

Enhancement of concrete performance via mixed limestone and granite powders

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1 **Enhancement of concrete performance via mixed limestone and granite powders**

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12

13 **Abstract:**

14 The granite and limestone powders are commonly exploited as a replacement for cement;
15 however, the effects of different mixing dosages of them on the mechanical properties and
16 durability of concrete have not been scrutinized carefully. Under different environmental
17 conditions, the compressive strength of the specimens is measured using cube compressive,
18 splitting tensile, freeze-thaw cycles, and sulfate immersion tests. The phase composition of
19 hydration products and microstructure is evaluated by SEM scanning analysis. The results
20 indicate that the composite mixture of granite and limestone powders shows a complementary
21 synergistic effect and improves the mechanical properties, freeze-thaw resistance, and sulfate
22 erosion resistance of the concrete. The best values for the mechanical properties and freeze-
23 thaw resistance are obtained when the dosages of granite and limestone powders in order are
24 10% and 5%. For the case of granite and limestone powders equal to 10% and 15%, respectively,
25 the best sulfate erosion resistance is reported.

26 **Keywords:** granite powder, limestone powder, concrete, mechanics, durability, microstructure.

27 **1. Introduction**

28 Concrete is one of the most important raw materials for infrastructure construction. By taking
29 into account green environmental protection and sustainable development, a serious
30 transformation from classical concrete to green one is underway. Stone powder is commonly
31 classified as waste materials produced from stone crushing as well as industrial production
32 process¹. Based on the available statistics, billions of tons of such materials are produced every
33 year. In the lack of proper treatment, a large amount of stone powder is accumulated in the open
34 air, causing severe damages to the environment. As a result, the application of waste stone

35 powder as concrete mineral admixture cannot only solve the resulted pollution problems, but
36 also would have important influences in promoting green and sustainable concrete²⁻⁴. At present,
37 limestone and granite powders are widely exploited in concrete. The use of Portland cement
38 containing limestone powder is a common implementation in European countries, particularly
39 in France. The Chinese standard GB175-2007 *General Portland Cement* stipulates that the
40 maximum dosage of limestone powder is limited to 5%⁵. Until now, researchers^{1, 6-9} have carried
41 out many studies on the mechanical properties, durability, and microstructure of concrete after
42 the addition of granite or limestone powder separately. Nevertheless, the influences of these
43 two kinds of commonly used stone powder compounds on the performance of concrete have
44 not been reported systematically. In this paper, the effects of different dosages of granite and
45 limestone powders on the mechanical properties, durability, and microstructure of concrete are
46 going to be investigated. It is hoped that this research works would provide a solid basis for
47 comprehensive exploitation of waste rock powder in concrete mixes shortly.

48 **2. Experimental**

49 **2.1 Raw materials**

50 The mixtures used were prepared with ordinary Portland cement PO 42.5 that meet the
51 Chinese standard GB 175-2007. Granite and limestone powders, produced from stone
52 processing enterprises (i.e., superfine limestone powder). In comparison with granite, limestone
53 has a lower hardness, is easier to grind, and is more economical¹⁰. The fine aggregates are
54 prepared from locally produced river sand, while the coarse aggregates are continuously graded
55 gravels that meet the Chinese standard GB/T 14685—2001. To reduce water usage, high-
56 performance naphthalene water-reducing agents are utilized, resulting in reduction of water

57 about by 20%. The exploited laboratory tap water meets the standard JGJ63-2206. The materials
58 properties of the concrete mix are provided in Table1 and the main chemical composition of
59 cement, granite powder, and limestone powder are also presented in Table2.

60 **2.2 Experimental scheme**

61 Limestone and granite powders are replaced as a part of the cement mixture in the concrete
62 such that the dosage of granite powder would be 10%(The current research shows that
63 controlling the dosage of granite powder at 10% is the most beneficial to the performance of
64 concrete¹¹), and the dosages of limestone powder are 0%, 5%, 10%, 15%, and 20%. The mix
65 ratio is made up according to C30 concrete, and group C is the reference group.The specific
66 mix ratios for various experimentally observed groups are now given in Table 3.

