

Reuse of light-emitting diode manufacturing process sludge in cement concrete

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The purpose of this study is to investigate the use of the sludge that is a by-product of the light-emitting diode (LED) manufacturing process to partially replace cement in cement concrete. Two water-to-cementitious-material ratios, $w/c = 0.5$ and 0.7 , are used. LED sludge powder replaced 5, 10 and 20 wt% of the cement in sludge-blended cement concrete (SBCC) specimens. These molded cylindrical specimens, 10 cm in diameter and 20 cm in height ($10\text{ cm } \Phi \times 20\text{-cm H}$), are subjected to compressive strength test and other mechanical properties tests. The properties of the LED sludge samples are determined using scanning electron microscopy/energy-dispersive X-ray spectrometry (SEM/EDS) and X-ray diffraction (XRD). The experimental tests reveal that the compressive strengths of SBCC specimens are comparable to those of ordinary Portland cement concrete (OPCC) specimens at all curing ages and that the other mechanical properties are also similar. These findings indicate that LED sludge can be converted into a useful resource, thereby avoiding the problems associated with its disposal and improving its environmental sustainability.

Keywords: Light-emitting diode (LED), Nanoparticles, LED sludge concrete, Compressive strength, Pozzolanic reaction

Light-emitting diode (LED) manufacturing consists of the following steps: polishing the sapphire, evaporating the adhesive and mirror layer, and electroplating the Ni substrate. The manufacturing process generates LED sludge that contains hazardous nanoparticles of SiO_2 , Al_2O_3 and SiC debris. These hazardous ingredients are harmful to the environment and endanger human health if improperly disposed¹⁻³. Moore⁴ noted that in aquatic environments, nanomaterials may harm the cells and tissues of aquatic animals after uptake via cell endocytotic routes and consequently may damage human health. Hazardous nanoparticles also threaten ecological food chains. Moore concluded that precautions must be taken when evaluating the effects of new nanomaterials on health and the environment. Improper disposal of the huge amounts of nanoparticles in sludge, 168,000 tons annually in Taiwan, can be disastrous. When dumping sites are close to reservoirs or underground water sources, these nanoparticles can pollute drinking water and cause lung-related diseases, among others⁴⁻⁶. Because human health is vulnerable to the presence of nanoparticles in sludge, the disposal of potentially hazardous sludge by immobilizing it as a useful

resource warrants investigation. Recent research has studied the addition of nano- SiO_2 materials into cement mortar and concrete to improve the filler effect and to promote the pozzolanic reaction⁷⁻¹⁰.

A similar sludge, a byproduct of chemical mechanical polishing (CMP) used in the integrated-circuit (IC) industries, is composed of hazardous materials such as nanoparticles of SiO_2 , Al_2O_3 , CaF_2 , and unknown organics. Lee *et al.*¹¹⁻¹³ found that CMP sludge can be utilized to replace a portion of the cement in cement mortar. They also found that through the hydration reaction and pozzolanic reaction, the originally hazardous nanoparticles of CMP sludge became part of the hydration compounds after curing for more than 28 days and could not be released into the environment, resulting in no release of heavy metal ions, according to TCLP tests. Because LED sludge is rich in nanoparticles such as SiO_2 , Al_2O_3 and SiC debris, taking advantage of these constituents may transform LED sludge into a useful resource and improve the environmental sustainability of LED manufacturing. To the best of our knowledge, the recycling of LED sludge in concrete has not yet been proposed. In this investigation, we conduct preliminary studies of LED sludge-blended cement concrete (SBCC) for use in construction.

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Materials and Methods

Materials

LED sludge for the investigations described in this paper were obtained from an LED manufacturing facility located at Hsinchu Science Park in Northern Taiwan. The sludge is a light grayish blue in color, with a specific gravity (SG) of 2.50 and a fineness of $12,000 \text{ cm}^2/\text{g}$.

The cement was Type I ordinary Portland cement (OPC) obtained from the Taiwan Cement Company. The SG and fineness of the OPC are 3.15 and $3380 \text{ cm}^2/\text{g}$, respectively. Its physical and chemical properties meet the ASTM C 150 standard.

Fine aggregate (sand) was obtained from the middle region of the Ta-an River in Miao-Li, Taiwan. The sand had a specific gravity of 2.57, and the fineness modulus was 2.69.

Coarse aggregate was also obtained from the middle region of the Ta-an River. Large stones were crushed, and those measuring 10 mm and 20 mm were mixed in a certain ratio and used as coarse aggregate. The coarse aggregate had a specific gravity of 2.59, and its fineness modulus was 6.72.

Methods

Scanning electron microscopy (SEM; JEOL, JSM-6700F, Japan) and energy-dispersive X-ray spectrometry (EDS; INCA_X-sight) coupled with the SEM was used to study the microstructure and chemical composition of the samples.

X-ray diffraction (XRD) analysis was conducted using an X-ray diffractometer (Rigaku, D/Max-2200, Japan) with Cu K_{α1} radiation and a 2θ scanning ranging between 5° and 100° . The XRD scans were run at 0.05° steps per second.

For the pozzolanic activity tests, LED sludge powder of 3 different maximum particle sizes,

namely, smaller than $150 \mu\text{m}$ (#100 sieve), smaller than $75 \mu\text{m}$ (#200 sieve and smaller than $38 \mu\text{m}$ (#400 sieve), were chosen. Mortar test cubes ($5 \times 5 \times 5 \text{ cm}^3$) were cast in standard molds, removed from the molds after 24 h, and unmolded and cured in a saturated calcium hydroxide solution. The ratio of cementitious materials to sand was maintained at 1 to 2.75 by weight, and the ratio of water to cementitious materials was maintained at 0.485. Portland cement was replaced by LED sludge at the rate of 20 wt% by weight of cementitious materials.

