

SP2 Report on

**Conversion and Its Effect on CAC Corrosion-
Based on Accelerated Corrosion Tests**

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Summary

Calcium aluminate cement is used as a protective lining against the corrosive attack of micro-organism and their metabolic products on reinforced concrete sewer pipes. The corrosion behaviour of CAC cement is related to the process of conversion of aluminate hydrates, where full conversion can result in as much as 1-4 times increase in the rate of corrosion. Therefore controlling and understanding the rate and the processes that affects conversion is critical in the effective use of CAC as a mitigation strategy against microbiological attack.

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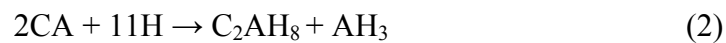
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1.0 INTRODUCTION

Calcium aluminate cements (CAC) is a specialty cement material with a main constituent of calcium aluminate. This material has a wide range of application in both the construction industry and as a sacrificial protective lining against corrosive environment because of its ability to gain early strength and its resistance to aggressive chemical environments and temperatures (Dumas 1990, Midgley 1990, Sand, Dumas et al. 1994, Scrivener, Cabiron et al. 1999, Chotard, Gimet-Breart et al. 2001, Lamberet 2008).

1.1 Hydration Reactions

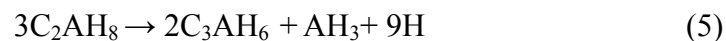
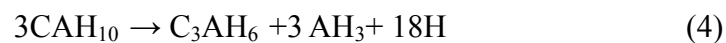
Calcium aluminate once hydrated undergoes a series of chemical reaction that influences both its physical, mechanical and chemical properties. Calcium aluminate cement reacts with water to form metastable (CAH_{10} and C_2AH_8) and stable hydrates (C_3AH_6) through time and temperature dependent reactions (Scrivener, Cabiron et al. 1999, Chotard, Gimet-Breart et al. 2001, Juenger, Winnefeld et al. 2011):



The designations for these chemical formulae are C - CaO, A – Al_2O_3 and H - H_2O .

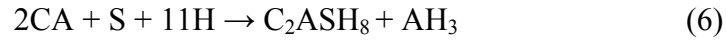
1.2 Conversion Reactions

Once hydrated CAC is subjected to further chemical change referred to as conversion that further transform the metastable phases to the stable phase (Bradbury, Callaway et al. 1976):



The key concern with conversion is that it leads to the formation, with time, of less dense precipitates and cement resulting in increased porosity, reduced hydraulic strength and increases susceptibility to corrosive attack (Bradbury, Callaway et al. 1976, Jambor and Skalny 1996, Chotard, Gimet-Breart et al. 2001). This process is thermodynamically driven and is therefore inevitable (Gosselin, Gallucci et al. 2010). The speed of conversion may take years at low temperature but could be accelerated with a high water/cement ratio and at elevated temperatures (Bradbury, Callaway et al. 1976, Barnes and Baxter 1978, Collepardi, Monosi et al. 1995, Pacewska, Wilinska et al. 2011). The continuous conversion of this material will adversely affect the long term durability of CAC.

The introduction of vitreous silica provides an alternative reaction to the converted C_3AH_6 phase to an aluminium hydrate with silica C-A-S-H phase, stratlingite (C_2ASH_8) or hydrated gehlenite (C_2AS) (Colleparidi, Monosi et al. 1995, Ding, Fu et al. 1995, Bensted 1996, Hidalgo, Garcia et al. 2009):



The aim of this aspect of the study is to examine the effect of conversion on the mineralogical transformation of CAC and its rate of corrosion.

2.0 EXPERIMENTAL

2.1 Materials

The materials used in this study included a commercially available calcium aluminate cement (Sewpercoat) sourced from Kerneos, Australia and fly ash from a power station. The chemical compositions of these materials were determined by X-ray fluorescence spectroscopy and are shown in Table 1.

Table 1. Composition of calcium aluminate cement and pozzolanic materials

Oxides (wt%)	Calcium Aluminate Cement	Fly Ash
Al_2O_3	41.3	6.86
CaO	37.4	9.53
Cr_2O_3	0.13	0.06
Fe_2O_3	9.54	2.03
K_2O	0.3	0.18
MnO	0.12	0.05
MgO	0.3	1.8
NiO	0.137	0.114
SiO_2	6.97	79.1
TiO_2	1.93	0.36
V_2O_5	0.05	--
ZrO_2	0.14	--

2.2 Preparation of the calcium aluminate cement specimens

The cement specimens were prepared according to manufacturer's instructions. The water to cement ratio used was 0.4. The cement mixture and water were mixed mechanically for 3 minutes and poured into 2 x 4 inch stainless steel molds, compacted and the surface smoothed manually. The molded specimens were cured for 24 hours at 21°C in 98% relative humidity environmental chamber, demolded and cured further in a water bath with a controlled temperature of 30°C at various periods of time up to 28 days.

2.3 Characterisation of cement specimen

The phase transformations during the curing and conversion of CAC were monitored with X-ray diffraction analysis using XRD Siemens D5000 and by thermal gravimetric analysis using Perkin Elmer TGA 4000. Thermal analysis was carried out by heating the samples from 30 to 1000°C with a heating rate of 10°C/min in nitrogen metered at 20 ml/min.

2.4 Accelerated and field corrosion tests

To simulate the corrosion of CAC in sewers, the cured specimens were immersed in 5 wt% sulphuric and citric acids maintained at 26-27°C. The pH and temperature of the solutions were monitored with pH-temperature probes coupled with a HNH19143 meter. Additional acids were added to adjust and maintain the pH. To measure the thickness loss, loose or soft corrosion products were scraped off. The specimen diameters were then measured using a vernier caliper. Field testing involved the installation of coupon specimens of CAC in the Southern and Western Suburbs Ocean Outfall Sewers (SWSOOS), South Barrel in Sydney for 364 days.