67 The appropriate mechanical properties tests were carried out following the standard GB/T
68 50081-2019. The dimensions of the understudy cubical specimens are
69 100mm×100mm×100mm. The compressive strength and splitting tensile strength of the
70 specimens are tested at curing ages of 7d, 14d, and 28d.

71 The freeze-thaw test was conducted by the quick-freezing method in accordance with GBJ
72 82—85. The 100mm×100mm×100mm specimens cured in 28d were immersed in water at the
73 temperature of 20±2°C for 4d. When the specimens are full of water, they are taken out until
74 the water is wiped on the surface, and then they are marked and weighted. For the numbers of
75 freeze-thaw cycles equal to 25, 50, 75, and 100 times, the specimens were taken out for
76 conducting the compressive strength test.

77 The sulfate immersion test was carried out in accordance with GB/T 50082—2009. The
78 cubic specimens subjected to standard curing at the age of 28d were immersed in the Na₂SO₄

79 solution of concentration 5% for 7d, 14d, and 28d, and then, they are taken out and dried by a
80 dryer at 80°C for 24h. Finally, the compressive strength of each specimen is measured.

81 The microstructure of specimens after 28 days of standard curing, 100 times freeze-thaw
82 cycles, and 28d of 5% Na₂SO₄ solution immersion was examined by SEM scanning. By
83 comparing the microstructure with the macro strength of concrete, the influence of the
84 microstructure on the strength of concrete is explored, and the corresponding mechanisms are
85 revealed.

86 For the sake of reducing the contingency due to existing differences between specimens,
87 each of the above-mentioned test groups is designated for three specimens, and their average
88 values are taken into account and reported.

89 **3. Results and discussion**

90 **3.1 Mechanical properties and microstructure**

91 The compressive strength of specimens at the curing ages of 7d, 14d, and 28d has been
92 demonstrated in Fig. 1. The obtained results indicate that the compressive strength of all
93 specimens increases with the curing age, but different dosages of LP and GP have different
94 influences on the compressive strength of concrete. The 28d compressive strength of G1, G2,
95 and G3 groups was higher than that of group C about 6.0%, 16.6%, and 2.7%, respectively. In
96 general, the strength growth rate of the first three groups is higher than that of group C in the
97 first 7days. The results show that when the dosages of GP was 10% and the LP was lower than
98 10%, the composite stone powder can enhance the compressive strength of concrete, especially
99 the early one. The research works of Yahia et al.¹²and Celik et al.¹³ also revealed that the LP
100 could raise the early compressive strength of concrete. When the dosages of GP and LP in order

101 were 10% and 5%, the benefit of the addition of LP was more apparent. However, the
102 compressive strength of the specimens in G4 and G5 groups at the age of 28d was significantly
103 lower than that of group C, and neither of them reached the strength value of C30 concrete.
104 This issue clearly indicates that when the dosages of GP and LP were 10% and more than 10%,
105 respectively, the composite stone powder has a deterioration influence on the compressive
106 strength.

107 **Fig. 1.** The compressive strength at different curing ages.

108 The results of splitting tensile strength of specimens at 7d, 14d, and 28d are presented in
109 Fig. 2. The obtained results show that the splitting tensile strength of all specimens would grow
110 with an increase of the curing age, and the GP and LP have a noticeable influence on the splitting
111 tensile strength of concrete. Among various groups under consideration, the G2 and G3
112 performed the best, and their splitting tensile strength at 28d increased by 27.3% and 22.7%
113 compared with that of group C. The pertinent strength of group G2 also grew about 17.4%
114 compared with group G1; however, the splitting tensile strength of G4 and G5 groups at 28d in
115 order was 4.5% and 13.6% lower than that of group C. This indicates that the splitting tensile
116 strength of concrete can be improved when the dosage of both LP and GP is no more than 10%.
117 Among different case studies, the addition of about 10% GP and 10% LP leads to a better
118 mechanical strength compared with the single mixture with 10% GP. Further, the excessive
119 addition of LP can degrade the splitting tensile strength of concrete.