Molding and curing of cement concrete specimens: The design proportions of the OPCC and SBCC specimens are given in Table 1. The cylindrical concrete specimens ($10\text{-cm } \Phi \times 20\text{-cm H}$) were molded according to ASTM C 39/C 39M-03 for compressive strength tests. Two types ratios of water to cementitious material, 0.5 and 0.7, were used to prepare the specimens. The LED sludge-blended cement concrete (SBCC) specimens are denoted SBCC (5%), SBCC (10%), SBCC (20%), where the percentage in parentheses is the cement replacement proportion in wt%. All of the specimens were placed in a chamber with programmable temperature and humidity for one day and then unmolded and cured in a saturated lime water at a temperature of $23.0 \pm 1.7^\circ\text{C}$ for 1-90 days. The fresh concrete mixes were subjected to tests for air content, chloride ion content and slump, and hardened concrete specimens were subjected to uniaxial compressive strength and ultrasonic pulse velocity tests.

Fresh cement concrete

Slump tests of the SBCC and OPCC mixes were performed in accordance with ASTM C 143. Air content tests were conducted in accordance with ASTM C 231. The variation in the volume of concrete

Table 1—constituent contents of the ordinary Portland cement concrete (OPCC) and LED sludge-blended cement concrete (SBCC) (design proportion weights per 0.06 m^3)

W/C Specimen	Cement (kg)	LED (kg)	Water (kg)	Fine aggregates	Coarse aggregates
OPCC ^a	16.92	—	11.82	41.52	67.8
W/C=0.5 SBCC ^b (5%)	16.07	0.85	11.82	41.52	67.8
SBCC (10%)	15.23	1.69	11.82	41.52	67.8
SBCC (20%)	13.54	3.38	11.82	41.52	67.8
OPCC	23.76	—	11.82	35.88	67.8
W/C=0.7 SBCC (5%)	22.57	1.19	11.82	35.88	67.8
SBCC (10%)	21.38	2.38	11.82	35.88	67.8
SBCC (20%)	19.01	4.75	11.82	35.88	67.8

^aordinary Portland cement concrete; the numbers in parentheses are the cement replacement percentages by weight.

^bsludge-blended cement concrete; the numbers in parentheses are the cement replacement percentages by weight.

due to pressure changes was measured with a pressure-type air content meter (Forney, USA). The chloride content was measured using a chloride content meter (CL-1B, Rikin, Japan) according to Taiwan National Standard CNS 3090. The initial and final setting times were measured in accordance with ASTM C 403 using a concrete penetration meter (H-4133, Humboldt Mfg. Co.).

Hardened cement concrete

Compressive strength tests were conducted in accordance with ASTM C 39. The average compressive strength of three specimens after each aging was determined. For the mass growth analysis, saturated-surface-dry (SSD) specimens were weighed with a precision electronic balance after curing for 1-91 days.

Measurements of mass growth in specimens

The specimens were weighed with a precision electronic balance (HJ-21KE, Shinko Denshi Corporation, Japan). The maximum reading was 21 kg, and the minimum was 0.1 g. The two groups of OPCC and SBCC specimens each comprised 6 specimens (10 cm diameter and 20 cm height cylindrical concrete) and were cured for 1, 3, 7, 14, 28, 56 and 91 days to measure weight variation during the curing period. Specimens were weighed under SSD conditions. The original weights of the OPCC and SBCC specimens were in the range of 3691.9-3838.2 g; thus, the measurement precision was approximately 1/36000, or 28 ppm, and the micro-growth of mass could be measured.

The porosity of the cured cement mortar was measured using mercury-intrusion porosimetry (MIP) tests. The plain cement mortar and sludge-blended cement mortar specimens preserved in methanol were removed, oven-dried at 100°C for 24 h, cooled in a dessicator, and evacuated. Then, the sample was put into a MIP facility (PoreMaster 60, Quantachrome, USA) and evacuated for porosity analysis.

The SBCC and OPCC specimens were removed from the saturated lime water at different curing ages and dried to SSD conditions for ultrasonic pulse velocity (UPV) measurement. The instrument (PUNDIT, Japan) used to measure pulse velocity generates elastic stress waves based on the piezoelectric effect and measures the path time of the waves through the material tested. The wave velocity can be obtained by dividing the path length by the path time. UPV data for the SBCC and OPCC specimens were collected at different curing periods.

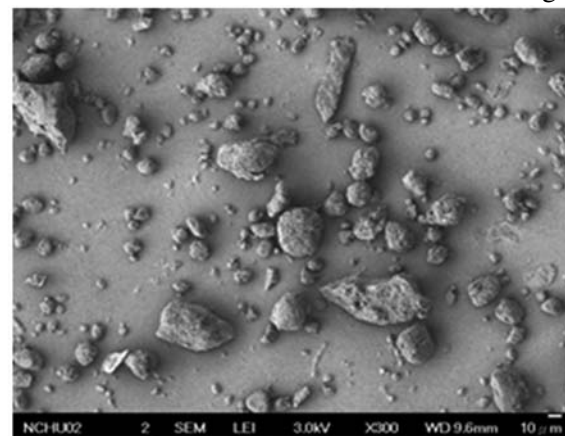
Results and Discussion

The characteristics of typical pozzolanic materials are high silicon content, high specific surface area, and an amorphous nature evident under X-ray diffraction. According to ASTM C 618, when the particle sizes of such materials are less than 38 μm (#400 sieve), they satisfy the requirements of pozzolanic materials¹⁴. Because LED sludge is rich in nanoparticles of SiO_2 , Al_2O_3 and SiC debris, it behaves as a filler and an activator that promotes hydration and thereby improves the microstructure of cured concrete and increases its compressive strength.

