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A Study of Elevated Temperatures on the Strength Properties of LCD Glass Powder Cement Mortars

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1. Introduction

The rapid increases in population, urbanization, and economic development, have been accompanied by an increase in the accidental fire risk. The fire redundancy of buildings can reduce the injury and damage, enhance the safety of residents, and increase the reusability of buildings. These are the prevailing concepts behind the development of fire proof buildings (Fang, 2006). The advancements in optoelectronic technology, software technology, and other high-tech production have made Taiwan a "green silicon island" over the global high-tech service and manufacturing industries. Unfortunately, these developments have also generated a considerable amount of industrial waste that, if handled improperly, will cause severe environmental damages. Recently, researchers have suggested that these industrial wastes are of high potential to be recycled, to generate economic benefits, and to reduce the dependency on national resources (Cheng, 2002). Rapid industrial development and high life standard have both increased the amount of waste glass, of which only a limited fraction is properly recycled and reused (Park et al., 2004; Mohamad, 2006). Liquid crystal products such as LCD screens and mobile phone panels have become increasingly popular in recent years. Taiwan's TFT-LCD panel manufacturing products have been ranked as the top 1st over the world, which account for 39.2% of the entire global output. The LCD waste glass generated from the manufacturing process is approximately 12,000 tons per year (Cheng, 2002; Fang, 2006). How to use LCD glass waste in producing concrete has therefore, become a highly attractive issue in Taiwan. Glass waste is considered as ecologically friendly and non-toxic, with qualified physical properties and a simple chemical composition. For example, soda-lime glass consists of approximately 73% of SiO_2 , 13% of Na_2O and 10% of CaO (Shi and Zheng, 2007.). This renders most glass wastes environmentally friendly as a recyclable material (Cheng and Chiang, 2003). The term "glass" comprises several chemical varieties, including binary alkali-silicate glass, boro-silicate glass, and ternary soda-lime silicate glass (Shayan and Xu, 2006). One solution to properly recycle these glass wastes is suggested by grinding the material into fine glass powder (GLP), and incorporating them into concrete as a pozzolanic agent. Laboratory experiments have shown that fine GLP is capable of suppressing the alkali reactivity present in coarser glass aggregates and naturally obtained reactive

aggregates. In addition, finer glass powders are beneficial to the pozzolanic reactions in concrete. It was reported that a replacing amount of 30% cement by glass powders in some mixes has shown to provide satisfactory mechanical strengths (Shayan and Xu, 2004).

Most reused glass is produced through the re-melting process. Therefore, not all waste glass is suitable for producing recycle glass, particularly for those beverage bottles. This is because they are mostly contaminated with paper and other undesired substances. For quality and security purposes, the outlets of waste glass must be properly identified, especially when using in the construction industry (Lin, 2006). Previous literature related to the functionality of waste glass in concrete production has focused on its application as a substituent for cement. Other successful examples of waste glass recycling projects include using recycled glass as a cullet in glass production, a raw material for the production of abrasives and fiberglass, an aggregate substituent in concrete (as a pozzolanic additive), an agent in sand-blasting, road beds, pavement and parking lots, a raw material for the production of glass pellets or beads used in the reflective paint of highways, and a fractionators for lighting matches and firing ammunition (Poutos et al. 2008; Zainab and Enas, 2009). Previous investigation shows that the compressive, flexural, indirect tensile strengths and Schmidt hardness of concrete would decrease as the content of waste glass aggregate increases, particularly when the content exceeds 20% (Bashar and Ghassan, 2008). Although the influence on the mechanical properties of concrete is not thoroughly characterized, the employment of recycled glass is still rapidly emerging, and can widely be found in many industries such as asphalt concrete (glasphalt), normal concrete, back-filling, sub-base, tiles, masonry blocks, paving blocks and other decorative employments (Jin et al., 2000; Dyer and Dhir, 2001; Xie et al., 2003; Topcu and Canbaz, 2004; Park et al., 2004).

Using waste glass as a finely ground mineral additive (FGMA) in cement is another potential application (Bashar and Ghassan, 2008). The primary concern regarding the use of glass in concrete is the chemical reaction that takes place between the silica-rich glass particles and the alkali environments in the concrete pores (alkali-silica reaction). This reaction is detrimental to the stability of concrete properties unless appropriate precautions are taken to minimize this negative effect. Preventative actions include the incorporation of suitable pozzolanic materials such as fly ash, ground blast furnace slag (GBFS), or met kaolin in the concrete mix (Al-Mutairi et al., 2004). Nevertheless, Shayan and Xu have found that a 30% content amount of glass powder could be incorporated as the fine aggregate or cement replacement in concrete without causing any long-term detrimental effects (Shayan and Xu, 2004). Other results have also revealed that there is an increase in the concrete compressive strength if waste glass of very fine grade is added (Federico and Chidiac, 2009). Glass contains large quantities of silicon and calcium, which is very similar to Portland material in nature. Its physical properties such as density, compressive strength, modulus of elasticity, thermal coefficient of expansion, and coefficient of heat conduction are also very close to those of concrete (Topcu and Canbaz, 2004). Previous research results have shown that the fluidity, air content, and unit weight of concrete would increase if glass sand is employed as the fine aggregate substituent (Zeng, 2005). In addition, researchers have reported that the compressive strength, flexural strength, and cleavage strength of concrete would increase with the amount of glass powder inclusion, while the optimum adding fraction is about 20% (Zeng, 2005; Wang et al., 2007). Hence, Chi Sing Lam et al. suggested that glass sand can be purposely used to economically design the strength, to effectively decrease the porosity, and to enhance the durability, ultrasonic velocity, and resistance to acid, salt, alkali, and chloride ion electric osmosis of concrete (Wang, 2010). In recent years,

recycling waste LCD glass has become an important issue in Taiwan (Wang, 2010). It has been reported that controlled low strength materials (CLSM) containing waste LCD glass would meet many engineering property requirements including the strength, high fluidity, high permeability, and low electrical resistivity. All these measures would thus usher in the innovative application of waste glass (Hsu, 2009).