2.5 Dissolved Metal Analysis

Dissolved metals were analysed by ICP-AES using standard procedures.

3.0 RESULTS

3.1 CAC Conversion

The conversion of CAC during curing was examined thermally by thermal gravimetric method. The CAC hydrates were identified according to Table 2. The DTG of CAC samples cured at various periods of time demonstrate the conversion of the metastable phase to the stable hyrogarnet phase (see Error! Reference source not found.1). As shown there is a rapid formation of the converted phases (AH_3 , CAH_{10} and C_2AH_8) after 1 day of curing followed by a slower conversion. The conversion continued for 28 days with distinct shift from

metastable phases (CAH_{10} and C_2AH_8) evident from loss of water from 50 to 180 °C to the
more stable converted phases

Table 2. Thermal decomposition of calcium aluminate hydrates.

Calcium Aluminate Hydrates						Heating Rate	Method	References
CAH ₁₀	C ₂ AH ₈	C ₃ AH ₆	AH ₃ gel	Al(OH) ₃	C ₂ ASH ₈			
	110, 175, 295					10°C/min	DTG	(Ukrainczyk, Matusinovic et al. 2007)
96				266		20°C/min	DTG	(Barnes and Baxter 1978)
110				267		5°C/min	DTG	(Guirado, Gali et al. 1998)
	170, 230, 275	320				10°C/min	DTG	(Das, Mitra et al. 1996)
110-120		320-350		295-310		25°C/min	DTA	(Bradbury, Callaway et al. 1976)
120		310		290		10°C/min	DTA	(Nilforoushan and Talebian 2007)
			90			10°C/min	DTA	(Calvo, Alonso et al. 2013)
137	137				280-350	10°C/min	DTA	(Hidalgo, Garcia et al. 2009)
					100, 138 and 240	10°C/min	DTG	(Kuzel 1976)
					120, 165 and 220	10°C/min	DTG	(Matschei, Lothenbach et al. 2007)

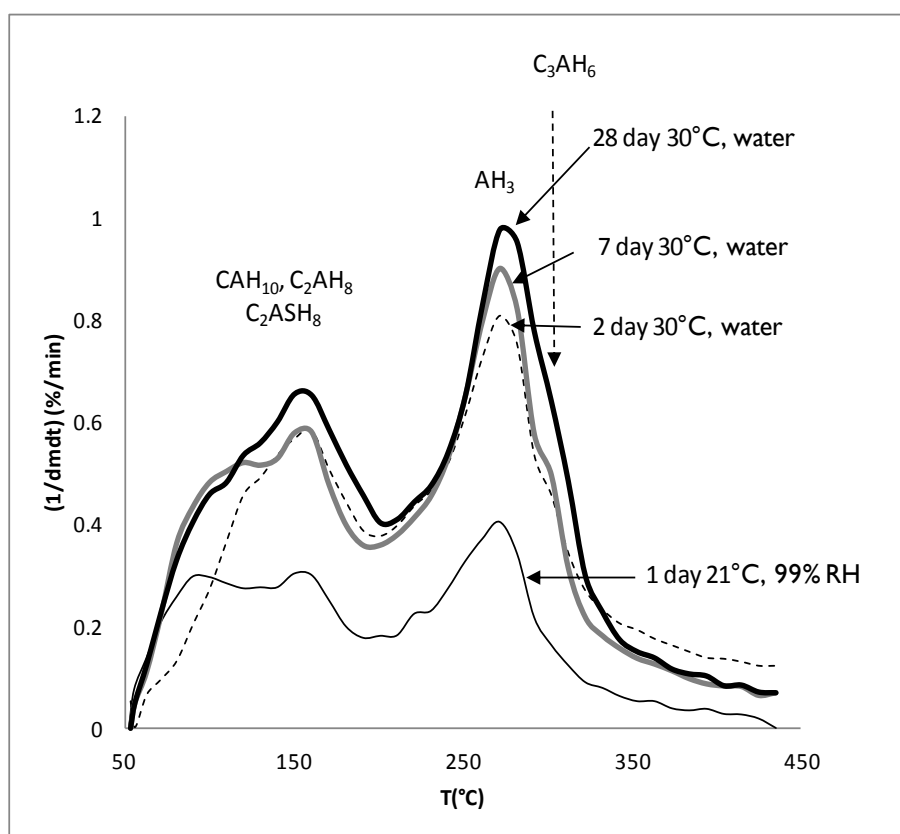


Figure 1. DTA of hydrated calcium aluminate cement.