120 **Fig. 2.** The splitting strength at different curing ages.

121 Fig. 3 shows the SEM scanning results of different groups of specimens at the standard

122 curing age of 28d. As shown in Fig. 3(C), the hydration products(C-S-H:Calcium silicate
123 hydrate, CH:Calcium hydroxide, Aft:ettringite.) of group C are relatively dense, dominated by
124 C-S-H gel and flake-like CH crystals. Additionally, a small amount of needle-stick Aft and
125 some pores are clearly detectable. After the addition of GP, the stone powder began to play
126 crucial roles in micro-filling^{12, 14}, crystal nucleation¹⁵, and active effects^{16, 17}, and thereby, the
127 pores became smaller and smaller. This fact is also supported by the performed study in Ref.¹⁸
128 showed that GP improves the concrete pore structure. The experimentally observed data also
129 demonstrate that the addition of GP making the distribution of hydration products to be more
130 uniform and the overall density of the specimens in group G1 be better than that of group C. At
131 the same time, there is more petite CH crystal in the hydration products. This is mainly because
132 of the fact that the active CaO and Al₂O₃ in the GP have a secondary hydration reaction with
133 the CH crystals, which further reduces the content of CH crystal. After simultaneous addition
134 of GP and LP, we can observe that the hydrates of group G2 are very dense and uniform, and
135 no obvious pores and cracks can be detected, which is greatly improved the mechanical
136 behavior of the specimens with respect to that of groups C and G1. This issue indicates that the
137 simultaneous addition of 10% GP and 5% LP provides a concrete mix with optimal grading,
138 and it maximizes the micro-filling, crystal nucleation, and stone powder active effects.
139 Furthermore, some performed studies^{19, 20} showed that because of the rough surface and
140 irregular shape of the composite stone powders, the above-mentioned fact would be beneficial
141 to enhance the absorption capacity of the cement slurry. Based on the test results for group G3,
142 we observed the precipitation of a large amount of unhydrated LP due to the increase of the
143 dosage of LP, and therefore, the densification of hydration products would lessen. For the

144 groups G4 and G5, this fact is more pronounced. The C-S-H gel in hydration products would
145 significantly lessen, the connection degree of hydration products would reduce, and a large
146 number of pores and micro-cracks would appear throughout the specimens. This fact is mainly
147 attributed to the replacement of the excessive cement by the addition of LP as well as the
148 coarsing effect^{16, 21, 22}. In fact, the LP itself is an inert substance²³, almost does not participate
149 in the hydration reaction, and its micro-filling, crystal nucleation, and active effects cannot
150 make up for the impact of a large drop in the cement hydration products. A large number of
151 pores and micro-cracks can seriously affect the performance of concrete. Such microscopic test
152 results are also consistent with the macroscopic mechanical properties of concrete.

153 **Fig. 3.** SEM scanning of concrete subjected to 28d standard curing.

154 **3.2 Freeze-thaw resistance and microstructure**

155 As one can see in Fig. 4, the compressive strength of concrete specimens decreases with
156 the increase of freeze-thaw cycles. Among various case studies, the compressive strength loss
157 of group G5 reduced about 50% when the number of freeze-thaw cycles was 75 times, and the
158 specimen had arrived at the failure standard. After 100 time freeze-thaw cycles, the strength
159 loss of the groups C-G5 was estimated to be 49.5%, 46.0%, 40.7%, 45.6%, 52.1%, and 59.9%,
160 respectively. The freeze-thaw resistance of the G1, G2, and G3 groups was better than that of
161 group C, and the strength loss of group G2 was reported to be the smallest one. These new
162 findings reveal that using 10% GP and 5% LP can provide the concrete with the best freeze-
163 thaw resistance. When the LP dosage exceeds 10%, the mechanical resistance of concrete
164 deteriorates, chiefly related to the increase of pores and micro-cracks within the concrete
165 specimens. Tikkanen et al.²⁴ also explained that the binding force after hydration becomes

