inner damage in the affected concrete. Therefore, these cracks while being closed over a compressive and cyclic test release a significant amount of energy, which results in a fast rise in SDI values. Moreover, mixtures incorporating Tx were found to dissipate more energy, since those samples have presented up to the date the highest expansion levels. Likewise, Tx and SPH mixtures designed through the cement replacement method displayed higher SDI values besides yielding lower average expansions than the fine aggregate replacement mixes. These results were somewhat surprising and not totally in agreement with previous research [34], where SDI values often increase as a function of the expansion level. However, in this research, the amount of water was kept constant to simulate the use of GUL cements. Therefore, the quality of the mixtures was indirectly changed depending upon the mix-design approach used to incorporate MF (i.e. better microstructure quality was achieved for sand replacement

when compared to PC replacement) and this might have impacted the SDI results. This should be further investigated. Moreover, an important envelope of SDI results (ranging from 0.19 to 0.22; expansions in between 0.5% and 0.6%) was found for mixtures incorporating MF through the sand replacement method whereas similar values of about 0.27% (and expansions between 0.39% and 0.50%) were observed for mixtures incorporating MF and replacing PC.

On the other hand, it has been found in the current research a quite interesting correlation between porosity and SDI values (Figure 4.21). The higher the porosity results, the higher the SDI values. Likewise, the cement replacement technique displayed general higher porosity and SDI values. Once more, these were likely found due to the lower quality microstructure of the mixtures that used PC replacement techniques due to the higher water-to-cement-ratio implemented.



**Figure 4.21: Correlation between porosity and SDI values for all concrete mixtures after 180 days of exposure to ASR development.**

To better understand the influence of the different types of fillers and the replacement procedures on the cement paste porosity, the Powers-Brownyard’s method was used to calculate the maximum porosity of the distinct pastes, considering 100% of cement hydration (Table 4.10). The theoretical results indicate clear differences on the total pores content, yet, mostly due to an increase of capillary pores. The analysis revealed that replacing 15% of the PC resulted in the increase of 8.1% in the total porosity, although 15C samples have 46% more capillary pores in the cement than control mixes. The latter may explain the mechanical and expansion results gathered in this research.

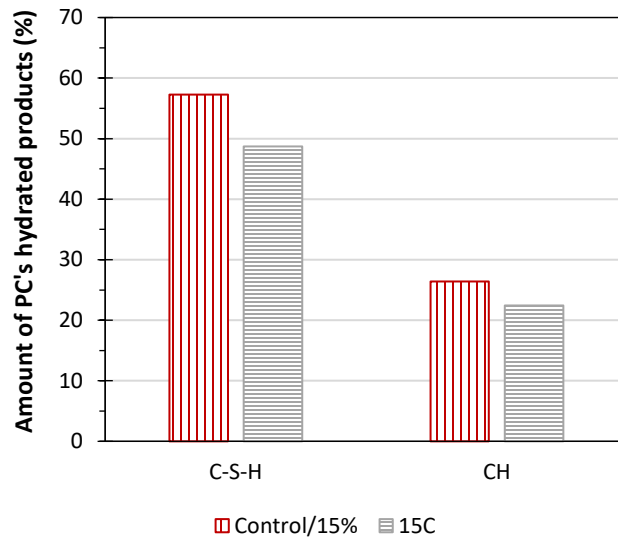
On the one hand, higher porosity may delay the expansion development caused by the ASR, since there is more empty space for the ASR-gel accommodation before causing internal pressures in the paste. On the other hand, higher porosity lessens the mechanical performance of material. Furthermore, replacing the fine aggregate by MF in this case is “translated” by adding fillers into the cement paste, decreasing its total porosity in 8.1% when compared to control mixes. Lower porosity enhances the mechanical performance of concrete, although it may accelerate AAR-induced development since less space to accommodate the reaction products will be available from the beginning of the reaction, which in turn will decrease the so-called induction period of the reaction process.

**Table 4.10: Total pores calculation**

Mixtures	Solids	Capillary	Gel	Total Pores
Control	63%	13%	24%	37%
O-15				
F-15	66%	12%	22%	34%
K-15				
S-15				
O-15C				
F-15C	60%	19%	20%	40%
S-15C				

Besides the above, it is known that higher PC contents lead to the formation of further hydration products (i.e. portlandite and C-S-H) which might also play an important role in the ASR-induced development. To evaluate this influence, a theoretical analysis was conducted combining Bogue’s equation and stoichiometric calculations as per [60]. It is worth noting that the values of CH and C-H-S are directly related to the cement content in the mixture since the fillers were considered as totally inert; the results obtained are displayed Figure 4.22.

Considering the products generated only in the cases when the PC was replaced by filler, the total amount of C-S-H and CH decrease of about 15%. Therefore, a slightly decrease in the compressive strength is expected, even though the filler effect contributes to the enhancement of the packing density of the mixture (Figure 4.22). Furthermore, the decrease of the CH formation may support the literature review since the presence of CH may increase the AAR expansion by modifying the viscosity of the AAR-gel. This background, may explain the lower expansion during cement replacement by filler in all mixtures, being supported by decrease on formation of CH.

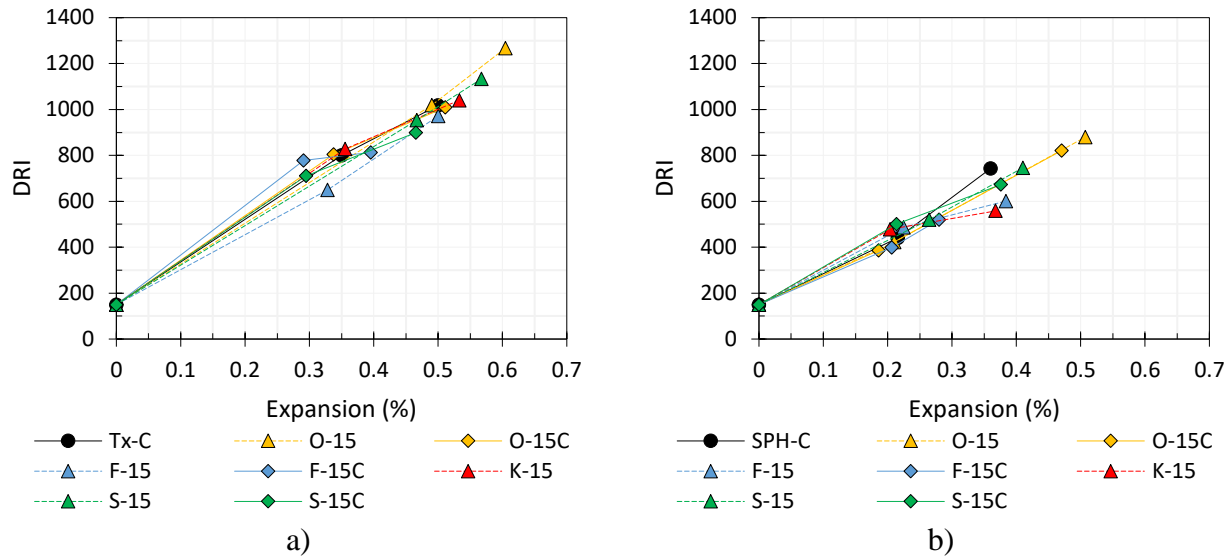


**Figure 4.22: Amount of Portland cement products formed considering 100% of PC's hydration for the different mixes proportioned.**

Figure 4.23 displays the overall microscopic evaluations and crack features observed through the DRI method. Besides the lower DRI numbers verified for SPH mixtures while PC replacement by MF, no other major difference was between the mixes made of Tx and SPH. In general, comparing the replacement procedure for the same type of fillers, the total number of open cracks in the aggregates (OCA) was higher for PC replacement; however, the number of cracks in the cement paste without and with gel (CCP and CCPG, respectively) decreases when compared to the sand replacement. This behavior was observed for all filler mixtures and may be explained by the aforementioned porosity in the cement paste matrix. Moreover, these findings could explain the higher expansion levels obtained in mixtures designs using the sand replacement approach, since the higher the number of cracks in the cement paste, the more severe AAR-induced deterioration

Figure 4.23 shows the DRI plots as a function of time for all mixtures evaluated in this research. In general, almost linear trends are presented for mixtures made of both Tx and SPH aggregates (Figure 4.23A and Figure 4.23B respectively). In the above plots, it is clear that the mixtures incorporating the limestone filler (O) yielded highest DRI (and expansion) results while mixes made of granite filler (F) showed the lowest DRI numbers (and thus expansion) at 180 days. Finally, the results gathered in this research seem to evidence that the type of MF may influence on ASR-induced expansion and damage; furthermore, these results demonstrate that limestone fillers are actually the ones displaying the worst performance against AAR-development. The latter raises concerns while the use of GUL

either for testing the potential reactivity of aggregates in the laboratory or for being used to field applications in mixtures incorporating reactive coarse or fine aggregates. Conversely, these results also seem to indicate that other types of filler such as granite, might also be considered to be blended with PC, since their impact on ASR-induced development were either positive or negligible.