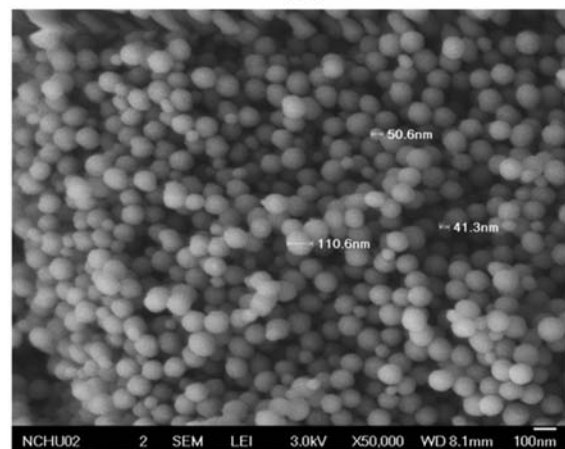
Characteristics of LED sludge

SEM/EDS analysis

SEM studies showed that the LED sludge powder used in this study appears as multiangular shapes, where particles less than 38 μm in size were SiC debris, a very hard and wear-resistant material, as shown in Fig. 1a.



(a)



(b)

Fig. 1 – SEM micrograph of (a) LED sludge under 300 $\times$ magnification and (b) LED sludge under 50000 $\times$ magnification

The other smaller agglomerates were nano-SiO₂ and nano-Al₂O₃ particles, which were the residues of the polishing process. For a better understanding of their composition, FE-SEM analysis was conducted. These studies showed that the LED sludge particles seemed to have more fine material attached to their surfaces, as shown in Fig. 1b. The nanoparticle sizes were 25-110 nm, as shown in Fig. 1b.

The EDS test results shown in Fig. 2 depict large amounts of Si in the LED sludge. Table 2 shows the composition of the LED sludge, which contains a large amount of SiC and SiO₂ and some CaO and Al₂O₃.

XRD analysis

Figure 3 shows the XRD spectra of the LED sludge powder. Comparisons between these spectra with data from JCPDS (Joint Committee on Powder Diffraction Standards) reveal that the principal constituents of LED sludge are SiC and SiO₂. The characteristic of a broad band between 18° and 28° without peaks

depicts the material rich in amorphous Si as a pozzolanic material; the other peaks were SiC and SiO₂.

Pozzolanic activity test

Table 3 shows the strength activity indices of sludge-blended cement mortar (SBCM) specimens for various curing ages. The results showed that at an age of 3 days, the strength activity index of SBCM specimens was 95.3-106.5% of that of the ordinary Portland cement mortar (OPCM) specimens; at an age of 7 days, it was 113.9-122.8%; and at ages of 28-90 days, it was 116.0-124.4%. These results indicate that although the strength activity index of the SBCM specimens was initially less than that of the OPCM specimens, with increasing curing time, the strength activity index of the SBCM specimens increased notably. The strength activity index was also observed to increase with the fineness of the LED sludge powder. This suggests that the LED sludge powder behaves as a very good pozzolanic material.

LED Sludge-blended cement concrete

To save on manpower and costs, we selected LED sludge with a maximum particle size passing the #100 sieve (less than 150 µm). Based on the pozzolanic activity test results, 5, 10 and 20 wt% cement was replaced by LED sludge in the LED concrete specimens.

Air content test

Table 4 shows that the air content increases with an increasing water to cementitious materials (W/C) ratio. For freshly prepared OPCC specimens, the air content was 0.6-0.9%, whereas for freshly prepared SBCC specimens with cement replacements of 5%, 10% and 20%, the air contents were 0.9-1.4%, 0.9-2.9% and 1.6-3.6%, respectively. These results

Table 2—Chemical compositions of cement and LED sludge

Composition (wt%)	Cement	LED sludge
Na ₂ O	—	0.62
MgO	2.71	—
Al ₂ O ₃	5.29	7.18
SiO ₂	21.21	35.95
SO ₃	2.08	0.61
Cl ⁻	—	—
K ₂ O	—	—
CaO	63.71	14.60
Fe ₂ O ₃	3.12	—
SiC	—	40.53

