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Engineering properties of controlled low-strength materials containing waste oyster shells



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HIGHLIGHTS

- We provide the feasibility of mixing WOS with recycled materials.
- The workability of the WOS CLSM was met the requirements of ASTM D6103.
- The early-stage compressive strength of WOS CLSM is achieves ASTM standard requirement.
- The optimal WOS replacement of 5% is suggested for concerning of construction.
- The WOS is a feasible material for CLSM.

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ABSTRACT

To evaluate the practical application of waste oyster shells (WOS) as controlled low-strength materials (CLSM), using a reference sample and four fine aggregate replacement 5%, 10%, 15% and 20% WOS sand, and the cement was replaced by 20% fly ash of the materials were tested. The hardened properties and the durability are tested and other various engineering properties are investigated. The experimental results demonstrate that there was no significant reduction in the compressive strength up to 20% of dosages of WOS sand instead of sand, and a proper amount of fly ash material and WOS sand for the replacement of the fine aggregate in cement mortar fills material pores, reduces the absorption rate. WOS sand can be resources of pure calcareous materials and effective in replacement of sand, indicating appropriate application of oyster shells, it is feasible to use in CLSM.

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1. Introduction

Over the past decade, more and more countries have begun to use controlled low-strength materials (CLSM). CLSM are not structural materials, so they cannot be called concrete. They are cementitious materials, mainly used as compacted fill materials. CLSM are also known as controlled low-density fills, flowable mortars, flowable fills and lean mix backfills [1]. A CLSM is a highly flowable material, usually composed of water, cement, fine aggregate, or fly ash or other byproduct materials. The simplest form is created by mixing sand with cement and water to make slurry and to add water accordingly to adjust its flowability. The American Concrete Institute ACI 229 R-99 [2] defines a CLSM as a self-compacting, flowable, durable and strong cementitious material mainly used to replace conventional backfilling soil and structural fillings; the compressive strength of the material is 8.3 MPa or lower. Compared with a conventional backfill, the advantages of a CLSM are no vibration, less field manpower, easy distribution in complex sites, no settlement, quickness, duration, flexibility, and the ability to be combined with non-conventional field materials [3]. To date, CLSM have been extensively used in channel backfill and road repair engineering in Taiwan and abroad.

The application of aggregates of CLSM faces certain limitations. Because governments are promoting public policies of sustainable development and green building, there have been numerous studies on recycled aggregates in recent years. The ACI 229 also suggests that any recycled granular material can be used as a substitute aggregate for CLSM as long as it is tested before it is used. Non-conventional materials such as boiler slag, recovered glass, cement kiln dust, incinerator bottom ash, chip ballast, crushed scrap-tire rubber particles, fluidized bed combustion ash, reclaimed concrete aggregate and other similar industrial byproducts have been used in CLSM as substitute fine aggregates





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throughout the past decade [4–9]. Chang replaced natural aggregate by recovered asphalt concrete to prepare a CLSM [10]. Sheen used oxidizing slag to replace fine aggregate and replaced cementitious material by reducing slag to investigate the effect on the strength of CLSM [11]. Razak et al. preliminarily studied the performance of CLSM with incinerator bottom ash mixed cement [12]. Sasha et al. used reclaimed concrete as coarse and fine aggregates and fly ash and slag to make CLSM without water [13]. Yao and Sun used duns and fly ash to make a new type of silicon–aluminumbased backfill material and used different temperatures to excite the pozzolanic reaction to study the backfill material systematically [14]. These studies indicate that recycled aggregates can be applied to CLSM after mix-design experiments.