The contamination, residue, and organic content of recycled waste glass sand may be disadvantageous for construction application because these will result voids generated within the concrete micro-structures, and consequently degrade the physical properties over time (Konstantin et al., 2007). However, observation from scanning electron microscope (SEM) has revealed a visible densification around the glass grains, due to their partial hydration and the formation of additional C-S-H gel. The SEM investigation has shown that the main difference between glass cement and Portland cement pastes was the shrinkage in the CH crystal size and amount. This is caused by the glass grains involved in the pozzolanic reaction, leading to the consumption of CH crystals (Konstantin et al., 2007). Very recently, researchers have proposed that using waste glass from liquid crystal panels to replace fine aggregate and cement in concrete is a pioneering step for waste recycling technology in Taiwan (Wang and Huang, 2010a, 2010b; Wang, 2009a, 2009b, 2011; Wang and Chen, 2008). When mixed at room temperature, the compressive strength of waste TFT-LCD glass cement mortar could achieve 211kgf/cm², while the specimens treated at elevated temperatures would behave even stronger. Although the alkali activators contain no sodium silicate, the compressive strength of TFT-LCD glass cement mortar can still be increased with adequate temperature treatment and mode of curing. All these are done for the purpose of reducing concrete production costs. Concrete made with LCD glass powder has a sharp aroma, but the inclusion of calcium hydroxide could eliminate the bad smell. Concrete slurry made with LCD glass powder has lower water permeability than that made with Portland cement, showing that glass cement mortar would generate a more compact micro-structure. The experimental results of alkali activators in waste glass cement slurry also indicate that glass sand could perform as well as the fine aggregate in forming the bonding agents, and could be used as the substituent for Portland cement (Ju, 2008). With all these promising outcomes presented, this study continues to address the influences of temperature on concrete strength, and the resistance of glass powder cement mortar to high temperature. We would demonstrate that the temperature resisting property of waste LCD glass cement mortar is a merit for enhancing the recycling value and the economic efficiency of waste LCD glass.

2. Experimental plan

2.1 Materials

This study used ASTM Type I Portland cement with the specific gravity of 3.15 and the Blaine fineness of 3519 cm²/g. The corresponding chemical composition are SiO₂ (22.01 %), Al₂O₃ (5.57%), FeO₃ (3.44%), CaO (62.80%), K₂O (0.78%), Na₂O (0.40%), and MgO (2.59%) with trace amounts of TiO₂. The waste LCD glass was provided by Chi-Mei Industrial Corp., Taiwan. To achieve the uniformity of particle size, the TFT-LCD waste glass was crushed, grinded, and passed through a #8 sieve, respectively. The grinded particles were then dried using a Planetary Mill (Pulversette 4). Table 1 shows the corresponding results of toxic chemical leaching procedure (TCLP). The fine aggregates used for the mortar mixtures were obtained from Ligang River, which have also been approved following the ASTM C295 standards.

Component	As	Cd	Cr	Cr6+	Hg	Pb	Se
LCD glasses powder	0.022	ND	ND	ND	0.0077	0.281	ND
Regulatory	5.0	1.0	5.0	2.5	0.2	5.0	1.0
Remark	ND : Not detected						

Table 1. TCLP of LCD glass (mg/L)

2.2 Experimental variables and mixtures

The glass powder cement mortars used in this study were mixed at three different W/B ratios - 0.47, 0.59, and 0.71. The fineness values of the glass powder were 1500, 4500, and 6000 cm²/g, and the replacement ratios were 0%, 10%, 20%, and 30% by weight, accordingly. The testing ages of the samples were 7 days, 28 days, 56 days, and 91 days. Elevated temperature at 105°C, 580°C, and 800°C were treated onto the specimens, with the detail procedure described later in 2.3. It should be mentioned that the water content used in this study had included the water absorbed by sand aggregates. As shown in Table 2, the water amount for W/B ratios of 0.47, 0.59, and 0.71 were 265, 325, and 385 g, respectively.

	NO.	Cement	Glass	Sand	Water	Glass powder fineness (cm ² /g)	
47 59 71	G0	500	0	1375	265	1500	
	G1	450	50				
	G2	F1	400				100
	G3		350				150
	G1	450	50				
	G2	F4	400				100
	G3		350				150
	G1	450	50				
	G2	F6	400				100
	G3		350				150

Table 2. Mixture proportions of cement mortars

2.3 Experimental methods

For the fresh property examination, flow test (according to the CNS 1176 standard) and setting time test (according to the CNS 785 standard) were both conducted. Specimens are prepared with the geometry of 25×25×25 mm for compressive strength test (CNS 1232 standard) and 40×40×100mm for flexural strength test (CNS 1233 standard). According to ASTM C1012 standards, the anti-sulfate attack test was also performed with the mortar specimens cured for 7 days. After removed from the curing cabinet, the specimens were

dried for 24 hours and then dipped with sulfates for another 24 hours; this was denoted as one cycle of sulfate corrosion attack. The weight loss of the specimens was measured and their appearance was simultaneously observed over the 5 cycles of corrosion attack. As for the high-temperature resistance test, compressive strengths of mortar specimens were investigated after several temperature treatments (105°C, 580°C, and 800°C). Each temperature treatment consists of three steps: constantly increase to the target temperature within 2 hours, maintain the temperature for another 2 hours, and then lower the temperature back to normal in the last 2 hours. The glass powder morphology and microstructures of the mortar specimens were examined using a scanning electron microscope (SEM), JEOL JSM-6700F Japan. Glass powders were spread on a conductive double-edged adhesive tape that would then be attached to an SEM sample stud. Loose particles were properly dislodged with air blast. Representative photographs were taken after each sample was thoroughly observed.

