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Properties of artificial lightweight aggregates made from waste sludge

I.J. Chiou*[1] and C.H. Chen[2]

[1]Graduate School of Materials Applied Technology, Department of Civil and Environmental Engineering, Nanya Institute of Technology, No. 414, Sec.3, Jhongshan E. Rd., Jhongli, Taoyuan 320, Taiwan, R.O.C.
[2]Department of Social and Regional Development, National Taipei University of Education, No. 134, Sec. 2, Heping E. Rd., Taipei City 106, Taiwan, R.O.C.

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Abstract. In this investigation, reservoir sediment and municipal sewage sludge were sintered to form the artificial lightweight aggregates. The sintered aggregates were compared with the commercialized lightweight aggregates to in terms of potential alkali-silica reactivity and chemical stability based on analyses of their physical and chemical properties, leaching of heavy metal, alkali-silica reactivity, crystal phase species and microstructure. Experimental results demonstrated that the degree of sintering of an aggregate affected the chemical resistance more strongly than did its chemical composition. According to ASTM C289-94, all potential alkali-silica reactivity of artificial lightweight aggregates were in the harmless zone, while the potential reactivity of artificial lightweight aggregates made from reservoir sediment and municipal sewage sludge were much lower than those of traditional lightweight aggregates.

Keywords: sewage sludge; reservoir sediment; sintering; lightweight aggregate (LWA); potential alkali-silica reactivity.

1. Introduction

For reasons of environmental protection, the cement industry adopts a dry process to manufacture cement. This process increases the alkalinity of the cement and its potential alkali-silica reactivity. Numerous examples of concrete deterioration from other parts of the world had been reported to show that the alkali-silica reaction might become one of the causes of distress in structures located in humid environments, such as dams, bridge piers, and sea walls (Mehta *et al.* 2006). Alkali-aggregate reactions could be classified into three groups- alkali-silica reactions, alkali-carbonate rock reactions, and the alkali-silicate reactions (Gillott 1975). The important factors that governed the activation of the alkali-aggregate reaction included the amount of reactive aggregate, the aggregate size, the ingredients of the aggregate, the potassium-sodium ratio and the alkali content in cement (Dolar 1984, Swamy 1992, Young 1999, Chatterji 2005, Khouchaf and Verstraete 2007, Multon 2008, Leemann 2008). Research performed by Taiwan Area National Expressway Engineering Bureau (MOTC) revealed that all the aggregates had the potential to exhibit an alkali-silica reaction with only a small amount of alkali, and could maintain long-term expansion (Su and Huang 1987, Wang 1989, Chu and Yen 1994). In addition, researches indicated that several Taiwanese aggregates had

* Corresponding author, Ph.D., E-mail: cij@nanya.edu.tw

potential alkali-silica reaction. Most could have a moderate reactivity in this respect. A high alkali content of cement could activate the alkali-silica reaction (Chu and Yen 1994, Young 1997). Artificial lightweight aggregate, made from recycled reservoir sediment, could be used to produced high-performance lightweight aggregate concrete, and could enhance performance characteristics and durability of concrete (Wang 2007, Wang and Sheen 2010). Curing conditions affected the relationship between pulse velocity and compressive strength of volcanic pumice concrete and normal concrete (Khandaker and Anwar 2009).

Extra-fine waste glass and expanding agent were mixed to sinter lightweight aggregates in a rotary kiln. The sintered lightweight aggregate not only exhibited a high potential to exhibit the alkali-silica reaction, but also could be a provider of extra alkali. However, no expansion or cracking of the cement mortar occurred (Ducman 2002). They studied the alkali-silica reactions of expanded vermiculite, expanded clay, expanded glass and perlite individually, and discovered that neither the expanded vermiculite nor the expanded clay had any potential alkali-silica reactivity. However, the texture of the expanded glass and perlite was seriously decomposed. No significant expansion of the mortar prism occurred in the acceleration experiment (Mladenovič 2004). The chemical composition of both sewage sludge and sewage sludge ash was similar to those of expansive clays. Using oil shale as an admixture could effectively reduced the expansion of cement mortars or concretes that was caused by the alkali-silica reaction. Additionally, they found that this oil shale could be used as a pozzolanic admixture (Yeinobali *et al.* 2006). The chemical composition of the expansive clay was as follows. SiO_2 48-70%; Al_2O_3 8-25%; $Fe_2O_3 + FeO$ 3-12%; $CaO + MgO$ 1-12%; K_2O+Na_2O 0.5-7% (Riley1950).