166 smaller when the dosage of LP is too large due to its limited vitality, which would be harmful
167 to the freeze-thaw resistance of concrete.

168 **Fig. 4.** The compressive strength at different freeze-thaw cycles.

169 According to Fig. 5, many micro-cracks appeared within specimens of group C as the
170 number of freeze-thaw cycles approaches 100 times. Additionally, a large number of needle-
171 stick Aft precipitate around the cracks, which seriously affect the compressive strength of
172 concrete. For specimens of groups G1, G2, and G3 (see their corresponding plots in Fig. 5),
173 numerous flower-cluster C-(A)-S-H gels would be formed and arranged in a close connection.
174 Although the resulting structure was loose, their pertinent effects on the compressive strength
175 were less than micro-cracks and pores. This is because of the fact that when the dosage of LP
176 is relatively small, its micro-filling effect improves the concrete densification, hinders water
177 from entering into the concrete²⁵, and reduces the generated stresses from ice expansion.
178 According to experimentally observed data, the densification of specimens of group G2 was
179 higher than that of G1 and G3 groups. For the specimens tested in G4 and G5 groups, the
180 reduction of cementitious materials leads to the increase of concrete pores, and the poor
181 connectivity of each part results in grave influence on the freeze-thaw resistance.

182 **Fig. 5.** SEM scanning of concrete after 100 freeze-thaw cycles.

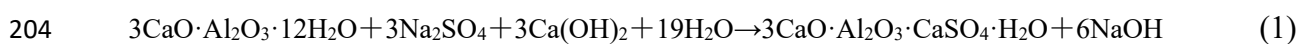
183 **3.3 Sulfate erosion resistance and microstructure**

184 Based on the plotted results in Fig. 6, the compressive strength of concrete specimens
185 decreases with the increase of soaking time. After soaking for 28 days, the compressive strength
186 loss of group C was about 16.1%, and that of group G1 was approximately equal to 14.3%,

187 indicating that the addition of GP improved the sulfate erosion resistance of concrete. In fact,
 188 the incorporation of GP into the concrete mix increases its compactness. Concerning the
 189 specimens of group G2, we found that the strength loss of group G2 was significantly lower
 190 than that of the G1 and C groups due to the addition of LP. Among different groups, group G4
 191 showed the best performance, whose compressive strength loss was only 8.9%. These
 192 investigations show that the addition of LP enhances the sulfate resistance of concrete.

193 **Fig. 6.** The compressive strength at different immersion times.

194 The performed study in Ref.²⁶ displayed that sulfate erosion is divided into ettringite and
 195 gypsum crystalline types. The crystal type of ettringite is the reaction of sulfate ions with
 196 sodium hydroxide and calcium aluminate hydrate to form the needle-like AFt precipitation,
 197 causing micro-crack and damage to the cement. The corresponding reaction equation has been
 198 provided in Eq. (1). For the gypsum crystal type, when the concentration of sulfate ions in the
 199 erosion solution is greater than 1000mg/L, AFt will also be produced plus to gypsum crystal
 200 precipitation through the chemical reaction. At this time, the volume of dihydrate gypsum
 201 formed inside the cement stone increases about 1.24 times the initial volume, causing damage
 202 to the cement stone due to excessive internal stress. The pertinent chemical reaction has been
 203 displayed by Eq. (2) .