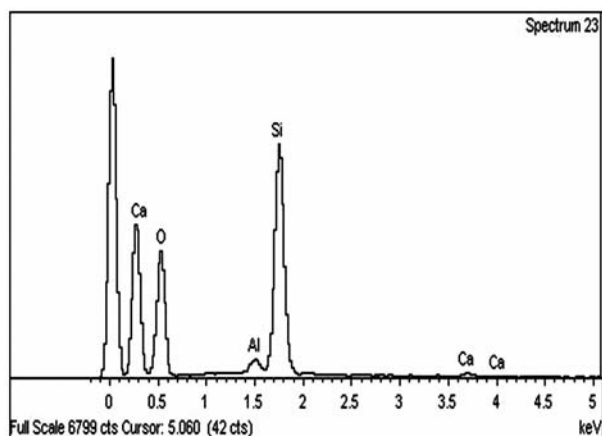


Fig. 2 – EDS spectra of LED sludge

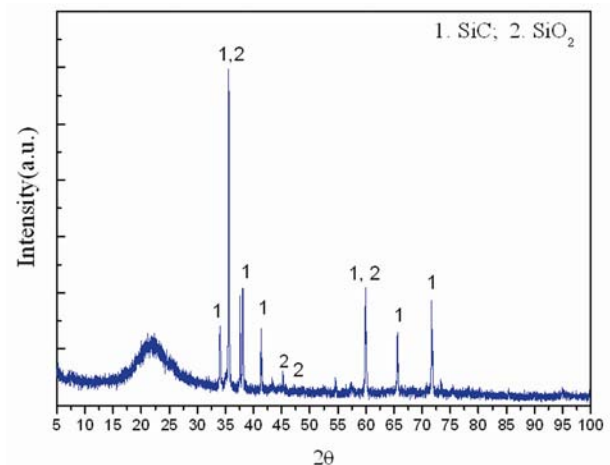


Fig. 3 – XRD diffraction patterns of LED sludge

indicate an increasing trend in two respects, i.e., the air content increases with increasing cement replacement and with increasing water-to-cementitious material ratio.

Chloride content test

According to the Taiwan National Standard CNS 3090 specification, for non-prestressed concrete, the chloride content should not be greater than 0.30 kg/m^3 , while for prestressed concrete, the chloride content shall not be greater than 0.15 kg/m^3 . Table 4 shows that the chloride ion content for the freshly prepared OPCC specimens was $0.011\text{--}0.034 \text{ kg/m}^3$, whereas for the freshly prepared SBCC specimens with cement replacements of 5%, 10% and 20%, the chloride contents were $0.010\text{--}0.033 \text{ kg/m}^3$, $0.009\text{--}0.031 \text{ kg/m}^3$ and $0.008\text{--}0.029 \text{ kg/m}^3$, respectively. The results indicate that the chloride contents were much lower than the specified limit and that the chloride content decreases with increasing cement replacement. Chloride content also decreases with increasing w/c ratio.

Slump test

Table 4 shows that the slump values of the OPCC specimens were 12.0–13.5 cm and that slump increases with an increasing w/c ratio. For the SBCC

specimens with cement replacements of 5%, 10% and 20%, the slump values were 9.0–12.0 cm, 7.0–11.5 cm and 5.0–8.5 cm, respectively. The results revealed that slump decreases with increasing cement replacement percentage. This was especially notable in the case of 20% cement replacement. The LED sludge consists of porous particles that absorb water in the mortar mix and reduce workability. This could be compensated for by adding water or a superplasticizer.

Setting time test

Table 5 shows that the initial and final setting times of the OPCC were 295–340 min and 400–500 min, respectively, and increased with an increasing w/c ratio. The initial and final setting times of SBCC with cement replacements of 5% to 20% were 315–370 min and 430–540 min, respectively. The results revealed that, for any given w/c ratio, the setting times of the specimens increased with increasing cement replacements by LED sludge.

Compressive strength tests

Figures 4 and 5 show the results of the compressive strength tests, along with equations for the trends derived using linear regression analysis. The linear regressions of compressive strength vs. curing ages of the cylindrical concrete specimens had correlation

Table 3—Pozzolanic activity indices of SBCC specimens

Specimens	Curing times (days)			
	3-day	7-day	28-day	90-day
OPCM	31.85 (100.00%)	34.19 (100.00%)	41.32 (100.00%)	49.62 (100.00%)
SBCC (#100) ^a	30.35 (95.29%)	38.94 (113.89%)	48.23 (116.72%)	57.55 (115.98%)
SBCC (#200) ^b	30.87 (96.92%)	40.82 (119.39%)	50.38 (121.93%)	59.28 (119.47%)
SBCC (#400) ^c	33.91 (106.47%)	41.98 (122.78%)	51.00 (123.43%)	61.75 (124.44%)

^aThe particle size of LED sludge smaller than $150 \mu\text{m}$ (#100 sieve)

^bThe particle size of LED sludge smaller than $75 \mu\text{m}$ (#200 sieve)

^cThe particle size of LED sludge smaller than $38 \mu\text{m}$ (#400 sieve)

Table 4—Physical properties of fresh concrete

	(W/C)	OPCC	SBCC (5%)	SBCC (10%)	SBCC (20%)
Air content (%)	0.5	0.6	0.9	0.9	1.6
	0.7	0.9	1.4	2.9	3.6
Chloride content (kg/m^3)	0.5	0.034	0.033	0.031	0.029
	0.7	0.011	0.01	0.009	0.008
Slump (cm)	0.5	12	9	7	5
	0.7	13.5	12	11.5	8.5

coefficient (R^2) values as high as 0.91-0.97. The results are as follows:

(i) $W/C = 0.5$

For the SBCC specimens with cement replacements of 5 and 10 wt% and curing ages of 1-7 days, the compressive strengths of the SBCC specimens are 79.55-95.92% of those of the OPCC specimens. For specimens at curing ages of 14, 28, 56 and 91 days, the compressive strengths of the SBCC specimens were 101.46-101.48%, 100.55-111.59%, 101.23-101.43% and 100.04-102.94%, respectively, of the compressive strengths of the OPCC specimens. For the SBCC specimens with cement replacement of 20 wt% and curing ages of 1-91 days, the compressive strengths were 57.54-86.43% of those of the OPCC. The results reveal similar strengths for the two groups at early curing stages, and at curing ages greater than 14 days, the strengths are higher for the SBCC specimens with cement replacements of 5 and 10 wt% than for the OPCC specimens. The strength evolution reveals the characteristics of the pozzolanic reaction. The results also reveal that the SiO_2 nanoparticles in the LED sludge behave as filler and as an activator promoting hydration, thereby improving the microstructure of

the concrete in SBCC specimens¹⁵⁻¹⁷. The nano- SiO_2 particles can absorb hydrated Ca(OH)_2 crystals, reducing their number and pore size and increasing the density of the binder in the paste matrix¹⁸. The phenomenon of reduced cement with greater strength growth is typical of the pozzolanic reaction. For the SBCC specimens with cement replacement of 20%, the results showed that the strengths are lower than the OPCC specimens at all points in time. The mixing water was limited at $W/C = 0.5$, and it was largely adsorbed by the nanoparticles of the LED sludge, resulting in a serious water shortage in the cement hydration. Without sufficient water, the cement cannot be fully hydrated, and the pozzolanic reaction of the LED sludge is weakened, which is the reason for the lower strengths of the SBCC specimens. These results demonstrate the importance of not using too much LED sludge; otherwise, a superplasticizer must be used to achieve adequate workability.

