# BEHAVIOUR OF SELF-COMPACTING CONCRETE INCORPORATING ALUM SLUDGE WITH OTHER POZZOLANS AS UNARY AND BINARY BLENDED CEMENTITIOUS SYSTEMS

**By**

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The conventional disposal (to landfill) of alum sludge produced from drinking water treatment plants is becoming a threat to the environment day by day as it contains hazardous substances. However, treated alum sludge (TAS), after calcination at temperature range of 100 to 950 ºC for 2 hours by an interval of 100 ºC, contains useful chemical compounds (like, silica and alumina) which are considered as main constituents of cement. This study explores the potential of treated alum sludge (TAS) at different percentages (with 5, 10, 15, 20, and 25%) to produce self-compacting concrete (SCC). Through strength activity index (SAI) 750ºC temperature was selected to produce TAS. The study also focused on the influence of TAS with silica fume (SF), fly ash (FA) and clinoptilolite (NZ) at different percentages (5, 10, and 15%) as binary blended pozzolans to produce SCC. Concrete mixes consist of total powder of 500 kg/m$^3$. The proportion of fine aggregate content was kept 50.46% of the total aggregate. The experimental work was divided into three stages that includes 48 mixes as well as three control mixes that contains three different water to powder (w/p) ratio (0.36, 0.38, and 0.4). In the first stage, TAS is utilized as a unary blended system at different percentages (with 5, 10, 15, 20, and 25%) to get the optimum level of replacement. Then in the second stage, other pozzolans are also used as unary blended system in SCC. Finally in the third stage, TAS is utilized as binary blended pozzolanic system together with FA, SF and NZ. In all stage, fresh properties of SCC are determined to meet the flowability requirements using the slump flow, v-funnel, v-funnel at T5 min, and L-box test. Bulk density, compressive strength, splitting tensile strength, flexural strength, and ultrasonic pulse velocity tests are conducted to evaluate the hardened properties. Group 3 (w/p ratio of 0.4) is selected to assess the durability properties of SCC. Shrinkage, initial surface absorption, water impermeability, water absorption tests; and tests on specimens exposed to elevated temperature and magnesium sulphate attack are also conducted to get the durability properties. Test results revealed that TAS can be utilized as a potential pozzolan to produce SCC. The early age (3 days) of compressive strength for all mixes ranges between 31.8 to 37.5 MPa for 100 mm standard cubes. The compressive strength at 28 days ranges between 49.5 to 85.8 MPa. Splitting tensile and flexural strengths are in between 3.91 to 5.68 MPa, and 5.59 to 8.96 MPa, respectively. The pore system of SCC is found refined in all mixtures with pozzolanic materials including TAS, SF, FA and NZ. Within pozzolanic activity index and characteristics of alum sludge, it can be considered it as natural pozzolanic. The experimental results showed an encouraging effect of TAS, SF, FA, and NZ on the fresh, hardened and durability properties of SCC up to 15%, 5%, 15%, and 10% replacement level, respectively. Therefore, alum sludge can be alternatively used in SCC either as unary or binary blended system with other pozzolans to achieve a greener and pollution free environment.

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# APPROVAL

This thesis was submitted to the Senate of Infrastructure University Kuala Lumpur (IUKL) and has been accepted as fulfilment of the requirement for the degree of (Doctor of Philosophy in Civil Engineering). The members of the Thesis Examination Committee were as follows:

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# DECLARATION

I, hereby, declare that this thesis, entitled ‘Behaviour of Self-Compacting Concrete Incorporating Alum Sludge with Other Pozzolans as Unary and Binary Blended Cementitious Systems’ is my own results except for quotations and citation which have duly acknowledged. I also declare that it has not been submitted anywhere for any award and is nor concurrently, submitted for any other degree at Infrastructure University Kuala Lumpur or at any other institution.

…………………………………

KHALID MOHAMMED BREESEM

Date:

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| ACI | American Concrete Institute |
| $Al_2O_3$ | Aluminium (III) oxide |
| ASTM | American Society for Testing and Materials |
| BS | British Standards |
| $C_2S$ | Di calcium silicate |
| $C_3A$ | Tri calcium aluminate |
| $C_3S$ | Tri calcium silicate |
| $C_4AF$ | Tetra calcium alumina-ferrite |
| CSH | Calcium Silicate Hydrate |
| EFNARC | European Guidelines for Self-Compacting Concrete |
| EPA | Environmental Protection Agency |
| $F_2O_3$ | Iron (II) oxide |
| FA | Fly Ash |
| GGBS | Ground granulated blast-furnace slag |
| ISI | Indian Standards Institution |
| ISAT | Initial surface absorption test |
| ITZ | Interfacial transition zone |
| IUKL | Infrastructure University Kuala Lumpur |
| LA | Los Angeles |
| LOI | Loss on Ignition |
| LPSD | Laser particle distribution size |
| $m^2/g$ | Square meter per kilogram |
| Mg | Magnesium |
| MgO | Magnesium oxide |
| $MgSO_4$ | Magnesium Sulphate |
| MK | Metakaolin |
| MnO | Manganese oxide |
| ND | No data |
| NVC | Non -vibrated concrete |
| NZ | Natural zeolite |

| | |
|---|---|
| OPC | Ordinary Portland cement |
| PAI | Pozzolanic activity index |
| PC | Portland cement |
| PSD | Pore size distribution |
| RAS | Raw alum sludge |
| RILEM | The International Union of Laboratories and Experts in Construction Materials, Systems and Structure |
| SAI | Strength activity index |
| SCC | Self-Compacting Concrete |
| SEM | Scanning electronic microscope |
| SF | Silica fume |
| SiO2 | Silicon dioxide (silica) |
| SP | Superplasticizer |
| SSA | Specific surface area |
| TAS | Treated alum sludge |
| TGA | Thermo gravimetric analysis |
| UKM | Universiti Kebangsaan Malaysia |
| UM | Universiti of Malaya |
| UPM | Putra Malaysia |
| UPV | Ultrasonic pulse velocity |
| VMA | viscosity-modifying agents |
| wt. | weight |
| WTP | Water treatment plant |
| XRD | X-ray diffraction |
| XRF | X-ray fluorescence |
| HWRA | High water reducing agent |
| SCM | supplementary materials |