**Figure 4.23: Correlations between the DRI values vs the expansion levels obtained for: a) Tx mixtures and b) SPH mixtures.**

## 4.8. Conclusion

The main objective of the current testing program was to understand the influence of different types of filler and replacement techniques (i.e. PC or sand replacement) on AAR-induced development. Through different analysis (i.e. expansion, physical, mechanical and microscopy test procedures), the main findings of this research are presented hereafter:

- Results indicate that in general, lower AAR-induced expansion levels are obtained for mixtures incorporating MF as a PC replacement when compared to both sand replacement approaches and control samples. Furthermore, with the exception of the limestone filler (O), all the other MF used in this research to replace PC either decreased or did not impact on the expansion levels reached.
- The use of distinct MF (i.e. nonreactive or reactive, distinct mineralogy, etc.), yield different behaviours related to the development of AAR-kinetics and final expansion. The use of granite filler (F) was found to be beneficial towards decreasing AAR-induced expansion. Moreover, the use of reactive fillers (i.e. crushed greywacke and dolomitic argillaceous limestone) did

not show significant influence on the expansion and deterioration of affected concrete. Conversely, limestone fillers were found to increase AAR-induced expansion regardless of the design approach selected (i.e. PC or sand replacement). These results demonstrate that careful consideration should be taken while the use of GUL cements for either assess the reactivity of aggregates in the laboratory or to be used in combination with reactive aggregates. However, the results also attested that different types of MF than limestone fillers might be selected to be blended with PC regarding their suitability against AAR-induced development.

- The PC replacement by MF may decrease the formation of CH, resulting in a less expansive gel and thus lowering the obtained expansion levels. These mixtures presented higher porosity than the ones designed through the sand replacement approach.
- The porosity of the concrete plays an important role in its mechanical properties and AAR-induced development. Exhibiting lower porosity due to the use of a lower water-to-cement-ratio (w/c), the sand replacement method mixtures displayed better mechanical results (i.e. lower SDI values along with lower CS and ME losses) than the cement replacement approach mixes (which employ higher w/c and thus porosity). Nevertheless, mixtures designed through the PC's replacement method yielded lower expansion levels
- Statistical analyses (analysis of variance - ANOVA) have been conducted with the data gathered in this research to validate all the above discussed research outcomes.

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## Chapter Five: Conclusion and Future Recommendations

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Concrete is likely the most common construction material used in critical infrastructure worldwide. However, the concrete industry currently faces crucial challenges of finding cost-effective strategies to reduce the carbon-footprint and embodied energy associated with the production of Portland cement (PC). One of the most adopted methods for reducing environmental impact of PC involves the use of “established” residues from other industries in concrete, the so-called supplementary cementing materials (SCMs) such as fly-ash, silica fume, blast-furnace slag, etc. Yet, the availability of these materials has become uncertain and limited worldwide, which indicates the need of finding alternatives to conventional SCMs.

In this research program, the use of alternative materials (i.e. wood ash - WA and mineral fillers - MF) was studied and their influence on the long-term behaviour of concrete mixtures particularly affected by alkali-aggregate reaction (AAR) was investigated. In order to thoroughly evaluate their potential use these finely ground materials (i.e. WA and MF) were physiochemically characterized through X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), particle size distribution and specific gravity tests. Afterwards, different concrete mixes were proportioned incorporating two highly reactive aggregates (i.e. greywacke coarse aggregate – SPH and polymictic fine aggregate - Tx), distinct WA amounts (i.e. 10%, 20%, 50% and 60% of PC replacement, by mass) or four different types of fillers (reactive and non reactive, with distinct mineralogy) replacing 15% (maximum allowed by CSA A3001 & ASTM C595), by mass, of either PC or fine aggregates. Concrete samples were fabricated from the various mixtures and were stored under controlled environmental conditions enabling AAR-induced development. At selected exposure periods, physical (i.e. porosity), microscopic (i.e. Damage Rating Index) and mechanical tests (i.e. Stiffness Damage Test, modulus of elasticity and compressive strength), were conducted for a comprehensive evaluation on the effect of WA and MF on the overall AAR-induced development (i.e. expansion and damage progress). The main findings of this experimental program are presented hereafter:

### *Influence of Wood Ash (WA) on AAR-induced development*

- The accelerated mortar bar test (AMBT) showed that the higher the replacement level of PC by WA, the lower the expansion levels reached by mixtures incorporating highly reactive Tx fine and SPH coarse aggregates. Mixtures made of 50% WA replacement were considered either non-reactive or just above the reactivity limit as per ASTM C 1260-1567 and CSA A23.2-25A
- The results obtained through the accelerated concrete prism test ACPT are not in agreement with the results obtained through the AMBT for both aggregates used. The latter seems to indicate an example of a “false negative” result yielded by the accelerated mortar bar test. The ACPT results found for the SPH aggregate seem to be counterintuitive since the higher the amount of WA replacement, the higher the induced expansion and distress obtained. Further analyses are still required to fully understand these results.
- The mechanical properties losses (i.e. compressive strength, modulus of elasticity and SDI) are in agreement with the expansion levels obtained and microscopic analyses conducted through the DRI method. Although the mechanisms observed while the use of WA seem to change as a function of the aggregate type and may be somewhat counterintuitive, the use of the multi-level assessment as per Sanchez. et al [44] clearly indicated that the induced expansion results obtained herein were correct.
- The preliminary results obtained in this research seem to indicate that WA is not a suitable material to be used as an SCM to mitigate ASR-induced development in concrete. Nevertheless, the data gathered attested the possibility of using WA as an inert filler, which could make a positive impact towards a greener future of the construction industry. Moreover, further treatment could be implemented in WA materials (or even while proceeding with the wood combustion process) to modify its chemical composition and thus make it more suitable to replaced established SCMs.
- A good linear correlation between porosity and compressive strength was verified in this research confirming that the addition of WA increased significantly the overall porosity of concrete materials; however, no correlation between porosity and expansion was observed.

### ***Influence of MF on AAR-induced expansion and deterioration***

- Results demonstrated that lower AAR-induced expansion levels are obtained for mixtures incorporating MF whenever it is used as PC's partial replacement. In addition, with the exception of the limestone filler (O), all the other MF types used in this research to replace PC either decreased or did not impact on the expansion levels obtained.
- The use of distinct MF types (i.e. nonreactive or reactive, distinct mineralogy, etc.) caused different impacts on AAR-kinetics and final expansion. The use of granite filler (F) was found to be beneficial towards lessening the induced expansion. Furthermore, the use of reactive fillers (i.e. crushed greywacke and dolomitic argillaceous limestone) did not show significant influence on the expansion and deterioration of affected concrete. These results attest that different types of MF (other than limestone) might be selected to be blended with PC regarding their suitability against AAR-induced development.
- Limestone fillers were found to increase AAR-induced expansion regardless of the design approach selected (i.e. PC or sand replacement). These results demonstrate that careful consideration should be taken while the use of GUL cements for either assess the reactivity of aggregates in the laboratory or to be used in combination with reactive aggregates.
- The PC replacement by MF may decrease the formation of CH, resulting in a less expansive gel and thus lowering the obtained expansion levels. These mixtures presented higher porosity than the ones designed through the sand replacement approach.
- Displaying lower porosity due to the use of a lower water-to-cement-ratio (w/c), the sand replacement method mixtures displayed better mechanical results (i.e. lower SDI values along with lower CS and ME losses) than the cement replacement approach mixes (which employ higher w/c and thus porosity). Nevertheless, mixtures designed through the PC's replacement method yielded lower expansion levels.
- Statistical analyses (analysis of variance - ANOVA) have been conducted with the data gathered in this research to validate all the above discussed research outcomes.