(ii) $W/C = 0.7$

For the SBCC specimens with cement replacements of 5 and 10 wt% and curing ages of 1-7 days, the compressive strengths of the SBCC specimens were 84.8-112.83% of those of the OPCC specimens. For specimens at curing ages of 14, 28, 56

Table 5—Setting times (in minutes) of ordinary Portland cement concrete (OPCC) and LED sludge-blended cement concrete (SBCC)

Setting time	W/C = 0.45		W/C = 0.55		W/C = 0.65	
	OPCC	SBCC	OPCC	SBCC	OPCC	SBCC
Initial	370	364	375	365	383	368
Final	462	458	474	460	480	464

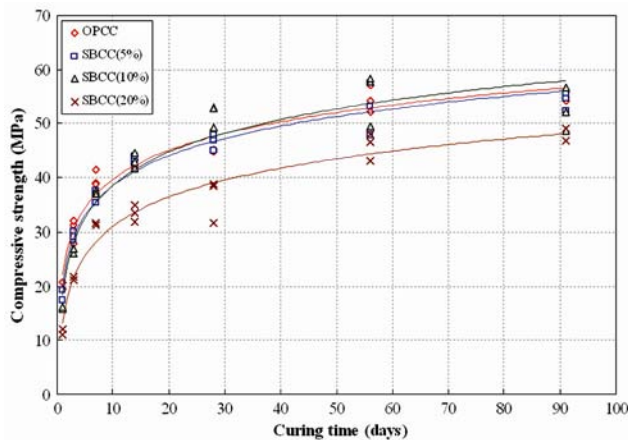


Fig. 4 – Regression analysis of compressive strength of OPCC and SBCC specimens ($w/c = 0.5$) as a function of curing age, 1-91 days

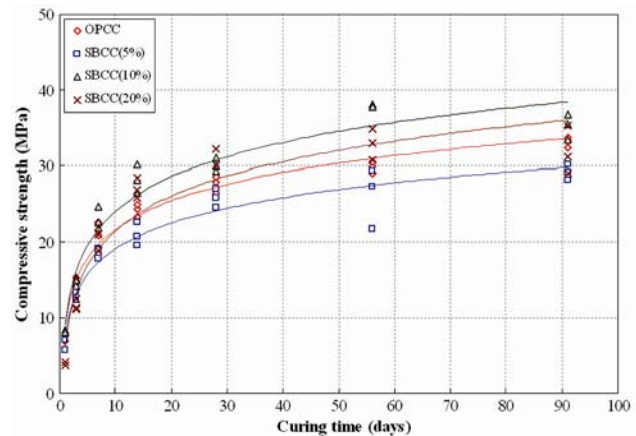


Fig. 5 – Regression analysis of compressive strength of OPCC and SBCC specimens ($w/c = 0.7$) as a function of curing age, 1-91 days

and 91 days, the compressive strengths of the SBCC specimens were 86.27-116.72%, 93.16-109.19%, 95.09-127.36% and 98.03-109.09%, respectively, of the compressive strengths of the OPCC specimens. The results reveal that the strengths of the SBCC specimens are comparable to those of the OPCC specimens at all curing ages. For SBCC specimens with cement replacement of 20 wt% and curing ages of 1-7 days, the compressive strengths of SBCC were 53.47-94.03% of those of the OPCC. For samples at curing ages of 14-91 days, the compressive strengths of the SBCC were 100.40-111.73% of those of OPCC. These test results are in agreement with previous strength activity index tests, as shown in Table 3. For the SBCC specimens with cement replacement of 10 wt%, the compressive strengths were 100.89-112.83% of those of the OPCC specimens at early curing ages of 1-7 days, but at curing ages of 14-90 days, the strengths were 109.19-127.36% of those of OPCC specimens. It is apparent that the pozzolanic reaction in this case is better and that the 10 wt% substitution is the best option within the range of cement substitution percentages considered. In Fig. 6a, the LED sludge cement mortar specimen with a 3-day curing age was observed in the 50,000 $\times$ magnification SEM micrograph. The nano-particles grow whiskery substances, which appears to be the initial phenomenon of pozzolanic reaction. Figure 6b shows the 28-day sample, in which the nano-SiO₂ particles become invisible and may gradually develop into hydration products⁷⁻¹⁰.

The above results prove that the nano-SiO₂ particles in LED sludge have a notably strong activation energy; thus, these nano-particles behave as fillers to fill the micro voids in the cement mortar specimens¹⁵⁻¹⁷. Their packing effect can increase the density of the structure and enhance the compressive strength of the SBCC specimens.