As an island country, aquaculture is one of the key industries in Taiwan. The southwestern coastal area mainly cultivates ovsters. According to the statistical data of the Fisheries Agency, the ovster vield was about 300.000 tons over the last five years [15], and the quantity of WOS was very considerable. WOS have little utility value; thus, they are randomly stacked, causing problems such as mosquitoes and environmental pollution [16]. If WOS can be recycled properly, the sources of pollution in the living environment can be reduced. In some countries, sand dug from the seabed is used in concrete without any treatment (e.g., screening); thus, the concrete contains high seashell content [17-20]. Most studies on WOS reutilization aim at biochemical technology, water-quality purification, and soil improvement and seldom on construction materials. Therefore, this study simulated the feasibility assessment of applying WOS sand to CLSM using recycled aquaculture waste

2. Experimental program

2.1. Materials

Ordinary Type I Portland cement conforming to ASTM C150 [21] Type I Portland cement standards was used in this investigation. The fly ash was Class F fly ash produced by a thermal power plant, the fineness + 325 meshes is 27.4%, satisfy the standard requirements of max 34%. The Activity Index on Day 28 is 76.4% satisfy the standard requirements of min 0.8%, and the properties conformed to Class F fly ash produced by a coal-fire power plant compliant with ASTM C618 [22]. The chemical admixture was calcium chloride of ASTM C494 [23] type C, and its specific gravity at room temperature was 1.24. The aggregate sused conformed to the ASTM C33 [24] concrete aggregate specification. The fine aggregate particle-size distribution curve is shown in Fig. 1. The oyster shells were crushed and sieved through a #4 sieve to obtain WOS sand and approach the particle size of natural fine aggregates. The aggregates were processed into a saturated surface dry state before preparing the CLSM. The aggregate physical properties and chemical composition of the WOS sand are shown in Table 1 and Table 2.

2.2. Mixture proportions and testing methods

Because there is no standard mix-design method for CLSM, this study reviewed the relevant literature and used the equal-weight-replacement for WOS sand to design the mix proportion of CLSM. It aimed to replace the partial normal-weight natural sand (0%, 5%, 10%, 15%, 20%) with WOS sand. It adopted a water-binder ratio



Fig. 1. Particle size distribution of WOS and sand materials.

Table 1

Aggregate and WOS sand physical properties.

Properties	Coarse aggregate	Sand	WOS sand
Specific gravity	2.65	2.61	2.1
Maximum size (mm)	1.2	_	-
Fineness modulus (FM)	-	3.02	2.0

Table	2
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Chemical compositions of Portland cement type I and WOS.

Chemical compositions (%)	Cement	WOS
SiO ₂	20.18	13.28
Al ₂ O ₃	4.57	-
Fe ₂ O ₃	3.07	-
CaO	63.34	77.81
MgO	3.21	-
SO ₃	3.00	1.09
K ₂ O	0.69	0.51
Na ₂ O	0.21	-
Cl	-	2.92

(W/B) of 1.3 and replaced 20% cementitious material with fly ash to enhance the workability to prepare and pour waste oyster shell CLSM specimens. The mix-design results are shown in Table 3. The CLSM mixtures featured 3% accelerating agent to accelerate the setting of the cement and the early development of materials strength, tested according to standard specifications at the ages of 1, 7, 28, 56 and 91 days.

For slump, slump flow and tube flow, this study aimed to determine the optimal replacement of WOS sand in CLSM. According to ASTM C143 [25] and ASTM D6103 [26], the slump and slump flow test values should reach above 20 cm and 40 cm, respectively, and the tube flow test value should reach above 15 cm. For penetration, construction can proceed if the penetration of CLSM is 2.74 MPa, according to the ASTM C403 [27] penetration resistance test. For ball-drop, according to ASTM D6024 [28], the ball-drop test is a destructive test used to determine workability. The diameter of the indentation formed during the drop test must be smaller than 7.6 cm for a mix proportion to have a sufficient bearing capacity. For compressive strength, according to ASTM D4832 [29], the most important index for the bearing capacity of CLSM is compressive strength. The ACI specifies that the 28 day uniaxial compressive strength of CLSM should not exceed 8.23 MPa. For ultrasonic pulse velocity, according to ASTM C597 [30], as a form non-destructive testing, the ultrasonic pulsing can be used to test the quality of concrete and detect the depths and widths of cracks so as to determine the density variation inside a CLSM. For absorption rate, according to ASTM C642 [31], porosity negatively affects strength and durability. High porosity generally leads to low compressive strength and durability. To evaluate the ability of external moisture to enter the specimen, the pore-size distribution in the CLSM specimen was identified by the absorption rate. For sulfate attack, according to ASTM C1012 [32], Air curing was carried out for the prepared CLSM specimen with waste WOS sand. The specimen was dried in an oven at 100 ± 5 °C for 24 h at the age of 28 days and then soaked saturated sodium sulfate solution was made from 350 g of the anhydrous salt (Na₂SO₄) per liter of water at 22 °C for 24 h. The specimen was then weighed. The procedures were repeated to evaluate the impact of sulfate attack on the CLSM with WOS sand.