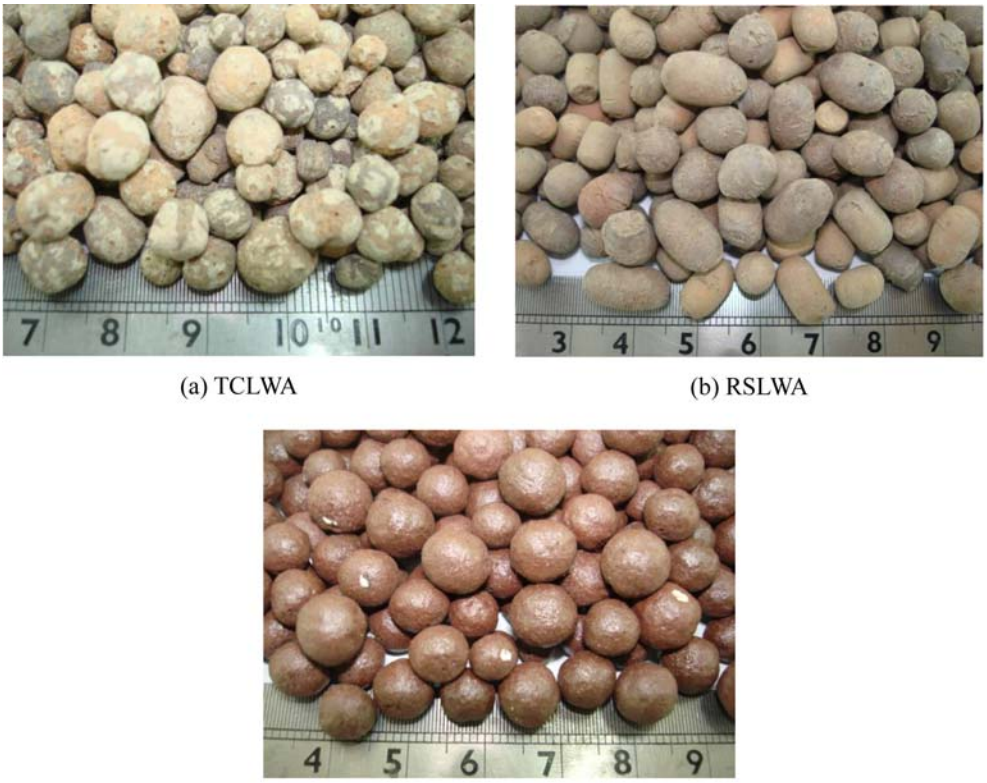
Therefore, sewage sludge and its ash could be sintered individually or together to produce normal-weight or lightweight aggregates. Moreover, sewage sludge could be added to adjust the chemical composition of sewage sludge ash (Chiou *et al.* 2006). In this investigation, reservoir sediment and municipal sewage sludge were utilized to sinter reservoir sediment lightweight aggregate (RSLWA) and sewage sludge lightweight aggregate (SSLWA). The basic characteristics and durability of RSLWA and SSLWA were compared with those of commercialized "Tai-Chin" lightweight aggregate (TCLWA), which was made of clay and shale. The potential alkali-silica reactivity of these three aggregates was investigated.

2. Methodology

2.1 Raw materials and production procedures of lightweight aggregate

Clay, expansive shale and reservoir sediment were used as the raw materials herein in this study. Clay and expansive shale were from the Chishan region, Kaohsiung County, sludge from the Shihmen Reservoir, Taoyuan County, and dehydrated sewage sludge from the Taipei City Wastewater Treatment Plant. These three raw materials were dried, ground and screened to yield dried expansive clay powder, dried reservoir sediment powder and dried sewage sludge powder. The dried sewage sludge powder was fired in a pilot-scale incinerator at 900ºC for 3 hours, ground for 3 hours, and screened with a #50-sieve (0.3 mm) to obtain the sewage sludge ash.

Three artificial lightweight aggregates were used; TCLWA, RSLWA and SSLWA. They were presented in Fig. 1. The approaches for producing these three aggregates were as follows. TCLWA was made of clay and expansive shale. It was shaped into spherical particles using water and some



(a) TCLWA (b) RSLWA

(c) SSLWA

Fig. 1 Appearance of lightweight aggregates (LWA)

additives in a titled rotating pan. The air-dried spherical coarse grains were fired and expanded in a rotary kiln (1100-1200ºC, for about 40-50 minutes). The TCLWA made in this way could be applied as normal lightweight concrete.

After the reservoir sediment was extracted from the water outlets of the reservoir, it was placed in settling ponds for deposition, which was followed by air drying. Next, the impurities of the dewatered reservoir sediment were screened out, and a pelletizing machine was used to make spherical pellets. The air-dried pellets were sintered in a rotary kiln at 1100-1200ºC for 40-50 minutes. In this process, RSLWA was produced. The mixing ratio of the municipal sewage sludge ash to the dried sewage sludge powder was 80:20. This mixture material was used to form pellets in a rotary pelletizing machine. The air-dried pellets were sintered in a furnace at 1100ºC for 10 minutes to yield SSLWA.

A #100-sieve (0.15 mm) machine was used to screen the sintered aggregates. The degree of sintering was evaluated from the amount of powder obtained, the weight loss of the aggregates upon washing, visual inspection and the amount of powder that adhered to the fingers when it was touched. The evaluation was used to adjust the sintering conditions.

2.2 Experiment

The raw materials used in this study were comprised clay, expansive shale, reservoir sediment, dehydrated sewage sludge and incinerated ash. The characteristics of expansive shale, reservoir sediment and sewage sludge ash were analyzed, by determining their chemical compositions (by Philips PW1606 X-ray fluorescence spectromete, XRF), their gross amounts of heavy metals (by flame atomic absorption spectrometer, FAAS, Hitachi Z-2000 Series) and their leaching concentrations (by the toxicity characteristic leaching procedure, TCLP as described in Taiwan EPA-SW 846-1311 method) of heavy metals.

The characteristics of artificial lightweight aggregates were determined by ignition loss, bulk density (ASTM C29), water absorption (ASTM C12), accumulative amount of power, weight loss in shaking and washing, point loading strength (failure load of the aggregate at a single point), Mohs hardness, gross amount of heavy metals, leaching concentrations of heavy metals (obtained using the toxicity characteristic leaching procedure, TCLP), chemical corrosion, potential alkali-silica reactivity (ASTM C289-03), crystal phase species and SEM microstructures.