206 Except for the cases presented in Figs. 7(C) and 7(G1), we can see obvious needle-like
 207 AFt in other cases. As it is seen in Fig. 7(C), there is almost no CH crystal, fewer hydration
 208 products, and apparent pores and micro-cracks. This indicates that CH crystal in the specimens

209 of group C is consumed in chemical reactions with sulfate ions. The GP in specimens of group
210 G1 plays a filling role to some extent, and its structural integrity is slightly better than that of
211 specimens in group C. Further, the fewer amount of AFt were produced in compared with group
212 C. This issue clearly displays that why the loss rate of the compressive strength of specimens
213 in group G1 is lower than that of group C. Ramadji et al.²⁷ found that existence of GP in the
214 concrete mix can reduce the heat of hydration and improve the resistance of the properly cured
215 structure to the acid attack due to its low participation in the hydration reaction. The obtained
216 results display that the addition of LP increased the amount of gypsum in the erosion products;
217 some scholars^{11, 28} believe that gypsum will cause the decalcification and softening of C-S-H
218 gel, resulting in the loss of concrete quality and the decrease of strength. However, no clear
219 gypsum crystals could be detected in Figs. 7(G2), (G3), and (G4). In these groups, the AFt could
220 be observed, but its quantity and density are significantly lower than those of groups C and G1.
221 Commonly, the structural integrity and compactness are optimized by increasing the LP's
222 dosage. Clustered prismatic gypsum crystals as well as granular sodium sulfate crystals can be
223 apparently observed in Fig. 7(G5). It is worth mentioning that the study of scholars in Ref.²⁹
224 revealed that the existence of sodium sulfate crystals causes concrete deterioration in sodium
225 sulfate solutions. Actually, the combined incorporation of LP and GP improves the compactness
226 of slurry and improves the coarsing effect caused by the excessive LP. In addition, LP can
227 consume the Al phase in the GP and reduce the number of AFt generated by sodium sulfate.
228 The Al₂O₃ and SiO₂ in the GP can also consume CH crystal^{30, 31}. Liu et al.³² also found that
229 calcium carbonate in LP could react with the acid such that a higher content of that leads to the
230 faster decrease of the concrete strength. When mixed with fly ash or silica fume, they could

231 both react with calcium hydroxide, and C-S-H is generated. This is good for enhancing the
232 paste's strength and weakens the degree of reactions between the hydration products and the
233 acid. When the dosage of GP was about 10%, and the dosage of LP was lower than 15%, they
234 result in a complementary synergistic effect and improve the sulfate resistance of concrete. The
235 optimal effect was achieved when the dosages of GP and LP in order were set equal to 10% and
236 15%. When the dosage of LP is too large, the dilution effect begins to dominate^{21, 22}, and the
237 complementary synergistic effect of composite stone powder begins to lose.

238 **Fig. 7.** SEM scanning of 5% Na₂SO₄ solution soaked for 28d.

239 **4. Conclusion**

240 In this study, the effects of different mixing dosages of granite and limestone powders on
241 the mechanical properties and durability of concrete were investigated. The main obtained
242 results from this experimental study are summarized in the following:

243 1) The simultaneous mix of GP and LP can improve both compressive strength and
244 splitting tensile strength of concrete, and the best effect is reported when 10% GP and 5% LP
245 are exploited. For such a mixing plan, the compressive strength and splitting tensile strength of
246 the specimens at the curing age of 28d increased about 16.6% and 27.3%.

247 2) The combination of both GP and LP can enhance the freeze-thaw resistance of concrete,
248 and the best effect is gained when 10% GP and 5% LP are exploited. After 100 times freeze-
249 thaw cycles, the compressive strength loss rate of the specimens was approximately 8.8% lower
250 than that of group C.

251 3) The compound mixing of 10% GP and no more than 15% LP results in an excellent
252 synergistic effect, enhancing the sulfate erosion resistance of concrete and resolving the

253 problem of poor sulfate erosion resistance of LP-based concrete. Among various cases, the best
254 results are observed for the case of 10% GP and 15% LP. After soaking in 5% Na₂SO₄ solution
255 for 28d, the strength loss of group G4 would be 7.2% lower than that of group C.

