

Continuing development of new supplementary cementitious materials from lithium production residues

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Abstract: Initial material concrete trials were carried out on lithium residues generated from processing of West Australian spodumene in 2012 and limited test data presented at the CIA Biennial Conference in 2013. Subsequently a more comprehensive testing program was carried out in 2017 and some of the test results were reported at the CIA Biennial Conference in 2017. The balance of test results not previously reported are included in this new paper. The 2017 paper also identified some areas where further development was required.

Development of these lithium residue products has continued since 2017 with a view to providing closure for these outstanding issues, such as the potential to mitigate alkali expansion, and the assessment of long-term concrete properties such as those related to durability. As there are no current Australian Standards applicable to these products, a process for development of Australian Standards for natural and processed pozzolans is also underway.

The results of these investigations confirm these lithium residues are pozzolans and that they meet acceptable performance requirements for use in typical concretes and therefore the lithium residues have potential for use as supplementary cementitious materials in both domestic and structural concretes.

Testing programs have also been initiated to assess these lithium residues in wider range of applications outside of traditional concretes with a view to increasing usage in other products that depend on the hydraulic binders with pozzolanic properties.

Keywords: lithium by-product, environmental, recycling, sustainability.

1. Introduction

Previous investigation carried out by BCRC in 2012 and 2013 and reported in a paper by Haigh et al. published at the 26th Biennial Conference in the Gold Coast, October 2013, concluded that TAS lithium residues have pozzolanic properties and could to be used as supplementary cementitious materials in concretes. Tianqi Lithium Australia Pty Ltd has taken over the development of the TAS lithium residues and proposed the use of two different products to those originally trialled. Tianqi Lithium are currently constructing a new processing plant in Kwinana, Western Australia that is expected to be producing the lithium residues in commercial quantities in 2019. When this plant becomes operational, Tianqi neutralized residue (TAS) or non-neutralized residue (TAS Ca) are expected to become available in the south west of Western Australia for use as supplementary cementitious materials (SCM) later in 2019.

Considering that there has been substantial development of products since the earlier trials, the TAS Ca and TAS residues were reassessed in a further testing program and partial results were reported in a paper by Munn et al published in the 27th Biennial Conference in Adelaide in October 2017. Results of testing TAS products for use in domestic concretes were reported in the former paper and will not be repeated. This new paper includes reporting of the basic properties of the TAS products and the properties of the structural concrete including longer term concrete properties not reported in the 2017 paper. It also reports additional testing carried out of new samples of TAS lithium residues subjected to testing for alkali expansion.

2. Compliance with AS 3582.1 and ASTM C618 Specification Requirements

The two new Tianqi lithium residue products (TAS Ca and TAS) have been assessed in order to ascertain compliance with current AS 3582.1 and ASTM C618 requirements and also to compare their

properties with those of Collie fly ash from Western Australia (WA) and Eraring fly ash from New South Wales (NSW). As there is no current Australian Standard Specification for natural pozzolan, the requirements of ASTM C618 “Coal fly ash and raw or calcined natural pozzolan for use as a mineral admixture in Portland cement concrete” have been included in the assessment.

The test methods referred to ASTM C618 are not identical to those used in the AS 3582.1, but are generally similar. As highlighted in one of our previous reports, the limits as required in ASTM C618 should therefore, be interpreted as providing an indication of properties only.

The two new Tianqi lithium residues assessed in this investigation are:

TAS Ca – neutralized residue containing some limestone and gypsum.

TAS – non-neutralized residue product.

The specific properties and specification limits of the two products assessed are presented in Table 1.

Table 1 – Specific product properties and specification limits

Properties		AS 3582.1	ASTM C618	Eraring Fly Ash, NSW	Collie Fly Ash, WA	TAS Ca	TAS
Fineness passing 45µm, [%]	Fine	min 75	min 66	87	85		
	Medium	min 65		-	-		64.6
	Coarse	min 55		-	-	55	
Moisture, [%]		max 1	max 3	0.1	0.2	13.4	11.9
LOI, [%]		max 4-6	6-10	1.3	0.5	4.3	4.74
Relative Density		-		2.11	2.65	2.5	2.54
Relative water [%]		-	105-115	94.3	-	105	107
Relative strength	7 days	-	Min 75% of control	98	-	97	103
	28 days	-		94	-	91	96
SO ₃ , [%]		max 3	max 5	2	0.4	2.28	4.69
Chloride [%]		-	-	0.001	0.01	0.014	0.005
Magnesia [%]		-	-	0.3-0.5	1.1	0.07	0.06
Available alkali [%]		-	-	0.17	0.8	0.1	<0.01
SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃ [%]		-	min 70%	91.88	90.3	88.19	81.82