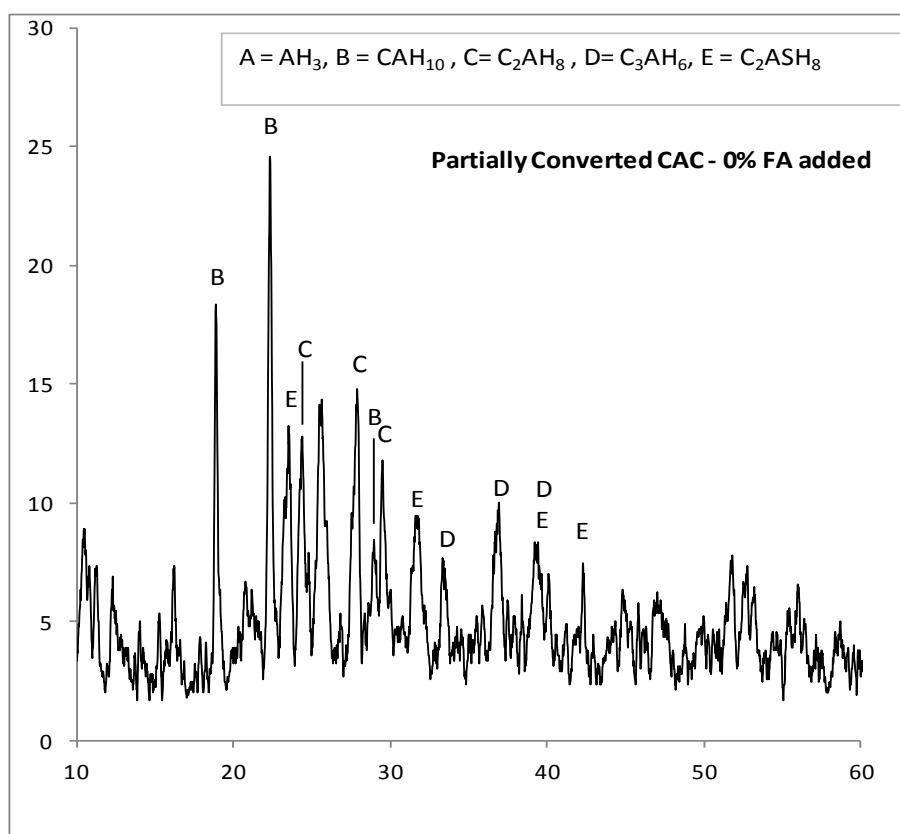


Figure 2. X-ray diffraction pattern for partially converted CAC (30 $^{\circ}C$ in water for 28 days).

(AH_3 and C_3AH_6) from 180 to 360°C from day 7. The X-ray diffraction pattern of CAC at day 28 confirms the conversion process with the presence of both metastable and stable phases. It is also apparent that in this particular CAC stratlingite (C_2ASH_8) also formed by day 28 of curing suggesting the composition of this CAC include pozzolanic material. Table 1, shows the raw CAC used in this study contained 6.97% SiO_2 . It was not clear however if all of this silica have pozzolanic activity.

3.2 Reactivity of converted phases

Most researchers appear to attribute the increased susceptibility of converted CAC to corrosion to increased porosity (Bradbury, Callaway et al. 1976, Fu, Ding et al. 1996). In this study, the relative reactivity of the converted phases to corrosion was examined. Corrosion tests were conducted both in abiotic or chemical acids and through installation of coupons in the sewer (see Figures 3 to 5). The abiotic acids chosen for the corrosion tests represents the biogenic acids typically metabolised by fungi and bacteria found in sewers (Valix, Zamri et al. 2012). Figure 3 shows the effect of CAC corrosion in 10% H_2SO_4 at 30°C for 75 days. As shown there is preferential corrosion of the converted hydrogarnet (C_3AH_6) phase and AH_3 whilst the metastable CAC phases and stratlingite (C_2ASH_8) remained as corrosion residues demonstrating their corrosion resistance. Figure 4 shows the effect of CAC corrosion in 10% citric acid at 30°C for 90 days. The shift of the corroded peak at 250-300°C to the left shows the hydrogarnet was preferentially removed with only partial removal of the AH_3 phase. These results suggest that in citric acid C_3AH_6 has lower corrosion resistance in comparison to AH_3 . The strong peaks from 50 to 200°C showed the stability of the metastable phases and of stratlingite. In leaching in citric acid, however there was very little soft corrosion products found as in sulphuric acid. Citric acid, a strong complexing agent prevents the formation for aluminium oxyhydroxide precipitate by forming stable Al-citrate complexes (Dabbs, Ramachandran et al. 2005, Xu, Hu et al. 2010). The products analysed on the surface of the CAC specimen corroded in citric acid was the harder component and maybe representative of both the remaining corrosion product and un-corroded CAC. The thermal analysis of a CAC coupon installed in the Southern and Western Suburbs Ocean Outfall Sewers (SWSOOS), South Barrel in Sydney for 364 days is shown in Figure 5. The average environmental conditions in this site is 2-3 ppm H_2S , 17-23°C and 91-97% relative humidity. Analysis of the CAC coupon showed a similar preferential corrosion of hydrogarnet (C_3AH_6) phase and AH_3 . These results demonstrate the higher corrosion susceptibility of the stable hydrogarnet phase and AH_3 relative to the metastable phases to both abiotic and biogenic acid attack from sewers. Because the conversion of metastable calcium aluminate hydrates to the more stable hydrogarnet phase is inevitable (Juenger, Winnefeld et al. 2011), these results suggest that the conversion could have an impact on the long term performance of CAC as a protective coating. This corrosion feature could make designing protective systems based on CAC challenging and complex.