3. Results and analysis

3.1 Chemical composition of waste LCD glass

Glass powders made from waste LCD glass consist of SiO₂ (62.48%), Al₂O₃ (16.76%), FeO₃ (9.41%), CaO (2.70%), K₂O (1.37%), Na₂O (0.64%), MgO (0.2%), and trace amounts of TiO₂, P₂O₆, and MnO. Table 1 presents the toxic chemical leaching procedure (TCLP) test results. As shown, the toxic contents of LCD glass powders were far below the statutory criteria, therefore meeting the certified standards for recycling hazardous industrial waste. These results suggest the recycled LCD glass, as a general industrial waste, could properly be used in concrete production.

3.2 Fresh properties

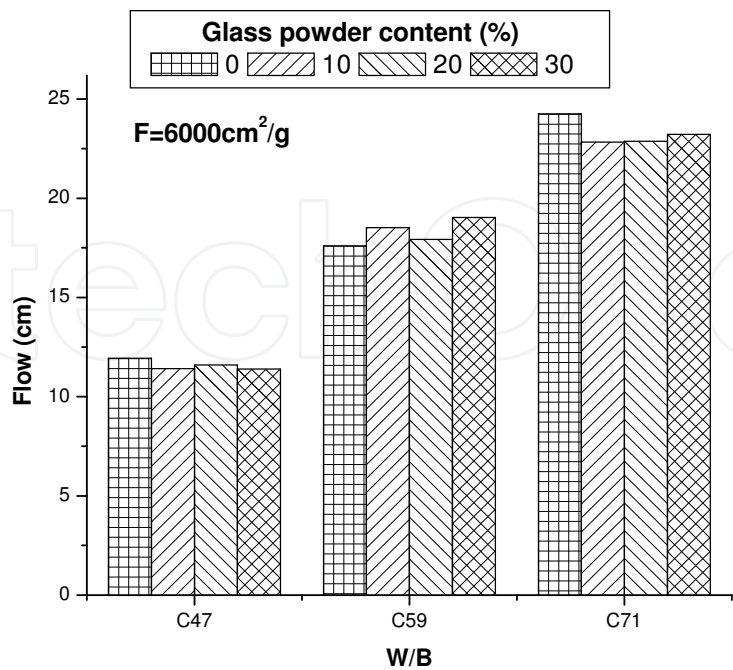


Fig. 1. Relationship between the flow and W/B ratio of waste LCD glass powder cement mortars with the powder fineness = 6000 cm²/g.

Figure 1 shows the fluidity versus W/B ratio of each group of the mortar specimens. As expected, the mortar fluidity increases with respect to a higher W/B ratio, suggesting that the amount of glass powder has no significant effect to the mortar fluidity. This is primarily caused by the blunt reactivity of glass powders to the hydration of mortar mixtures, even with the powder fineness of $6000 \text{ cm}^2/\text{g}$.

Figure 2 shows the relationship between glass powder content and final setting time of the mortar mixtures. As illustrated, the setting time was about 210 to 395 minutes (with glass powder fineness of $6000 \text{ cm}^2/\text{g}$). Higher W/B ratio would result in a longer setting time, which was caused by the delay in the hydration process when glass powders were added. It was also observed that the mortar setting time with W/B ratios of 0.59 and 0.71 increased with respect to higher glass powder content. This result suggested that the low water absorption capability of glass powders may have influenced the hydration process of cement mortars. In particular, when a sufficient moisture condition was present ($W/B \geq 0.59$), the setting time was significantly extended.

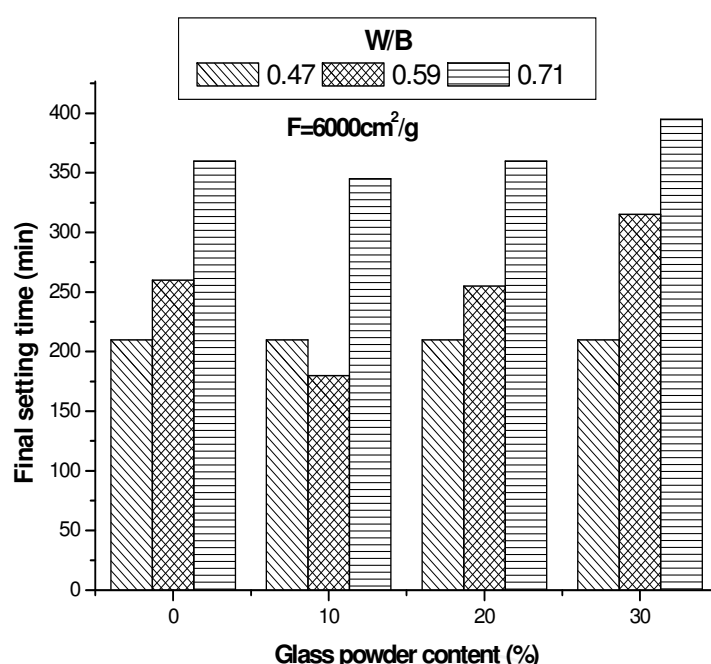


Fig. 2. Relationship between the glass powder content and final setting time of waste LCD glass powder cement mortars with the powder fineness = $6000 \text{ cm}^2/\text{g}$.