Notes (1) AS 3582.1 allow variations from reference limits if different testing methods are being used;

(2) In some areas in Australia, there are limited quantities of reactive fly ash available.

(3) The TAS Ca and TAS samples moisture contents were tested pre-drying. The products were oven dried for 24 hours before the trial mixes were carried out.

(4) Relative water demand and relative strength were determined by comparing the value obtained by testing with relevant values obtained by testing Portland cement mortar only.

The TAS residues are natural products that have been treated and probably composed of a specific type of zeolites. The TAS residues products are not fly ash but have good pozzolanic properties based on these tests and therefore are potential SCMs. The comparison with fly ash has been made for the purpose of assessing the two lithium residue products against a relevant Australian Standard.

Fineness - Both TAS Ca and TAS residues are coarse when compared to AS 3582.1 and ASTM C618 limits and also when they are compared with Eraring fly ash (NSW) and Collie fly ash (WA). The TAS lithium residue is finer than TAS Ca lithium residue. Finer TAS residue products would be expected to be more reactive and have better pozzolanic activity.

Moisture – Both TAS Ca and TAS lithium residue products possess high moisture contents prior to drying, with TAS Ca moisture content being slightly higher than that of TAS. Drying of the TAS products before packing/delivery, should be considered in order to assist the commercialization of product.

LOI – The loss on ignition properties are well within the AS 3582.1 and ASTM C618 limits and lower than previous TF 13 and TF 14 products tested. The source of the LOI is considered to be mainly combined water that is released above 600°C.

Relative density – The densities of TAS Ca and TAS residues are in the normal range for fly ash.

Relative water – Both TAS Ca and TAS products exhibit higher water demand when compared with Eraring fly ash. This is likely to result mainly from the differences in fineness and particle shape. However, the relative water demands are less than the ASTM C 618 maximum limit. There is no significant difference in water demand between the two products assessed.

Relative strength – At 7 days, the relative strength is well within ASTM C618 Specification limits. The results are also similar to Eraring fly ash relative strength at the same age. At 28 days the relative strengths are also within the ASTM C618 specification limits, with better performance from the TAS lithium residue product.

SO₃ – The results for TAS Ca is below the maximum limit of AS 3582.1. The TAS result is slightly higher but less than the ASTM C618 maximum limit of 5% that may be a more appropriate upper limit.

Chlorides – The test result is low for TAS lithium residue and higher for TAS Ca lithium residue.

Magnesia – Both TAS Ca and TAS lithium residue products contain low magnesia when compared with Eraring and Collie fly ashes. There is no need for an autoclave expansion test as the MgO content is well below the 4% requirement for the test to be performed.

Available alkali – The test is not mandatory in the AS 3582.1 but may be specified, tested and reported. The two TAS Ca and TAS lithium residue products exhibit low alkali content.

SiO₂+Al₂O₃+Fe₂O₃ – It is well known that high SiO₂ content in SCM products assist in the strength development and control of the alkali silica reaction (ASR). Furthermore, in order to ensure that a high pozzolanic activity is present, the ASTM C618 requires a minimum of 70% of SiO₂+Al₂O₃+Fe₂O₃. The TAS Ca and TAS lithium residue products satisfy these requirements.

The SiO₂ content of TAS Ca lithium residue was 65% and for TAS lithium residue, 59% that are regarded as silica contents that support pozzolanic activity.

3. Assessment of the TAS Ca and TAS lithium residues in structural, high durability concretes

High durability concretes play an important role in WA concrete industry and in particular in the mining industry, gas and port projects, railway, bridges, etc. The assessment of the TAS Ca and TAS products in high durability, structural concrete was carried out as required for a typical bridge concrete mix in NSW as per B80-B2 Class exposure. The design strength grade was 40 MPa, 20 mm maximum sized aggregate was used and nominal slump was 120 mm. The results for fresh concrete properties are presented in Table 2 and for hardened concrete properties presented in Table 3.