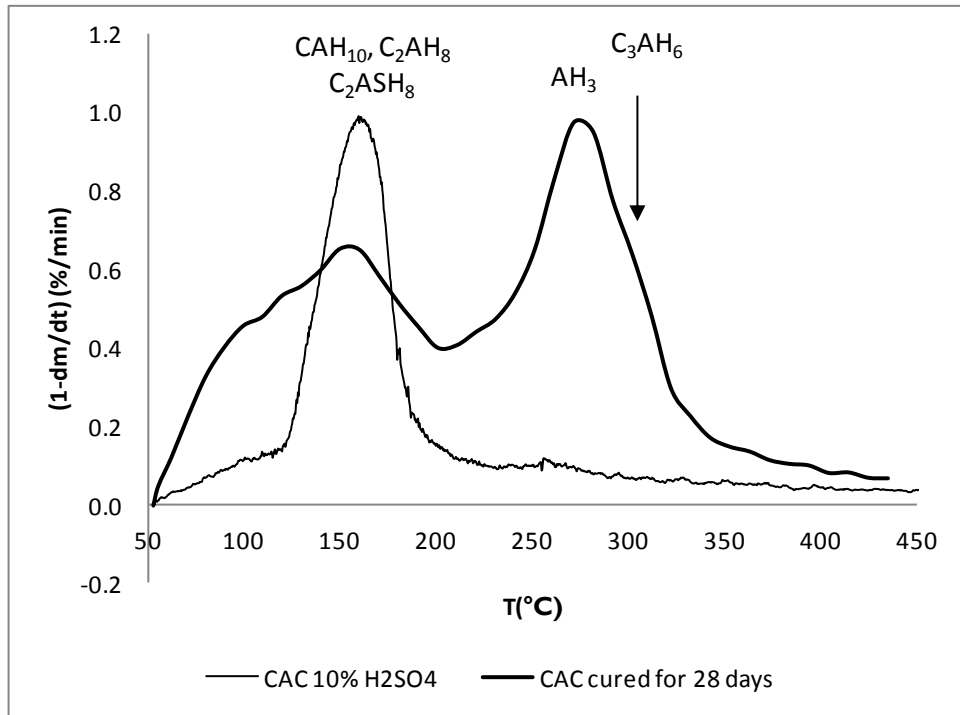


Figure 3. DTG of cured CAC and CAC corroded in 10% H₂SO₄ at 30 $^{\circ}C$ for 42 days.

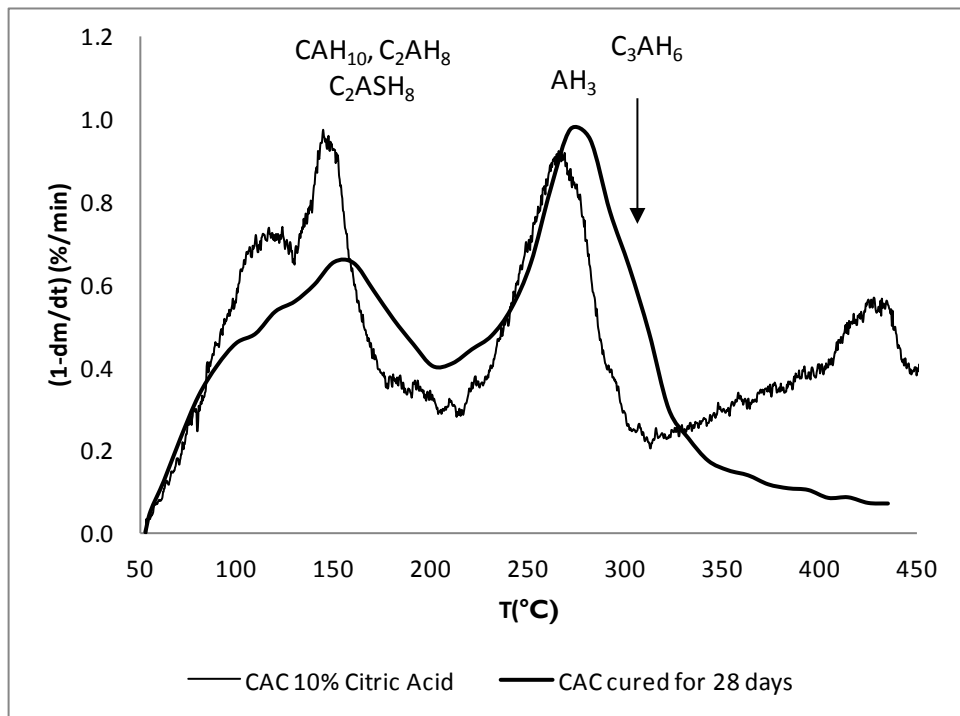


Figure 4. DTG of cured CAC and CAC corroded in 10% Citric acid at 30 $^{\circ}C$ for 42 days.

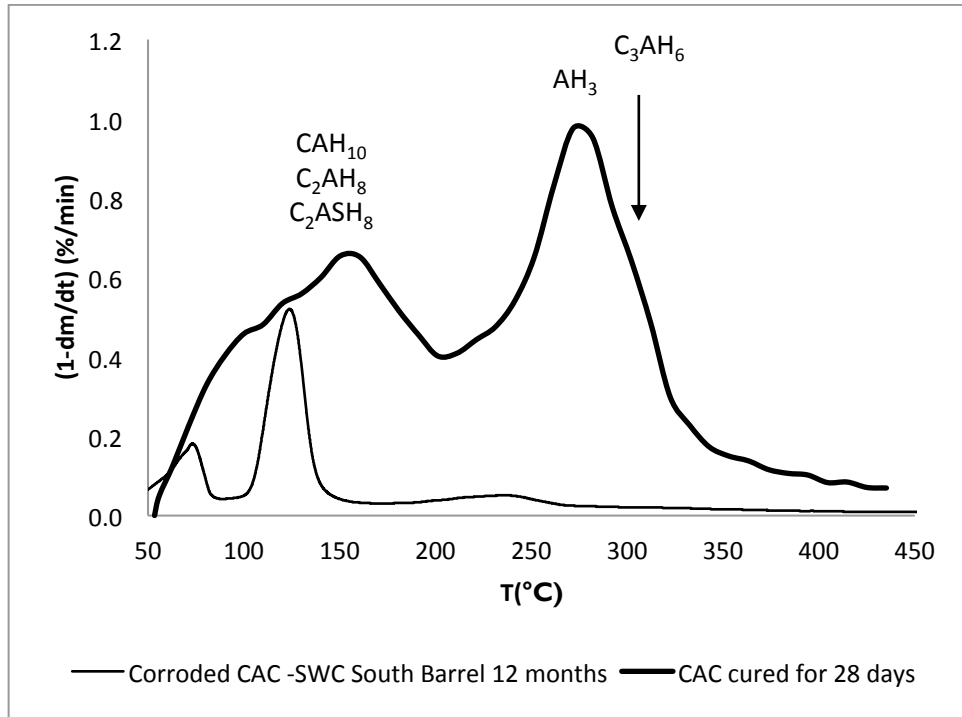


Figure 5. DTG of cured CAC and CAC corroded in the SWSOOS South Barrel sewer for 364 days.

