

Research Article

Properties of Calcium Acetate Manufactured with Etching Waste Solution and Limestone Sludge as a Cementitious High-Early-Strength Admixture

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Concrete is one of the most widely used construction materials. There are several methods available to improve its performance, with one of them being the use of high-early-strength admixtures (HESAs). Typical HESAs include calcium nitrate, calcium chloride, and calcium formate (CF). Industrial by-products, such as acetic acid and lime stone sludge (LSS), can be used together to produce calcium acetate (CA), which can subsequently be used as a cementitious HESA. In this study, calcium carbonate and LSS were mixed with cement in weight ratios of 1:1,1:1.5, and 1:2, and the properties of the as-produced CA were evaluated. CA and CF were mixed with cement in different weight ratios (0, 1, 2, and 3 wt%) to obtain CA- and CF-mortars, respectively. The flow behavior, setting time, pH, and compressive strength of these mortars were evaluated, and their X-ray diffraction patterns were also analyzed. It was found that as the CF content in the CF-mortar increased, the initial strength of the mortar also increased. However, it impaired its long-term strength. On the other hand, when 1% CA was mixed with cement, satisfactory early and long-term strengths were achieved. Thus, CA, which is obtained from industrial by-products, can be an effective HESA.

1. Introduction

Several methods such as accelerated curing and the use of precast concrete and construction robots are used for increasing the production rate in ferroconcrete construction [1–7]. In particular, it is important to reduce the concrete curing time to reduce the overall concrete construction period. The overall performance and development of concrete are significantly influenced by both the curing process and the subsequent cast removal. Some of the methods currently used to improve these characteristics include steam curing and the use of high-early-strength admixtures (HESAs) [8-13]. Accelerated curing however requires the use of costly equipment. On the other hand, in the case of HESAs, an admixture addition of just 1-3 wt% is required to enhance the concrete performance, rendering this method more costeffective. In addition, with an increase in the use of blended cements and cementitious composites, the use of various HESAs as a supplement to low-early-strength cements such

as the ground-granulated blast-furnace slag has increased to improve their performance [14–17]. The primary mechanisms for such HESAs include the enhancement of the production of calcium silicate hydrate (C-S-H) using highly soluble calcium ions or increasing the hydration of amphoteric aluminum ions [18–21]. Generally, calcium nitrate, calcium chloride, and highly soluble calcium formate (CF) are used as HESAs to produce calcium salts, and amine mixtures are used to enhance the hydration of aluminum ions [9, 22, 23].

The semiconductor and steel industries produce a wide array of by-products including acetic acid and calcium salts. The amount of chemical substances used for etching thin film transistor-liquid crystal display boards in the semiconductor industry continues to increase. Etching is used to corrode a material with chemicals such as nitric acid and acetic acid to polish its surface and create a product with desired properties. The chemicals used for etching can be recycled by distillation [24]. Such substances have a lower solid content than the pure substances and use water as a solvent. According to the World

TABLE 1: Chemical composition of CaCO₃ and LSS (%).

	CaO	SiO ₂	MgO	Al_2O_3	SO3	Fe ₂ O ₃	P_2O_5	K ₂ O	Na ₂ O
CaCO ₃	96.08	0.98	2.06	0.39	0.02	0.36	0.00	0.11	0.00
Limestone sludge	90.46	3.81	1.06	2.06	0.08	1.35	0.70	0.45	0.04

Steel Association, the total global steel production in 2014 was over 12,700,000,000 tons [25]. The process of steelmaking commences by cleansing limestone, which is obtained by dehydration of the by-products produced during desulfurization, with water to produce quicklime. The quicklime so obtained is used as a raw material to produce steel. During this process, small limestone particles (<1 mm) are separated by precipitation and thus lime stone sludge (LSS) can be manufactured through a filter press process. LSS contains large quantities of water, which renders it uneconomical for recycling [26].

In this study, we manufactured a calcium-containing HESA (calcium acetate, CA), which is a source of calcium ions, by reacting acetic acid with LSS, which is a by-product of various industrial processes and evaluated the performance of the admixture so obtained. For a thorough evaluation, we examined the viscosity, density, pH, calcium content, heavy metal content, and solid content of the resultant admixture. We also added this admixture in different weight ratios to cementitious composites or mortars and examined their fluidity, setting time, pH, and compressive strength. We also carried out an X-ray diffraction (XRD) analysis of the cementitious composites or mortars containing this admixture.