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Figures

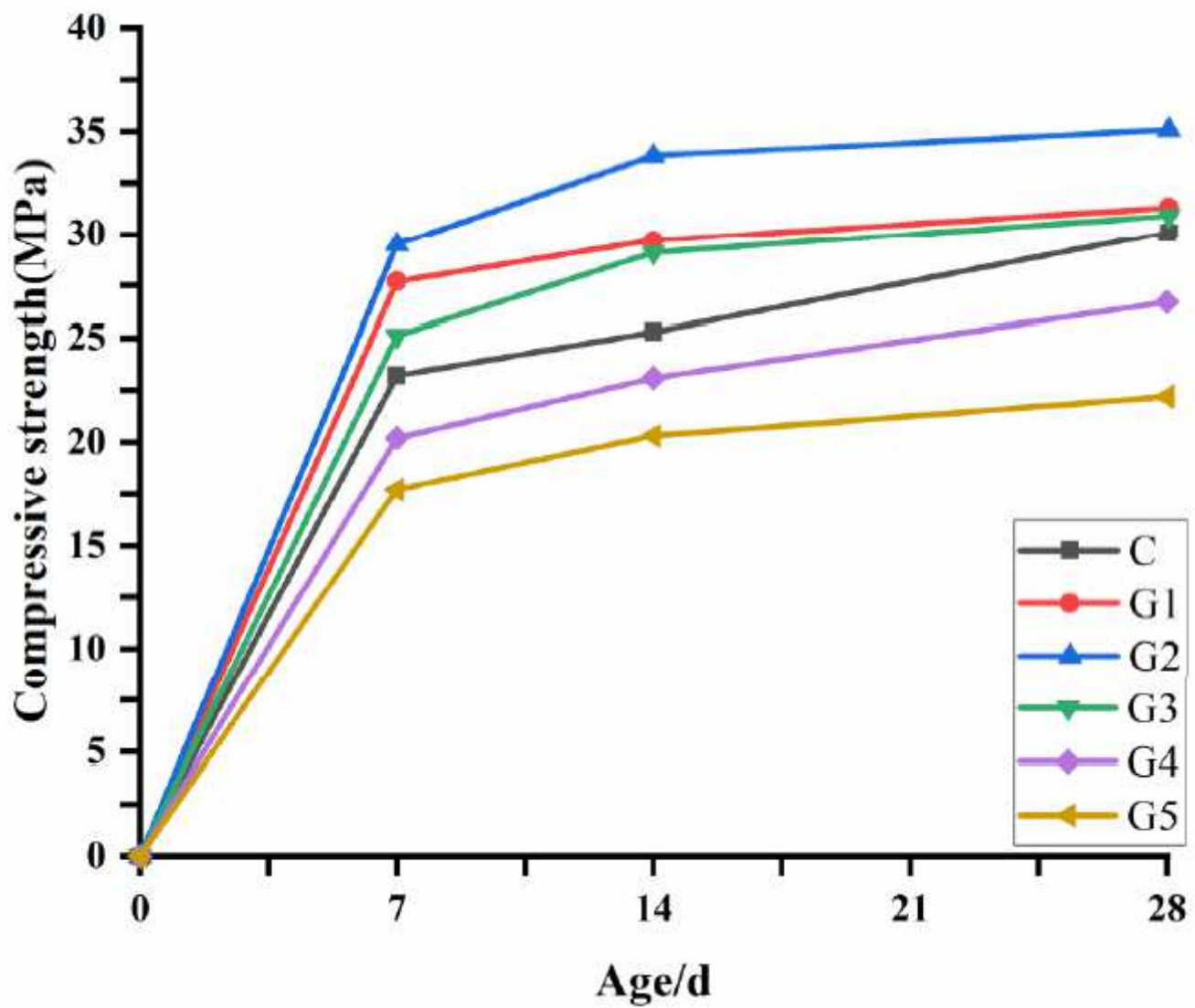


Figure 1

The compressive strength at different curing ages.