3.3 Accelerated Conversion of CAC

The impact of CAC conversion on the acid resistance was examined by accelerating the conversion process. Although it is widely recognised that conversion can affect the structural strength of CAC concrete, the effect of conversion on the acid resistance of CAC and thus its impact on its service life is not as well understood (Midgley 1990, Taylor 1990). The use of high temperatures, from 30-80°C and higher are known to accelerate the process of conversion (Scrivener, Cabiron et al. 1999, Fryda 2008, Kirca, Yaman et al. 2013). In this study, the conversion of CAC was conducted by curing the specimen at temperatures of 98-100°C. Our study established that faster conversion of 2 inch diameter CAC cores could be achieved within 2 hours at this temperature. A comparison of the thermal analysis of partially converted (30°C, 28 days) and CAC cores cured at 100°C is shown in Figure 6. The conversion is evident from the shoulder above 300 °C resulting from the formation of the cubic hydrogarnet (C_3AH_6) phase accompanied by the reduction of the peaks at 50-250°C attributed primarily to the metastable phases. X-ray diffraction analysis of the specimens in Figure 7 confirmed the greater formation of cubic hydrogarnet (C_3AH_6) in the converted CAC. It is apparent however that some metastable phases, in particular C_2AH_8 , still exist in

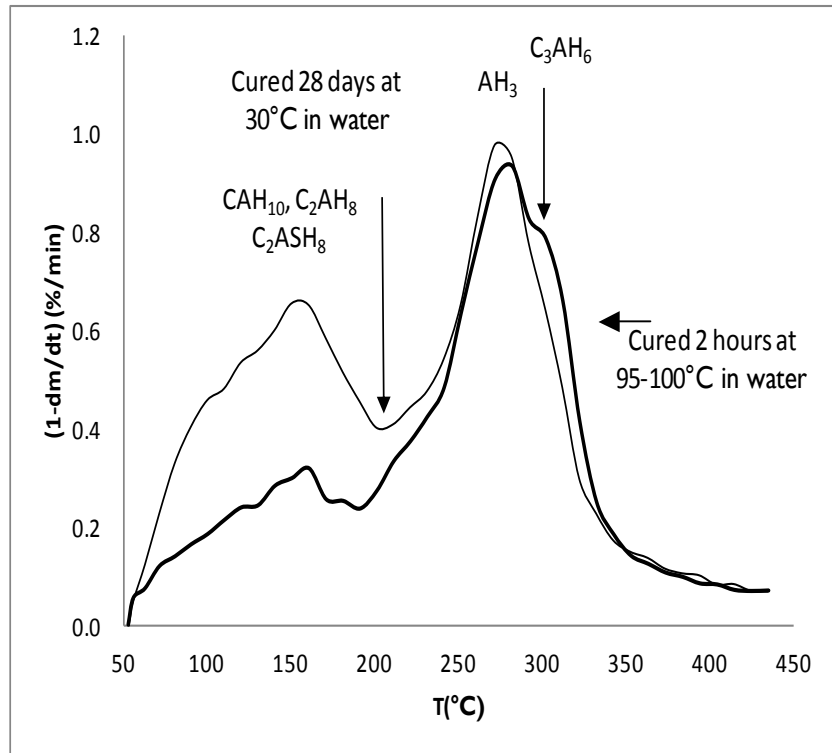


Figure 6. Thermal analysis of partially cured (30°C, water, 28 days) and CAC converted at 98-100°C in water for 2 hours.

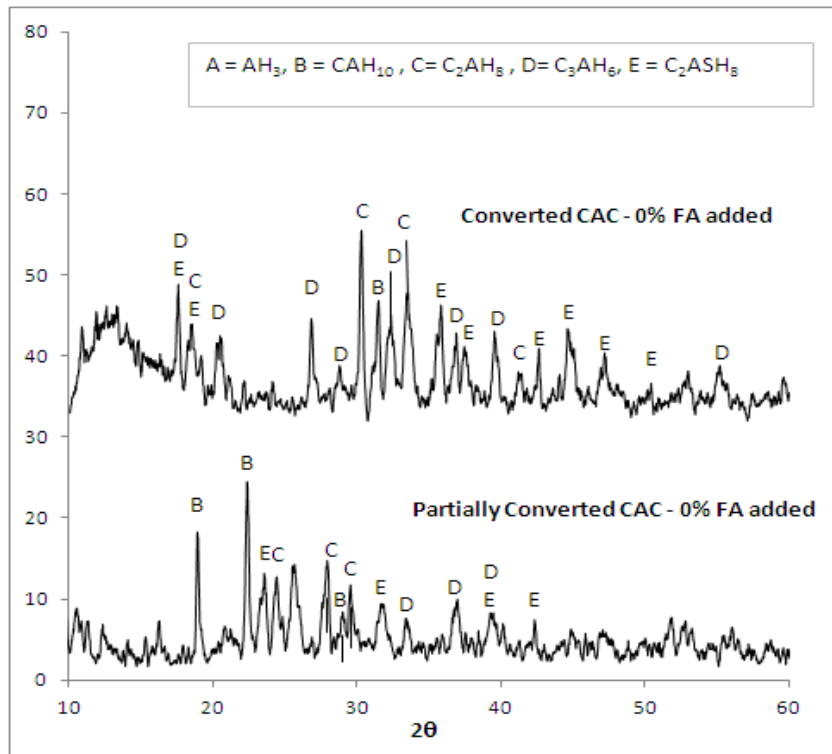


Figure 7. X-ray diffraction pattern for partially converted (30°C, water, 28 days) and converted CAC (98-100°C, water, 2 hours).

the converted phase and is accompanied by the formation of stratlingite. This is consistent with proposed progression of conversion through the $CAH_{10} \rightarrow \alpha C_2AH_8 \rightarrow \beta C_2AH_8 \rightarrow C_3AH_6$ pathway (Rashid, Barnes et al. 1994)

3.4 Effect of Conversion on Resistance of CAC to Acid Attack

The effect of conversion on the corrosion resistance of CAC was examined by corroding partially converted (28 days at 21°C) and converted CAC (2 hours, 100°C) in 1, 5 and 10% citric and sulphuric acids at 30°C. The rate of corrosion was monitored by measuring from the rate of thickness loss and dissolved Al. The rates of corrosion based on thickness loss are shown in Figures 8 and 9.

