DEVELOPMENT OF HIGH PERFORMANCE SHRINKAGE RESISTANT CONCRETE, USING NOVEL SHRINKAGE COMPENSATING ADMIXTURES

by

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This thesis is submitted in fulfilment of the requirements for the degree of Doctor of Philosophy

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Certificate of Authorship / Originality

I certify that the work in this thesis has not previously been submitted for a degree nor has it been submitted as part of requirements for a degree except as fully acknowledged within the text.

I also certify that the thesis has been written by me. Any help that I have received in my research work and the preparation of the thesis itself has been acknowledged. In addition, I certify that all information sources and literature used are indicated in this thesis.

Pedram Hamedanimojarrad

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List of Abbreviations

APP	Ammonium Polyphosphates
AS	Australian Standards
ASTM	American Society for Testing and Materials
C_2S	Dicalcium Silicate
C ₃ A	Tricalcium Aluminate
C ₃ S	Tricalcium Silicate
C ₄ AF	Tetracalcium Aluminoferrite
СН	Calcium Hydroxide (Portlandite)
C-S-H	Calcium Silicate Hydrate
DTA	Differential Thermal Analysis
DTG	Differential Thermogravimetry
EnAPP	Encapsulated Ammonium Polyphosphates
EnAPP HAp	Encapsulated Ammonium Polyphosphates Hydroxyapatite
НАр	Hydroxyapatite
HAp HWR	Hydroxyapatite High-range Water Reducer
HAp HWR ITZ	Hydroxyapatite High-range Water Reducer Interfacial Transition Zone
HAp HWR ITZ Ke	Hydroxyapatite High-range Water Reducer Interfacial Transition Zone Keratin
HAp HWR ITZ Ke LOI	Hydroxyapatite High-range Water Reducer Interfacial Transition Zone Keratin Loss on Ignition
HAp HWR ITZ Ke LOI Me	Hydroxyapatite High-range Water Reducer Interfacial Transition Zone Keratin Loss on Ignition Melamine
HAp HWR ITZ Ke LOI Me MePP	Hydroxyapatite High-range Water Reducer Interfacial Transition Zone Keratin Loss on Ignition Melamine Melamine Polyphosphates

RH	Relative Humidity
SCA	Shrinkage Compensating Admixture
SDT	Simultaneous Differential Thermal
SEM	Scanning Electron Microscopy
SRA	Shrinkage Reducing Admixture
TG	Thermogravimetry
TGA	Thermogravimetric Analysis
Wo	Wollastonite
w/b	Water / binder
w/c	Water / Cement
wt. %	Weight Percent

Abstract

Shrinkage in cementitious materials can bring about major durability issues, beginning with a crack in a restrained section. This phenomenon causes contraction and consequently tensile stresses in the sample, and since the concrete and all conventional cementitious materials are not capable enough to bear tensile stresses, they crack. In conventional concrete, the major contribution to shrinkage strain belongs to drying shrinkage. Therefore, reducing the amount of drying shrinkage in concrete and cement mortars can result in lower total shrinkage strain and therefore, lessen the risk of cracking and ion invasion into the sample.

Shrinkage reducing admixtures (SRA), expansive agents and fibres are currently used in research and industry, in order to reduce the amount of shrinkage in one or more aspects; or at least decrease the risk of shrinkage cracking. Having been used in industry, SRAs and many other shrinkage compensating admixtures have been showing adverse side effects on the physical and mechanical properties of concrete and cement mortars; such as strength reduction and change in rheology.

Therefore, this research project aimed to introduce, investigate and develop new drying shrinkage compensating admixtures with no or limited reduction in mechanical strength, in order to be used in mortar, concrete and cementing compounds. The methodology of current research project was based on examining engineering physical and mechanical properties of OPC mortar samples associated with the addition of these new admixtures. Cementitious material used in this research project was ordinary Portland cement (OPC) mortar instead of concrete samples, in which the coarse aggregates provide more resistant to any expansion or contraction.