# LIST OF SYMBOLS

| | |
|---|---|
| & | And |
| µm | Micrometre (10-6 metre) |
| ºC | Degree Celsius |
| Å | Angstrom (10-6 meter) |
| C | Cement content by weight |
| cm | Centimetre |
| $d_{50}$ | The median diameter or the medium value of the particle size distribution |
| $f_{c'}$ | Compressive strength |
| $F_V$ | V-funnel flow time in mortar and concrete tests |
| h | Hour |
| $H_2O$ | Water |
| kg | Kilogram |
| $kg/m^3$ | Kilogram per cubic metre |
| kN | Kilo Newton |
| m | metre |
| m/s | metre per second |
| min | Minute |
| mL | Millilitre |
| mm | Millimetre |
| MPa | Mega Pascal (N per mm2) |
| $R^2$ | Coefficient of correlation |
| sec | Second |
| SP | Superplasticizer |
| $T_{50}$cm | Time for a concrete flow to 50 cm in diameter in slump flow test |
| w/c | Water/cement ratio |
| w/p | Water/powder ratio |

# CHAPTER 1

# INTRODUCTION

## 1.1 Background of the Study

A Large quantity of alum sludge is generated each year from water treatment plants in Malaysia. An estimated over 2.0 million tonnes of water treatment sludge or residue (WTS) is produced annually by the water operators throughout the Malaysia. There is no doubt, alum sludge (AS) will continue to generate for a long time in future. There are no specific standards for drinking water treatment sludge (i.e. AS), but Act 672 :Solid Waste And Public Cleansing Management 2007 (Quality, 2007) categorised sludge that contains one or more metals including that alum sludge. For that matter, AS as the waste material will continue to be generated. This would result in massive quantities and require a high cost for disposal, and there would also be environmental effects as these wastes continue to accumulate. Therefore, this research aims to explore the potential reuse of alum sludge in the production of self-compacting concrete (SCC) as a partial replacement of cement which is so far remained unused and has not been studied.

## 1.2 Problem Statement

Existing methods for the disposal of alum sludge that is produced from drinking water treatment plants by landfill became unsafe and unfriendly to the environment as it contains heavy metals. In Malaysia, an estimated over 2.0 million tonnes of water treatment sludge or residue (WTS) is produced per year by the water operators throughout the country as a by-product of the process of purifying water for human consumption (National Water Services Commission (SPAN), 2010). The large quantities of alum sludge and the scarcity of land area are significant with growing problem of alum sludge disposal can be alleviated if new disposal options other than the landfilling can be found. New studies emerging worldwide spotlight on reusing the alum sludge. Alum sludge can create a greener environment in construction as it contains useful chemical compounds similar to that of cement composites. Treatment

## REFERENCES


Abdul Razak, H., & Wong, H. S. (2005). Strength estimation model for high-strength concrete incorporating metakaolin and silica fume. Cement and Concrete Research, 35(4), 688–695.

ACI 363R-92. (1992). State- of -the- Art Report on High- Strength Concrete. ACI Committee report363. American Concrete Institute.

ACI Committee 116. (2000). Cement and Concrete Technology, ACI 116R-00, ACI Committee 116 Report (p. 73 pages.). Farmington Hills, Michigan.

ACI Committee 234. (2000). Guide for the Use of Silica Fume in Concrete. In ACI 234R-96 Reapproved 2000 (pp. 1–51).

ACI Committee 318., & American Concrete Institute. (2011). Building code requirements for structural concrete (ACI 318-11) and commentary. American Concrete Institute.

Ahmaruzzaman, M. (2010). A review on the utilization of fly ash. Progress in Energy and Combustion Science, 36(3), 327–363.

Akçaözoğlu, K., Fener, M., Akçaözoğlu, S., & Öcal, R. (2014). Microstructural examination of the effect of elevated temperature on the concrete containing clinoptilolite. Construction and Building Materials, 72, 316–325.

Al-Akhras, N. M. (2006). Durability of metakaolin concrete to sulfate attack. Cement and Concrete Research, 36(9), 1727–1734.

Alexander, M., Bertron, A., & Belie, N. De. (2012). Performance of Cement-based Materials in Aggressive Aqueous Environments (Vol. 10).

Alqam, M., Jamrah, A., & Daghlas, H. (2011). Utilization of Cement Incorporated with Water Treatment Sludge. Jordan Journal of Civil Engineering, 5(2), 268–277.

American Public Health Association, American Water Works Association, & Water Environment Federation. (1999). Standard Methods for the Examination of Water and Wastewater (p. 541).

Amrutha, N., Narasimhan, M., & Rajeeva, S. (2011). Chloride-ion impermeability of self-compacting high volume of fly ash concrete mixes. International Journal of Civil & Environmental Engineering IJCEE, (August), 29–33.

Anagnostopoulos, N., Sideris, K. K., & Georgiadis, A. (2009). Mechanical characteristics of self-compacting concretes with different filler materials, exposed to elevated temperatures. Materials and Structures, 42(10), 1393–1405.

Annerel, E., Taerwe, L., & Vandevelde, P. (2007). Assessment of temperature