3.3 Compressive strength

Figure 3 shows the growth of compressive strength of the mortar specimens. There were three plots in the figure, with each plot showing different fineness grade of glass powder inclusion – 1500, 4500, and $6000 \text{ cm}^2/\text{g}$, respectively. Cement mortars with different glass powder contents (10%, 20%, and 30%) and identical powder fineness were compared with the control specimen (plain cement mortar), as shown in each plot. All the mortar specimens had a consistent W/B ratio of 0.47. For the mortars with $F = 1500$ and $4500 \text{ cm}^2/\text{g}$, specimens showed a lower compressive strength as compared to the control specimen at early age (7 days), while the difference was not significant. The mortar strengths with glass powder fineness = $1500 \text{ cm}^2/\text{g}$ were shown to closely approach the control group at the age of 91 days, as shown in the left plot. For the group with powder fineness = $4500 \text{ cm}^2/\text{g}$

(middle plot), the mortar strength started to surpass the control group after 28 days. In particular, the cement mortar with 10% glass powder replacement has exhibited a compressive strength as high as 63MPa at 91 days. For the mortars with powder fineness = 6000 cm²/g (right plot), all the testing groups have shown higher compressive strengths than plain cement mortars. Based on the data presented, it is concluded that the inclusion of finer glass powder could significantly enhance the compressive strength of cement mortars, while the optimal powder content is suggested as 10%.

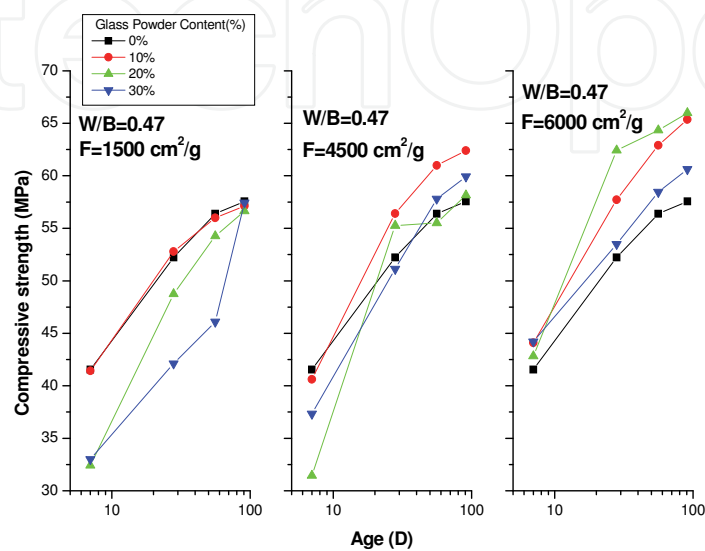


Fig. 3. The compressive strengths of waste LCD glass powder cement mortars ($W/B = 0.47$) at room temperature.

3.4 Flexural strength

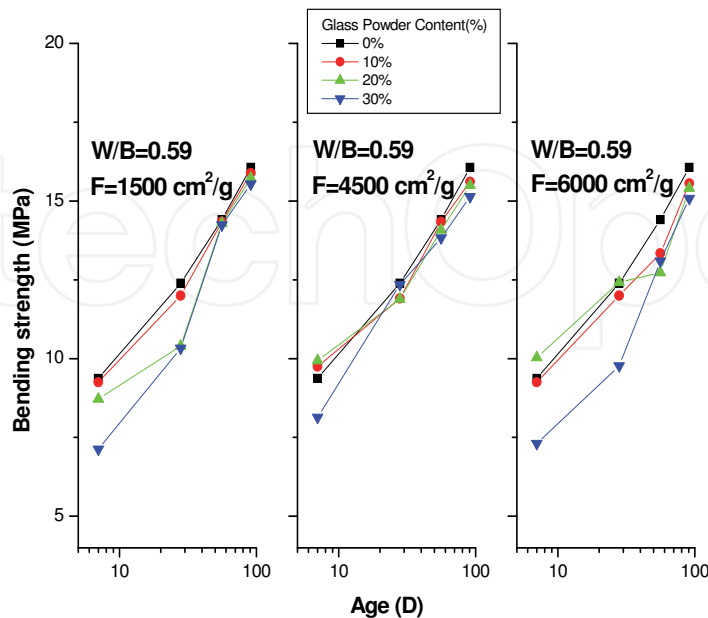


Fig. 4. The flexural strengths of waste LCD glass powder cement mortars ($W/B = 0.59$) at room temperature.

Similarly, Figure 4 shows the growth of flexural strength of cement mortar specimens with three grades of glass powder fineness (1500, 4500, and 6000 cm^2/g) and glass powder content (0%, 10%, 20%, and 30%) under a consistent W/B ratio of 0.59. As seen, the inclusion of glass powder would slightly lower the mortar flexural strength; however, the effect was nearly nullified as the curing age increases. The amount of flexural strength drop depends on the amount of glass powder added (i.e. cement replaced), while the maximum strength drop was shown to be less than 8% even at the age of 91 days.