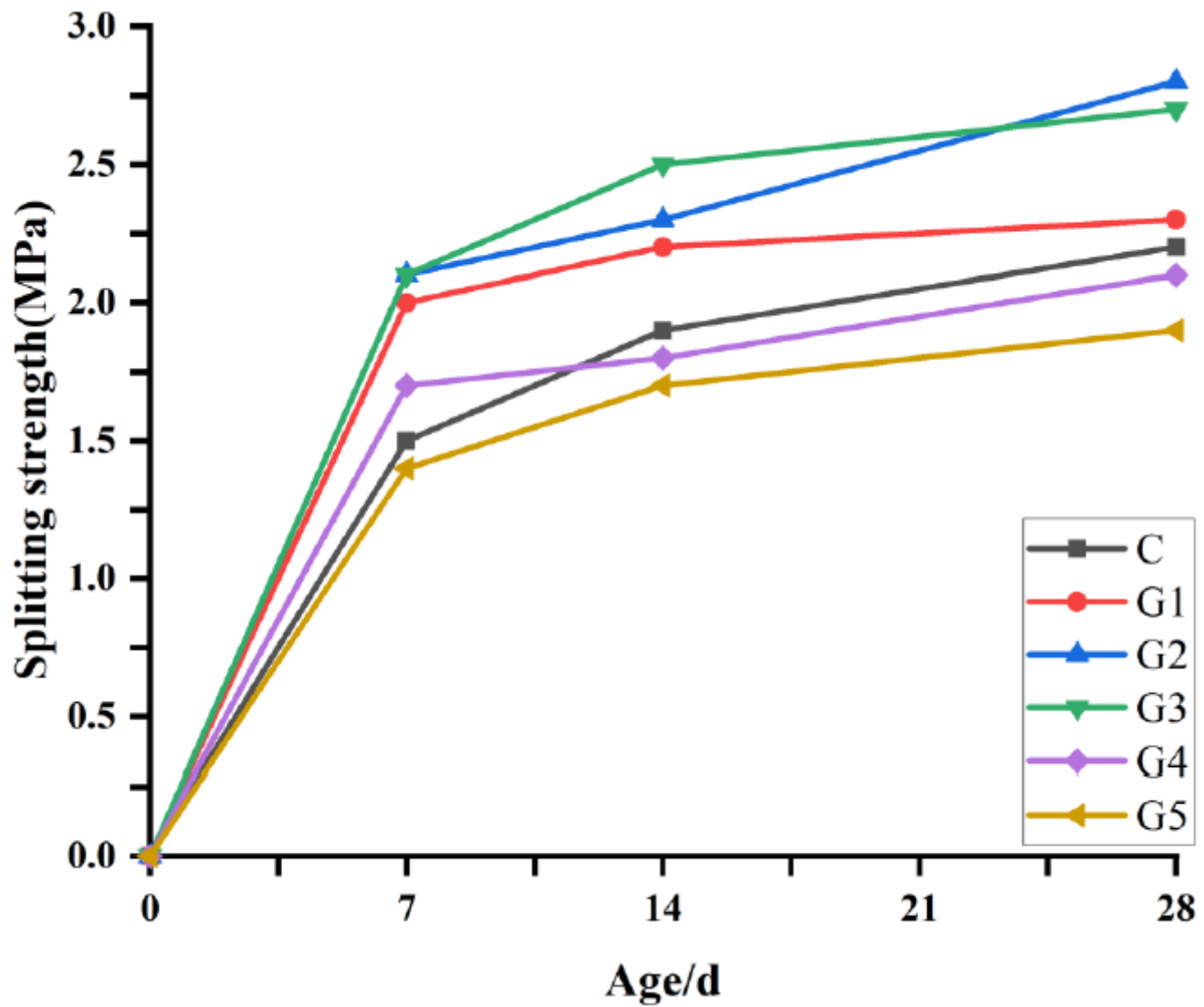


Figure 2

The splitting strength at different curing ages.