The rates of corrosion were fitted to the following equation:

$$x = \left(\frac{2D_i C_o}{C_a} t \right)^{1/2} = kt^{1/2} \quad (7)$$

Where $k = \sqrt{\frac{2D_i C_o}{C_a}}$

C_a = concentration of Al_2O_3 and CaO in CAC solid (moles/cm³)

C_o = concentration of the sulphate or corresponding acid anion in the bulk solution (moles/cm³)

X = depth of corrosion (mm)

D_i = acid anion diffusion coefficient (cm²/s)

t = time (days)

The corresponding parameters are reported in Table 3.

Table 3. Corrosion parameters at 30°C Based on Thickness loss.

Acidic Environment	Partially Converted		Converted		Ratio k%
	t_{lag} (days)	k (mm/years ^{1/2})	t_{lag} (days)	k (mm/years ^{1/2})	
1% H ₂ SO ₄	6.95	0.68	0.00	4.18	614
5% H ₂ SO ₄	11.96	6.63	0.00	5.60	85
10% H ₂ SO ₄	7.99	13.70	0.00	8.28	60
1% Citric Acid	0.0	5.4	0.00	5.9	110
5% Citric Acid	2.34	12.68	0.00	7.30	58
10% Citric Acid	2.34	29.24	0.00	7.51	26

The rate of corrosion based on dissolved Al are shown in Figures 10 and 11. Comparison of these results shows that the thickness loss is subjected to loss of material by corrosion and expansion. The evidence for expansion is evident in the thickness gained with leaching from

the 1% H₂SO₄ leaching in Figure 8. The lag phase observed in the early phase of corrosion of partially converted CAC and in comparison to the continued dissolution of metals in Figures 10 and 11 also demonstrate the expansion of these coupons. Given these results, the rate of corrosion of CAC would be more accurately measured from the dissolved Al. However field monitoring could make such an analysis challenging.

The two sets of corrosion results show that conversion does impart a higher rate of corrosion. It is apparent from the rates measured from Al dissolution that partially converted CAC and converted CAC dissolves at the same rate initially. However with time, it appears the partially converted CAC passivates whilst the converted phase continues to corrode at the same rate it begins to corrode.

The relative corrosivity of CAC in sulphuric and citric acids are also evident in these results. Although the thickness loss demonstrate greater loss of thickness in citric acid compared to sulphuric acid in Figures 8 and 9, the rate measured by Al dissolution shows the rate of corrosion in sulphuric acid is generally higher. The reduced rate of thickness loss in sulphuric acid could be attributed to greater expansion in this acid resulting from the formation of ettringite.

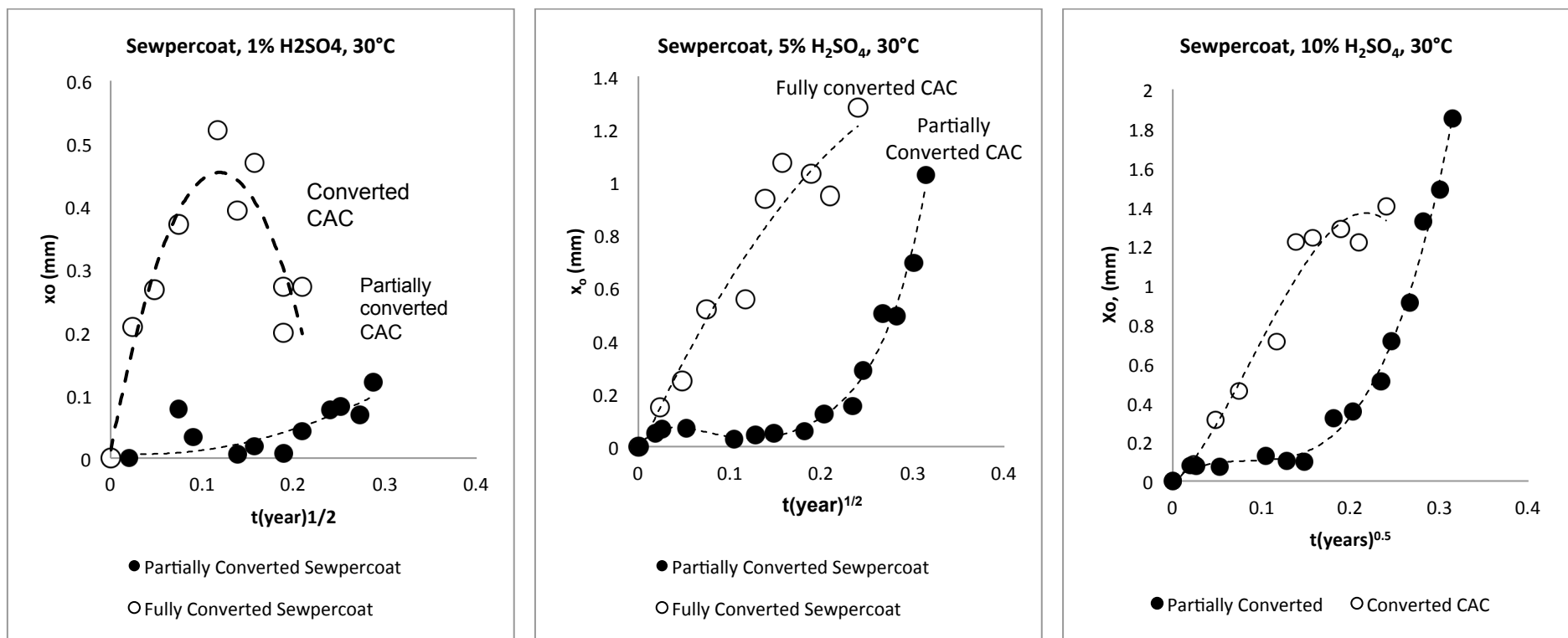


Figure 8. Effect of conversion on corrosion of CAC in H₂SO₄ based on thickness loss.