3.5 Anti-sulfate attack

Figure 5 compares the corrosive weight loss of various glass powder cement mortars with the W/B ratio fixed at 0.59. A complete corrosion cycle has been previously described in section 2.3. Each mortar specimen has experienced five cycles of test. The measurements were taken after each cycle was completed. As expected, the rate of weight loss increased with the cycle of corrosion. Among them, the 20% glass powder group exhibited the most durability (least weight loss) as compared to the other groups. In particular when the powder fineness is 6000 cm^2/g , as illustrated in the right plot of the figure, the long-term durability was shown to be even more promising than plain cement mortars.

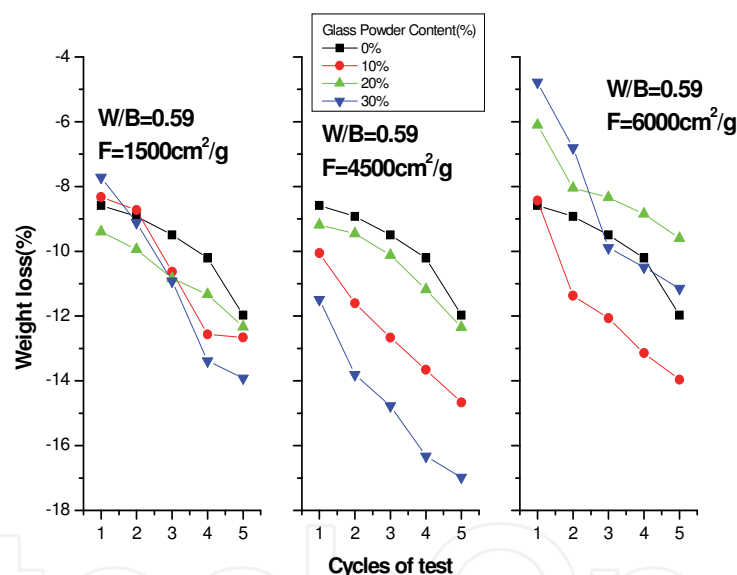


Fig. 5. Weight loss of the sodium sulfate corrosion tests of waste LCD glass powder cement mortars.

3.6 Compressive strength after elevated temperatures

Figure 6-8 show the results of elevated temperature resisting capacity of glass powder cement mortars. Here the powder content was fixed at 20% for all the tests. Although the W/B ratio was shown to have a negative influence to the mortar strength, the inclusion of glass powder appeared to provide a compensating effect to it. As shown in the middle and right plot of Figure 6, the 91 day compressive strengths of the mortars with W/B = 0.59 ($F = 6000 \text{ cm}^2/\text{g}$) and W/B = 0.71 ($F = 6000 \text{ cm}^2/\text{g}$) were 1.8% and 15% higher as compared to their corresponding control groups. Similar to the results discussed in section 3.3, finer glass particles would tend to enhance the compressive strength after experiencing an elevated temperature of 105°C. When the thermal treatment was increased to 580°C, as shown in

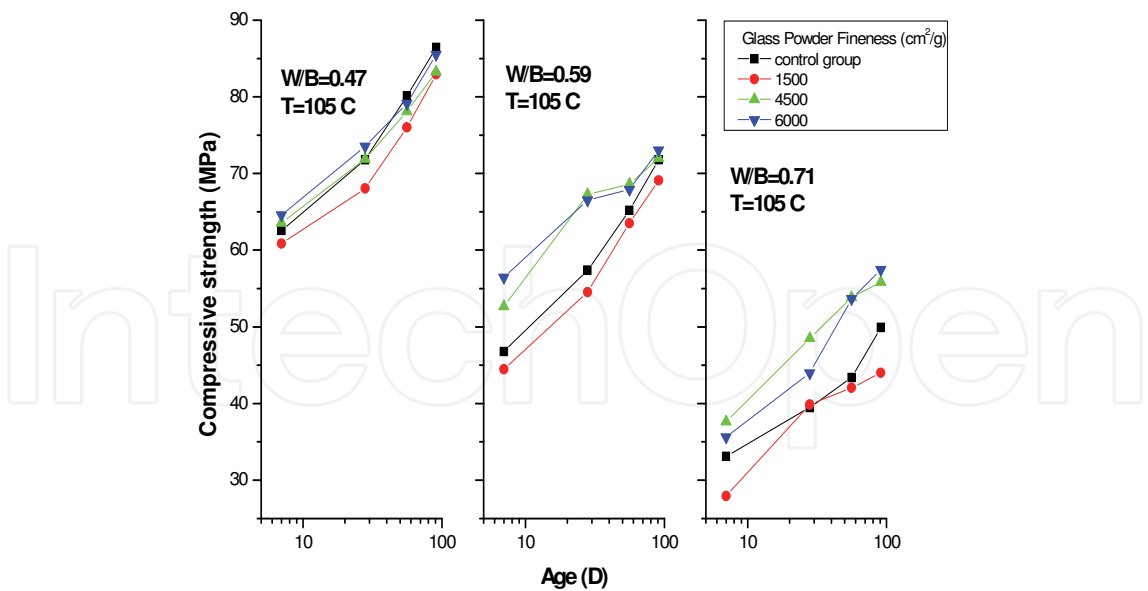


Fig. 6. The compressive strengths of waste LCD glass powder cement mortars after an elevated temperature treatment of 105°C.

