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Resistance of alkali-activated binders to organic acids found in agri-food effluents

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Abstract

Organic acids such as acetic and lactic acid are prevalent in agricultural and food effluents. They pose a considerable pollution threat and must be collected and stored safely before treatment and release. They cause significant damage to cementitious materials, reducing the service life of structures. In this study the resistance of alkali-activated fly ash and slag blended binders to organic acids was studied and a comparison with ordinary Portland cement binders was carried out. Findings demonstrate that alkali-activated binders with increased fly ash content have marginally better resistance to acetic acid but mixes with increased slag content have better resistance to lactic acid. This is due to the solubility of the calcium and aluminium salts of acetic and lactic acid. Overall the performance of the alkali-activated binders was better than that of the ordinary Portland cement binder with lower mass and strength losses observed. This was attributed to their lower calcium content with less vulnerable phases such as calcium hydroxide and ettringite. Instead the C-S-H type gels in alkali-activated binders suffered decalcification and dealumination but left behind a silicon rich gel which helped to resist further acid attack.

Author keywords: Alkali-activated binder; lactic acid; acetic acid; durability; degradation; fly ash; slag

Introduction

The agriculture and food industries produce an abundance of effluents or waste waters which are pollutants if released directly into the environment. This means they need to be collected, stored and treated before being released (Bertron and Duchesne 2013). Therefore, these effluents regularly come into contact with concrete causing considerable damage and reducing the service life of structures. Fig. 1 shows silage effluent leaching out from grass stored in a concrete silo pit as winter feed for livestock. This photograph was taken only a few hours after the grass was initially stored. The effluent continues to flow slowly over the concrete floor for around 6 months per year. After only six years in-service the concrete has suffered considerable damage. The surface has been scaled, with cement paste removed, exposing large aggregates and leaving behind a rough surface. This exposure has also reduced the depth of cover to steel reinforcement and mostly likely caused a reduction in strength and eventually will lead to a shorter than expected service life. The silage effluent mainly consists of acetic and lactic acid and has pH of around 4.0 (Bertron et al. 2005a; Gruyaert et al. 2012).

Silage effluent is only one example of numerous organic acid media which commonly attack concrete. There are many others including animal feed, vinasse, distillates, molasses, whey and liquid manure (De Belie et al. 1997; Bertron 2013; Bertron et al. 2005b; Castillo Lara and Chagas Cordeiro 2019). These cover a broad range of organic acids including acetic, lactic, propionic, butyric, malic, citric and oxalic. These organic acids are often described as weak acids compared with strong acids such as hydrochloric or nitric. However, in some cases the attack on cementitious materials is more aggressive (Shi and Stegemann 2000). This is because the pH increase of an organic acid solution in contact with cementitious materials is slower than for strong acids, meaning samples are exposed to a lower pH for longer. Additionally, at a given pH, the acid concentration is higher for an organic acid solution than for a strong acid solution (Castillo Lara and Chagas Cordeiro 2019).

In recent decades the cement and concrete industry has been under increased pressure to reduce CO₂ emissions and the consumption of natural resources. This has led to increased attention for alkali-activated binders which involve the reaction of an aluminosilicate source with an alkali activator to

form a hardened binder (Shi et al. 2019). The most commonly investigated aluminosilicate sources are fly ash (FA) and slag as they are residues from coal combustion and steel production, respectively and require limited further processing before they can be used (Rakhimova and Rakhimov 2019). The alkali-activation of FA and slag as single precursors have resulted in niche problems specific to that binder type. Alkali-activated FA normally requires high temperature curing to achieve reasonable strength which is a barrier to widespread use in the concrete industry (Bakharev 2005). Whilst alkali-activated slag does not require high temperature curing, there are issues regarding its fast setting time (Collins and Sanjayan 1999; Nath and Sarker 2014; Shi et al. 2019). The blending of FA and slag together has reduced the aforementioned issues. Alkali-activated FA/slag blends can be cured at ambient temperature and are less susceptible to fast setting (El-Hassan and Elkholy 2019; Rafeet et al. 2019). Therefore, blended FA/slag systems have generated significant interest in recent years.