After conducting a very comprehensive experimental campaign (with over 1000 samples) and evaluating the main research outcomes, further investigations can be drafted as future research recommendations, as presented hereafter:

- Further research and investigations should be performed on the use of WA in concrete; Chemical analyzes on the secondary products formed while AAR development (i.e. AAR-gel) in the presence of WA should be further investigated due to the likely changes in viscosity and chemical composition when compared to conventional AAR products. This might give us some key information to fully understand the results gathered in this research.
- Although the results found in this research suggest that WA is not quite suitable to be used as an SCM in concrete for mitigating AAR, the results clearly demonstrated that WA could be indeed used as an MF in cement-based materials towards a greener future of the concrete industry.
- Studies to implement strategies to conduct wood combustion processes and/or WA post-treatment to change the chemical components of the material, and thus make it more suitable to replace established SCMs would be extremely beneficial.
- Other durability mechanisms (e.g. sulphate attack, corrosion, freezing and thawing, etc.) might be performed on WA made concrete in order to gather a more holistic understanding on the impact of WA on the durability-related properties of concrete.
- The research on MF developed in this Thesis is an ongoing work. Results will be gathered until 365 days and a much better understanding of the results gathered so far along with the main research outcomes will definitely be achieved.
- There is a need of testing reactive MF materials using a wide variety of types and replacement levels in concrete incorporating non-reactive coarse and fine aggregates. The latter will be very helpful to appraise the capacity of MF to trigger and maintain AAR-induced development.
- The study of systems combining MF and SCM's might be useful. Maybe, the amount of conventional SCMs to mitigate AAR-induced development might be largely reduced while combined with MF.
- There is a need of assessing the performance of concrete mixtures incorporating numerous MF types with distinct replacement levels against other durability-related mechanisms than AAR, such as internal/external sulphate attack, freeze-thaw cycles, chloride penetration, carbonation, steel corrosion of etc.

## APPENDIX

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### Appendix A: Data of expansion level measurement induced by AAR (ACPT)

**Table 11: Texas control samples at 15 days**

Sample	Measurement			AVERAGE	Mass	Expansion
	1	2	3			
<b>1</b>	-2.896	-2.880	-2.890	162.111	3963	0.25
<b>2</b>	-3.915	-3.919	-3.916	161.083	3946	0.24
<b>3</b>	-4.071	-4.072	-4.074	160.928	3986	0.16
<b>4</b>	-4.594	-4.592	-4.596	160.406	3926	0.22
<b>5</b>	-2.635	-2.632	-2.637	162.365	4011	0.32
<b>6</b>	-3.586	-3.587	-3.588	161.413	3968	0.29
<b>7</b>	-7.206	-7.203	-7.205	157.795	3933	0.29
<b>8</b>	-6.153	-6.155	-6.156	158.845	3900	0.27
<b>9</b>	-4.851	-4.853	-4.855	160.147	3921	0.27
<b>10</b>	-3.961	-3.962	-3.972	161.035	3927	0.27
<b>11</b>	-3.183	-3.171	-3.174	161.824	3982	0.29

**Table 12: Texas sand mixtures with 20% of WA at 15 days**

Sample	Measurement			AVERAGE	Mass	Expansion
	1	2	3			
<b>1</b>	-3.643	-3.641	-3.645	161.357	3941	0.38
<b>2</b>	-1.790	-1.791	-1.792	163.209	3994	0.32
<b>3</b>	-4.458	-4.464	-4.451	160.542	3904	0.32
<b>4</b>	-1.349	-1.364	-1.351	163.645	3918	0.33
<b>5</b>	-3.031	-3.027	-3.029	161.971	3919	0.27
<b>6</b>	-3.650	-3.648	-3.655	161.349	3933	0.29
<b>7</b>	-0.292	-0.295	-0.301	164.704	4078	0.37
<b>8</b>	-3.017	-3.020	-3.022	161.980	3892	0.31
<b>9</b>	-3.214	-3.213	-3.210	161.788	3945	0.40
<b>10</b>	-2.274	-2.276	-2.281	162.723	3933	0.36
<b>11</b>	0.070	0.072	0.073	165.072	4050	0.33
<b>12</b>	-3.402	-3.407	-3.403	161.596	3925	0.36

**Table 13: Texas sand mixtures with 60% of WA at 15 days**

Sample	Measurement			AVERAGE	Mass	Expansion
	1	2	3			
1	-0.015	-0.013	-0.016	164.985	3740	0.10
2	-0.286	-0.288	-0.286	164.713	3803	0.14
3	-3.692	-3.687	-3.696	161.308	3719	0.08
4	0.357	0.352	0.354	165.354	3798	0.12
5	-2.415	-2.414	-2.409	162.587	3773	0.17
6	-2.141	-2.145	-2.146	162.856	3830	0.12
7	-4.218	-4.214	-4.220	160.783	3757	0.19
8	-0.401	-0.402	-0.404	164.598	3804	0.20
9	-3.750	-3.758	-3.753	161.246	3829	0.21
10	-3.320	-3.322	-3.324	161.678	3763	0.25
11	-2.984	-2.985	-2.982	162.016	3791	0.20
12	2.580	2.582	2.584	167.582	3861	0.18

**Table 14: SPH mixture samples control at 15 day**

Sample	Measurement			AVERAGE	Mass	Expansion
	1	2	3			
1	-4.636	-4.634	-4.641	160.363	3975	0.12
2	-6.061	-6.067	-6.068	158.935	3906	0.10
3	-5.630	-5.631	-5.632	159.369	3912	0.11
4	-7.234	-7.235	-7.236	157.765	3915	0.09
5	-4.993	-4.987	-4.986	160.011	3941	0.11
6	-4.876	-4.879	-4.881	160.121	3933	0.09
7	-6.016	-6.019	-6.020	158.982	3922	0.01
8	-5.149	-5.150	-5.151	159.850	3977	0.11
9	-4.578	-4.571	-4.593	160.419	3918	0.08
10	-4.974	-4.976	-4.979	160.024	3913	0.07
11	-5.724	-5.730	-5.728	159.273	3917	0.08
12	-4.695	-4.698	-4.701	160.302	3993	0.11

**Table 15: SPH mixtures with 20% of WA at 15 days**

Sample	Measurement			AVERAGE	Mass	Expansion
	1	2	3			
1	-1.475	-1.48	-1.484	163.520	3865	0.05
2	-4.29	-4.295	-4.3	160.705	3855	0.13
3	-0.546	-0.554	-0.555	164.448	3914	0.17
4	2.937	2.936	2.929	167.934	3939	0.14
5	-3.075	-3.077	-3.082	161.922	3852	0.16
6	-0.382	-0.385	-0.393	164.613	3860	0.12
7	-2.007	-2.01	-2.019	162.988	2873	0.22
8	-0.627	-0.631	-0.631	164.370	3870	0.15
9	-2.981	-2.983	-2.987	162.016	3904	0.16
10	-4.432	-4.437	-4.44	160.564	3865	0.22
11	-3.997	-3.999	-4.001	161.001	3869	0.16
12	-3.901	-3.608	-3.604	161.296	3879	0.12

**Table 16: SPH mixtures with 60% of WA at 15 days**

Sample	Measurement			AVERAGE	Mass	Expansion
	1	2	3			
1	-0.162	-0.163	-0.164	164.837	3824	0.18
2	0.792	0.788	0.789	165.790	3831	0.14
3	-4.113	-4.114	-4.118	160.885	3748	0.18
4	-2.562	-2.567	-2.568	162.434	3711	0.15
5	-2.668	-2.674	-2.677	162.327	3722	0.19
6	0.427	0.421	0.421	165.423	3815	0.21
7	-1.601	-1.613	-1.607	163.393	3807	0.21
8	-3.322	-3.325	-3.330	161.674	3771	0.18
9	-3.409	-3.415	-3.407	161.590	3772	0.13
10	-0.536	-0.537	-0.539	164.463	3768	0.14
11	-2.058	-2.071	-2.064	162.936	3774	0.14
12	-2.192	-2.193	-2.189	162.809	3692	0.10