2. Materials and Methods

2.1. Materials

2.1.1. Limestone Sludge. In this study, LSS, which is obtained from industrial by-products, was used to manufacture the calcium-containing HESAs. The XRD pattern and chemical composition analyses of LSS are shown in Figure 1 and Table 1, respectively. The XRD patterns of both $CaCO_3$ and LSS showed the presence of a $CaCO_3$ peak at approximately 29°. The structure of LSS appears to be identical to that of $CaCO_3$. However, considering the chemical composition, the CaO content in LSS was approximately 5.84% lesser than that in $CaCO_3$. In addition, LSS also contained SiO₂ and metallic salts such as Al_2O_3 and Fe_2O_3 .

2.1.2. Recycled Acetic Acid. Recycled acetic acid (RAA) was obtained from the previous etching processes by extraction via an acid separation process. In this study, approximately 60% RAA was used. Figure 2 shows the Fourier transform infrared (FT-IR) spectrum of RAA. In the FT-IR spectrum of RAA, the peaks in the range of $3300-2500 \text{ cm}^{-1}$ correspond to organic acids containing carboxylic groups. The peaks associated with C=O groups (1760–1690 cm⁻¹) and C-O groups (1320–1210 cm⁻¹) can also be observed in this spectrum. Some examples of carboxylic acids include acetic acid,



FIGURE 2: FT-IR spectrum of RAA.

formic acid, gluconic acid, and propionic acid. Hence, it was confirmed that RAA contained carboxylic acid groups [27].

2.1.3. Binder and Sand. A comparison between the properties of the conventional HESAs and the admixture produced in this study was performed by using mortar specimens. The mortar specimens consisted of class I ordinary Portland (OPC). The chemical compositions and physical properties of these materials are given in Tables 2 and 3, respectively. A description of the sand used in this study is given in Table 4.

2.2. Methods

2.2.1. Overview. In this study, we manufactured both the CA and the mortars containing CA in different weight ratios (0, 1, 2, and 3 wt%) and evaluated the performance of these mortars. The viscosity, density, and pH of these mortars were evaluated. Their inductively coupled plasma mass spectroscopy

	TABLE 2. One mean composition of binder (10).							
CaO	SiO ₂	MgO	Al_2O_3	SO ₃	Fe ₂ O ₃	P_2O_5	K ₂ O	Na ₂ O
63.85	21.09	3.32	4.84	3.09	2.39	0.36	0.73	0.14

TABLE 2: Chemical composition of binder (%)

	TABLE 3: Physical proj	perties of ordinary	Portland cement (OPC).		
Specific gravity	Blaine specific surface (cm^2/g)	Setting	g time	Compressive strength of mortar (MPa)		
	Diame specific surface (cm /g)	Initial set (min)	Final set (hour)	3 days	7 days	28 days
3.15	3400	400	7	20	23	38

TABLE 4: Properties of fine aggrega

Maximum size (mm)	Unit weight (kg/ℓ)	Specific gravity	Water absorption (%)	Solid volume percentage (%)
<1.2	1.5	2.62	0.40	58.1

TABLE 5: Measurements and methods.

Experiment	Measurement sample	Properties measured	Standards
xperiment alcium acetate using limestone udge and recycled acetate ement matrix using accelerator		Viscosity (cps)	ASTM D 2196 [28]
	Calcium acetate	Density (kg/m ³)	ASTM D 4052-11 [29]
sludge and recycled acetate	Galefulli accuate	pН	Properties measuredStandardsViscosity (cps)ASTM D 2196 [28]Density (kg/m³)ASTM D 4052-11 [29]pH—ICP-MS analysisASTM D 6800-12 [30]Setting timeASTM C 191 [31]pH—XRD—FlowASTM C 230/230M-14 [32]Compressive strengthASTM C 109 [33]
sludge and recycled acetate	ICP-MS analysis	ASTM D 6800-12 [30]	
		Setting time	ASTM C 191 [31]
Calcium acetate using limestone Calcium acetate Calcium acetate Density (kg/m ⁻) Calcium acetate pH ICP-MS analysis Setting time pH Cement paste pH Cement matrix using accelerator XRD	Cement paste	pН	
	_		
	Cement mortar	ICP-MS analysis ASTM D 6800-12 [30] Setting time ASTM C 191 [31] nent paste pH XRD - Flow ASTM C 230/230M-14 [32] Compressive strength ASTM C 109 [33]	
Cement paste pH ement matrix using accelerator XRD Cement mortar Flow Compressive strength	ASTM C 109 [33]		

(ICP-MS) analyses were also performed. In addition, an existing HESA (CF) was also added to mortar in different weight ratios (0, 1, 2, and 3 wt%) to obtain CF-mortars and their CF content, setting time, XRD patterns, and pH were also investigated. The flow behavior and compressive strength of the mortars were also measured. Table 5 summarizes these experiments. The flow chart for the synthesis of CA is shown in Figure 3.