The majority of studies which have considered the acid resistance of alkali-activated FA/slag systems have focused on strong acids such as sulfuric and nitric acid (Allahverdi and Škvára 2001, 2005; Lee and Lee 2016; Lloyd et al. 2012). The mechanism of acid attack due to sulfuric acid is unique due to the formation of gypsum which causes expansion and mass increases. Gypsum can also act as barrier for further acid attack (Aiken et al. 2018). Therefore, results need careful interpretation and cannot be correlated with the performance of other acid types. As discussed above the nature and behaviour of cementitious materials differs during exposure to weak organic acids. Therefore, this study focuses on the resistance of alkali-activated FA/slag blends to acetic and lactic acid. A comparison with ordinary Portland cement is also presented. In order for the use of alkali-activated binders to become more widely adopted, increased understanding of their durability is needed.

Materials and experimental methods

The FA used was supplied by Power Minerals Ltd., Drax Power Station, North Yorkshire, UK. The slag used was obtained from Civil and Marine Ltd-Hanson Company, member of the Heidelberg Cement Group, Essex, UK. The Portland cement (CEM I 42.5N) used, was produced by Quinn Cement in Northern Ireland. The FA, slag and Portland cement conform to the standards of BS EN

450-1:2012 (British Standards Institution 2012), BS EN 15167-1:2006 (British Standards Institution 2006) and BS EN197-1:2011 (British Standards Institution 2011), respectively. Table 1 shows the specific gravity and oxide composition of each powder binder component which was obtained by X-ray florescence (XRF). The particle size distribution of each powder binder component is shown in Fig. 2. Their X-ray diffraction (XRD) patterns are displayed in Fig. 3. Quartz, mullite and hematite were the main phases found in the FA. The slag was almost completely amorphous with a broad peak or hump between 25 and $35^\circ 2\theta$. The Portland cement had numerous crystalline phases including alite, belite, aluminate, brownmillerite and gypsum. The FA/slag mixes were activated by solutions of sodium silicate and sodium hydroxide. The sodium silicate solution was supplied by Fisher Scientific and consisted of 12.8% Na_2O , 25.5% SiO_2 and 61.7% water. It had a specific gravity of 1.4. The sodium hydroxide solution was prepared at 30% w/w by the dissolution of solid commercial grade (99% purity) sodium hydroxide and was allowed to cool to room temperature prior to preparation of mortars and pastes. It had a specific gravity of 1.33.

The aggregate used in this study was 0-5 mm lough sand, supplied by Creagh Concrete, Northern Ireland. The oven-dry particle density of the sand was 2695 kg/m^3 and its water absorption was 0.9 and 1.1% after 1 and 24 h, respectively [BS 812-2 (British Standards Institution 1995)]. The particle size distribution of the sand is shown in Fig. 2 and was determined according to BS 812-103.1:1985 (British Standards Institution 1989). Prior to mixing, moisture was removed from the sand by oven-drying at 100°C for a minimum of 24 h. It was then allowed to cool to room temperature before mixing began.

Laboratory grade acetic (99-100%) and lactic acid ($\geq 85\%$) were used to prepare 0.10, 0.31 and 0.52 mol/L acid solutions of each acid type by mixing in the required proportions with distilled water. Acetic and lactic acids have dissociation constants pK_a of 4.76 and 3.86, respectively at 25°C (Bertron and Duchesne 2013).

The mix proportions used are presented in Table 2. The mixes were based on initial mix design optimisation carried out by The Geopolymer Team at Queen's University Belfast (Kwasny et al. 2018c; Rafeet et al. 2017, 2019; Vinai et al. 2016). The alkali-activated mixes have increased FA content from 30% (FA/S-30/70) to 80% (FA/S-80/20). The alkali dosage and alkali modulus were fixed at 7.5 and 1.25, respectively. The alkali dosage is defined as the percentage of Na₂O in the binder and the alkali modulus is the ratio of Na₂O and SiO₂ (sometimes referred to as silica modulus). The paste content of all mortar mixes was fixed at 50% volume. The equivalent pastes were designed with the same composition as mortars, except without the presence of sand. The absorption water is the amount of water needed to bring the sand to saturated surface dry condition. The total added water is the total amount of water needed plus the absorption water, less the water contained within activating solutions. The water/solid ratios were chosen based on recommendations from previous studies (Rafeet 2016; Rafeet et al. 2017) which found that mixes with increased slag content had an increased water demand. Mortar samples were used to assess physical changes due to acid attack as they are more representative of in-service exposure due to the presence of sand. Paste samples were used to assess microstructural changes on the paste itself due to acid attack. Sand can complicate microstructural analysis techniques due to the presence of quartz. Therefore, the mortar samples were used to quantify the extent of the attack while paste samples were used to assess the nature of the attack.