## Appendix B: Data of expansion level measurement induced by AAR (AMBT)

Table 17: AMBT results for Tx sand mixtures

Control															
Day	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	23-May	24-May			27-May	28-May	29-May	30-May	31-May	1-Jun	2-Jun	3-Jun	4-Jun	5-Jun	6-Jun
<b>B1</b>	0.014	0.204			1.500	1.658		2.032	2.108				2.468	2.508	2.562
<b>B2</b>	- 0.174	0.000			1.258	1.376		1.752	1.830				2.166	2.206	2.258
<b>B3</b>	- 0.078	0.106			1.414	1.540		1.898	1.978				2.328	2.364	2.418
<b>Expansion</b>		0.13%			0.59%	0.64%	0.06%	0.77%	0.80%	0.06%	0.06%	0.06%	0.93%	0.94%	0.96%
		0.03%			0.47%	0.51%	0.03%	0.64%	0.67%	0.03%	0.03%	0.03%	0.79%	0.80%	0.82%
		0.04%			0.50%	0.54%	0.00%	0.67%	0.69%	0.00%	0.00%	0.00%	0.82%	0.83%	0.85%
<b>Average</b>	0.00 %	0.07%	0.25%	0.40%	0.52%	0.56%	0.62%	0.69%	0.72%	0.75%	0.78%	0.81%	0.84%	0.86%	0.88%
Wood Ash 10%															
Day	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	23-May	24-May			27-May	28-May	29-May	30-May	31-May	1-Jun	2-Jun	3-Jun	4-Jun	5-Jun	6-Jun
<b>B1</b>	0.444	0.730			1.578	1.668		1.932	1.986				2.222	2.246	2.302
<b>B2</b>	1.798	2.084			2.960	3.060		3.328	3.368				3.624	3.656	3.698
<b>B3</b>	0.154	0.462			1.338	1.434		1.706	1.762				2.008	2.032	2.08
<b>Expansion</b>		-0.37%	- 0.63%	- 0.63%	-0.08%	-0.05%	-0.63%	0.05%	0.07%	- 0.63%	- 0.63%	- 0.63%	0.15%	0.16%	0.18%
		0.68%	- 0.05%	- 0.05%	0.98%	1.02%	-0.05%	1.11%	1.13%	- 0.05%	- 0.05%	- 0.05%	1.22%	1.23%	1.24%

		0.16%	0.00%	0.00%	0.47%	0.50%	0.00%	0.60%	0.62%	0.00%	0.00%	0.00%	0.70%	0.71%	0.73%
<b>Average</b>	0.00%	0.16%	0.20%	0.30%	0.46%	0.49%	0.48%	0.59%	0.60%	0.57%	0.59%	0.62%	0.69%	0.70%	0.72%
<b>Wood Ash 20%</b>															
Day	<b>0</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>	<b>11</b>	<b>12</b>	<b>13</b>	<b>14</b>
	<b>23-May</b>	<b>24-May</b>			<b>27-May</b>	<b>28-May</b>	<b>29-May</b>	<b>30-May</b>	<b>31-May</b>	<b>1-Jun</b>	<b>2-Jun</b>	<b>3-Jun</b>	<b>4-Jun</b>	<b>5-Jun</b>	<b>6-Jun</b>
<b>B1</b>	2.682	3.030			3.610	3.684		3.860	3.896				4.043	4.08	4.094
<b>B2</b>	0.626	0.970			1.498	1.556		1.718	1.746				1.912	1.942	1.952
<b>B3</b>	0.024	0.364			0.932	0.994		1.158	1.192				1.36	1.384	1.396
<b>Expansion</b>		0.84%	-0.22%	-0.22%	1.04%	1.07%	-0.22%	1.13%	1.14%	-0.22%	-0.22%	-0.22%	1.20%	1.21%	1.21%
		0.33%	-0.01%	-0.01%	0.52%	0.54%	-0.01%	0.59%	0.60%	-0.01%	-0.01%	-0.01%	0.66%	0.67%	0.68%
		0.13%	0.00%	0.00%	0.33%	0.35%	0.00%	0.41%	0.42%	0.00%	0.00%	0.00%	0.48%	0.49%	0.49%
<b>Average</b>	0.00%	0.43%	0.15%	0.20%	0.63%	0.65%	0.38%	0.71%	0.72%	0.43%	0.44%	0.45%	0.78%	0.79%	0.79%

<b>Wood Ash 50%</b>															
Day	<b>0</b>	<b>1</b>	<b>2</b>	<b>3</b>	<b>4</b>	<b>5</b>	<b>6</b>	<b>7</b>	<b>8</b>	<b>9</b>	<b>10</b>	<b>11</b>	<b>12</b>	<b>13</b>	<b>14</b>
	<b>28-Jun</b>	<b>29-Jun</b>	<b>30-Jun</b>	<b>1-Jul</b>	<b>2-Jul</b>	<b>3-Jul</b>	<b>4-Jul</b>	<b>5-Jul</b>	<b>6-Jul</b>	<b>7-Jul</b>	<b>8-Jul</b>	<b>9-Jul</b>	<b>10-Jul</b>	<b>11-Jul</b>	<b>12-Jul</b>
<b>B1</b>	0.542				0.734	0.814		0.834			0.858	0.85	0.828	0.84	
<b>B2</b>	-0.230				-0.024	0.014		0.050			0.082	0.062	0.07	0.068	
<b>B3</b>	0.452				0.654	0.706		0.744			0.772	0.754	0.746	0.756	
<b>Expansion</b>					0.34%	0.37%	0.08%	0.37%	0.08%	0.08%	0.38%	0.38%	0.37%	0.38%	0.08%
					-0.17%	-0.15%	-0.16%	-0.14%	-0.16%	-0.16%	0.16%	0.13%	0.14%	0.13%	0.13%
					0.23%	0.25%	0.00%	0.26%	0.00%	0.00%	0.27%	0.26%	0.26%	0.27%	0.00%

<b>Average</b>	0.00%	0.02%	0.04%	0.06%	0.13%	0.15%	0.09%	0.16%	0.10%	0.10%	0.17%	0.17%	0.17%	0.17%	0.11%
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**Table 18: AMBT results for SPH mixtures**

Control															
Day	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	13-Jul	14-Jul	15-Jul	16-Jul	17-Jul	18-Jul	19-Jul	20-Jul	21-Jul	22-Jul	23-Jul	24-Jul	25-Jul	26-Jul	27-Jul
<b>B1</b>	0.052	0.126		0.210		0.416				0.612		0.684			0.804
<b>B2</b>	0.306	0.378		0.466		0.694				0.93		1.004			1.126
<b>B3</b>	0.124	0.186		0.268		0.472				0.69		0.762			0.878
<b>Expansion</b>		0.11%	0.06%	0.13%	0.06%	0.21%	0.06%	0.06%	0.06%	0.28%	0.06%	0.30%	0.06%	0.06%	0.34%
		0.16%	0.03%	0.19%	0.03%	0.27%	0.03%	0.03%	0.03%	0.35%	0.03%	0.38%	0.03%	0.03%	0.42%
		0.07%	0.00%	0.09%	0.00%	0.17%	0.00%	0.00%	0.00%	0.24%	0.00%	0.27%	0.00%	0.00%	0.31%
<b>Average</b>	0.00%	0.11%	0.04%	0.14%	0.09%	0.21%	0.15%	0.18%	0.19%	0.29%	0.22%	0.32%	0.25%	0.26%	0.36%

Wood Ash 10%															
Day	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	13-Jul	14-Jul	15-Jul	16-Jul	17-Jul	18-Jul	19-Jul	20-Jul	21-Jul	22-Jul	23-Jul	24-Jul	25-Jul	26-Jul	27-Jul
<b>B1</b>	0.138	0.230		0.324		0.520				0.7		0.748			2.848
<b>B2</b>	2.082	2.186		2.290		2.512				2.7		2.748			0.844
<b>B3</b>	-1.218	-1.114		-1.010		-0.772				-0.576		-0.53			-0.43
<b>Expansion</b>		0.03%	-0.05%	0.06%		0.13%				0.19%		0.21%			0.94%
		0.77%	0.00%	0.80%		0.88%				0.95%		0.96%			0.30%
		-0.39%	0.00%	-0.35%		-0.27%				-0.20%		-0.19%			-0.15%
<b>Average</b>	0.00%	0.13%	0.05%	0.17%	0.11%	0.25%	0.17%	0.19%	0.20%	0.31%	0.22%	0.33%	0.25%	0.26%	0.36%