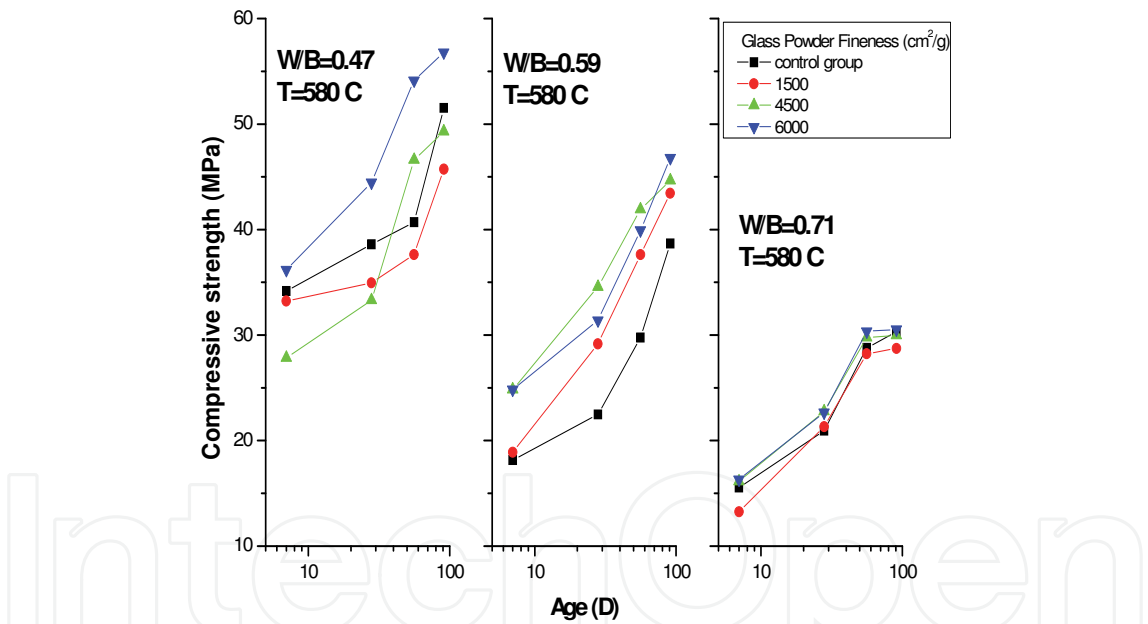


Fig. 7. The compressive strengths of waste LCD glass powder cement mortars after an elevated temperature treatment of 580°C.

Figure 7, the 91 days compressive strengths of the mortars with $W/B = 0.47$ ($F = 6000 \text{ cm}^2/\text{g}$) and $W/B = 0.59$ ($F = 6000 \text{ cm}^2/\text{g}$) were 10% and 21% higher as compared to their control groups, respectively. The mortar specimens with the highest W/B ratio of 0.71, on the contrary, exhibited no significant strength enhancement, while the one with $F = 6000 \text{ cm}^2/\text{g}$ still behaved a slightly higher strength than others. Figure 8 shows the results when the temperature treatment was further increased to 800°C. As seen, the effects of W/B ratio and fineness grade to the compressive strength were similar to the case of 580°C. When the W/B ratio is as high as 0.71, however, the inclusion of glass powder appeared of no

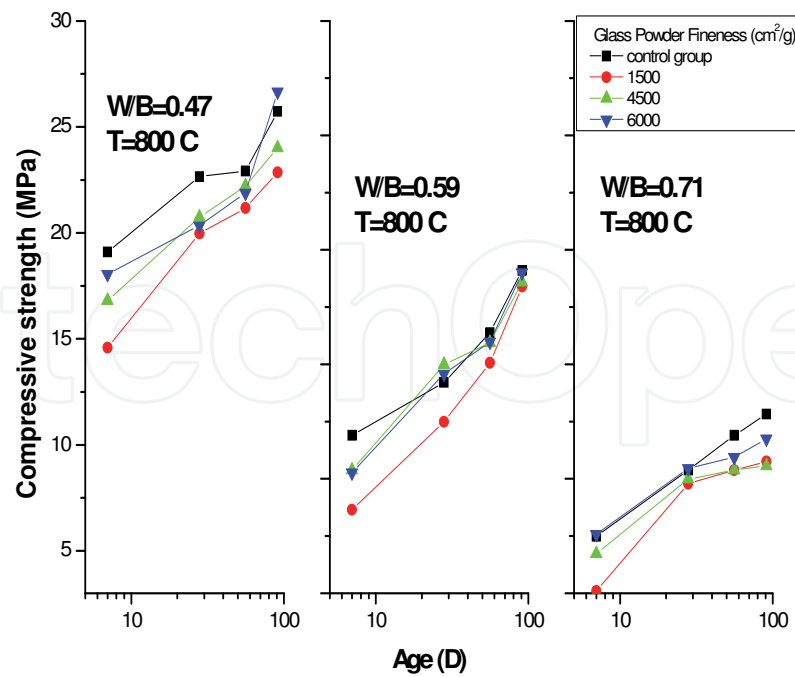


Fig. 8. The compressive strengths of waste LCD glass powder cement mortars after an elevated temperature treatment of 800°C.