3. Results and discussion

3.1 Chemical properties of lightweight aggregates

The moisture, ash and combustible material contents of municipal sewage sludge were 52.5%, 32.8%, and 11.7% respectively. Table 1 presented the chemical components of the expanded clay, reservoir sediment and sewage sludge ash. The chemical components of these three raw materials were similar. The major component was silica oxide (SiO_2), which represented at least 61.7% of each; the next most important components were aluminum oxide and ferric oxide. Riley noted that the chemical constituents of expansive clay were SiO_2 48-70%, Al_2O_3 8-25%, Fe_2O_3+FeO 3-12%, CaO+MgO 1-12% and K_2O+Na_2O 0.5-7% (Riley 1950). Furthermore, the content of SiO_2 in the high expansive clay must be less than 60%, with Al_2O_3 14-20%, CaO+MgO less than 7%, Fe_2O_3+FeO 6-10% and K_2O+Na_2O 3-5% (Riley 1950). Based on Riley's assertion, reservoir sediment and sewage sludge ash could only be classified as normal expansive clay, not highly expansive clay, because their SiO_2 contents both exceed 60%.

Table 2 presented the chemical composition of the artificial lightweight aggregates. The major component of all these three lightweight aggregates was silica oxide (SiO_2), whose content ranged between 51.2 and 56.3%. The ignition losses of these three lightweight aggregates (TCLWA, RSLWA, and SSLWA) were 0.8%, 0.3%, 0.1% individually, indicating that most of the organic matter contained in the aggregates vaporized upon sintering. Therefore, the aggregates were similar to inorganic substances and exhibited superior long-term stability. SSLWA did not have an unpleasant odor,

Table 1 Chemical composition of experimental materials

Composition (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	P_2O_5	SO_3
Expansive clay	61.7	16.8	6.97	1.02	2.25	1.68	3.07	-	<0.01
Reservoir sediment	62.3	17.5	9.24	0.72	1.37	1.22	2.85	-	<0.01
Sewage sludge ash	62.8	15.4	6.81	1.80	1.03	0.70	1.51	7.02	<0.01

Table 2 Chemical composition of lightweight aggregates (LWA) (%)

Aggregates	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	P_2O_5	LOI
TCLWA	51.2	14.8	4.79	1.59	6.72	<0.01	2.97	ND	0.73
RSLWA	54.8	16.1	5.71	1.51	1.72	<0.01	2.94	ND	0.20
SSLWA	56.3	17.6	7.88	2.93	1.89	0.93	2.59	0.23	0.13

*LOI: Loss on ignition; Tai-Chin lightweight aggregate (TCLWA); Reservoir sediment lightweight aggregate (RSLWA); Sewage sludge lightweight aggregate (SSLWA).

unlike sewage sludge.

Sewage sludge ash contained 7.02% P_2O_5, but SSLWA contained only 0.23% P_2O_5, revealing that P_2O_5 vaporized at 1100ºC. The generation of gas by expansive clay, reservoir sediment and sewage sludge ash at high temperatures was investigated. XRF could not detect the sulfide constituents (such as SO_3) of the three raw materials. Since the sintering temperatures in this investigation all exceeded 1100ºC, gas was generated by the expansion of the aggregate that was caused by the decomposition of CO_2 and O_2 from $CaCO_3$ and Fe_2O_3 individually. Table 1 indicated that almost no sulfur oxide (such as SO_2 or SO_3) was decomposed from FeS, $Fe(SO_4)_2$ and $CaSO_4$ upon sintering, and therefore no potential corrosion problem. The pH values of TCLWA, RSLWA, and SSLWA were 7.61, 6.83 and 6.69 individually, revealing that all of these aggregates were approximately pH neutral materials.

3.2 Distribution of heavy metals in lightweight aggregates

Tables 3 and 4 presented the results of the analysis of the total amount of heavy metals in the raw materials and lightweight aggregates, and of the Toxicity Characteristic Leaching Procedure (TCLP). The results showed that the leaching concentrations of heavy metals in the sewage sludge, sewage sludge ash, reservoir sediment, and the three lightweight aggregates were all very low, and all satisfied the requirements of the Environmental Protection Administration, Taiwan. With reference to the total amounts of heavy metals in Table 3, it could be observed that firing at 900ºC increased the concentrations of Pb, Cd, Cr, Cu, and Zn. However, according to the leaching concentrations of heavy metals in Table 4, after firing at 1100ºC, only Cu and Zn were detected; those of Pb, Cd, and Cr were too low to be detected, suggesting that the leaching of heavy metals is limited when firing at 1100ºC.

Table 3 Results of concentrations of heavy metals and leaching in sewage sludge and reservoir sediment

Material	Heavy metals	Pb	Cd	Cr	Cu	Zn
SSP	Total amount (mg/kg)	0.28±0.09	1.60±0.13	9.51±0.11	33.32±1.28	176.57±2.97
SSA	Total amount (mg/kg)	1.20±0.013	4.81±0.03	21.96±1.84	89.64±3.77	567.3±4.85
	TCLP (mg/L)	0.017±0.006	ND	0.10±0.05	3.81±0.13	ND
RSP	Total amount (mg/kg)	44.35±0.02	3.61±0.11	ND	27.37±1.12	176.2±4.41
	TCLP (mg/L)	0.065±0.001	0.037±0.001	ND	0.271±0.003	0.707±0.022
Regulatory of TCLP (mg/L)		5	1	5	15	-

*SSP: Sewage sludge powder; SSA: Sewage sludge ash; RSP: Reservoir sediment powder; ND: Not detected.