Wood Ash 20%															
Day	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	13-Jul	14-Jul	15-Jul	16-Jul	17-Jul	18-Jul	19-Jul	20-Jul	21-Jul	22-Jul	23-Jul	24-Jul	25-Jul	26-Jul	27-Jul
<b>B1</b>	2.082	2.242		2.316		2.442				2.552		2.56			2.612
<b>B2</b>	-0.118	0.026		0.104		0.220				0.334		0.354			0.408
<b>B3</b>	0.256	0.418		0.494		0.624				0.728		0.744			0.812
<b>Expansion</b>		0.79%	0.00%	0.81%		0.86%	0.00%	0.00%	0.00%	0.90%			0.00%	0.00%	0.92%
		0.01%	0.00%	0.04%		0.08%	0.00%	0.00%	0.00%	0.12%			0.00%	0.00%	0.14%
		0.15%	0.00%	0.17%		0.22%	0.00%	0.00%	0.00%	0.26%			0.00%	0.00%	0.28%
<b>Average</b>	0.00%	0.31%	0.06%	0.34%	0.10%	0.38%	0.13%	0.14%	0.15%	0.42%	0.16%	0.17%	0.17%	0.18%	0.45%

Wood Ash 50%															
Day	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14
	13-Jul	14-Jul	15-Jul	16-Jul	17-Jul	18-Jul	19-Jul	20-Jul	21-Jul	22-Jul	23-Jul	24-Jul	25-Jul	26-Jul	27-Jul
<b>B1</b>	2.534	2.778		2.834		2.894				2.908		2.926			2.946
<b>B2</b>	0.232	0.480		0.534		0.596				0.608		0.626			0.64
<b>B3</b>															
<b>Expansion</b>		0.97%	0.00%	0.99%	0.00%	1.02%	0.00%	0.00%	0.00%	1.02%	0.00%	1.03%	0.00%	0.00%	1.03%
		0.17%	0.00%	0.19%	0.00%	0.21%	0.00%	0.00%	0.00%	0.21%	0.00%	0.22%	0.00%	0.00%	0.22%
<b>Average</b>	0.00%	0.57%	0.10%	0.59%	0.12%	0.61%	0.13%	0.13%	0.13%	0.62%	0.13%	0.62%	0.14%	0.14%	0.63%

**Appendix B: Wood Ash - Complementary data results**

**Table 19: Tx mixtures porosity**

<b>1 month</b>						
<b>Mix</b>	<b>Local</b>	<b>Dry</b>	<b>Sub</b>	<b>SSD</b>	<b>AP</b>	<b>AP Total</b>
<b>WT-C</b>	<i>Top</i>	1212.4	653.95	1264.5	9%	7.5
	<i>Mid</i>	1235.3	665.43	1282.4	8%	
	<i>Bot</i>	1243.7	665.3	1283.5	6%	
<b>WT-20</b>	<i>Top</i>	1183.3	623.65	1232.75	8%	7.2
	<i>Mid</i>	1222.1	649.05	1266.03	7%	
	<i>Bot</i>	1285.6	690.38	1326.7	6%	
<b>WT-60</b>	<i>Top</i>	1125.1	591.57	1192.5	11%	11.2
	<i>Mid</i>	1143.5	602.31	1215.25	12%	
	<i>Bot</i>	1226.9	654.94	1296.53	11%	

**3 months**

<b>Mix</b>	<b>Local</b>	<b>Dry</b>	<b>Sub</b>	<b>SSD</b>	<b>AP</b>	<b>AP Total</b>
<b>WT-C</b>	<i>Top</i>	742.1	371.68	776.89	9%	8.1
	<i>Mid</i>	702.6	347.1	733.47	8%	
	<i>Bot</i>	709.3	347.8	739.32	8%	
<b>WT-20</b>	<i>Top</i>	465.6	203.22	493.27	10%	8.4
	<i>Mid</i>	449	193.55	472.42	8%	
	<i>Bot</i>	437.5	187.8	457.59	7%	
<b>WT-60</b>	<i>Top</i>	427.5	181.15	464.39	13%	13.2
	<i>Mid</i>	381.6	154.15	417.95	14%	
	<i>Bot</i>	454.8	192.98	493.18	13%	

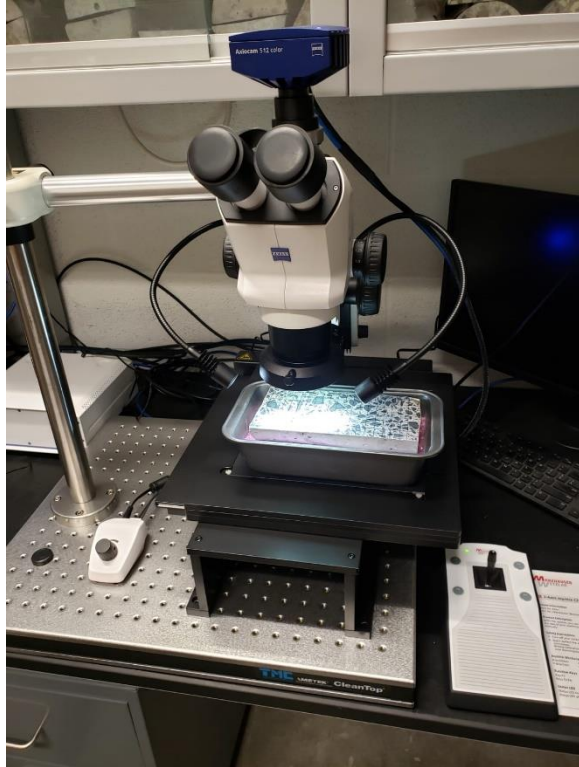
**Table 20: SPH mixture porosity**

<b>1 month</b>						
<b>Mix</b>	<b>Local</b>	<b>Dry</b>	<b>Sub</b>	<b>SSD</b>	<b>AP</b>	<b>AP Total</b>
<b>WS-C</b>	<i>Top</i>	1229.2	652.1	1263.9	6%	5.2
	<i>Mid</i>	1228.2	648	1260.03	5%	
	<i>Bot</i>	1281	681.1	1310.99	5%	
<b>WS-20</b>	<i>Top</i>	1140.8	608.1	1206.5		7.8
	<i>Mid</i>	1202.8	639.05	1254.53	8%	
	<i>Bot</i>	1298.9	698.3	1346.65	7%	
<b>WS-60</b>	<i>Top</i>	1086.7	582.17	1177.35	15%	14.4
	<i>Mid</i>	1128	603.34	1216.59	14%	
	<i>Bot</i>	1232.7	667.57	1323.2	14%	

3 months						
Mix	Local	Dry	Sub	SSD	AP	AP Total
WS-C	Top	690.8	334.95	717.74	7%	6.72
	Mid	714.5	346.46	741.15	7%	
	Bot	751.4	370.95	777.29	6%	
WS-20	Top	411.4	166.72	434.93	9%	9.7
	Mid	432.6	179.2	460.59	10%	
	Bot	498.8	221.09	531.09	10%	
WS-60	Top	453.1	198.2	495.35	14%	13.8
	Mid	410.1	169.9	446.77	13%	
	Bot	434.8	185.95	476	14%	