Mass growth analysis

More hydration products fill the voids of concrete specimens as curing time elapses, and thus, the specimen's density and compressive strength increase. Cured specimens were weighed under SSD conditions. The weight of each specimen at an age of 1 day was normalized as 1.0000, and the weights at other ages were normalized to the weight at 1 day of age by dividing the weight for a specific curing age by the weight after 1 day of curing. The compressive strength of each group of specimens at 28 days of age was also normalized to 1.0000 for comparison with

the strengths at other curing ages. The results show that the weights of the specimens increased with curing time for all of the concrete specimens.

(i) $W/C = 0.5$

The 28-day weights of the OPCC and SBCC specimens were 1.0045 and 1.0045–1.0047, respectively, normalized to the 1-day weights, while the 91-day weights were 1.0054 and 1.0053–1.0057, respectively, normalized to the 1-day weights. The weight gains observed arise from the increase in the amount of hydration and pozzolanic reaction products with curing time. The 28- and 91-day weights of the SBCC specimens were similar to and somewhat higher than those of the OPCC specimens. This phenomenon of reduced cement content with increased mass growth is typical of the pozzolanic reaction. Figures 7a and 7b

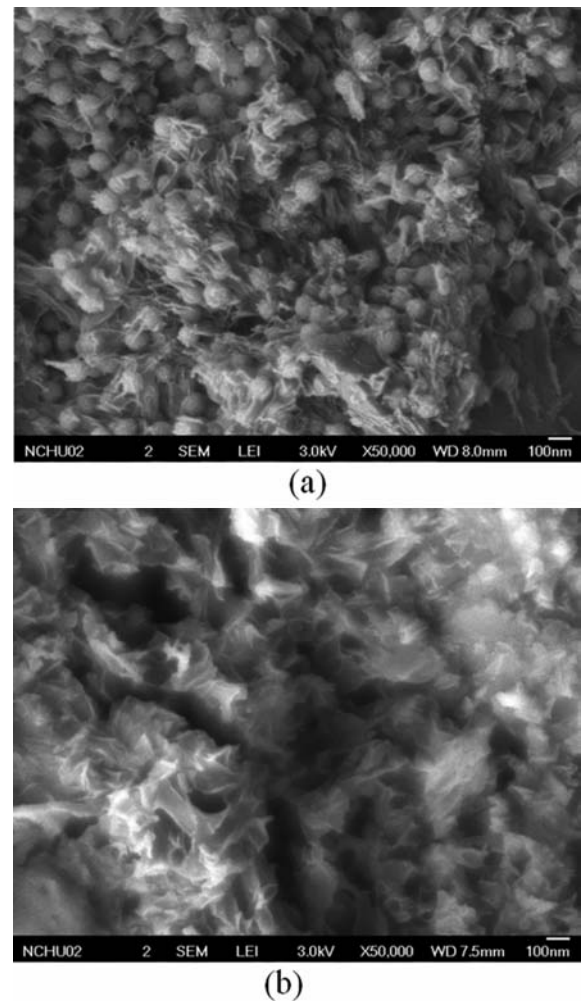


Fig. 6 –SEM micrographs of (a) an SBCC sample at 3-day curing age under 50,000 $\times$ magnification and (b) an SBCC sample at 28-day curing age under 50,000 $\times$ magnification.

show the evolution of compressive strength and normalized weight in OPCC and SBCC (10%) specimens.

(ii) $W/C = 0.7$

The 28-day weights of the concrete specimens were 1.0046-1.0049, normalized to the 1-day weights, while the 91-day weights were 1.0053-1.0063, normalized to the 1-day weights. The results for $W/C = 0.7$ were similar to those described above for $W/C = 0.5$.

The shape of the growth curve of normalized weight is comparable to that of normalized compressive strength, and the trend is consistent with the compressive strength test results. The precise mechanisms remain unknown but most likely are related to the hydration and pozzolanic reactions.

Pore size distribution

Porosity decreased in all of the samples as hydration progressed because cementitious materials

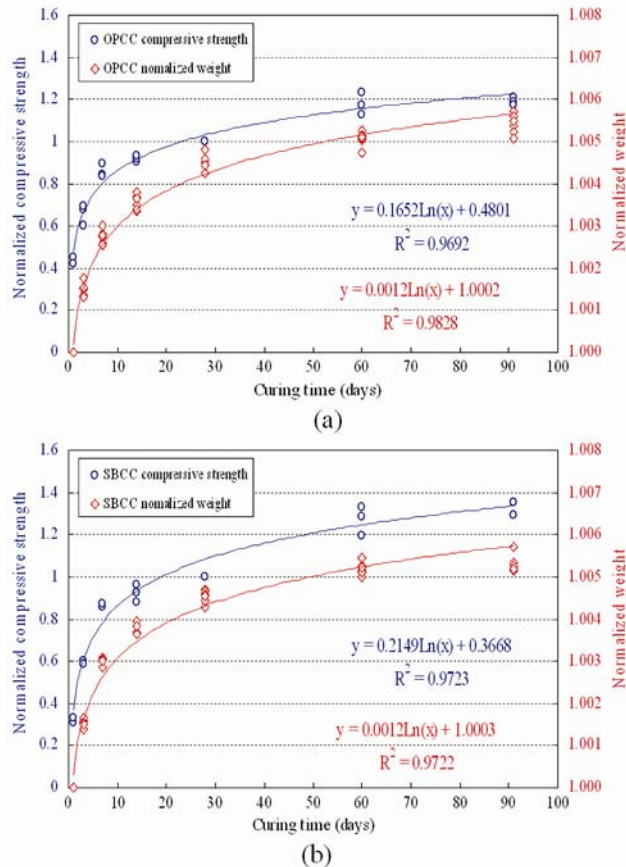


Fig. 7 – Evolution of normalized compressive strength and normalized weight in (a) OPCC cylindrical concrete specimens and (b) SBCC (20%) cylindrical concrete specimens

gradually fill the large pores with hydration products¹⁹, as shown in Fig. 8. The increase in compressive strength with respect to curing time (Figs 4 and 5) was also found to correspond well to the decrease in the total number of pores or capillary pores versus curing time. Although the total pore volume of OPCM and SBCCM were similar, the volume of pores smaller than 50 nm in size was greater for SBCCM than for OPCM specimens, which may be due to the LED sludge contain nanoparticles.