Both mortar and paste samples were prepared with a 10 l capacity planar-action high-shear mixer. Firstly, the oven dried sand was placed in the mixing bowl along with half of the total added water and mixed together for 1 min. After 15 mins the powder component (FA and slag or Portland cement) of each mix was added. Finally, the remaining water (and activating solutions if appropriate) was added and mixing continued for 6 mins before casting.

The samples were cast in two layers into 50 mm three-gang cubic moulds and 100 mm two-gang cubic moulds, each layer was compacted using a vibrating table. After casting the samples were wrapped in cling film for 24 h to prevent moisture evaporation. To prevent leaching of alkalis,

140 samples were cured at 20 ± 1 °C and stored on 15 mm high spacers above 5 mm of water in sealed
141 containers throughout where the relative humidity was $> 90\%$ (Kwasny et al. 2018b). Samples were
142 demoulded after 24 h and returned immediately to their curing containers.

143
144 Organic acid resistance of mortars was assessed using an accelerated method, based on the guidelines
145 outlined in ASTM C267 (American Society for Testing and Materials 2012) and previous studies
146 (Aiken 2017; Aiken et al. 2017; Kwasny et al. 2018a). After 21 days curing, the 50 mm mortar cubes
147 were placed into a water bath (20 ± 1 °C) until 28 days when their mass was recorded. Subsequently
148 four mortar cubes from each mix were put into individual plastic containers together with acid
149 solutions with concentrations of 0.10, 0.31 and 0.52 mol/L of acetic and lactic acid. The acid solution
150 to sample volume ratio in each container was maintained at approximately 0.9 throughout. Each week,
151 visual inspection was carried out, the mass of the mortar cubes was recorded and the acid solution
152 replenished. Prior to their mass being recorded in air, the samples were removed from the container
153 and their surface was gently wiped to remove any excess testing solution. Additionally the pH of the
154 acid solution was monitored at intervals during each week. After 8 weeks, the average compressive
155 strength was obtained by testing three cubes from each box with a constant loading rate of 50 kN/min.
156 The loading zones were protected with softboard to reduce the effect of uneven surfaces. This method
157 was used instead of capping or grinding to allow immediate testing following acid attack, avoiding
158 testing delays which would have caused issues regarding intermediate sample storage conditions and
159 drying during processing. Prior to testing each sample was removed from the container and its surface
160 was gently wiped to remove any excess testing solution. Control samples which were not exposed to
161 acid were also tested for comparison (stored in water). The fourth specimen was used to visually
162 inspect the alkalinity loss. This was achieved by splitting the cubes and applying phenolphthalein
163 solution on the split surface which highlights (deep pink/purple) the region where the pH is greater
164 than approximately 9 (Chang and Chen 2006; Chinchón-Payá et al. 2016; Liu et al. 2017).

165
166 Mercury intrusion porosimetry (MIP) was used to obtain the porosity of each mortar mix. At 28 days
167 mortar fragments measuring approximately $8 \times 8 \times 20$ mm were sectioned from the core of each cube

using a diamond saw. Hydration/reaction was stopped by oven drying the samples for 24 h at 60 ± 1 °C, then immersing them in acetone for 4 h and subsequently drying them in a desiccator (20 ± 1 °C) for a further 24 h. A temperature of 60 °C was used as previous research (Gallé 2001; Ma 2014) has indicated that using a temperature in this region is more realistic and reduces the effect on pore structure compared with higher temperatures. The pore structure of the samples was determined using a Pascal 140/240 mercury intrusion porosimeter from ThermoFisher Scientific. The mercury contact angle was taken to be 140°.

The volume of voids (VPV) of mortars was assessed according to the standard procedure given in ASTM C642 (American Society for Testing and Materials 2006). This involves recording the mass of samples in various conditions which are given below. This test was carried out on 100 mm long and 75 mm diameter cores which were extracted from 100 mm cubes. Three samples were tested for each mix and the VPV was calculated according to:

$$VPV (\%) = \frac{C - A}{C - D} \times 100$$

where:

A = Mass in air after oven drying, g

C = Mass in air after immersion and boiling, g

D = Apparent mass in water after immersion and boiling, g

The standard recommends an oven drying temperature between 100 and 110 °C. In this study, an oven drying temperature of 40 °C was used to avoid excessive drying which can cause changes to binding phases such as calcium aluminium silicate hydrate gel within alkali-activated concrete (Ismail et al. 2013).