**Table 21: Wood ash - Particle Size Distribution (PSD)**

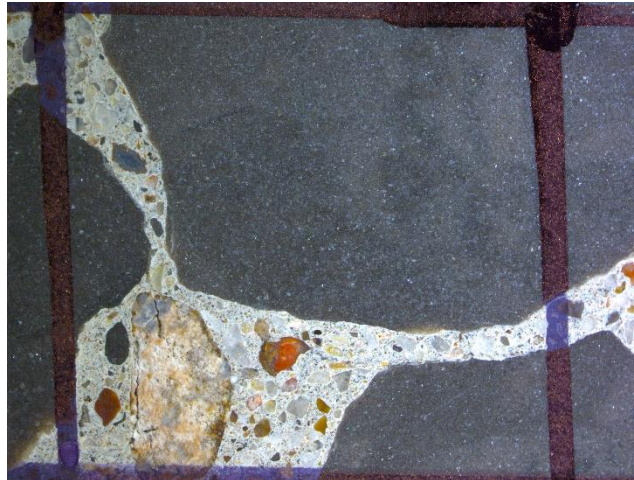
No	Size (µm)	Mean In%	1*S.D.	No	Size (µm)	Mean In%	1*S.D.	No	Size (µm)	Mean In%	1*S.D.	No	Size (µm)	Mean In%	1*S.D.
1	0.010	0.00	0.00	27	0.363	0.16	0.01	53	13.183	1.28	0.07	79	478.630	2.61	0.41
2	0.011	0.00	0.00	28	0.417	0.21	0.01	54	15.136	1.55	0.09	80	549.541	2.43	0.45
3	0.013	0.00	0.00	29	0.479	0.24	0.01	55	17.378	1.88	0.10	81	630.957	2.07	0.46
4	0.015	0.00	0.00	30	0.550	0.25	0.01	56	19.953	2.26	0.13	82	724.436	1.57	0.49
5	0.017	0.00	0.00	31	0.631	0.24	0.01	57	22.909	2.69	0.15	83	831.764	1.02	0.48
6	0.020	0.00	0.00	32	0.724	0.22	0.01	58	26.303	3.18	0.17	84	954.993	0.57	0.41
7	0.023	0.00	0.00	33	0.832	0.19	0.01	59	30.200	3.68	0.19	85	1096.478	0.29	0.32
8	0.026	0.00	0.00	34	0.955	0.15	0.01	60	34.674	4.18	0.21	86	1258.925	0.17	0.23
9	0.030	0.00	0.00	35	1.096	0.11	0.01	61	39.811	4.63	0.22	87	1445.440	0.10	0.16
10	0.035	0.00	0.00	36	1.259	0.08	0.01	62	45.709	4.99	0.23	88	1659.587	0.05	0.09
11	0.040	0.00	0.00	37	1.445	0.07	0.01	63	52.481	5.21	0.23	89	1905.461	0.01	0.02
12	0.046	0.00	0.00	38	1.660	0.06	0.02	64	60.256	5.27	0.24	90	2187.762	0.00	0.00
13	0.052	0.00	0.00	39	1.905	0.07	0.01	65	69.183	5.14	0.24	91	2511.886	0.00	0.00
14	0.060	0.00	0.00	40	2.188	0.09	0.01	66	79.433	4.84	0.26	92	2884.032	0.00	0.00
15	0.069	0.00	0.00	41	2.512	0.10	0.01	67	91.201	4.39	0.27	93	3311.311	0.00	0.00
16	0.079	0.00	0.00	42	2.884	0.13	0.01	68	104.713	3.84	0.27	94	3801.894	0.00	0.00
17	0.091	0.00	0.00	43	3.311	0.16	0.01	69	120.226	3.25	0.27	95	4365.158	0.00	0.00
18	0.105	0.00	0.00	44	3.802	0.20	0.01	70	138.038	2.68	0.24	96	5011.872	0.00	0.00
19	0.120	0.00	0.00	45	4.365	0.25	0.02	71	158.489	2.20	0.21	97	5754.399	0.00	0.00
20	0.138	0.00	0.00	46	5.012	0.31	0.02	72	181.970	1.87	0.16	98	6606.934	0.00	0.00
21	0.158	0.00	0.00	47	5.754	0.39	0.02	73	208.930	1.73	0.12	99	7585.776	0.00	0.00
22	0.182	0.00	0.00	48	6.607	0.47	0.03	74	239.883	1.76	0.11	100	8709.636	0.00	0.00
23	0.209	0.00	0.00	49	7.586	0.58	0.03	75	275.423	1.93	0.15		10000.000		
24	0.240	0.00	0.00	50	8.710	0.70	0.04	76	316.228	2.18	0.22				
25	0.275	0.01	0.00	51	10.000	0.86	0.05	77	363.078	2.43	0.29				
26	0.316	0.11	0.01	52	11.482	1.05	0.06	78	416.869	2.60	0.36				
	0.363				13.183				478.630						



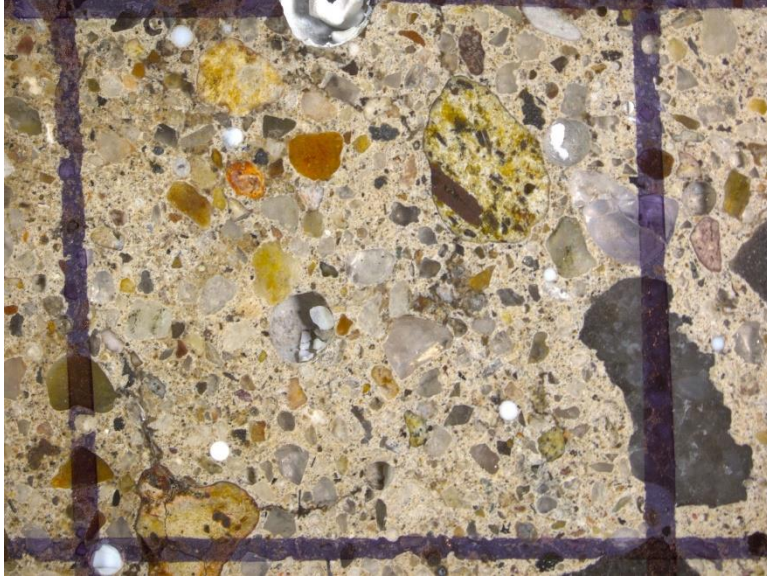
**Figure 24: Stereomicroscope to perform the DRI.**



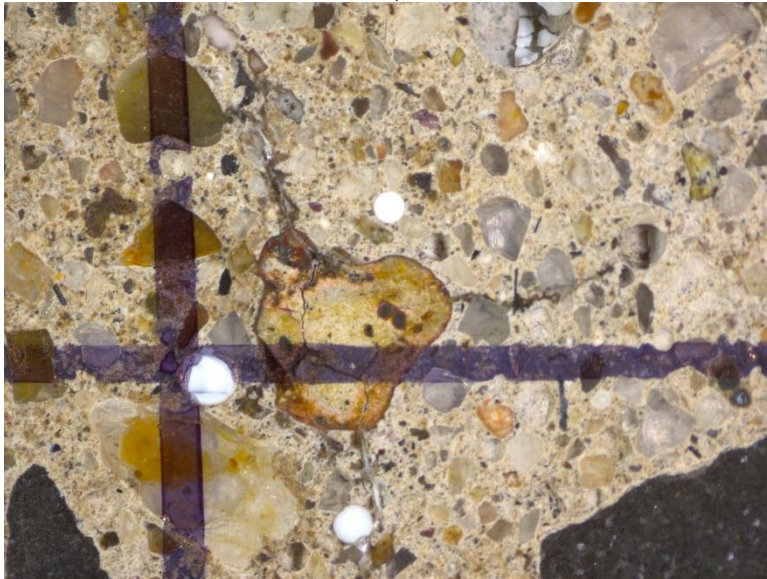
**A)**



**B)**

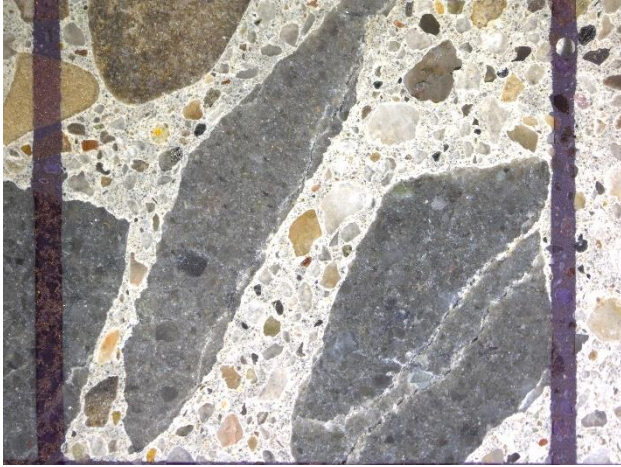


C)



D)

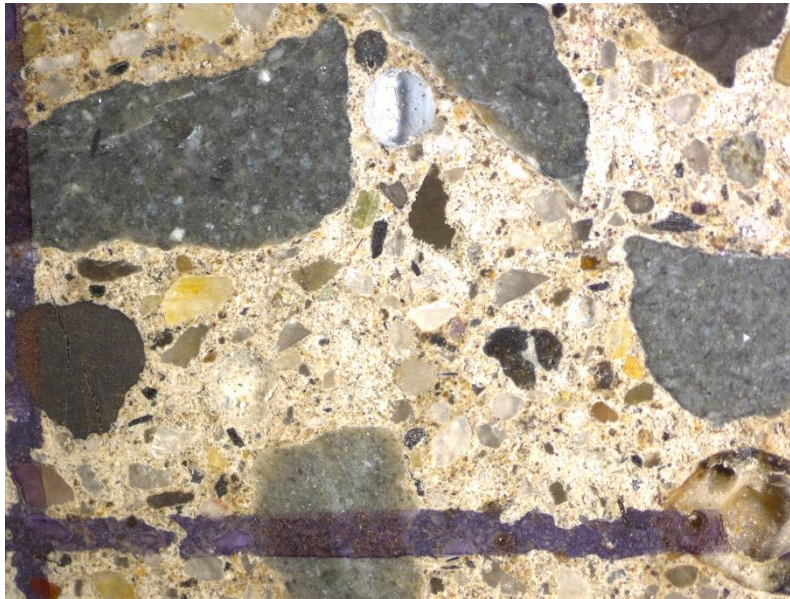
**Figure 25: Microscopic images for Tx sand mixtures: A) Texas control, B) WT-20%, C and D) WT-60%**



A)



B)



C)

**Figure 26: Microscopic images for SPH coarse mixtures: A) SPH control, B) WS-20% and C) WS-60%**

## Appendix C: Mineral Fillers (MF) - Complementary data results

Illustration of some CPT reading results: this procedure and results were obtained for each mixture presented in the document at selected time periods (i.e. 30,90,180 days).