The trial mix program included:

- a typical control mix (B80/B82 class exposure) used in Australia with fly ash cement;
- two concrete mixes using 25% of TAS Ca and 25% TAS residues as fly ash replacement.

3.1 Comments on fresh properties of structural, high durability concretes

- **Slump** – No difficulty was experienced in mixes containing TAS Ca & TAS lithium residues achieving the slump requirements of the mix design (120 ±20mm).
- **Water/Cement** – The results are not significantly different to control for both TAS residue concretes.
- **Bleeding** – There is no negative impact on bleeding and the TAS mix shows reduced bleeding.
- **Air content** – TAS mixes are not significantly different to the control mix.
- **Setting time** – TAS Ca lithium residue setting time is slightly shorter than control (both initial and final setting), but overall there is no significant difference from the control mix.

As a general comment, there are no negative impacts on fresh concrete properties when TAS Ca and TAS residue products as used as SCM (25%) replacing fly ash.

Table 2 – Fresh concrete properties

Concrete Properties	Control 25% fly ash	TAS Ca 25%	TAS 25%
Slump [mm]	125	120	110
Water/cement	0.44	0.45	0.45
Bleeding [%]	1.6	1.5	0.9
Air Content [%]	1.4	1.2	1.2
Setting time [min]	365/460	355/450	375/465

3.2 Comments on hardened concrete properties of structural, high durability concretes

- **Compressive strength**

Age 1 day - TAS and TAS Ca mixes are similar to control.

Age 7 days - TAS Ca and TAS lithium residue mixes are similar to control.

Age 28 days - The compressive strengths of both TAS and TAS Ca mixes increase significantly and are approximately 25% greater than the control concrete.

Age 56 days - The compressive strengths of both TAS and TAS Ca mixes increase significantly and are approximately 6% to 12% greater than the control concrete.

Testing of the structural concretes confirms that both TAS Ca and TAS concretes exhibit good pozzolanic activity. The strength growth profile indicates that, due to the coarser particle size distribution of both residues compared with fine grade fly ash, the pozzolanic activity takes some time to take effect. Reducing the mean particle size of the TAS products may accelerate pozzolanic reactivity.

- **Drying shrinkage** – The test results at 21, 28 and 56 days for the TAS and TAS Ca concretes are lower when compared with control concrete.
- **Sorptivity** – The results for TAS Ca and TAS lithium residue concretes are better than control concretes. All results for TAS Ca and TAS are well below the specifications requirements of maximum 25 mm. Both TAS Ca and TAS contribute to better concrete durability.
- **Chlorides and sulphates** – In general chloride and sulphate contents for TAS and TAS Ca concretes are low.
- **Chloride ion diffusion** – The Nordtest Method NT Build 443 was used to assess chloride ion diffusion coefficients after 35days immersion of concrete samples in 16.5% sodium chloride solution. When compared with the control concrete containing 25% Eraring fly ash in the binder, the concrete containing 25%TAS Ca exhibited a significant reduction of 41% in the chloride ion diffusion coefficient and the concrete containing 25%TAS, a significant reduction of 33% in the chloride ion diffusion coefficient.
- **General** - These test results show that concretes containing TAS products have generally superior performance in strength, serviceability and durability related properties when used in similar proportions and comparable concretes made with well recognised fly ash.

Table 3 – Hardened concrete properties

Concrete Properties		Control 25% fly ash	TAS Ca 25%	TAS 25%
Compressive strength, [MPa]	1 day	14.4	13.7	14.7
	3 days	30	27.1	28.1
	7 days	38.2	37.8	39.4
	28 days	49.4	61.6	62.3
	56 days	60.9	64.7	69.1
Drying Shrinkage [µm]	21 days	450	430	430
	28 days	490	460	470
	56 days	580	540	540
Sorptivity at 7 days [mm]	wet	14	11.5	12.4
	bagged	18	14.9	14.7
	waxed	18.9	14.2	13.9
Chlorides [%]		0.007	0.011	0.009
Sulphates [%]		0.8	0.78	0.71
NT443 Chloride Diffusion [E-12m ² /sec]		3.35	1.98	2.26

4. Initial assessment of the TAS lithium residues (TAS Ca and TAS) to control alkali silica expansion.

Certain aggregates used in Perth and elsewhere in Western Australia are known as having potential for ASR, with associated long-term expansion in concrete structures. It is well established that an effective mitigation measure for ASR expansion in concrete is the inclusion of SCM such as fly ash, silica fume, blast furnace slag at appropriate levels in concrete mixes.