2.2.2. Manufacturing CA with LSS and RAA. Here, RAA and LSS reacted to yield CA in accordance with (1). The yield was maximum when the ratio of acetic acid to $CaCO_3$ was 2:1 with 1 mol each of CA, water, and carbon dioxide per mol of $CaCO_3$:

$$2CH_{3}COOH + CaCO_{3}$$

$$= Ca (CH_{3}COO)_{2} + H_{2}O + CO_{2}$$
(1)

Calcium carbonate and RAA were used to compare the solubility of the LSS and acetic acid together. To 60% solid acetic acid, LSS was added in different weight ratios (1, 1.5, or 2) as shown in Table 6. This mix was reacted for 12 h. Upon



FIGURE 3: Flow diagram for CA synthesis.

mixing, the unreacted particles were removed via filtering and acetic acid was separated for future use.

2.2.3. Cementitious Composites with HESAs. The mix proportions of the cementitious composites (cement paste and mortar) that were mixed with HESAs are given in Table 7. The



FIGURE 4: Viscosity and density of calcium acetate.

CA (used as a HESA) was weighed such that the weight ratio of LSS to acetic acid was 2:1 and the pH of the overall mixture was 5.9. The manufactured specimen underwent steam curing at a temperature of 20°C and a relative humidity of 60%.

The paste specimens were produced by mixing 0, 1, 2, and 3% of CA and CF each with 100 g of OPC at a water to binder ratio of 0.32 for 3 min. The setting time and pH of these specimens were evaluated. Their XRD patterns were also analyzed. The setting time was measured according to ASTM C191. The pH measurements were performed by collecting 10 mL of each material after binding and subsequently immersing them in 100 g of distilled water. The results were recorded at 60 min and 24 h, in triplicate. For the XRD analysis, 30 g of the cured paste specimen was collected after 18 and 24 h, following which it was broken down into particles of <300 μ m diameter of the particles and finally immersed in acetone. The X-ray diffractometer was operated at 45 KV and 40 mA with Cu radiation. A scan rate of 5°/min was maintained over a range of 5–60°.

The cement mortar was manufactured by adding 135 g of sand to the paste composites. The sand and paste composites were mixed for 3 min, allowed to rest for 2 min, and finally mixed for additional 2 min. The flow of the mixed specimen was measured according to ASTM C230. For each mixture, molds with the dimensions of $4 \text{ cm} \times 4 \text{ cm} \times 16 \text{ cm}$ were formed, cured for 18 h, and were immediately removed. The compressive strength was measured according to ASTM C109 at the intervals of 1, 3, 7, and 28 days, in triplicate.

3. Results and Discussion

3.1. CA

3.1.1. *pH and Density of CA*. The results of the viscosity and density measurements of the manufactured CA are shown in Figure 4. The viscosity and density of the CA solutions (containing $CaCO_3$ and LSS in weight ratios) showed similar trends. The viscosity and density of the CA solutions

TABLE 6: Reaction ratio according to weight ratio of RAA : CaCO₃.

RAA: CaCO ₃	1:0.5	1:1	1:1.5	1:2
Reaction yield (%)	52	42	31	24.97
Reacted acetic acid (%)	42.4	80.8	91.6	99.86

TABLE 7: Mixtures for paste and mortar specimens.

Mixture number	OPC	Water	Calcium acetate	Calcium formate	Sand
OPC	100	32	0		135
OPC-A1 ^(A)	100	32	1		135
OPC-A2	100	32	2		135
OPC-A3	100	32	3		135
OPC-F1 ^(B)	100	32		1	135
OPC-F2	100	32		2	135
OPC-F3	100	32		3	135

 $^{(A)}A = \text{calcium acetate}, 1 = \text{cement} \times 1 \text{ wt\%}.$

 $^{(B)}$ F = calcium formate, 1 = cement × 1 wt%.

increased with an increase in the weight ratios of $CaCO_3$ and LSS. The density of acetic acid is 1.049 g/cm³ and that of the CA is 1.509 g/cm³. Thus, as a greater quantity of acetic acid reacts with $CaCO_3$ to produce the CA, the density of this reactant increases.

3.1.2. pH and Ca Ion-ICP Analysis. Figure 5 shows the results of the pH measurement and Ca Ion-ICP analysis of the manufactured CA. The pH appeared to increase slightly when calcium carbonate was used. Conversely, the pH was low when LSS was used as it contains small quantities of Ca. However, when the weight ratio of LSS was greater than 2, the pH of the CA produced using CaCO₃ was similar to that obtained when LSS was used for producing the CA.