**Table 22: CPT results for SPH mixture control zero reading**

Sample	Measurement			AVERAGE	Mass
	1	2	3		
1	- 3.910	- 3.922	- 3.920	161.083	3857
2	- 0.535	- 0.543	- 0.545	164.459	3951
3	0.792	0.795	0.790	165.792	3937
4	- 2.759	- 2.763	- 2.767	162.237	3866
5	3.230	3.225	3.227	168.227	3933
6	1.322	1.318	1.315	166.318	3955
7	- 3.746	- 3.748	- 3.750	161.252	3851
8	- 1.705	- 1.715	- 1.712	163.289	3928
9	- 1.952	- 1.955	- 1.964	163.043	3891
10	- 2.530	- 2.533	- 2.535	162.467	3842
11	2.120	2.125	2.117	167.121	3972
12	- 0.535	- 0.550	- 0.552	164.454	3907
13	- 0.770	- 0.775	- 0.780	164.225	3914
14	- 2.540	- 2.542	- 2.545	162.458	3851
15	- 2.777	- 2.788	- 2.790	162.215	3787
16	2.290	2.292	2.287	167.290	3921
17	2.235	2.230	2.232	167.232	3954
18	- 1.180	- 1.182	- 1.185	163.818	3935
19	0.965	0.960	0.957	165.961	3892
20	- 5.680	- 5.688	- 5.695	159.312	3898
21	3.816	3.810	3.807	168.811	3953
22	- 2.692	- 2.697	- 2.699	162.304	3886

23	0.785	0.780	0.770	165.778	3905
24	- 0.175	- 0.179	- 0.185	164.820	3937

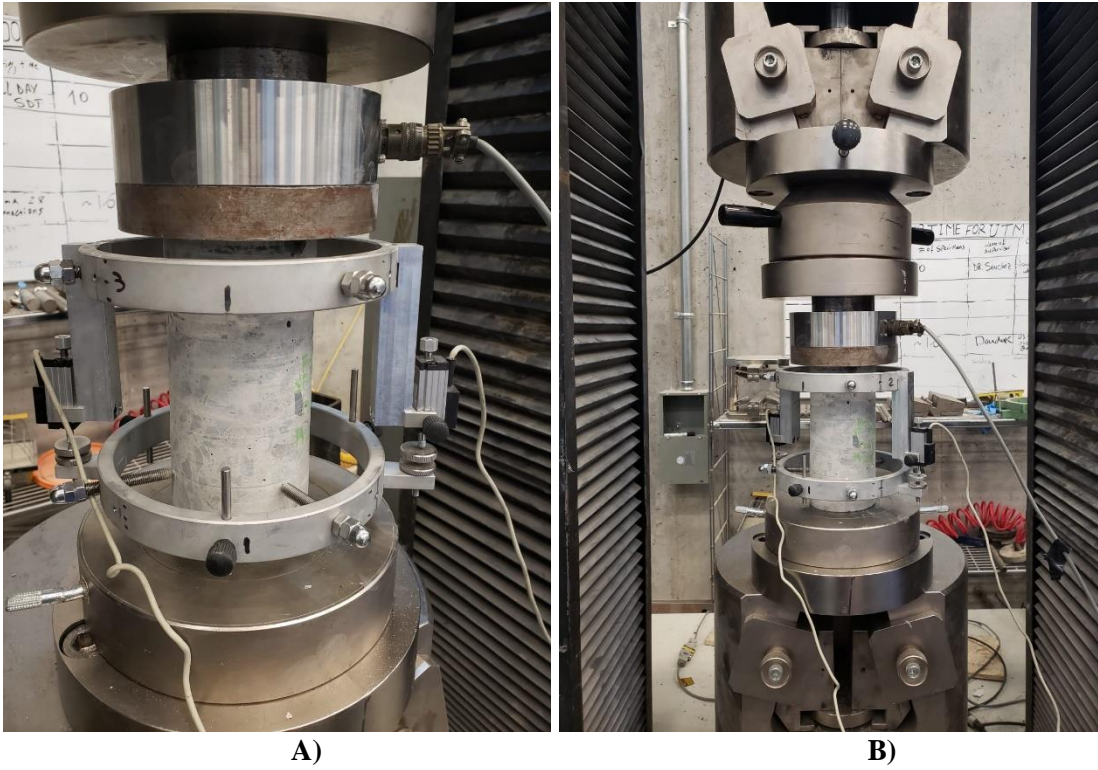
**Table 23: CPT results for SPH mixture control reading at 180 days.**

Sample	Measurement			AVERAGE	Mass	Expansion
	1	2	3			
1	- 3.398	- 3.399	-3.4	161.731	3941	0.40
2	0.01	0.004	0.002	165.135	4030	0.41
3	1.068	1.074	1.075	166.172	4015	
4	- 2.374	- 2.375	- 2.378	162.754	3957	0.32
5				#DIV/0!		
6	1.805	1.801	1.802	166.933	4048	0.37
7	- 3.256	- 3.257	- 3.258	161.873	3943	0.39
8	- 1.187	- 1.188	-1.19	163.942	4014	0.40
9				#DIV/0!		
10				#DIV/0!		
11	2.631	2.63	2.625	167.759	4070	0.38
12	- 0.153	-0.15	-0.15	164.979	3997	
13	- 0.375	- 0.375	-0.38	164.753	3997	
14	- 2.043	- 2.041	-2.04	163.089	3928	0.39
15	- 2.327	- 2.328	-2.33	162.802	3865	0.36
16	2.808	2.8	2.8	167.933	4009	0.38
17				#DIV/0!		
18				#DIV/0!		
19				#DIV/0!		
20				#DIV/0!		
21				#DIV/0!		
22				#DIV/0!		
23	1.364	1.366	1.365	166.495	3999	0.43
24				#DIV/0!		
						0.36

Table 24: Summary of some MF results

Mix						0 (day)		1 Month						3 Months				6 Months					
Filler Replacem ent	Co ars e	Fi n e	Nam e	Co mb o	Fa mili es	M ass	Resis tivity	Expa nsion	S D	Resis tivity	Mas s	Mass (Loss)	Stre ngth	Expa nsion	SD	Resis tivity	Mas s	Mass (Loss)	Expa nsion	S D	Resis tivity	Ma ss	
CONTR OL - Tx	X	O	Tx	7	Tx	39 64. 2	18.2	0.13	0. 0 4	34.50	398 3.52 9	0.49%	40.5	0.35	0.0 4				0.50	0. 0 2			
CONTR OL - SH	O	X	C	6	C	39 05. 1	24.3	0.09	0. 0 2	27.17	395 6.75	1.32%	42.6 7	0.22	0.0 2	28.2 5	397 8.94 7	1.89%	0.36	0. 0 3			
SH-15 %	O	X	S-15-OX	2	4	39 70. 5	12.2	0.09	0. 0 5	22.70	399 8.25	0.70%	46.0 0	0.26	0.0 2	20.4 5	402 6.11	1.40%	0.41	0. 0 2		22.00	403 3.3 3
	X	O	S-15-XO	3	6	39 02. 6	12.3	0.10	0. 0 2	28.50	392 6.54	0.61%	43.0 0	0.47	0.0 4	33.1 0	395 9.50	1.46%	0.57	0. 0 3		31.67	397 7.2 9
SH-15 %-C	O	X	S-15C-OX	2	7	39 37. 2	11.5	0.08	0. 0 5	19.90	396 6.22	0.74%	41.6 7	0.21	0.0 5	17.4 3	399 3.61	1.43%	0.38	0. 0 3		17.25	400 9.2 7
	X	O	S-15C-XO	6	9	39 25. 9	36.0	0.15	0. 0 4	30.30	397 8.17	1.33%	40.6 7	0.29	0.0 4	32.2 5	400 4.95	2.01%	0.47	0. 0 4		27.00	403 3.6 3
O-15 %	O	X	O-15-OX	1	13	39 65. 7	18.0	0.11	0. 0 4	20.79	398 4.45	0.47%	46.0 0	0.21	0.0 6	22.9 7	401 0.61	1.13%	0.51	0. 0 4		18.25	402 3.7 5
	X	O	O-15-XO	4	15	39 78. 0	12.4	0.11	0. 0 5	25.80	400 1.44	0.59%	43.6 7	0.49	0.0 4	28.8 3	403 7.33	1.49%	0.60	0. 0 4		27.33	405 4.2 7
O-15 %-C	O	X	O-15C-OX	1	16	39 81. 1	12.0	0.10	0. 0 4	20.89	400 1.76	0.52%	45.3 3	0.19	0.0 3	22.4 3	402 1.78	1.02%	0.47	0. 0 6		18.75	404 5.6 9
	X	O	O-15C-XO	5	18	39 13. 5	31.8	0.12	0. 0 3	25.35	393 5.42	0.56%	39.0 0	0.34	0.0 4	23.3 3	399 6.05	2.11%	0.51	0. 0 3		21.50	310 6.4 4
F-15 %	O	X	F-15-OX	2	22	39 63. 0	11.5	0.10	0. 0 3	19.63	398 9.14	0.66%	43.0 0	0.22	0.0 4	17.3 3	400 5.78	1.08%	0.38	0. 0 6		0.38	401 6.8 7