3. Results and discussion

3.1. Slump, slump flow and tube flow

As shown in Fig. 2. When the *W*/*B* was 1.3, the slump/flow of the CLSM contained 20% fly ash, the slump value was 26.5–21.5 cm, the slump flow value was 60–41 cm, and the tube flow value was 21–15 cm, all of which conformed to the performance requirements for general flowability grade for CLSM. When the WOS sand replacement was 20%, the tube flow reached the minimum requirement of 15 cm specified for CLSM, but different replacements still showed good workability. The WOS sand replacement did not improve the slump flow. The slump decreased gradually, the slump flow decreased by a few degrees and the flowability declined as the WOS sand replacement increased, making

Table 3				
Mixture	proportions	of CLSM	(unit:	kg/m ³).

NO.	Binding materials		Coarse aggregate	Fine aggregate		Water	W/B	Admixture (Type C)
	Cement	Fly ash		Sand	WOS			
00 05 010 015 020	160	40	400	1285 1220.75 1156.5 1092.25 1028	- 64.25 128.5 192.75 257	254	1.3	6



Fig. 2. Effect of WOS content on flowability.

construction difficult. Similar to the results of reference [33], when the material replacement is 5%, the flowability higher than the control group flowability. However, when the replacement is increased to 10%, 20% or 30%, the flowability decreases. The following are the causes of these phenomena:

- (1) The workability of the CLSM declines as the WOS sand replacement increases. This is due to the high water absorption of WOS sand. The replacement of WOS sand increases and adsorbs the mixing water and the flowability declines as the mixing water is lost, making the mortar viscous when mixing the CLSM. For the same mixing water, more replacement means that the workability is more likely to be lost.
- (2) In addition, the fineness modulus (FM) of WOS sand is 2.0, which is lower than the 3.02 FM of fine aggregates. When the particles are smaller, the specific surface area is larger. The WOS sand replacement increases the amount of surface-adsorbed water such that the CLSM flowability is reduced, and self-compaction is lost. The increase in the WOS sand replacement ratio affects the workability of the CLSM significantly.

3.2. Penetration

The penetration results of this study are shown in Fig. 3. When the WOS sand replacement increased from 0% to 5%, the penetration became 2.74 MPa at 210 to 260 min, conforming to the required 3–5 h for early-strength CLSM. When the replacement increased to 10%, 15% and 20%, the penetration was 2.74 MPa at 330 to 465 min, conforming to the required 12–36 h for general CLSM.

In this study, the time required to reach a penetration of 2.74 MPa increased with the WOS sand replacement. The amount of WOS sand added affected the time of penetration. This was primarily due to the large pores of the WOS sand, which led to high water absorption, postponing hydration such that the initial setting time was extended. In addition, because the oysters are cultured in seawater, the WOS sand carries an excess amount of organic substances that result in slow setting [18].



Fig. 3. Effect of WOS content on ball drop and reached penetration 2.74 MPa of the time.

3.3. Ball-drop

The drop test was carried out in 1 day, and the results are shown in Fig. 3. The indentation diameter was 4.9–5.9 cm, which is less than 7.6 cm, indicating that the mix proportion has a sufficient bearing capacity for further construction. The drop test value is only a preliminary index of the bearing capacity of a material. Subsequent tests should be conducted to accurately determine the bearing capacity of a material. When the WOS sand replacement was 5%, the diameter of the drop indentation was 4.9 cm, which is smaller than that produced on the other replacements. Furthermore, the compressive strength was higher: the 1 day compressive strength was 1.67 MPa. When the WOS sand replacement exceeded 5%, the indentation diameter increased gradually with the replacement ratio.