FIGURE 5: pH and Ca ion concentration of calcium acetate.

TABLE 8: ICP Analysis of heavy metals (m
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	Decycled eastic eaid	Limest	one sludge : recycled ace	tic acid	Ston doud(A)
	Recycled acetic acid	2:1	2:1.5	2:2	Standard
Cd	Not detected	Not detected	Not detected	Not detected	< 0.1
Cr	Not detected	Not detected	Not detected	Not detected	<2
Cu	3	1.5	1.62	1.61	<3
Hg	Not detected	Not detected	Not detected	Not detected	< 0.005
Pb	Not detected	Not detected	Not detected	Not detected	<1
Mn	Not detected	Not detected	Not detected	Not detected	_
Zn	3	1.5	1.7	1.9	<5
As	Not detected	Not detected	Not detected	Not detected	< 0.5
Solid contents	62%	22%	27%	29%	

^(A)Water quality standards of Japan.

3.1.3. Microelement Analysis. Table 8 shows the results of the ICP analysis of heavy metals of the manufactured CA. The microelements and solid contents of the manufactured CA are listed in Table 8. Heavy metals were not detected, indicating that the CA manufactured in this work is less hazardous to the environment than the other similar materials [34].

3.2. Cement Composites

3.2.1. Flow and Setting Time of Mortar. Figures 6 and 7 display the flow and setting times, respectively, of the cement mortar obtained by mixing CA with cement. It was found that, for the CA-mortar, the fluidity increased with an increase in the CA content, as shown in Figure 6. However, in the case of the CF-mortar, the fluidity decreased with an increase in the CF content.

From Figure 7, it can be observed that when CF was used and the mixing ratio was identical for the CA- and CF-mixed mortars, the initial and finish setting times were reduced. In the case of the CA-mortar, the initial setting time was reduced when the mixing ratio increased. At a CA content of 1%, a minimum final setting time was obtained, which increased



FIGURE 6: Flow test results.

as the mixing ratio increased. In case of the CF-mortar, no reversible change in the setting time was observed with a change in the mixing ratio, and the setting time decreased proportionally with a decrease in the mixture ratio.



FIGURE 7: Setting time tests results.

HESA Calcium acetate	Admixture proportion (cement \times %)	pH of cemer	pH of cement paste as a function of time			
		Immediate	60 minutes	24 hours		
	OPC	12.2	12.4	12.4		
	OPC-A1	11.8	12.2	12.3		
Calcium acetate	OPC-A2	10.2	11.0	12.1		
	OPC-A3	9.0	10.2	11.9		
	OPC-F1	12.3	12.2	12.4		
Calcium formate	OPC-F2	12.2	12.3	12.3		
	OPC-F3	12.1	12.2	12.1		

TABLE 9: pH of cement paste as a function of tim	ie.
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3.2.2. Change in pH of Cement Paste. The pH values of the cement pastes, as a function of time, are given in Table 9. In the case of the paste containing the CA, the pH initially decreased with an increase in the CA content. On the other hand, in the case of the paste containing CF, the pH remained relatively constant regardless of the CF content. When the CA content exceeded 1%, the setting time was reduced. Bortoluzzi et al. reported that when $CaCl_2$ (pH = 4.4) is mixed with cement, the pH of the cement decreases. The pH subsequently recovers because of the production of Ca^{2+} , which reduces the setting time [35]. When 3% CA is mixed, the hydration time decreases to 24 h and a retarding phenomenon can be observed. When CA content is more than 2%, both initial pH decrease and setting time delay were observed because of the high solubility of CA and residual acetic acid.

3.2.3. Compressive Strength of Mortar. Figure 8 shows the compressive strengths of the CA- and CF-mortar specimens at different curing times. When a CA content of 1% was used, the compressive strength of the resulting mixture was 22% greater than that obtained when no admixture was added (i.e., for a standard specimen). When the CF content in the mortar was 2%, similar strengths were obtained. When 3%

CF was mixed and the mix was cured for seven days, the strength of the resulting mortar was found to be greater than that of the standard material. However, the strength started decreasing after 28 days. When 1% CA was mixed and the mix was cured for three days, the strength of the resulting mortar was over 40% greater than that of the standard specimen and was still 14% greater even after 28 days. However, when the CA content exceeded 2%, the compressive strength of the material diminished because of the reduction in both the pH of the cement paste and the hydration of acetic acid.