Equivalent paste samples were exposed to the same cyclic exposure regime for 3 weeks using the highest concentration (0.52 mol/L) of both acetic and lactic acid. Inductively coupled plasma mass

spectroscopy (ICP) was used to analyse the acid solution at the end of each exposure cycle (every week). This provided the concentration of metals such as Al, Ca, Na and Si that were leached from the specimens and found in each testing solution. At the end of 3 weeks, the paste samples were used for microstructural analysis. They were compared with control pastes which were submerged in water instead of acid but otherwise processed in the same way. Powdered samples were obtained by crushing and grinding the outside layer of the paste samples in a pestle and mortar. The powders were dried in a desiccator at 35 °C for 24 h prior to analysis. XRD was carried out with a PANalytical X'Pert PRO MPD diffractometer, applying CuK α radiation of wavelength 1.54Å. Diffraction patterns were collected between 5 and 65° 2 θ with a step size of 0.02°. PANalytical X'Pert Highscore software with the powder diffraction file (PDF) database was used to analyse the diffraction patterns. Fourier transform infrared spectroscopy (FTIR) data was obtained using Jasco 4100 series FTIR Spectrometer with attenuated total reflectance attachment. The spectra were gathered between 650 and 2000 cm⁻¹ wavenumber at 8 cm⁻¹ resolution. Scanning electron microscopy (SEM) with elemental analysis was carried out on polished paste samples coated with gold. An accelerating voltage of 5 kV and a working distance of 10 mm were used for imaging. The equipment used was QUANTA FEG250 with OXFORD X-Act as chemical composition analyser, which was run by Aztec version 2.0 software for analysis. Paste samples were sectioned and polished in preparation for SEM and elemental analysis.

Results and discussion

Physical properties

Fig. 4 shows photographs of each mortar mix after immersion in solutions of acetic and lactic acid for 8 weeks. Unexposed samples are also shown for comparison. Lactic acid appears to cause more damage to the surface of each mix compared with acetic acid. The alkali-activated mixes have suffered the loss of paste following immersion in the most concentrated lactic acid solutions and the loss of paste seems to have increased as the FA content increased. Following immersion in 0.31 and 0.52 mol/L solutions of lactic acid, the OPC mix has also suffered the loss of cement paste, exposing sand particles.

The mass change for each mix during 8 weeks of immersion in solutions of acetic and lactic acid is shown in Fig. 5. For each mix the samples mass decreased with each consecutive week of immersion in acid solutions. The lactic acid solutions caused larger mass losses than the acetic acid solutions for each mix investigated. This is likely due to the higher dissociation constant of lactic acid compared with acetic acid (Bertron and Duchesne 2013). As the concentration of acid increased the mass loss also increased. In general the alkali-activated mortars displayed a rapid mass loss initially which decelerated during each consecutive exposure cycle. Conversely the OPC mix displayed smaller mass losses initially which accelerated with prolonged exposure, eventually having larger mass losses than the alkali-activated mortars after 8 weeks. This was particularly the case when exposed to the higher concentrations of each acid solution. The accelerating behaviour of the mass loss of the OPC mix is likely due to the dissolution of phases causing an increase in porosity of the matrix allowing a faster onset of further degradation. As the FA content increased the mass loss of the alkali-activated mortars also increased following immersion in lactic acid. However, the opposite is true for acetic acid immersion as shown in Fig. 6, but to a much lesser extent. The mass loss decreased as the FA content increased. This suggests mixes with increased FA content are more resistant to acetic acid, but less resistant to lactic acid. This phenomenon is discussed in more detail in the sections on leaching behaviour and microstructural changes due to organic acid attack.