The ASR assessment was carried out for TAS products on mortars as per the accelerated mortar bar test method, AS1141.60.1 -2015, using a highly reactive aggregate:

- Control mix 1, with a known highly reactive aggregate (Teven Quarry) and GP cement.
- Control mix 2, with the same aggregates and 75% GP cement plus 25% fly ash (Ering).
- TAS Mix 3, fully replacing the fly ash in control 2 with TAS Ca lithium residue.
- TAS Mix 4, fully replacing the fly ash in control 2 with TAS lithium residue.
- The results are presented in Table 5.

According to AS 1141.60.1, the aggregate reactivity classification is as presented in Table 4 below:

Table 4 – Classification of aggregates for potential expansion

Mortar Bar Expansion -E (% in 1M NaOH @ 80°C)		Classification
10 days	21 days	
-	$E < 0.10^*$	Non - reactive
$E < 0.10^*$	$0.10^* < E < 0.30$	Slowly reactive
$E > 0.10^*$	-	Reactive
-	$0.30 < E$	Reactive

*0.15% for fine aggregates

A summary of ASR assessment test results for TAS Ca and TAS is presented in Table 5 below:

Table 5 – Mortar bar expansion (% in 1M NaOH at 80°C)

Age/Cement type	GP cement only (control 1)	75%GP + 25% fly ash (control 2)	75% GP + 25% TAS Ca	75% GP + 25% TAS
10 days	0.56	0.01	0.05	0.1
21 days	1.04	0.02	0.14	0.39

The test results were compared with the acceptable limits for ASR according to the AS 1141.60.1 and from the data obtained above the following comments are made:

- TAS Ca at 10 days is classified non-reactive and at 21 days is slowly reactive.
- TAS at 10 days is classified slowly reactive and at 21 days is reactive.
- Both TAS Ca and TAS lithium residue products have assisted in reducing the ASR reactivity level from 0.56 at 10 days to 0.05 TAS Ca and 0.10 TAS, a reduction of 82 – 91%.
- At 21 days the reduction in ASR reactivity for TAS Ca product is 86%, while the reduction for TAS product is 62%.

- Based on the initial assessment TAS would be expected to be useful to control slowly reactive aggregates at 25% binder replacement but not fully control expansion of a highly reactive aggregate. Further testing at different replacement levels for GP cement was recommended.

5. Further assessment of the Tianqi lithium residue (TAS Ca) to control alkali silica expansion.

Considering the limited TAS lithium residue potential for ASR mitigation in concretes shown in the initial assessment, the following ASR assessment was carried out using three TAS Ca products with different fineness in mortars with different cement replacement levels. The fineness of the TAS Ca was altered by screening the coarser fractions from the initial product by a process similar to classification of fly ash:

- Control 1 mortar with GP cement
- Control 2 mortar with 75% GP cement and 25% fly ash cement replacement
- Three mortar mixes with coarse grained TAS lithium residue at 3 levels of GP cement replacement – 25%, 30% and 35%.
- Three mortar mixes with medium grained TAS lithium residue at 3 levels of GP cement replacement – 25%, 30% and 35%.
- Three mortar mixes with fine grained TAS lithium residue at 3 levels of GP cement replacement – 25%, 30% and 35%.

Accelerated mortar bar tests to 21 days as per AS 1141.60.1 were carried out on the 11 mixes, with GP cement, fly ash and highly reactive aggregates (glassy basalt) being sourced by BCRC.

A summary of ASR assessment results is presented in the following Tables 6, 7 and 8 for the TAS products with different fineness and levels of GP cement replacement, together with reactivity classification according to Table 4.