Table 4 Gross amount and leaching concentration of heavy metals in lightweight aggregates (LWA)

Lightweight aggregates		Heavy metals				
		Pb	Cd	Cr	Cu	Zn
Total amount (mg/kg)	TCLWA	N.D	N.D	N.D	1.19	117.7
	RSLWA	N.D	N.D	N.D	N.D	62.95
	SSLWA	3.96	N.D	15.09	155.7	245.1
TCLP (mg/L)	TCLWA	N.D	N.D	N.D	N.D	1.23
	RSLWA	N.D	N.D	N.D	0.035	1.16
	SSLWA	N.D	N.D	N.D	0.392	1.22
Regulatory of TCLP (mg/L)		5	1	5	15	-

*Tai-Chin lightweight aggregate (TCLWA); Reservoir sediment lightweight aggregate (RSLWA); Sewage sludge lightweight aggregate (SSLWA); ** ND: Not detected.

3.3 Mechanical properties of lightweight aggregates

From Fig. 1 (appearance of the aggregates), the total amount of powder that passed through a #100-sieve (0.15 mm) and weight loss upon washing, the degree of sintering was determined. The experimental results in Table 5 revealed that the ratio of powdered TCLWA, RSLWA and SSLWA that passed through a #100-sieve (0.15 mm) were 8.09%, 7.35%, 6.87% respectively. The weight losses of these three aggregates upon washing were 1.34%, 0.35% and 0.31%, individually. Both the powder amount and weight loss of SSLWA were the least. This result demonstrated that the degrees of sintering of these three artificial aggregates differed. That of SSLWA was the greatest, followed by that of RSLWA and that of TCLWA, which exhibited the least sintering.

Table 5 displayed the properties of TCLWA, RSLWA, and SSLWA. The bulk densities of these three aggregates were 383 kg/m^3, 817 kg/m^3, and 649 kg/m^3 individually, indicating that all three

Table 5 Characteristics of physical and chemical properties of lightweight aggregates (LWA)

Properties		TCLWA	RSLWA	SSLWA
Bulk density (kg/m^3)		383	817	649
Water absorption (%)		37.8	11.0	8.87
Loss on ignition (%)		0.8	0.3	0.1
Potential alkali-silica reactivity	Sc (mmol/L)	422	305	25
	Rc (mmol/L)	715	870	885
	Sc/Rc	0.59	0.35	0.03
Point loading strength (kgf)		12.4±2.4	23.1±4.1	61.4±5.5
pH		7.61	6.83	6.69
Mohs hardness		2~3, Gypsum	6~7, Feldspar	7~8, Quartz
Accumulative powder amount passing #100-sieve (0.15 mm) (%)		8.09	7.37	6.87
Weight loss through washing (%)		1.34%	0.35%	0.31%

*Tai-Chin lightweight aggregate (TCLWA); Reservoir sediment lightweight aggregate (RSLWA); Sewage sludge lightweight aggregate (SSLWA).

were lightweight but to different extents. In water absorption, that of TCLWA reached 37.8%, but those RSLWA and SSLWA were only 11.0% and 8.87% individually. The water absorption of these three aggregates was determined from the brilliance of the dry aggregates surface, shown in Fig. 1.

The weight losses of TCLWA, RSLWA, and SSLWA upon firing at 900ºC for 3 hours were determined to evaluate their degrees of sintering and long-term stabilities. The losses on ignition of these three lightweight aggregates were all less than 0.8%; that of SSLWA was only 0.1%. The glossiness (Fig. 1), water absorption and weight loss of these three aggregates demonstrated that the RSLWA and SSLWA were more sintered and stable than TCLWA.

The mechanical properties of the lightweight aggregates were evaluated using Mohs scale of mineral hardness and point loading strength. Table 3 presented the results. The Mohs hardness scales of TCLWA, RSLWA, and SSLWA were 2-3, 6-7 and 7-8 individually. These three aggregates therefore had the hardness of gypsum, feldspar, and quartz, respectively. This result indicated that the hardness of TCLWA was much lower than those of RSLWA and SSLWA. However, those of RSLWA and SSLWA were similar. "Point loading strength" meant the failure loading of aggregate in one single point. Fig. 2 showed the point loading strengths of TCLWA, RSLWA, and SSLWA were 10.5-17.3 kgf (average 12.4kgf), 17.7-28.2kgf (average 23.1 kgf), and 58.2-66.4 kgf (average 61.4 kgf) individually; TCLWA had the lowest values. The coefficients of variation (CV) of the point loading strength of the three lightweight aggregates were 19.35%, 17.75%, and 8.96% individually. TCLWA had the largest coefficient of variation of point loading strength. Accordingly, coefficients of variation of point loading strength were negatively related to degree of sintering.