The compressive strength of each mix following immersion in solutions of acetic and lactic acid for 8 weeks is shown in Fig. 7. The compressive strength of unexposed samples (kept in water) are also shown for the comparison and the percentage strength loss values are also provided. Regardless of the acid concentration the strength loss was similar for the same mix and acid type. This could be because the strength loss is related to the acid penetration depth which may be similar regardless of acid concentration. As the FA content of the alkali-activated mixes increased their strength decreased. Following immersion in each acid the percentage strength loss values are between 31 and 47% for each mix and acid concentration investigated. Following immersion in lactic acid, FA/S-30/70 displayed the best performance with a strength loss of around 32%. As the FA content increased the strength loss values also increased to values between 40 and 45%. Comparing the effect of the two

acid types on alkali-activated mortars shows that mixes with increased FA content (FA/S-80/20) had greater resistance to acetic whereas mixes with increased slag content had greater resistance to lactic acid. This agrees with the mass loss findings. The OPC mix experienced the largest strength losses following immersion in lactic acid of around 48%.

Fig. 8 shows the cross-section of each mix after the application of phenolphthalein solution. Samples that were unexposed (kept in water) and immersed in 0.52 mol/L solutions of acetic and lactic acid for 8 weeks are shown for comparison. The entire cross-section of the unexposed samples was coloured pink/purple, whereas the samples which were immersed in solutions of acetic and lactic acid have an outer layer which is not coloured indicating alkalinity loss. As the FA content increased the alkalinity loss also increased and can be related to the porosity and volume of voids of each mix which also increased as the FA content increased and is shown in Table 3. The increased porosity allows the acid solution to penetrate further, causing ion exchange and a loss of alkalinity. The alkalinity loss for OPC appeared similar to that of FA/S-30/70.

Leaching behaviour

Fig. 9 shows the pH of acetic and lactic acid solutions during immersion of mortar mixes for 8 weeks. At the beginning of each cycle the pH is low because the acid solution has been replenished with fresh acid. During each exposure cycle the pH increased as leaching occurred from the highly alkaline samples. For each acid type and concentration, the increase in pH was larger for OPC compared with the alkali-activated mortars. This indicates that the OPC mix has a higher acid neutralisation capacity than the alkali-activated mixes. This likely explains the smaller mass loss observed for OPC mixes compared with alkali-activated mixes exposed to the lowest concentration (0.10 mol/L) of each acid used. During exposure to the higher concentrations of each acid the ability of OPC to neutralise the acid decreased. This likely explains the small mass losses initially which accelerated with increased exposure cycles (Fig. 5). For the alkali-activated mortars the mixes with increased FA content had less of a pH increase during each exposure cycle. This could indicate less leaching occurred. With an

increased number of exposure cycles, the pH of the acid solutions decreased suggesting less leaching occurred with subsequent cycles.

Fig. 10 shows the cumulative concentrations of Al, Ca, Na and Si in solutions of water, acetic acid and lactic acid during immersion of alkali-activated and OPC paste mixes for 3 weeks. The main component leached into the acid solutions was Ca. The concentration was highest for OPC and decreased as the FA content increased in the alkali-activated pastes, likely due to the lower Ca content of FA compared with slag (Table 1). When organic acids interact with a cementitious matrix, various Ca and Al salt complexes are formed (Bertron and Duchesne 2013). The Ca concentrations found in the acid solutions were larger in acetic acid compared with lactic acid. This is likely due to the higher solubility of the Ca salt of acetic acid compared with the Ca salt of lactic acid meaning more Ca went into solution. On the other hand, the Al salt of lactic acid is more soluble than the Al salt of acetic acid (Bertron and Duchesne 2013). This explains the leaching of Al into the lactic acid solution. The concentration of Al in solution increased as the FA content increased (Fig. 10c). This is likely due to the increased Al content of FA compared with slag (Table 1). The amount of Na leached in the water and acid solutions was similar. In the case of Si, only small quantities were found in the acid solutions, likely due to the preferential leaching of Ca and Al during acid attack.

Microstructural changes due to organic acid attack

The XRD pattern of each mix following immersion in 0.52 mol/L solutions of acetic and lactic acid for 3 weeks is shown in Fig. 11. The patterns of unexposed (kept in water) samples are displayed as a reference. Mullite and quartz were identified in the unexposed alkali-activated mixes. These were present due to unreacted FA particles (Fig. 3) and the intensity of these peaks increased as the FA content increased. There was also a broad peak centred between 29 and 30° 2 θ and has been related to the presence of poorly crystalline calcium silicate hydrate (C-S-H) type gel by previous authors (Ben Haha et al. 2011; Wang and Scrivener 1995). Additionally, the peak assigned to quartz at around 50° 2 θ is broad in nature, likely due to the contribution of a broad peak at around 49.5° 2 θ indicating the presence of the same C-S-H type gel (Ben Haha et al. 2011). This may be calcium aluminium silicate