Firstly, a novel chemical composition of polyphosphates, Ammonium Polyphosphates (APP) that was designed and produced in Science Faculty of UTS, was adopted in current research. Based on its unique composition, this agent was expected to produce a cementing product (binder) via reaction with calcium hydroxide in the mix, where can enhance mechanical characteristics of the cement mortar and at the same

time generate ammonia gas (or volatile amine, as an anti-corrosion product), and might end up in generating expansion in the mix.

APP was used as an admixture inside OPC mortar samples and resulted in less shrinkage strain and enhanced compressive strength and workability. However, after more investigation it was concluded that the shrinkage compensation of APP could not only be due to the ammonia agent, but it was mainly related to the robust structure of calcium polyphosphate as one of the primary products of APP and calcium hydroxide reaction. This assumption was tested by the means of expansion studies and microstructural tests, and then comprehensively explained in Chapter 4.

On the other hand, APP showed some undesirable aspects such as ammonia odour during mixing. This downside aspect of APP was firstly addressed by encapsulating APP with a superplasticiser, and then by changing the chemical composition of APP to melamine polyphosphate or even testing melamine as an admixture. Encapsulation did not eliminate the ammonia release from the mix completely, but remarkably reduced the rate of ammonia release. Other admixtures however, were subjected to different results, due to different behaviours involved in them, which were not similar to the mechanism of APP in cementitious materials.

As mentioned earlier, fibres are mainly known for their effects on crack resistance rather than their ability to limit the shrinkage. This dissertation has a close look at microfibres as shrinkage resistant admixtures, as expected to be more efficient for their micro-crack bridging. This micro-bridging parameter was supposed to lock in shrinkage immediately as soon as it begins due to moisture loss.

Therefore, polyethylene (PE) microfibre was tested and used as an addition to OPC mortar samples before mixing. Results showed reduction in drying shrinkage, workability and compressive strength. Reduction in moisture loss of PE reinforced specimens suggested that the hydrophobicity of PE microfibres make it a shrinkage reducing admixture, since with considering their dimensions, they can be located in a way to block capillary pores and accordingly avoid moisture loss. Whereas, loss in strength might reveal this phenomenon that PE microfibre could not be efficient in micro-bridging of particles under stress. Another conclusion was that micro-bridging is not the mechanism behind the shrinkage reduction of PE microfibre.

Another admixture used was microfibre of an inosilicate mineral, wollastonite. Its micro-bridging characteristics and its positive influence on some mechanical properties of concrete were the incentives for being used in this research. Wollastonite microfibre reduced the amount of drying shrinkage and for some concentrations; it even enhanced the compressive strength, with no adverse effect on workability. Behavioural test results showed that micro-bridging of wollastonite microfibre can possibly be a reason behind its shrinkage reduction.

Early age behaviour of these admixtures has also been investigated. Current early age experiments and the information that each can give, were studied. A simple experiment was adopted from the literature, and mortar specimens were cast using optimum concentrations of these admixtures. The specimens were stored in a controlled drying environment and monitored for 24 hours. The number of major cracks, the cracking time and the dimensions of cracks have been assessed and analysed. Based on these experiments, APP, PE microfibre and wollastonite microfibre can significantly reduce the number and the size of early age cracks in cementitious materials. APP modified mortar could also delay the cracking time significantly.

This dissertation concludes that APP, PE microfibre and wollastonite microfibre reduce the drying shrinkage and early age cracking potential. APP and wollastonite microfibre have no side effect in mechanical characteristics of OPC mortars. These admixtures not only reduce the drying shrinkage in a cementitious material, but also control the early age cracking. By examining some other chemical compositions of polyphosphates, this thesis concludes that there are still new shrinkage compensating admixtures and mechanisms, which need to be examined and introduced to the industry.

Throughout this PhD research project, a number of conference and journal articles were submitted and published, while there is also one article still under peer review. These publications have been listed under Appendix B of this dissertation and copies of published papers have been attached.