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Characterization of a mortar made with cement and sludge from the light-emitting diode manufacturing process

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HIGHLIGHTS

- The proposed method provides a new application of reused LED sludge as a resource.
- LED sludge powder contains nano-SiO_2 particles and is a pozzolanic material.
- LED sludge enhances the mechanical properties of cement mortar.
- Sludge-blended cement mortar has superior strength relative to plain cement mortar.

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ABSTRACT

This study uses sludge, which is a by-product of the light-emitting diode (LED) manufacturing process and an industry waste (called LED sludge), to replace some weight percentage of cement. The LED sludge powder was used to replace 5, 10, 20, and 30 wt.% of the cement, and the ($5 \times 5 \times 5$-cm^3) sludge-blended cement mortar (SBCM) specimens were molded for a compressive strength test and other engineering property tests. The properties of the LED sludge sample were checked using a scanning electron microscope/energy dispersive X-ray spectrometry (SEM/EDS) analysis, an X-ray diffraction (XRD) analysis, and a solid-state nuclear magnetic resonance spectroscopy (NMR) analysis. The compressive strength test shows that the SBCM specimens have comparable compressive strength relative to ordinary Portland cement mortar (OPCM) specimens in the early curing age of 1–7 days, and 103–115% of the compressive strength of OPCM specimens after curing for 14–90 days. The test results reveal that the nano-particles promoted the pozzolanic reaction and enhanced the strength evolution. LED sludge can be converted into a useful resource by exempting the difficulty of disposal problems and appealing to the environmental sustainability.

1. Introduction

In this decade, waste resources for construction have become increasingly indispensable in developing feasible methods to increase recycling incentives and avoid pollution. In recent years, both public and private organizations in Taiwan have strongly advocated the use of light-emitting diode (LED) lighting to reduce the electricity consumption and the carbon emission. LED sludge, which is generated from the LED manufacturing process, contains hazardous nano-particles of SiO_2, Al_2O_3 and SiC debris. These hazardous ingredients are harmful to the environment and endanger human health if improperly disposed of [1–3]. When the dumping sites are near reservoirs or underground water sources, these nano-particles can pollute drinking water and cause lung-related diseases [4–6]. Such hazardous nanoparticles may also harm the cells and tissues of aquatic animals and plants and consequently threaten ecological food chains [4]. Moore concluded that precautions must be taken when evaluating the effects of new nanomaterials on health and the environment because human health is vulnerable to the presence of nano-particles in the sludge. Improper disposal of the huge amount of nano-particles in sludge, which is 168,000 tons annually in Taiwan, can be disastrous. Therefore, efforts to immobilize the potentially hazardous sludge to make it a useful resource are warranted. The addition of nano-SiO_2 materials into cement mortar and concrete to improve the filler effect and to promote the pozzolanic reaction had been studied [7–10]. Lee et al. [11–13] replaced a portion of the cement with a chemical mechanical polishing (CMP) sludge in cement mortar, a byproduct composed of hazardous materials such as the nanoparticles of SiO_2, Al_2O_3, CaF_2 and unknown organics of the integrated-circuit (IC) industries. They found that through the

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hydration reaction and the pozzolanic reaction, the originally hazardous nano-particles of CMP sludge became parts of the hydration compounds after curing for more than 28 days and could not be released into the environment, which results in no release of heavy metal ions, according to the 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 may improve the environmental sustainability of LED manufacturing. Therefore, this paper attempts to conduct preliminary studies of LED sludge-blended cement mortar (SBCM) for use in construction.

2. Materials and methods

2.1. Materials

The cement was Type I ordinary Portland cement (OPC), which was obtained from the Taiwan Cement Company. The S.G. and the fineness of OPC are 3.15 and 3380 cm^2/g, respectively. The reference sand was Ottawa standard sand that adhered to ASTM C778 [14] specifications. This sand has a specific gravity of 2.63.

The investigated LED sludge in this paper was obtained from an LED manufacturing facility at Hsinchu Science Park in northern Taiwan. The sludge has a light grayish blue color, a specific gravity (S.G.) of 2.50 and a fineness of 12,000 cm^2/g. The main content of the LED sludge is SiC (Silicon carbide, chemical formula SiC). SiC is a synthetic crystal with a density of 3.1 g/cm^3 and excellent physical and chemical properties.

2.2. SEM/EDS analysis

The only required pretreatment was drying. To make the cured cement mortar specimen, the cut samples were first oven-dried at 100 °C for 24 h and then cooled in a desiccator. Then, the samples were secured in a sample holder and coated with a thin platinum layer, which was no thicker than 20 nm to prevent interference with the EDS analysis). Scanning electron microscopy (SEM; JEOL, JSM-6700F, Japan) and energy-dispersive X-ray spectrometry (EDS; INCAX-sight) coupled with the SEM were used to study the microstructure and the chemical composition of the samples.

2.3. XRD analysis

Based on Bragg's Law, the X-ray diffraction (XRD) analysis was conducted using an X-ray diffractometer (Rigaku, D/Max-2200, Japan) with Cu Kα1 radiation and a 2θ scanning range of 5–100°. The XRD scans were performed at a 0.05° interval per second.