Ultrasonic pulse velocity measurement

The plain concrete OPCC and SBCC specimens were removed at 1, 3, 7, 14, 28, 56, and 91 days of curing age for UPV measurements. The wave velocities in both OPCC and SBCC specimens were found to increase with increasing age. Changes in the wave velocities and normalized strengths of the specimens at different curing ages were plotted as shown in Fig. 9. For the OPCC (control group)

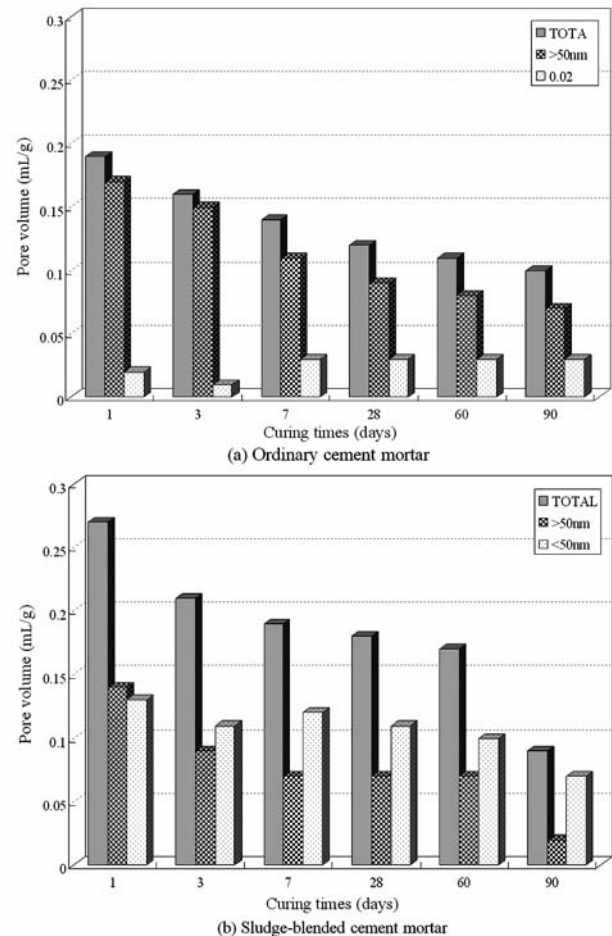


Fig. 8 – Pore size distribution in cured ordinary cement mortar and sludge-cement mortar

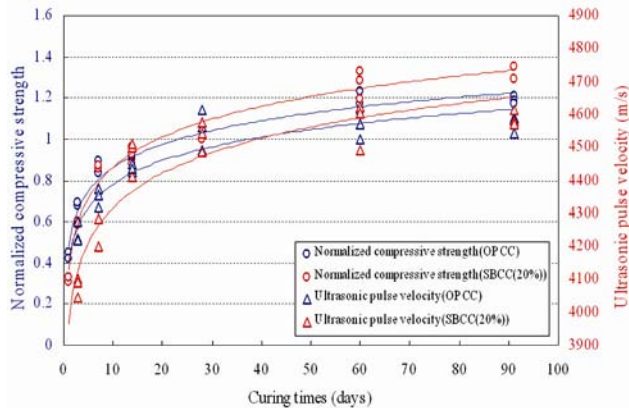


Fig. 9 – Evolution of normalized compressive strength and normalized weight in OPCC and SBCC(20%) cylindrical concrete specimens

specimens, the curve of the wave velocities and that of the normalized strengths were similar and in close proximity. Similar results were obtained for the SBCC (experimental group) specimens. Overall, the OPCC and SBCC results were similar. The compressive strength of concrete is related to its organizational structure density, which is, in turn, closely related to the velocity: the higher the density was, the faster the wave propagation velocity was. The results of MIP tests confirmed the densification phenomenon, i.e., the porosity decreases as the curing time increases. Regression analysis of the compressive strength and UPV measurements yielded linear regression relations with R^2 values in the range of 0.9071-0.9518, indicating a strong correlation between measured compressive strength and measured UPV for both OPCC and SBCC specimens. The physical and chemical properties of SBCC suggest that LED sludge replacement has the potential to benefit the concrete industry.

Conclusions

In this study, cylindrical specimens of LED sludge-blended cement concrete (SBCC, the experimental group) were cast with 5, 10, 20 wt% of cement replaced by LED sludge; cylindrical specimens of the same size were cast of ordinary Portland cement concrete (OPCC, the control group) for comparison. The specimens were subjected to compressive strength tests and related tests and analyses. The following conclusions can be drawn:

- (i) The properties of fresh SBCC and OPCC are similar. In the tests conducted for this study, the air content and chloride content were comparable; the initial setting time of SBCC was somewhat

greater than that of OPCC (20-30 min longer), and the final setting time of SBCC was 30-40 min longer than that of OPCC. The minor differences observed in the properties of the two have little impact on practical applications.