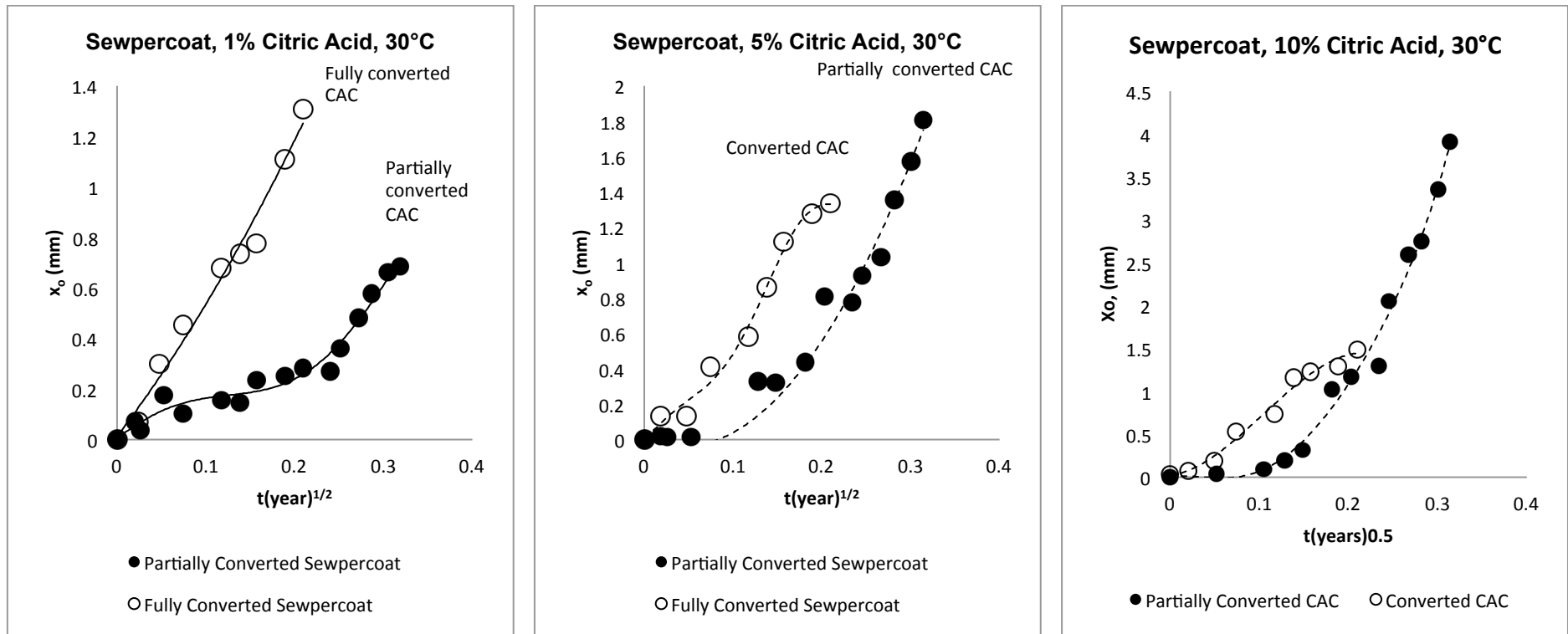


Figure 9. Effect of conversion on corrosion of CAC in Citric Acid based on thickness loss