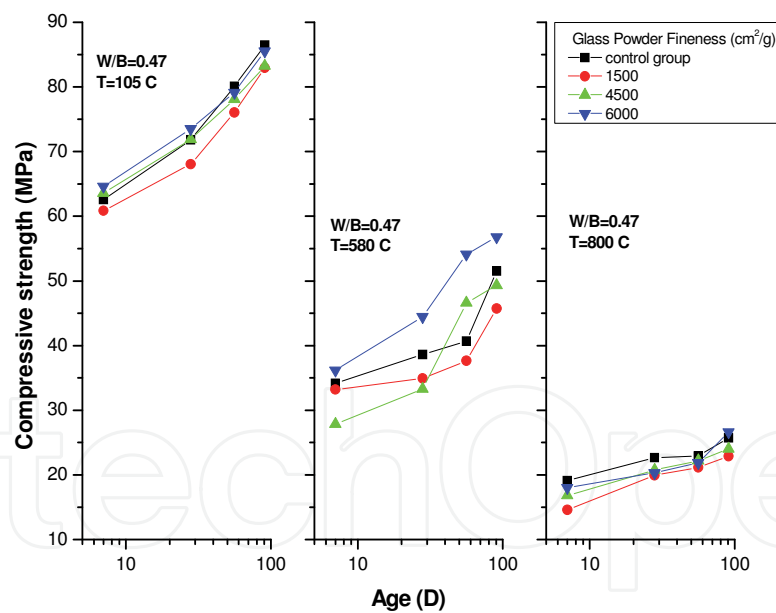


Fig. 9. The compressive strengths of waste LCD glass powder on cement mortars after elevated temperature treatments.

benefit to the mortar strength. Figure 9 summarized the effects of elevated temperatures to the compressive strengths of glass powder cement mortars. It is fairly obvious that higher temperature treatment would result in a lower strength development of cement mortars. This phenomenon was attributed to the cracks and pink spots generated on the surface of the mortar specimens under higher temperature, which would greatly reduce the compressive strength. The 91 days compressive strengths at 105°C, 580°C and 800°C, were

86.44, 51.53 and 25.73MPa, respectively. Although Figure 9 merely summarized the mortars with $W/B = 0.47$, the other two cases should exhibit similar results.

3.7 Interfacial microstructure (SEM)

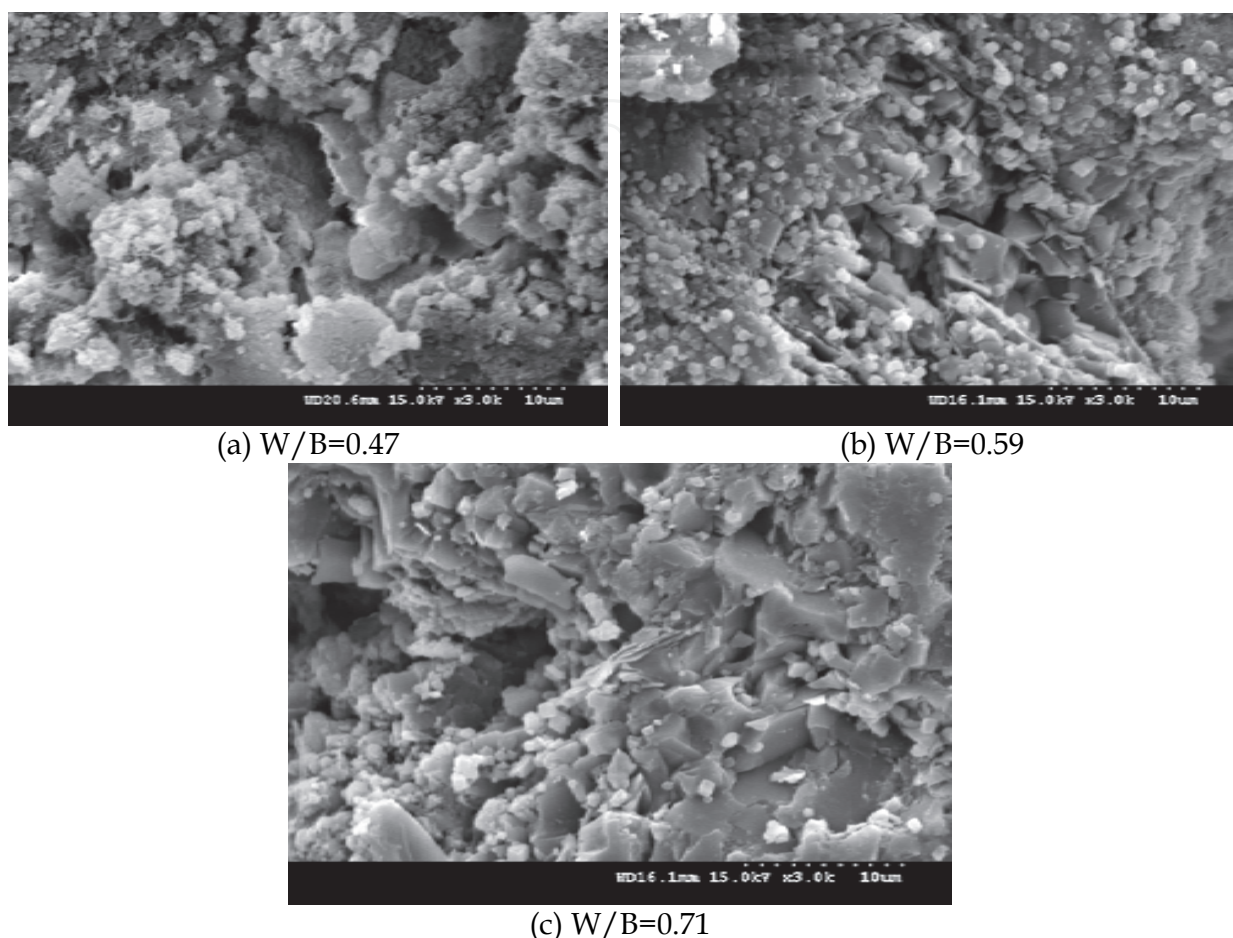
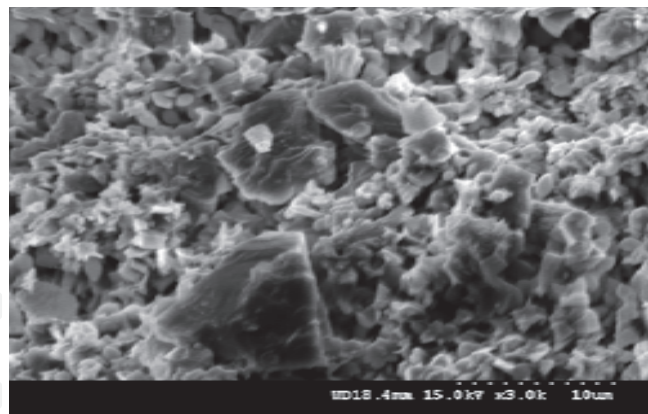
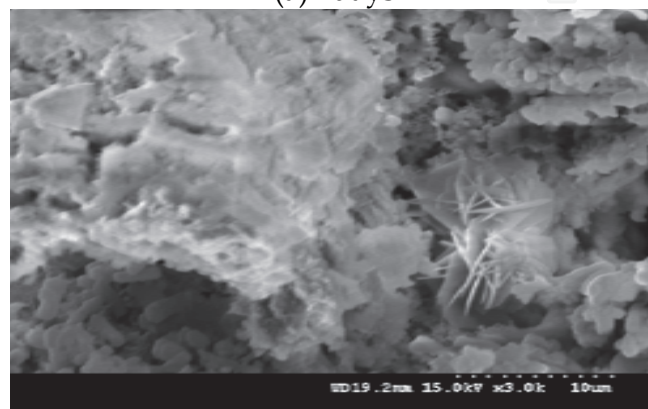