hydrate (C-A-S-H) gel or calcium sodium aluminium silicate hydrate (C-N-A-S-H) gel, depending on the proportions and composition of FA and slag used (Aragón et al. 2020; Ismail et al. 2014). As the FA content increased, the intensity of this broad peak between 29 and 30° 2 θ decreased, suggesting less C-S-H type gel was present. Following immersion in acid, this peak was no longer observed, indicating its decalcification or dealumination. This is confirmed by the presence of Ca and Al in the acid solution (Fig. 10). Additionally, following immersion in acid the broad hump centred between 15 and 35° 2 θ shifted left and was larger which indicated the formation of Si rich gels (Bernal et al. 2013; Provis et al. 2005; Shearer et al. 2016). This is in agreement with the limited Si found leached into the acid solutions and suggested that while Ca and Al were removed Si rich gels remained.

The main minerals identified in the unexposed OPC mix were calcium hydroxide and ettringite. Following immersion in acid these were no longer present due to their dissolution. Calcite was observed instead, similar to previous studies, and was likely formed due to the carbonation of calcium available following the dissolution of phases with high calcium content such as calcium hydroxide and ettringite (Beddoe and Dorner 2005; Bernal et al. 2012; Bertron et al. 2004). After exposure to lactic acid, calcium lactate hydrate was also observed in OPC. This is the Ca salt of lactic acid and is less soluble than the Ca salt of acetic acid (Bertron and Duchesne 2013). This explains why lactic acid resulted in less Ca being drawn into the acid solution than acetic acid (Fig. 10).

The FTIR spectra of each mix following immersion in 0.52 mol/L solutions of acetic and lactic acid for 3 weeks is shown in Fig. 12. The patterns of unexposed (kept in water) samples are displayed as a reference. The band shown at around 1640 cm⁻¹ in each sample was due to the bending mode of H-O-H from water molecules (Burciaga-Díaz and Escalante-García 2012; Ismail et al. 2013). The main band in each alkali-activated mix which was not exposed to acid was found between 953 and 970 cm⁻¹. This band is assigned to asymmetrical T-O stretch vibrations (T = Si or Al) and indicates the presence of C-S-H type gel (García Lodeiro et al. 2009). Following acetic and lactic acid attack this main band shifts to higher wavenumbers between 1012 and 1045 cm⁻¹. This indicates the decalcification and dealumination of C-S-H types gels such as C-A-S-H and C-N-A-S-H (Bascarevic

et al. 2013; Bernal et al. 2012). A shoulder was also observed at around 1160 cm^{-1} which was identified as a Si rich gel product by previous authors (Bernal et al. 2012; García-Lodeiro et al. 2008). For each alkali-activated mix the main peak shifted less due to acetic acid attack (between 44 and 61 cm^{-1}) than it did due to lactic acid attack (between 70 and 79 cm^{-1}). This is likely due to the combination of decalcification and dealumination occurring due to lactic acid attack. In the case of acetic acid only decalcification occurred as evidenced by the leaching behaviour shown in Fig. 10. This is confirmed by FTIR as aluminosilicate gels which are centred at higher wavenumber have less Al present (Gu et al. 2020). The alkali-activated mixes which were attacked by acetic acid had their main band centred between 1012 and 1014 cm^{-1} , while following lactic acid attack they were centred between 1032 and 1045 cm^{-1} . This may explain the lower mass loss due to acetic acid as only decalcification occurred whereas lactic acid caused both decalcification and dealumination of the main binding gel and resulted in larger mass losses being observed (Fig. 6).

Following acetic and lactic acid attack the main band in OPC shifted from 960 to 1039 and 1030 cm^{-1} , respectively. This indicated decalcification of C-S-H and like the alkali-activated mixes a shoulder was observed at around 1170 cm^{-1} which suggested the formation of Si rich gels. The band at 1105 cm^{-1} , corresponding to ettringite was no longer observed after acid attack suggesting its decomposition (Bernal et al. 2012). Additional bands were observed at around 875 and 1425 cm^{-1} and have been attributed to the presence of calcite which was also observed by XRD (Bascarevic et al. 2013; Yu et al. 2004). An additional peak was also observed at around 1580 cm^{-1} following exposure to lactic acid and was previously related to carboxylic acid salts, in particular calcium lactate hydrate (National Institute of Standards and Technology 2018; Sedlarik et al. 2006; Song et al. 2014; Tian et al. 2015). This is in agreement with our XRD findings. Previous authors (De Belie et al. 1996; Singh et al. 1986) also observed the formation of calcium lactate hydrate following the interaction of calcium hydroxide and lactic acid.