3.4 Microstructure of lightweight aggregates

Fig. 3 presented the XRD patterns of TCLWA, RSLWA, and SSLWA. The main peak represented SiO_2; the next represented Al_2O_3. The species in the XRD patterns were consistent with the chemical components of the aggregates that were listed in Table 2. Moreover, the peak intensities of SiO_2 in RSLWA and SSLWA were much higher than that of TCLWA, because the SiO_2 contents in both RSLWA and SSLWA exceeded that in TCLWA. In addition to $SiO_2{\cdot}BAl_2O_3$, the lightweight

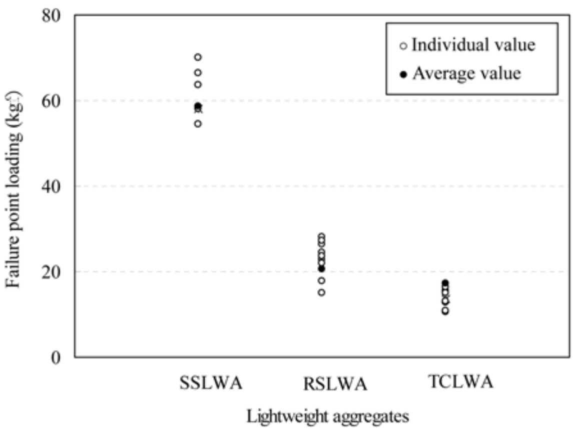


Fig. 2 Point loading strength of lightweight aggregates (LWA)

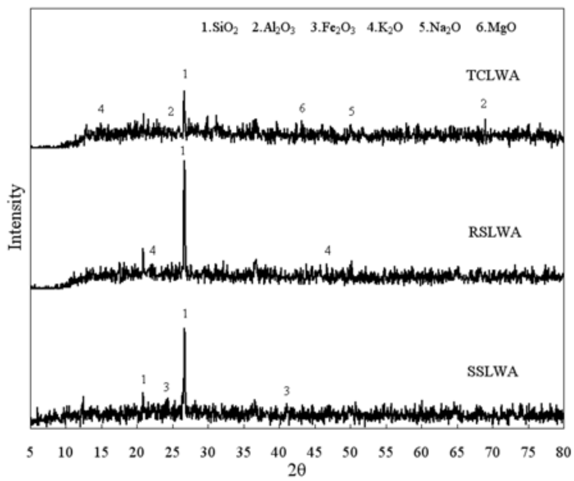


Fig. 3 XRD patterns of lightweight aggregates (LWA)