3.4. Compressive strength

The compressive strength of the CLSM in this study is shown in Fig. 4. The mix proportion increased with age, and the compressive strength increased accordingly. The 1 day compressive strength was 0.96–1.67 MPa, and the 1 day compressive strength of various mix proportions was higher than 0.69 MPa. The compressive



Fig. 4. Effect of WOS content on compressive strength.

strength was 4.21-6.63 MPa at the age of 28 days, conforming to the strength requirement for digs. As the age increased, the cement hydration was gradually completed, and the development of strength became smoother.

The compressive strength developed best when the WOS sand replacement was 5%-indeed, better than the control group. This result is identical to that reported by Chu [33-34]. When the WOS sand replacement was 5%, the pores could be filled. When the replacement exceeded 5%, the pore-filling function was lost. The compressive strength decreased when the WOS sand replacement was 10%. When the WOS sand replacement is large, the compressive strength is low, which is due to the porous structure and absorption rate of WOS sand. In addition, as mentioned in Section 3.1. the fineness modulus of WOS sand was lower than that of fine aggregate: therefore, at the same water-binder ratio, the fineness modulus of fine aggregate decreased as the WOS sand replacement increased. The compressive strength of the CLSM was reduced as the fineness modulus of fine aggregate decreased.

3.5. Ultrasonic pulse velocity

The results are shown in Fig. 5. The wave velocity of the groups increased with age. Low cementation was used in designing the mix proportion of the CLSM, so the C-S-H gel was weak, lacking colloids to fill the internal pores. Therefore, the measured ultrasonic pulse velocity was much lower than that of normal concrete. The overall ultrasonic pulse velocity was 1723-2603 (m/s); the trends associated with the wave velocity and compressive strength was similar. Higher wave velocity indicates a denser internal structure of concrete as well as higher compressive strength.

3.6. Absorption rate

An absorption rate test was carried out at the age of 28 days, and the results are shown in Fig. 6. The absorption rate was 12.1-14.0%. The specimen with 5% replacement of WOS sand had the lowest absorption rate of 12.1%, which was better than the absorption rate of 12.4% for the specimen with 0% replacement. Because the pozzolanic reaction of fly ash occurs continuously production of the C-S-H gel fills up the pores in the specimen, the absorption rate of the specimen decreases remarkably [33]. The absorption rates of the replaced WOS sand are increased by 1.1-1.6% when compared to the control group, because WOS sand has a higher porosity and absorption rate; the water absorption of the specimen and the water loss increased with the replacement percentage.

3.7. Porosity parameter

2700

2500

Fig. 6 shows that the porosity of the CLSM specimen was 22.4-23.7%. The porosity average increase of about 0.4 to 5.5%, when the



Fig. 5. Effect of WOS content on ultrasonic pulse velocity.



Fig. 6. Effect of WOS content on absorption and porosity.

increase in the quantity of WOS sand. According to the results of this study, the volume density of the CLSM specimen decreased as the replacement of WOS sand increased. The quantity of fly ash is not sufficient to excite the pozzolanic reaction, plus WOS sand has a high absorption rate and porosity, and the high water-binder ratio resulted in a loose distribution of mortar and aggregates. Therefore, the internal pores of the specimen could not be filled effectively, and the porosity of the CLSM was thus high. Overall, the engineering properties of cement replaced by fly ash are improved.

3.8. Shrinkage

A high water-binder ratio of 1–1.5 requires more batched water for CLSM, and the free-water content generated after grouting affects the volume shrinkage of CLSM significantly. The results pertaining to the variation in the volumes of the specimens are shown in Fig. 7. The shrinkage was observed to be 0.03% to -0.123%; the variation in volume shrank and increased with age. The 0% replacement specimen showed the lowest variation (-0.063%), nearly 50% of the shrinkage of the 20% replacement (-0.123%). Larger replacement percentages resulted in greater shrinkage. WOS sand has a high absorption rate; thus, the moisture content increased in the internal pores of the specimen, and the mortar structure was loose. As water filled pores, the number of capillary pores converted from water and enveloped air not taking part in the hydration decreased gradually such that the volume shrinkage grew with age. In addition, because the Young's modulus of WOS sand is lower than that of natural fine aggregate [17], the aggregate with a high Young's modulus was replaced by the aggregate with a low Young's modulus, and the shrinkage of the specimen increased accordingly.