Coarse grained TAS Ca lithium residue with approximately 65% passing 45 micron sieve

Table 6. Mortar bar expansion and classification of coarse grained TAS lithium residue

Cement replacement %	Expansion %		Reactivity Classification
	10 days	21 days	
25	0.03	0.07	Non-reactive
30	0.02	0.05	Non-reactive
35	0.02	0.04	Non-reactive

The GP cement replacement by coarse grained TAS in the mortar mixes with 25%, 30% and 35% TAS lithium residue (65% passing 45 micron), has resulted in sufficient mitigation of ASR potential expansion to safely permit use of a highly reactive glassy basalt aggregate in concrete. Potential expansion decreased as the proportion of TAS replacement increased.

Medium grained TAS Ca lithium residue with approximately 85% passing 45 micron sieve

Table 7. Mortar bar expansion and classification of medium grained TAS lithium residue

Cement replacement %	Expansion %		Reactivity Classification
	10 days	21 days	
25	0.02	0.06	Non-reactive
30	0.02	0.03	Non-reactive
35	0.02	0.03	Non-reactive

The GP cement replacement by medium grained TAS in the mortar mixes with 25%, 30% and 35% TAS lithium residue (85% passing 45 micron), has resulted in greater mitigation of ASR potential expansion than the coarse grained TAS to safely permit use of a highly reactive glassy basalt aggregate in concrete. Potential expansion decreased as the proportion of TAS replacement increased and expansion was reduced to levels similar to those reported for fly ash in control mix 2.

Fine grained TAS Ca lithium residue with approximately 98% passing 45 micron sieve

Table 8. Mortar bar expansion and classification of fine grained TAS lithium residue

Cement replacement%	Expansion %		Reactivity
	10 days	21 days	Classification
25	0.02	0.05	Non -reactive
30	0.02	0.04	Non-reactive
35	0.02	0.04	Non-reactive

The GP cement replacement by fine grained TAS in the mortar mixes with 25%, 30% and 35% TAS lithium residue (98% passing 45 micron), has resulted in sufficient mitigation of ASR potential expansion to safely permit use of a highly reactive glassy basalt aggregate in concrete. Potential expansion decreased as the proportion of TAS replacement increased however the magnitude of expansion at higher levels of replacement was similar to that of the medium grained TAS.

Mortars tested with all grades of TAS confirmed mitigation of ASR with highly reactive aggregate, however it appears that no significant benefit has resulted from the using the fine grade product compared with the medium grade TAS. Hence the medium grade TAS Ca is recommended for use at a GP cement replacement rate of 25%.

6. Accelerated carbonation testing

6.1 Test Method

Accelerated carbonation testing was carried out on cylinder samples from the structural concretes testing program made with TAS Ca and TAS discussed in Section 3, following wet curing for 28days and dried at 50% relative humidity for 21 days at 23oC. Testing for carbonation was subsequently undertaken on these samples according to the method developed by CSIRO in the Boral Technical Services Laboratory at Baulham Hills. The test samples were wax coated with only one end face exposed to CO₂ in the testing chamber. The conditions in the testing chamber were maintained at 23oC, relative humidity of 60% and CO₂ concentration of 4%. The depth of carbonation was determined progressively by measurement of depth of carbonation of three samples from each concrete mix by spraying with phenolphthalein as per RILEM Procedure CPC -18.

The performance of the two 40MPa TAS concretes was compared with the control concrete mix using 25% replacement of GP cement with Eraring fly ash. Hence the three concretes assessed were:

- 40MPa control concrete with GP cement replacement of 25% Eraring fly ash
- 40MPa concrete with GP cement replacement with 25% TAS Ca residue
- 40MPa concrete with 25% cement replacement with TAS residue

6.2 Results of accelerated carbonation testing

Table 9 sets out the average depths of carbonation of the three samples tested for each concrete mix at ages of 7, 28, 63 and 112days exposure to the 4% CO₂ environment.

Table 9. Accelerated depth of carbonation

Concrete Type	Control 25% fly ash	TAS Ca 25%	TAS 25%
Age	Depth (mm)	Depth (mm)	Depth (mm)
7days	3.6	2.9	3.0
28days	6.4	6.2	5.4
63days	10.5	8.6	9.0
112days	12.3	11.5	11.6

The test results show that the depth of carbonation under this accelerated test for the 40 MPa TAS Ca and the 40MPa TAS concretes are lower than that of the control concrete containing fly ash at each respective age. The TAS product concretes would therefore be expected to have lower carbonation depths than the control concrete at all ages subject to normal carbonation rates in field exposure.