2.4. Molding the cement mortar specimens and curing

The mortar cubes ($5 \times 5 \times 5$-cm^3) were molded according to ASTM C109 [15] to test the compressive strength. A water-to-cement ratio (W/C) of 0.485 and a sand-to-cement ratio of 2.75 were used. The acronym OPCM is used for the ordinary Portland cement mortar, and SBCM is used for the sludge-blended cement mortar. The percentage in the parentheses following the SBCM notation denotes the weight percentage of cement that was replaced by the sludge powder, e.g., SBCM(5%), SBCM(10%), SBCM(20%) and SBCM(30%). The design proportions of the OPCM and SBCM specimens are listed in Table 1. The mortar test cubes were cast in standard molds, which were placed in a chamber with programmable temperature and humidity for 1 day. Then, the specimens were removed from the molds and cured in a saturated lime water, which was maintained at 23.0 ± 1.7 °C, for 1, 3, 7, 14, 28, 60 and 90 days. Afterwards, the compressive strength of these specimens was examined according to ASTM C109.

Table 1
The design proportions of the OPCM and SBCM specimens (for nine specimens).

	LED sludge powder particle <#100 (150 μm)				
	OPCM	SBCM(5%)	SBCM(10%)	SBCM(20%)	SBCM(30%)
Cement (g)	740	703	666	592	518
Sand (g)	2035	2035	2035	2035	2035
LED sludge (g)	0	37	74	148	222
Water (g)	359	359	359	359	359

2.5. Fresh cement mortar

The fluidity test was performed according to ASTM C1437 [16]. The setting time test was performed according to ASTM C187 [17] to obtain the required water quantity for the cement. The VICAT needle method was used for the cement paste setting time test according to ASTM C191 [18] to obtain the initial and final setting times of the ordinary Portland cement paste (OPCP) and sludge-blended cement pastes (SBCP).

2.6. Pozzolanic activity index

For the pozzolanic activity tests, the LED sludge powders of three different maximum particle sizes were chosen: smaller than 150 μm (<sieve #100), smaller than 75 μm (<sieve #200) and smaller than 38 μm (<sieve #400). The mortar test cubes ($5 \times 5 \times 5$-cm^3) were molded according to ASTM C109. Portland cement was replaced by LED sludge at the ratio of 20 wt.% of the cementitious materials. We obtained the pozzolanic activity index according to ASTM C311-013 [19].

2.7. Compressive strength test

The compressive strength tests for the cured OPCM and SBCM specimens were performed according to the ASTM C109 standard. The average compressive strengths of three specimens of each age were used as the compressive strength for the specimens of that age.

2.8. Measurements of mass growth in specimens

The specimens were weighed using a precision electronic balance (AJ-6200E, SHINKO Corporation, Japan). The maximum reading was 6200 g, and the minimum was 0.1 mg. One group of OPCM and four groups of SBCM specimens, with each group comprising six specimens ($5 \times 5 \times 5$-cm^3), were cured for 1, 3, 7, 14, 28, 60 and 90 days to measure the weight variations during the curing period. The specimens were weighed in a saturated-surface-dry (SSD) condition. The minimum of the original weights for the OPCM and SBCM specimens were in the range of 263.65–282.76 g. Thus, the measurement precision is approximately 1/26,360, and the micro-growth of the mass can be measured. More hydration products filled the voids of the cement mortar specimens as the curing time elapses; thus, the specimen density and the compressive strength increased. The weight of each 1-day-old specimen was normalized to 1.0000 for comparison with the weights at other curing ages.

2.9. NMR analysis

In this study, for the solid-state nuclear magnetic resonance (NMR) spectroscopy, a German spectrometer (BRUKER AVANCE III 400 NMR SPECTROMETER) and a solid-state NMR spectroscopy superconductor (Spectrometer) were used. The superconducting magnetic field was 9.4 Tesla, the hydrogen nuclear resonance frequency was 400 MHz, and the spectrum range of records was from 178.1555 to −345.9393 ppm. Computer software calculated the signal peak integral intensity under the curve (i.e., the signal area that was surrounded by the baseline).

3. Results and discussion

3.1. SEM/EDS observation and analysis of the LED sludge

The LED sludge powder micro samples were analyzed using SEM. The scanning microscopic images show that the specimens have irregular angular particles and a size of approximately 1–20 μm, as shown in Fig. 1(a). The EDS analysis of the sample showed the spectra of Si, Ca, Al, and O, as shown in Fig. 1(b). The oxides obtained using the EDS analysis were SiO_2, CaO, SiC, Al_2O_3 and other ingredients, as shown in Table 2. At 300 times magnification, the LED sludge was found on the surface of SiC particles, as observed in Fig. 2(a). These finer particle agglomerations were then observed at 50,000 times magnification. Fig. 2(b) shows that the aforementioned tiny particles are composed of spherical aggregates of nano-SiO_2 particles, whose size is approximately 40–110 nm.

3.2. XRD analysis of the LED sludge

Fig. 3 shows the XRD spectra of the LED sludge powder. Comparisons between these spectra with the data from JCPDS (Joint Committee on Powder Diffraction Standards) reveal that the