- (ii) The compressive strengths of SBCC(10%) specimens at ages less than 7 days were comparable to those of OPCC specimens, while those at 14-91 days were approximately 2-27% higher than those of OPCC specimens.
- (iii) The weight measurements of concrete specimens revealed that the weights increased with curing age. The weight increase of SBCC was somewhat greater than that of OPCC, which is indicative of the pozzolanic reaction, and coincided with increased compressive strength.
- (iv) The mass growth of both OPCC and SBCC specimens increased over time for all specimens. The shape of the growth curve of normalized weights is comparable to that of compressive strength. This phenomenon is consistent with the compressive strength test results.
- (v) The cement hydration products of the hydration and pozzolanic reaction processes increase the density of the concrete structure. Therefore, the total porosity of the specimens decreased as curing progressed. Compressive strength and UPV increased with increasing curing time. These results offer cross-verification.
- (vi) The experimental tests reveal that the compressive strength of SBCC is comparable to that of ordinary Portland cement concrete (OPCC) at all curing ages and that the other engineering properties of the two materials are similar. Hence, LED sludge can be converted to a useful resource by partially replacing Portland cement in concrete mixes, thereby avoiding problems with disposal and improving the environmental sustainability of the LED manufacturing process.

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Table 1 – constituent contents of the ordinary Portland cement concrete (OPCC) and LED sludge-blended cement concrete (SBCC) (design proportion weights per 0.06 m³)

W/C Specimen	Cement (kg)	LED (kg)	Water (kg)	Fine aggregates	Coarse aggregates	
W/C=0.5	OPCC ^a	16.92	—	11.82	41.52	67.8
	SBCC ^b (5%)	16.07	0.85	11.82	41.52	67.8
	SBCC (10%)	15.23	1.69	11.82	41.52	67.8
	SBCC (20%)	13.54	3.38	11.82	41.52	67.8
W/C=0.7	OPCC	23.76	—	11.82	35.88	67.8
	SBCC (5%)	22.57	1.19	11.82	35.88	67.8
	SBCC (10%)	21.38	2.38	11.82	35.88	67.8
	SBCC (20%)	19.01	4.75	11.82	35.88	67.8

^aordinary Portland cement concrete; the numbers in parentheses are the cement replacement percentages by weight.^bsludge-blended cement concrete; the numbers in parentheses are the cement replacement percentages by weight.

Table 2 – Chemical compositions of cement and LED sludge

Composition (wt%)	Cement	LED sludge
Na ₂ O	—	0.62
MgO	2.71	—
Al ₂ O ₃	5.29	7.18
SiO ₂	21.21	35.95
SO ₃	2.08	0.61
Cl [−]	—	—
K ₂ O	—	—
CaO	63.71	14.60
Fe ₂ O ₃	3.12	—
SiC	—	40.53

Table 3 – Pozzolanic activity indices of SBCM specimens

Specimens	Curing times (days)			
	3-day	7-day	28-day	90-day
OPCM	31.85 (100.00%)	34.19 (100.00%)	41.32 (100.00%)	49.62 (100.00%)
SBCM (#100) ^a	30.35 (95.29%)	38.94 (113.89%)	48.23 (116.72%)	57.55 (115.98%)
SBCM (#200) ^b	30.87 (96.92%)	40.82 (119.39%)	50.38 (121.93%)	59.28 (119.47%)
SBCM (#400) ^c	33.91	41.98	51.00	61.75

	(106.47%)	(122.78%)	(123.43%)	(124.44%)
^a The particle size of LED sludge smaller than 150 μm (#100 sieve)				
^b The particle size of LED sludge smaller than 75 μm (#200 sieve)				
^c The particle size of LED sludge smaller than 38 μm (#400 sieve)				

Table 4 – Physical properties of fresh concrete

	(W/C)	OPCC	SBCC (5%)	SBCC (10%)	SBCC (20%)
Air content (%)	0.5	0.6	0.9	0.9	1.6
	0.7	0.9	1.4	2.9	3.6
Chloride content (kg/m^3)	0.5	0.034	0.033	0.031	0.029
	0.7	0.011	0.01	0.009	0.008
Slump (cm)	0.5	12	9	7	5
	0.7	13.5	12	11.5	8.5

Table 5 – Setting times (in minutes) of ordinary Portland cement concrete (OPCC) and LED sludge-blended cement concrete (SBCC)

Setting time	W/C = 0.45		W/C = 0.55		W/C = 0.65	
	OPCC	SBCC	OPCC	SBCC	OPCC	SBCC
Initial	370	364	375	365	383	368
Final	462	458	474	460	480	464

Figure captions

Fig. 1 – SEM micrograph of (a) LED sludge under 300 $\times$ magnification and (b) LED sludge under 50000 $\times$ magnification

Fig. 2 – EDS spectra of LED sludge

Fig. 3 – XRD diffraction patterns of LED sludge

Fig. 4 – Regression analysis of compressive strength of OPCC and SBCC specimens ($w/c=0.5$) as a function of curing age, 1-91 days

Fig. 5 – Regression analysis of compressive strength of OPCC and SBCC specimens ($w/c=0.7$) as a function of curing age, 1-91 days

Fig. 6 –SEM micrographs of (a) an SBCC sample at 3-day curing age under 50,000 $\times$ magnification and (b) an SBCC sample at 28-day curing age under 50,000 $\times$ magnification.

Fig. 7 – Evolution of normalized compressive strength and normalized weight in (a) OPCC cylindrical concrete specimens and (b) SBCC (20%) cylindrical concrete specimens

Fig. 8 – Pore size distribution in cured ordinary cement mortar and sludge-cement mortar

Fig. 9 – Evolution of normalized compressive strength and normalized weight in OPCC and SBCC(20%) cylindrical concrete specimens