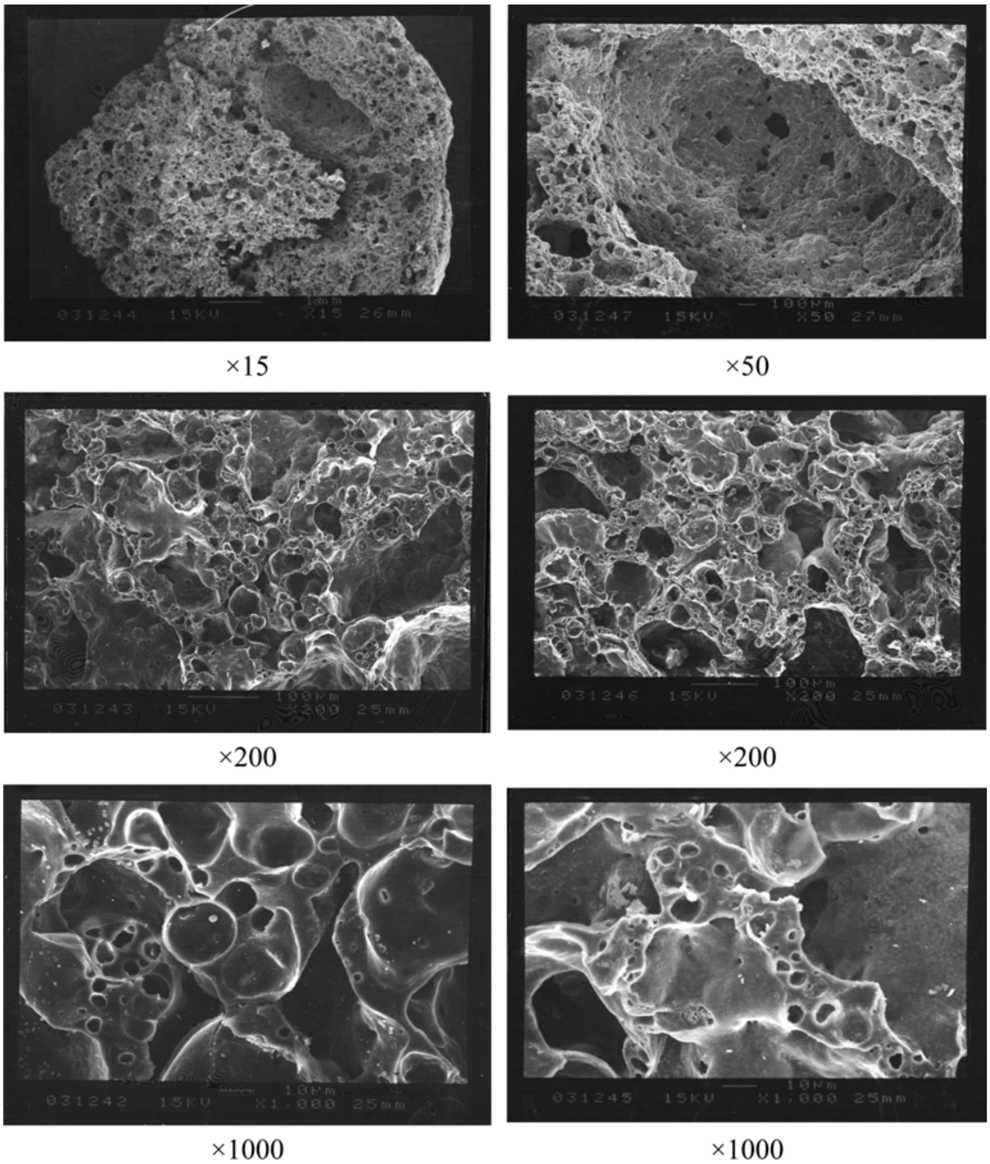


Fig. 4 SEM micrograph of TCLWA

aggregates contained other amorphous, such as Fe_2O_3, CaO, MgO, Na_2O, and K_2O.

In this investigation, the lightweight aggregates were cut into two pieces, and the degree of sintering and pore structure were then determined from the cut profile using a scanning electronic microscope (SEM). Figs. 4, 5 and 6 presented SEM images. As shown in Fig. 1, the powder remained presented on the surface of TCLWA; moreover, the SEM images under 15x and 50x magnification, displayed in Fig. 4, clearly revealled that TCLWA exhibits bloating and lightening. The 200x and 1000x SEM images of TCLWA clearly indicated that the interior of the said lightweight aggregate had undergone an initial period of sintering. In Fig. 1, some powder was presented on the surface of RSLWA, but the amount was much less than that on the surface of TCLWA, as could be easily

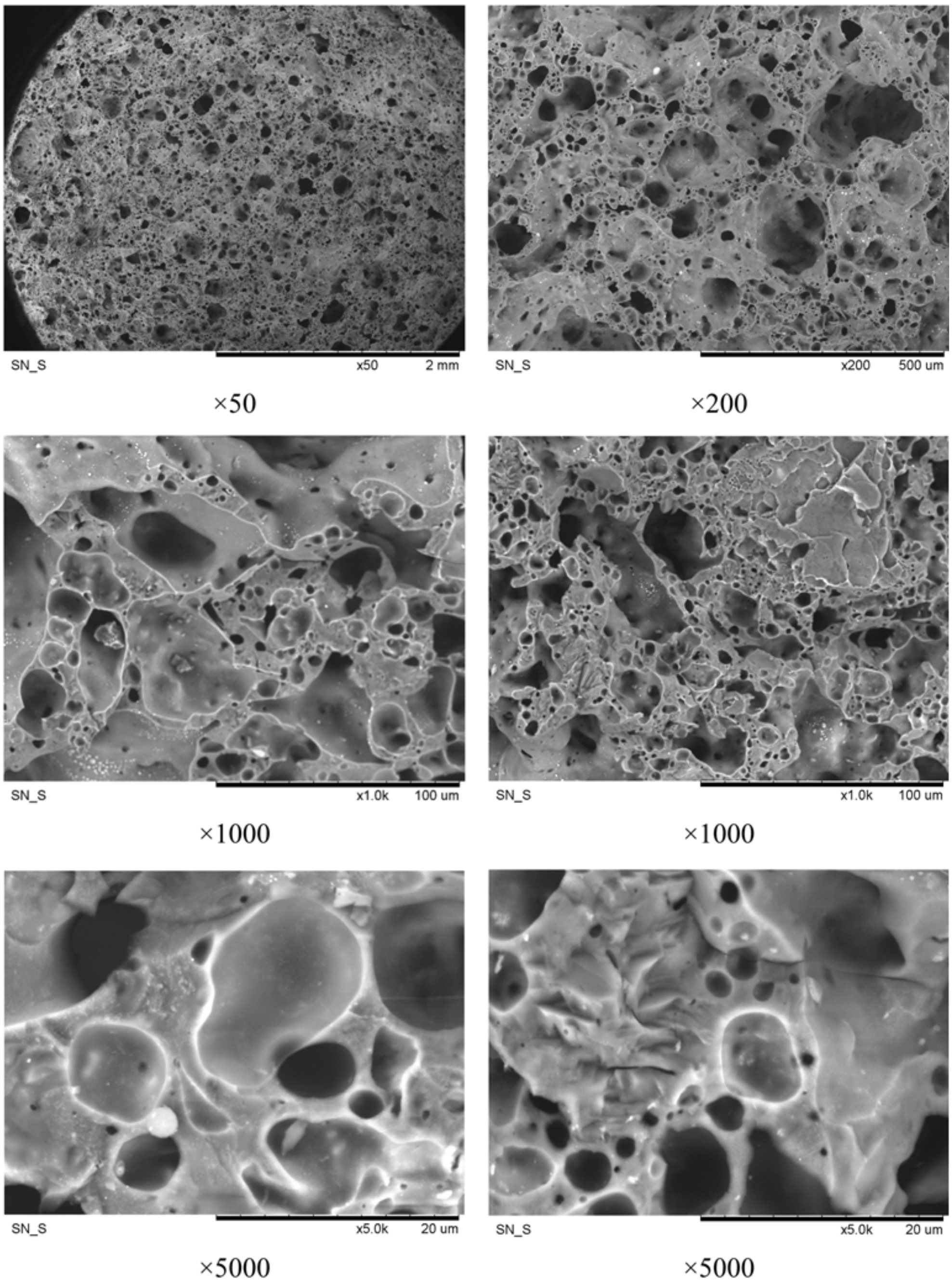


Fig. 5 SEM micrograph of RSLWA

determined by touching the powder with a finger and observing the powder amount passing a #100-sieve (0.15 mm). Fig. 5 presented SEM images at a magnification of 50x. These images clearly showed that RSLWA became bloating and lightened. The 1000x and 5000x SEM images clearly indicated that the interior of RSLWA had undergone some sintering. In Fig. 1, no powder was on