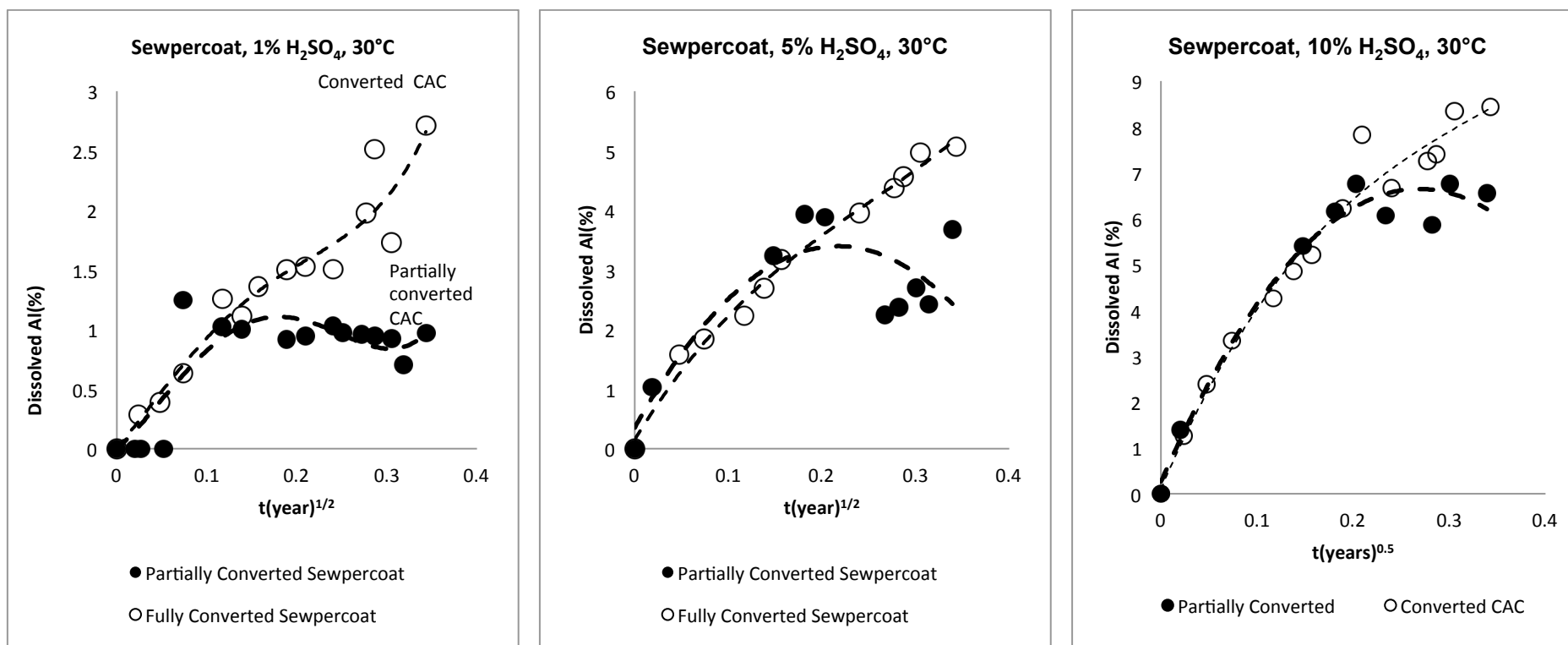


Figure 10. Effect of conversion on corrosion of CAC in H_2SO_4 based on dissolved Al.

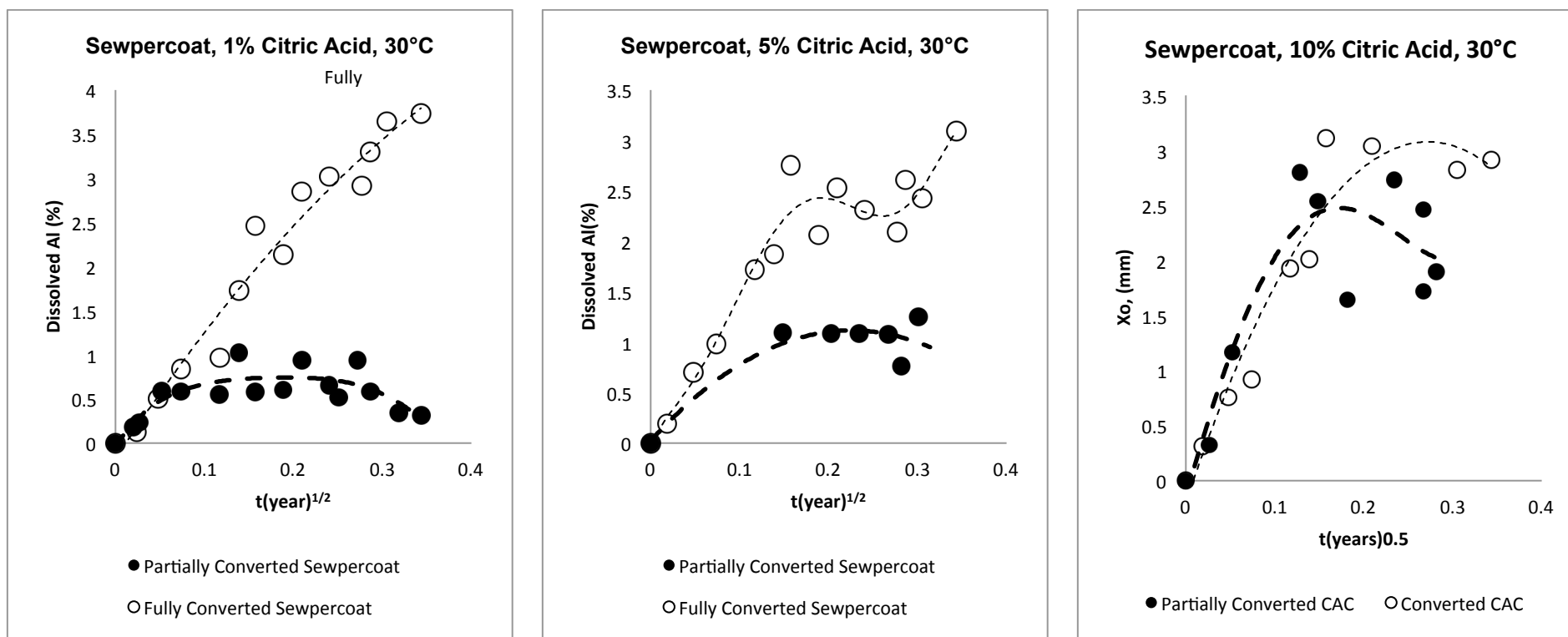


Figure 11. Effect of conversion on corrosion of CAC in Citric Acid based on dissolved Al.

4.0 CONCLUSIONS

This study has shown the metastable phases of CAC has a higher corrosion resistance to both biotic and biogenic acids generated in sewers. The stable phase (C_3AH_6) and AH_3 are preferentially corroded in both abiotic and biogenic acids.

The effect of conversion is evident in both greater thickness loss and greater dissolved Al. The rate measured by thickness loss however could not accurately measure the rates of corrosion because of the initial effect of expansion.

Measured rates based on dissolved Al show the initial rates partially and fully converted CAC are similar but with time, the partially converted CAC appear to passivate whilst the fully converted CAC continues to corrode at the same rate at it began.

Conversion appear to increase the rates of corrosion by 100-400% from the measured rates based on dissolved Al.

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