Interpretation of these accelerated carbonation test results requires reference to performance of concretes in the field as the carbonation concentration in the test chamber is approximately 100 times greater than that of typical field exposure. According to AM Neville, Properties of Concrete, the carbonation depth is a function of compressive strength and the typical environment of the concrete. For a concrete with water/cement of 0.6, a typical depth of carbonation of 15mm after 3 months could be expected under these accelerated test conditions. Neville indicates that for a similar concrete under field exposure, the depth of carbonation would be expected to be 15mm after 15 years.

In this case, the water/cement is approximately 0.45 and the measured carbonation depths in the accelerated test in an atmosphere of 4%CO₂ were approximately 12mm after 112days (or 0.3years). Using the equation for conversion recommended by Neville of:

$$t_i / t_s = c_s / 100,$$

where t_i is the period of field exposure, t_s is the accelerated test period and c_s is the CO₂ content used in the accelerated test.

The above equation predicts that time in excess of 80years will be required for TAS field concrete to reach a carbonation depth of 12mm and a period of approximately 125years will be required for a carbonation depth of 15mm to be realised. As the TAS concretes have lower accelerated carbonation depths than the fly ash concrete, field carbonation depths of the TAS concretes are also predicted to be proportionately lower than those of the fly ash concrete at the same age.

7. Conclusions

Both TAS Ca and TAS lithium residue products show good pozzolanic activity with positive effects on fresh and hardened concrete properties and so could be used as partial GP cement, or fly ash replacement in concrete mixes, despite being coarser than typical fly ash. The two products were assessed for their basic properties and performances against current AS 3852.1 for fly ash and references were made to ASTM C618 specification, applicable to natural pozzolan.

The TAS Ca and TAS lithium residue product have higher moisture content than AS 3582.1 and ASTM C618 specification limit. Consideration should be given to significantly reduce the moisture in order to permit adequate handling of the bulk product in practice.

The TAS Ca lithium residue meets SO₃ limits for AS3582.1 and ASTM C618 limits. Chlorides are very low for TAS lithium residue and slightly higher in TAS Ca residue, magnesia and available alkali are also low and well within specified limits. The SiO₂ + Al₂O₃ + Fe₂O₃ of both TAS residues meet the requirements of the ASTM C618 for a natural pozzolanic material.

TAS Ca and TAS lithium residues are good SCMs in concrete and improve both concrete strength and durability. Drying shrinkage results for TAS and TAS Ca in 40MPa concretes are also slightly lower than the fly ash control concrete. The sorptivity tests and chloride diffusion tests to NT Build 443 in 40MPa concretes also recorded significantly lower results in all cases compared with the fly ash control mix, and well within the typically specified limits.

Considering the limited TAS lithium residue potential for ASR mitigation in concretes shown in the initial assessment, the following ASR assessment was carried out using three TAS Ca products with different fineness in mortars with different cement replacement levels. Mortars tested with all grades of TAS confirmed mitigation of ASR with highly reactive aggregate, however it appears that no significant benefit has resulted from the using the fine grade product compared with the medium grade TAS. Hence the medium grade TAS Ca is recommended for use to mitigate ASR at 25% rate.

Accelerated carbonation tests predict that time in excess of 80years will be required for TAS field concrete to reach a carbonation depth of 12mm and a period of approximately 125years will be required for a carbonation depth of 15mm to be realised. As the TAS concretes have lower accelerated carbonation depths than the fly ash concrete, field carbonation depths of the TAS concretes are also predicted to be proportionately lower than those of the fly ash concrete at the same age.

Both TAS and TAS Ca lithium residues have been found to be good SCMs in in 40MPa concrete with improved strength, serviceability and durability compared with good quality fly ash. In addition, the TAS products successfully mitigate ASR expansion, reduce sorptivity, reduce chloride diffusion and reduce depth of carbonation. These TAS products have potential for widespread use to partly replace GP cement and replace other SCMs in both domestic and structural concretes.

8. Acknowledgements

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