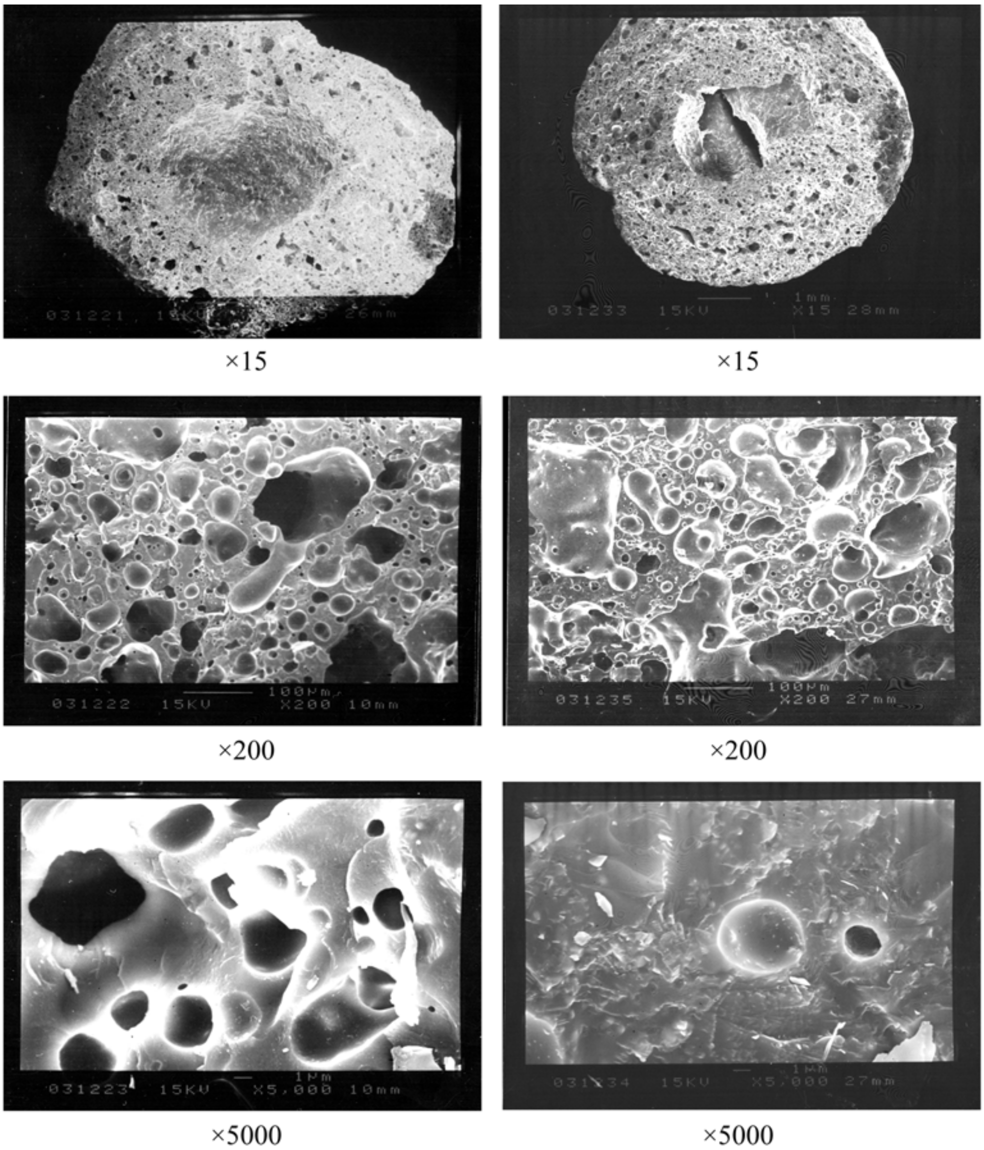


Fig. 6 SEM micrograph of SSLWA

the surface of SSLWA, which appeared noticeably glassy. The 15x SEM images in Fig. 6 indicated that SSLWA exhibited significant bloating and lightening. The 200x and 5000x SEM images showed that the interior of SSLWA had reached sintering.

3.5 Chemical corrosion and alkali-silica reaction of lightweight aggregates

Acid and alkali-resistance and alkali-silica reaction were applied to evaluate the chemical stability of the lightweight aggregates. Table 6 presented the results. Three acid solutions, 5 wt% HNO_3 (0.54M HNO_3, pH = 1.7), 5 wt% HCl (0.51M HCl, pH = 1.7) and 5 wt% CH_3COOH (0.84M CH_3COOH, pH = 3.3), and one alkali solution, 5 wt% NaOH (1.32M NaOH, pH = 12.4), were used in the experiments on the acid and alkali resistance. Fig. 7 showed that the SSLWA had the highest chemical resistance. The weight loss of SSLWA was lower than 0.04%; RSLWA had the next lowest weight loss, which was 0.48-0.64%. TCLWA had the highest weight loss, 1.52-2.75%.