SEM images of the outmost layer (approximately 2 mm) of FA/S-30/70 and OPC paste mixes after immersion in lactic acid for 3 weeks are shown in Fig. 13. The distribution of Al, Ca, Na and Si is

also shown and the arrow indicates the direction of acid attack. It was not possible to examine via SEM all the combinations of mixes and acids investigated in this study. Therefore, one alkali-activated mix (FA/S-30/70) and the OPC mix were examined following lactic acid attack as lactic acid was found to be more aggressive for both mixes. FA/S-30/70 has two distinct layers visible. The outer layer (1) which is darker in colour and has cracks and voids where alkali-activated paste has been removed. The inner layer (2) is lighter in colour and appears more intact with the exception of a few large cracks. The elemental analysis shows that the outer layer (1) has suffered depletion of Al and Ca, while the concentration of Na and Si appears to have increased in the outer layer. This is in agreement with the leaching behaviour observed and the results obtained by XRD and FTIR. It appears the C-A-S-H type gel present suffered decalcification and dealumination resulting in a relatively thick layer of Si rich gel which also contained Na.

The OPC mix appeared to have three main layers. The outer layer (1) appears very porous and has suffered a significant depletion of Ca. This is in agreement with the decomposition of calcium hydroxide and ettringite observed by XRD and FTIR following acid attack and the leaching of Ca into solution (Fig. 10). Similar to FA/S-30/70, the concentration of Si appears to have increased in the outer layer (1). The second layer (2) contains several cracks parallel to the outer surface and has suffered some depletion of Ca. This suggests that the outer layer (1) of Si rich gel has not been able to resist further acid attack. The innermost layer (3) appears intact and unaffected by acid attack.

In OPC the outer layer appears more porous than the outer layer in FA/S-30/70. This is likely due to the vulnerability of phases such as calcium hydroxide and ettringite which are not present in FA/S-30/70. Therefore, the outer layer of FA/S-30/70 is more dense and able to resist further acid attack. This is evidenced by the slightly decalcified second layer (2) of paste observed in OPC which is not observed in FA/S-30/70. Furthermore, the mass losses for OPC are accelerating but are decelerating for FA/S-30/70 (Fig. 5). This can be related to the more dense Si gel observed in the outer layer of FA/S-30/70.

Conclusion

The following conclusions have been drawn from this study.

- Alkali-activated mixes with increased FA content had similar or better resistance to acetic acid than that of mixes with increased slag content. This was evidenced by slightly decreased mass and compressive strength losses as the FA content increased.
- The main mechanism of attack by acetic acid was the decalcification of C-S-H type gel due to the high solubility of the Ca salt of acetic acid. The mixes with increased FA content had lower Ca content meaning their main binding gel was more resistant to acetic acid attack. However, their increased porosity allowed further ingress of acid which meant the overall performance was similar regardless of FA content.
- In the case of lactic acid attack the alkali-activated mixes with increased FA content have lower resistance to lactic acid attack. This was evidenced by larger mass losses and compressive strength losses. The alkalinity loss was also increased due to the increased porosity with increased FA content.
- Lactic acid attack was overall more aggressive than acetic acid as it caused both decalcification and dealumination of the main binding gel in alkali-activated mixes.
- The alkali-activated binders displayed better acetic and lactic acid resistance than ordinary Portland cement. Smaller mass and percentage compressive strength losses were observed. The ordinary Portland cement mix was susceptible to decomposition of its ettringite and calcium hydroxide phases leaving behind a more porous outer layer which was more susceptible to further acid attack, particularly when its initial acid neutralisation capacity was diminished.

This study suggests alkali-activated FA/slag binders have the potential to provide increased service life for concrete structures exposed to organic acids in the agri-food industries. Findings also indicate that the type of organic acid should dictate the composition of alkali-activated binder which is most suitable. Further work is needed on other types of organic acid prevalent in these industries.