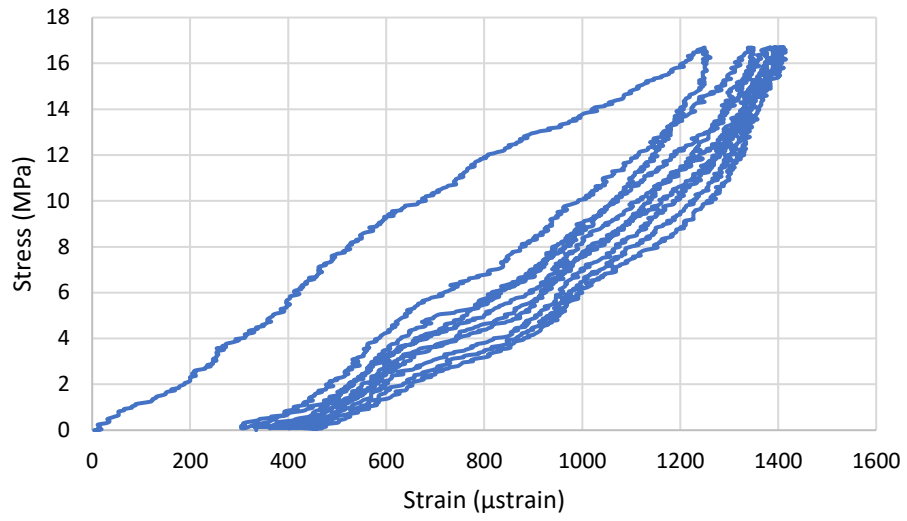
	X	O	F-15-XO	5	24	3900.7	31.3	0.10	0.03	27.63	392.017	0.50%	43.00	0.33	0.04	33.00	394.2.68	1.08%	0.50	0.04	29.83	396.2.63
F-15%-C	O	X	F-15C-OX	2	25	3887.2	10.5	0.09	0.03	19.13	391.4.52	0.70%	38.00	0.21	0.04	15.97	394.1.83	1.40%	0.28	0.05	13.25	396.1.53
	X	O	F-15C-XO	5	27	3888.6	16.1	0.10	0.05	25.70	390.7.83	0.49%	35.67	0.29	0.04	32.83	404.0.42	3.90%	0.40	0.03	29.00	394.7.50
K-15%	O	X	K-15-OX	3	31	3969.4	11.7	0.07	0.03	#DIV/0!	399.1.22	0.55%	48.33	0.20	0.02	20.45	403.1.71	1.57%	0.37	0.04	19.67	404.4.67
	X	O	K-15-XO	6	32	3935.0	29.1	0.16	0.03	30.32	398.4.04	1.25%	39.67	0.36	0.05	33.50	401.4.68	2.02%	0.53	0.03	34.33	404.4.73



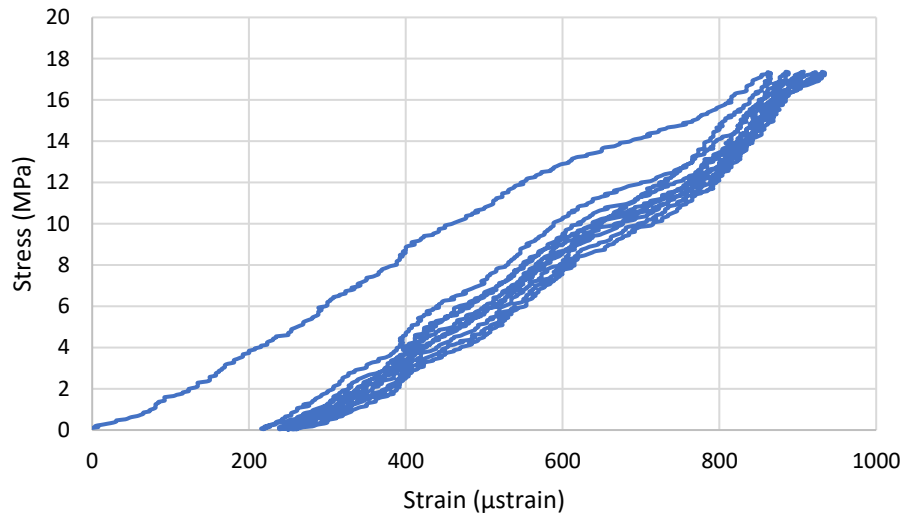
**Figure 27: Mechanical analyses – A and B) SDT and C) Compressive strength.**

**Table 25: SDT outcomes (i.e. SDI, PDI and ME)**

# Mixture s		SDI			PDI			%ME (loss)		
		SDI I	SDI II	SDI Avera ge	PDI I	PDI II	PDI Averag e	ME I	ME II	Avera ge
<b>C-TX</b>	<b>Tx</b>	<b>0.20</b>	<b>0.22</b>	0.21			#DIV/0!	<b>50</b>	<b>41</b>	46
<b>C -SPH</b>	<b>C</b>	<b>0.17</b>	<b>0.15</b>	0.16	<b>0.12</b>	<b>0.15</b>	0.135	<b>18</b>	<b>25</b>	21.5
<b>4</b>	<b>S-15- OX</b>	0.13	0.17	0.15	0.18	0.16	0.17	10.6	35.7	23.15
<b>6</b>	<b>S-15- XO</b>	0.2	0.2	0.2	0.08		0.08	41.1		41.1
<b>7</b>	<b>S-15C- OX</b>	0.21	0.21	0.21	0.3	0.16	0.23	31.4	31.8	31.6
<b>9</b>	<b>S-15C- XO</b>	0.29	0.25	0.27	0.23	0.14	0.185	44	42.2	43.1
<b>13</b>	<b>O-15- OX</b>	0.18	0.13	0.155	0.21	0.15	0.18	25.4	27.6	26.5
<b>15</b>	<b>O-15- XO</b>	0.2	0.18	0.19	0.13	0.19	0.16	28.6	27	27.8
<b>16</b>	<b>O-15C- OX</b>	0.20 5	0.20 5	0.20	0.08		0.08		32	32
<b>18</b>	<b>O-15C- XO</b>	0.26	0.27	0.265	0.22	0.13	0.175	38.4	41.5	39.95
<b>22</b>	<b>F-15- OX</b>	0.16	0.14	0.15	0.18	0.13	0.155	10.1	24.6	17.35
<b>24</b>	<b>F-15- XO</b>	0.23	0.21	0.22	0.14		0.14	44.5	36	40.25
<b>25</b>	<b>F-15C- OX</b>	0.25	0.17	0.21	0.23	0.27	0.25	36.2	12.3	24.25
<b>27</b>	<b>F-15C- XO</b>	0.28	0.25	0.265	0.27	0.17	0.22	37.6	37.6	37.6
<b>31</b>	<b>K-15- OX</b>	0.15	0.1	0.125	0.18	0.15	0.165	11.9		11.9
<b>32</b>	<b>K-15- XO</b>	0.18	0.2	0.19	0.09	0.09	0.09	23.8	31.7	27.75



**Figure 28: SDT stress-strain curve - Texas sand mixture**



**Figure 29: SDT stress-strain curve – SPH coarse mixture**