The potential alkali-silica reactivities of the aggregates were evaluated using the ASTM C289-94 Chemical Method Judgment Chart, from amount of dissolved silica (Sc Value) and the amount of depleted alkali (Rc Value). Reactivity was classified as harmful, potentially harmful or harmless, as shown in Fig. 8. In Table 3, the concentrations of dissolved silica (Sc Value) in TCLWA, RSLWA, and SSLWA were 422 mmol/L, 305 mmol/L, and 25 mmol/L individually; the amounts of depleted alkali (Rc Value) were 715 mmol/L, 870 mmol/L and 885 mmol/L, individually. Hence, the Sc/Rc values were 0.59, 0.35, and 0.03 individually: SSLWA had the lowest value; RSLWA had the middle value, and TCLWA had the highest. These results showed that the potential alkali-silica reactivity of each aggregate was below the critical value of 1.0. Therefore, the potential alkali-silica reactivities of these three aggregates were all within the harmless zone, as displayed in Fig. 8. These results proved that the degree of sintering of the artificial aggregate was significantly related to its chemical resistance. A higher degree of sintering of an aggregate was associated with greater resistance to acid and alkali solutions and lower potential alkali-silica reactivity. Furthermore, degree of sintering had a stronger affect than chemical composition on the chemical resistance of the aggregates.

Table 6 Chemical stability of lightweight aggregates (LWA) (weight loss, wt%)

Lightweight aggregates	5wt%-HNO_3 (0.54NHNO_3) (pH=1.69)	5wt%-HCl (0.51N HCl) (pH=1.74)	5wt%-CH_3COOH (0.84N CH_3COOH) (pH=3.33)	5wt%-NaOH (1.32N NaOH) (pH=12.41)
TCLWA	2.75	2.66	1.73	1.52
RSLWA	0.48	0.46	0.65	0.49
SSLWA	0.02	0.02	0.02	0.04

*Tai-Chin lightweight aggregate (TCLWA); Reservoir sediment lightweight aggregate (RSLWA); Sewage sludge lightweight aggregate (SSLWA).

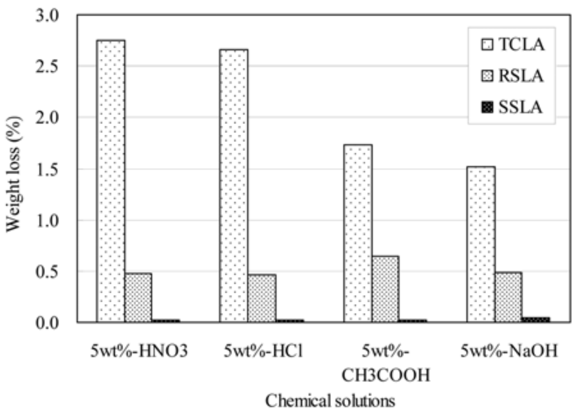


Fig. 7 Chemical stability of lightweight aggregates (LWA)

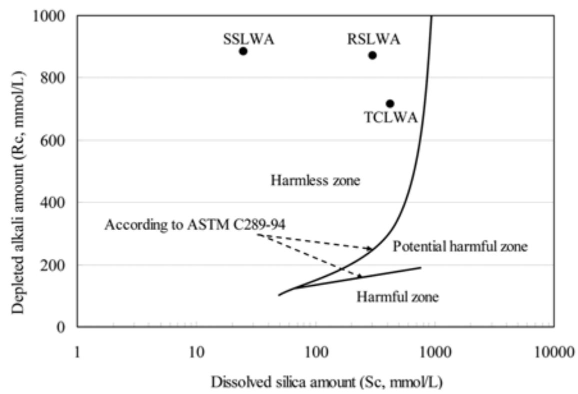


Fig. 8 Potential alkali-silica reactivity of lightweight aggregates (LWA)

Using reservoir sediment or industrial sludge to replace the expansive clay and shale to produce lightweight aggregates is a future tendency. In addition, studies on chemical composition of different types of sludge to produce lightweight aggregates, expansive mechanism of aggregates in sintering, the performance of aggregates, and the durability of concrete with waste-sludge-made aggregates are highly required to achieve the targets of waste reduction and recycling.

4. Conclusions

(1) X-ray fluorescence analysis (XRF) and the work of Riley (Riley 1950) demonstrated that the chemical components of both reservoir sediment and sewage sludge ash were similar to those of expansive clay, but did not meet the requirements of highly expansive clay.

(2) RSLWA and SSLWA had a high point loading strength, low water-absorption, and a much lower leaching concentration of heavy metals than allowed by the standards in Taiwan. RSLWA and SSLWA had high resistance against chemical corrosion, and potential alkali-silica reactivities within the harmless zone; their Sc/Rc values were less than 1.0. That of SSLWA was only 0.1%.

(3) The degree of sintering of lightweight aggregates was closely related to their chemical resistance. Greater sintering on the surface of the aggregate was associated with greater chemical resistance and lower potential alkali-silica reactivity. The effect the degree of sintering on the chemical resistance of the aggregate was stronger than that of its chemical components.

(4) Though aggregates made from waste sludge on potential alkali-silica reactivities were within the harmless zone, attention to the potassium-sodium ratio or alkali content in cement was still required when applying RSLWA and SSLWA in concrete to prevent the occurrence of alkali-silica reaction.

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