3.2.4. XRD. The XRD analysis was performed on the CAand CF-mortars. Figure 9 shows the XRD patterns of the CA-mortars with different CA contents after a curing period of one day. When 1% CA was mixed, the intensity of the Ca(OH)₂ peak decreased, while that of the calcium aluminosilicate hydrate (C-A-S-H) and C-S-H peaks increased, indicating rapid hydration. The CA-mortar having a CA content of 3% showed a weaker Ca(OH)₂ peak than that of the mortar containing no admixture. However, in the case of the CA-mortar containing 1% admixture, this peak was more intense, and the intensity of the C-A-S-H and C-S-H peaks



FIGURE 8: Compressive strength of specimens.



FIGURE 9: Specimen incorporating calcium acetate (1 day).

was also lower. In contrast, the intensity of the ettringite peak increased as the CA content increased.

The XRD patterns of the CF-mortars with different CF contents after a curing period of one day are shown in Figure 10. When 1% CF was mixed, the intensity of both the Ca(OH)₂ and C-A-S-H peaks increased. When 3% CF was mixed, the intensity of both the Ca(OH)₂ and C-A-S-H peaks decreased, while that of the ettringite and C-S-H peaks increased. As reported by Heikal [36], the diffusion rate of $\rm HCOO^-$ ions is greater than that of $\rm Ca^{2+}$ ions. Therefore, it is possible for HCOO⁻ ions to penetrate into the protective layer of C-S-H. As a result, the C-S-H decomposition is directly proportional to the deposition of Ca(OH)₂. In addition, Ramachandran [37] stated that the presence of COOH⁻ ions promotes the hydration of C₃S by interfering with the protective layer of cement particles, which ultimately leads to the formation of ettringite. It was found that the structure of the CA- and CF-mortars containing 1% CA and CF, respectively, was similar, and both the mortars enhanced



FIGURE 10: Specimen incorporating calcium formate (1 day).

the production of C-A-S-H, ettringite, and C-S-H during the initial early stages of concrete formation [38].

The XRD patterns of the CA- and CF-mortars cured for three days are shown in Figures 11 and 12, respectively. In the case of the CA-mortar containing 1% CA, ettringite, the Ca(OH)₂, and C-A-S-H peaks were more intense, as shown in Figure 11. When 3% CA was mixed, the intensity of the Ca(OH)₂ and C-A-S-H peak decreased, while that of the ettringite and C-S-H peaks increased. In the case of the CAmortar containing 1% CF, ettringite, the Ca(OH)₂, and C-A-S-H peaks were more intense, as shown in Figure 11. When 3% CA was mixed, the intensity of the Ca(OH)₂ and C-A-S-H peak decreased, while that of the ettringite peaks increased as shown in Figure 12.

When Al ion content is the same, $Ca(OH)_2$ and C-A-S-H increase according to the increase of Ca ion [39]. Thus, when CA and CF contents are 1%, $Ca(OH)_2$ and C-A-S-H increased. On the other hand, according to the increase of CA and CF contents, $Ca(OH)_2$ and C-A-S-H peaks decreased.



FIGURE 11: Specimen incorporating calcium acetate (3 days).



FIGURE 12: Specimen incorporating calcium formate (3 days).

While the pH of CF is 7.0 and its solubility is 166 g/L, the pH of CA is about 5.9 and its density is 247 g/L. High solubility of CA affected both ettringite generation and decrease of $Ca(OH)_2$ and C-A-S-H peaks [40].

4. Conclusions

CA was manufactured using acetic acid and LSS obtained from industrial by-products, and its characteristics were examined. The performances of the cementitious composites mixed with this manufactured CA were evaluated. On the basis of the results obtained, the following conclusions were made:

(1) The pH of the CA manufactured via the reaction of $CaCO_3$ and LSS was found to be the same as that of the CA manufactured using $CaCO_3$ (5.9). The microelemental analysis showed that the heavy metal content in the manufactured CA was lower than the detection limits.

- (2) The compressive strength of the CA-mortars was maximum when 1% CA was mixed. This value of compressive strength was more than that obtained when no admixture was added even when the specimens were cured for 28 days. In particular, on the 28th day, the CA-mortar showed a higher compressive strength than that of the CF-mortar.
- (3) For the cement pastes mixed with CF, the pH remained constant at all the mixing ratios. However, when the CF content exceeded 1%, CA, which is more acidic, had a greater influence on the mixture, leading to a reduction in the pH and strength of the cement, for the hydrates of the hardened cement pastes mixed with the CA and CF, which is advantageous for the enhancement of the C-S-H and ettringite.
- (4) Finally, we can conclude that the CA is manufactured using the recycled industrial by-products functions as an effective HESA for cement composites.

Competing Interests

The authors declare that there are no competing interests regarding the publication of this paper.

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