3.9. Sulfate attack

Fig. 8 shows that the weight loss was observed to 1.170-4.822% after soaking in sulfate 5 times. As shown, the weight lost due to



Fig. 7. Effect of WOS content on shrinkage.



Fig. 8. Effect of WOS content on sulfate attack weight loss.

sulfate attack increased as the WOS sand replacement in the mix proportion increased, the test results are similar to the results in the literature [33]. This CLSM is a type of porous and low-strength cement material. SO_4^{2-} was carried by water through the specimen. WOS sand is a fine aggregate with a high absorption rate and porosity and is likely to be corroded by strong acids due to its high content of CaCO₃; thus, the addition of WOS sand to the CLSM cannot suppress sulfate attack effectively.

3.10. Microscopic interfacial properties

As shown in Fig. 9a, a part of the unreached hydrates resulted in the loose structure of the hydration product at the age of 7 days, and the flaky CH crystals and irregular rose crystalline phase AFm was dispersed everywhere. The C–S–H gel colloid hydrate providing strength was not apparent. Because the high water-binder ratio resulted in an insufficiently dense mortar structure, black pores and cavities were clearly observed. Fig. 9b shows that the hydration proceeded at the age of 28 days, and the C–S–H gel colloid filled a portion of the pores. The porosity was reduced significantly, suggesting that the compressive strength grew with age and that the grout developed with age.

Fig. 10 (a) shows that the top of the specimen was spread with laminar CH and C–S–H gel floccose net-structured colloids at the age of 7 days. The overall structure was loose, and the stacked flakes on the right were WOS sand. A few pores were observed in the flake stack, indicating that the interfacial cementation was not good. At the curing age of 28 days, as shown in Fig. 10b, the surface of the WOS sand was textured with strips featuring indentations along parallel rows and cracks occurred along the upper right, the weak plane of the material. This proves that the binding effect between WOS sand and mortar was poor, such that the compressive strength of the CLSM specimen with WOS sand decreased as the amount of added sand increased.

4. Conclusion

- (1) When the W/B was 1.3, the slump/flow of the CLSM contained 20% fly ash, the slump value was 26.5–21.5 cm, the slump flow value was 60–41 cm, and the tube flow value was 21–15 cm, all of which conformed to the performance requirements for general flowability grade for CLSM.
- (2) The control group reached the penetration value of 2.74 MPa the fastest (210 min), but the time required to reach 2.74 MPa increased with the WOS sand replacement.
- (3) The compressive strength of the CLSM developed more favorably than that of the control group when the WOS sand replacement was 5%, indicating that the 5% replacement with WOS sand produced an excellent pore-filling effect. For convenience in digging, the delayed development of strength in CLSM is desirable. The results of this study show



Fig. 9. SEM image of CLSM (0%).



(a) 7 days

(b) 28 days

Fig. 10. SEM image of WOS CLSM (20%).

that the overall compressive strength of CLSM decreased as the WOS sand replacement increased. The 1 day strength was 0.69 MPa, and the 28 day compressive strength did not exceed 8.23 MPa, indicating that the WOS sand is a feasible material for CLSM.

- (4) The ball-drop indentation diameter of the mix proportion designed in this study was 4.9–5.9 cm, within the acceptable range specified by ASTM D6024.
- (5) The minimum absorption rate occurred when the WOS sand replacement was 5%, when the replacement to 20%, the absorption rate increased by only 1.1–1.6%, a very small influence.
- (6) The volume variation exhibited shrinkage behavior in the CLSM with WOS sand and increased with age and WOS sand replacement.
- (7) The CLSM with WOS sand was less resistant to sulfate attack, and the replacement was proportional to the weight-loss rate.

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