Data availability statement

All data, models, and code generated or used during the study appear in the submitted article.

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Tables

Table 1. Oxide composition and loss on ignition (LOI) of FA, slag and Portland cement.

Oxide composition (%)	FA	Slag	Portland cement
SiO ₂	46.78	29.38	20.21
Al ₂ O ₃	22.52	11.23	4.79
Fe ₂ O ₃	9.15	0.36	2.78
CaO	2.24	43.72	63.01
MgO	1.33	6.94	1.93
MnO	0.05	0.51	0.08
TiO ₂	1.05	0.67	0.27
Na ₂ O	0.89	1.05	0.19
K ₂ O	4.09	0.93	0.59
SO ₃	0.90	1.76	2.60
LOI (%)	3.57	2.40	3.16
Specific gravity	2.42	2.92	3.13

Table 2. Mix proportions (kg/m³) for alkali-activated and ordinary Portland cement mortars

Mix composition		FA/S-30/70	FA/S-60/40	FA/S-80/20	OPC
Binder composition	Fly ash (%)	30	60	80	-
	Slag (%)	70	40	20	-
	OPC (%)	-	-	-	100
Paste content (vol. %)		50	50	50	50
Water/solid ratio		0.42	0.40	0.38	-
Water/cement ratio		-	-	-	0.42
Alkali dosage (M+)		7.5	7.5	7.5	-
Na ₂ O/SiO ₂		1.25	1.25	1.25	-
Fly ash (kg/m ³)		172	344	463	-
Slag (kg/m ³)		401	229	116	-
Portland cement (kg/m ³)		-	-	-	676
Sodium silicate (kg/m ³)		135	135	136	-
Sodium hydroxide (kg/m ³)		111	111	112	-
Sand (kg/m ³)		1348	1348	1348	1348
Absorption water (kg/m ³)		12	12	12	12
Total added water (kg/m ³)		125	112	100	296

Table 3. Porosity and volume of permeable voids for each mortar mix at 28 days.

Mix	Porosity (%)	VPV (%)
FA/S-30/70	5.3	8.5
FA/S-60/40	11.5	12.3
FA/S-80/20	15.8	19.0
OPC	10.1	12.5

Figure Captions List

Fig. 1. Photograph of a) grass stored in a concrete silo with brown effluent leaching out and b) damage to a concrete floor caused by silage effluent.

Fig. 2. Particle size distribution of fly ash, slag, Portland cement and sand.

Fig. 3. XRD patterns of FA, slag and Portland cement.

Fig. 4. Photographs of each mortar mix after immersion in solutions of acetic and lactic acid for 8 weeks.

Fig. 5. Mass change of a) FA/S-30/70, b) FA/S-60/40, c) FA/S-80/20 and d) OPC following immersion in solutions of acetic and lactic acid for 8 weeks.

Fig. 6. Mass change of alkali activated mortars following immersion in solutions of acetic and lactic acid for 8 weeks.

Fig. 7. Compressive strength of alkali-activated and OPC mortars following immersion in solutions of a) acetic and b) lactic acid for 8 weeks. The percentage compressive strength loss values are also provided.

Fig. 8. Cross-sections of alkali-activated and OPC mortar mixes after application of phenolphthalein solution following immersion in water (unexposed) and 0.52 mol/L solutions of acetic and lactic acid.

Fig. 9. pH of acetic and lactic acid solutions during immersion of mortar mixes for 8 weeks.

Fig. 10. Cumulative concentration of Al, Ca, Na and Si in solutions of water, acetic acid and lactic acid during immersion of alkali-activated and OPC paste mixes for 3 weeks.

Fig. 11. XRD patterns of each paste mix after immersion in solutions of acetic and lactic acid for 3 weeks.

Fig. 12. FTIR spectra of each paste mix after immersion in solutions of acetic and lactic acid for 3 weeks.

Fig. 13. SEM images and elemental analysis of the outmost layer of a) FA/S-30/70 and b) OPC paste mixes after immersion in lactic acid for 3 weeks.

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	Unexposed	Acetic acid concentration (mol/L)			Lactic acid concentration (mol/L)		
		0.10	0.31	0.52	0.10	0.31	0.52
FA/S-30/70
FA/S-60/40
FA/S-80/20
OPC

Fig. 4. Photographs of each mortar mix after immersion in solutions of acetic and lactic acid for 8 weeks.

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