Fig. 10. SEM crystal structure diagrams of waste LCD glass powder cement mortars with various W/B ratios, powder fineness = $6000\text{cm}^2/\text{g}$, cured at room temperature for 28 days, and thermal treated at 105°C .

Figure 10 shows the SEM images ($\times 3000$) of the cement mortar with the glass powder fineness = $6000\text{ cm}^2/\text{g}$. The specimens were cured for 28 days and treated with an elevated temperature of 105°C before SEM observation was performed. As seen, the primary hydration products of the mortar specimens were C-S-H gel and CH hydrolyzed spiked balls. If the temperature continued to increase, water exiting in the C-S-H gel would be forced to release from the pore structures. When the temperature was raised to 800°C , as shown in Figure 11, the porous structures were mostly granulated and the porosity was significantly enhanced. In particular, a complete hydration was shown to be achieved after 7 days of curing followed by an elevated temperature treatment of 800°C . It was observed that there still exists certain amount of calcium sulphotoaluminate hydrate even when the mortar specimen was cured for 28 days accompanied with a 800°C thermal treatment. A granulated coarse surface also generated on the CH appearance due to water volatilization at high temperatures.



(a) 7days



(b) 28days

Fig. 11. SEM crystal structure diagrams of waste LCD glass cement mortars with W/B ratio = 0.71, powder fineness = 6000cm²/g, and treated with an elevated temperature of 800°C.

4. Conclusions

1. The TCLP test result shows that waste LCD glass powder fairly meets the certification criteria of hazardous industrial waste, suggesting that it could be properly recycled and used for concrete production. For the fresh property examination of the mortars, glass powder content shows no effect to the mortar fluidity, while would increase the final setting time when the W/B ratio exceeds 0.59.
2. Experimental results indicate that substituting 10% of cement by glass powder would gain a very promising compressive strength of the mortars, particularly when the added glass has a powder fineness $\geq 4500 \text{ cm}^2/\text{g}$. In real practices, this amount of glass powder substituent could be suggestively used to replace cement.
3. Although the W/B ratio had a negative influence to the mortar strength, the inclusion of glass powder appeared to provide a compensating effect to it. This effect is more prominent when the mortars experienced an elevated temperature. After a thermal treatment of 105°C, the compressive strengths with W/B = 0.59 ($F = 6000 \text{ cm}^2/\text{g}$) and W/B = 0.71 ($F = 6000 \text{ cm}^2/\text{g}$) were 1.8% and 15% higher as compared to their corresponding control groups (plain cement mortars). When the temperature is further raised to 580°C, the compressive strengths with W/B = 0.47 ($F = 6000 \text{ cm}^2/\text{g}$) and W/B = 0.59 ($F = 6000 \text{ cm}^2/\text{g}$) were then 10% and 21% higher as compared to their control groups, respectively.

4. When cured at room temperature for 91 days and treated with an extreme temperature of 800°C, the group with 20% glass powder content, fineness grade = 6000 cm²/g, and W/B = 0.47 exhibited the highest compressive strength, exceeding the control group by 3.6%. It should be mentioned that under this circumstance (91 days of curing, 800°C thermal treatment), the other groups have shown slight to fair decreases in compressive strength.
5. The sulfate corrosion test results indicate that cement mortars with W/B ratio = 0.59 and glass powder content = 20% would behave the best durability performance. In particular when the powder fineness is as fine as 6000 cm²/g, the long-term durability was shown to be even better than the plain cement mortar.
6. The microstructure observation indicates that the cement mortars would achieve a complete hydration after 7 days of curing and then treated with an elevated temperature of 800°C. The corresponding SEM image shows that under this circumstance, the porous structures were mostly granulated and the porosity was significantly enhanced.

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