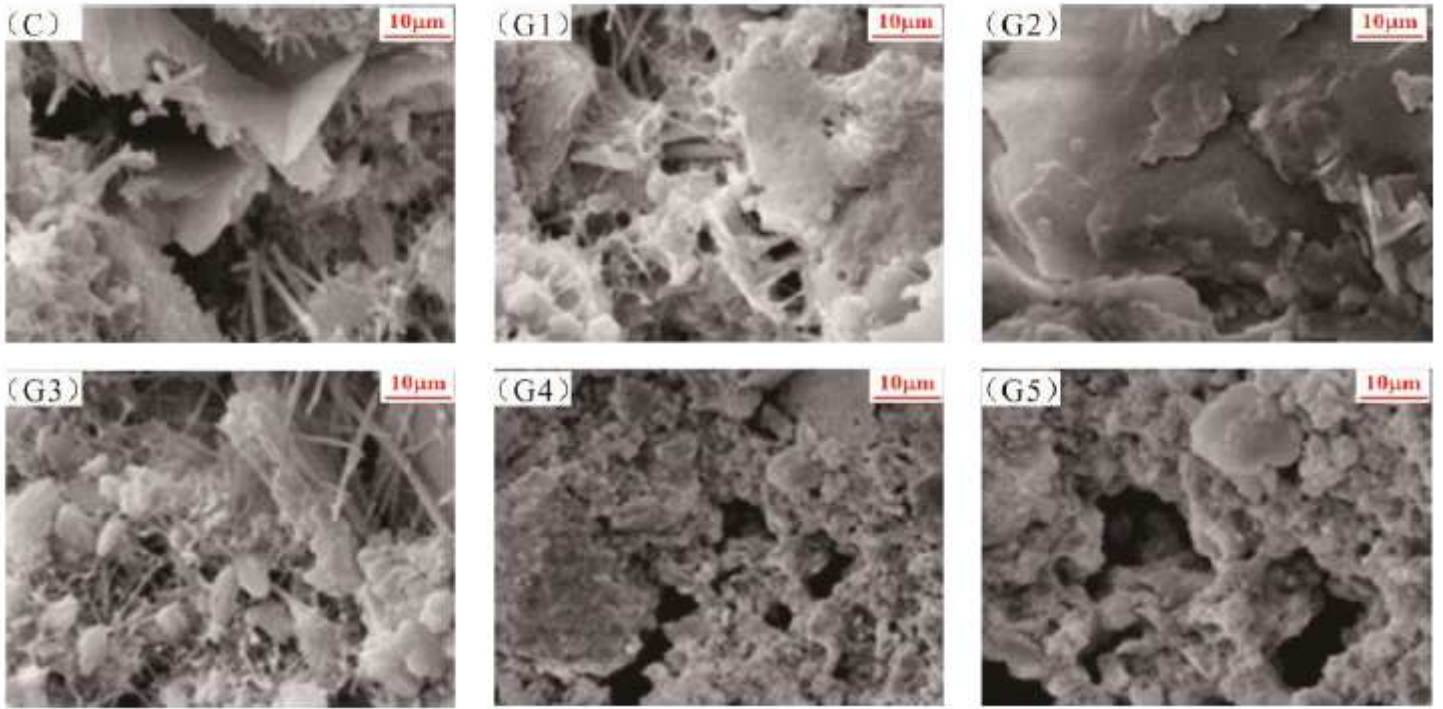


Figure 3

SEM scanning of concrete subjected to 28d standard curing.

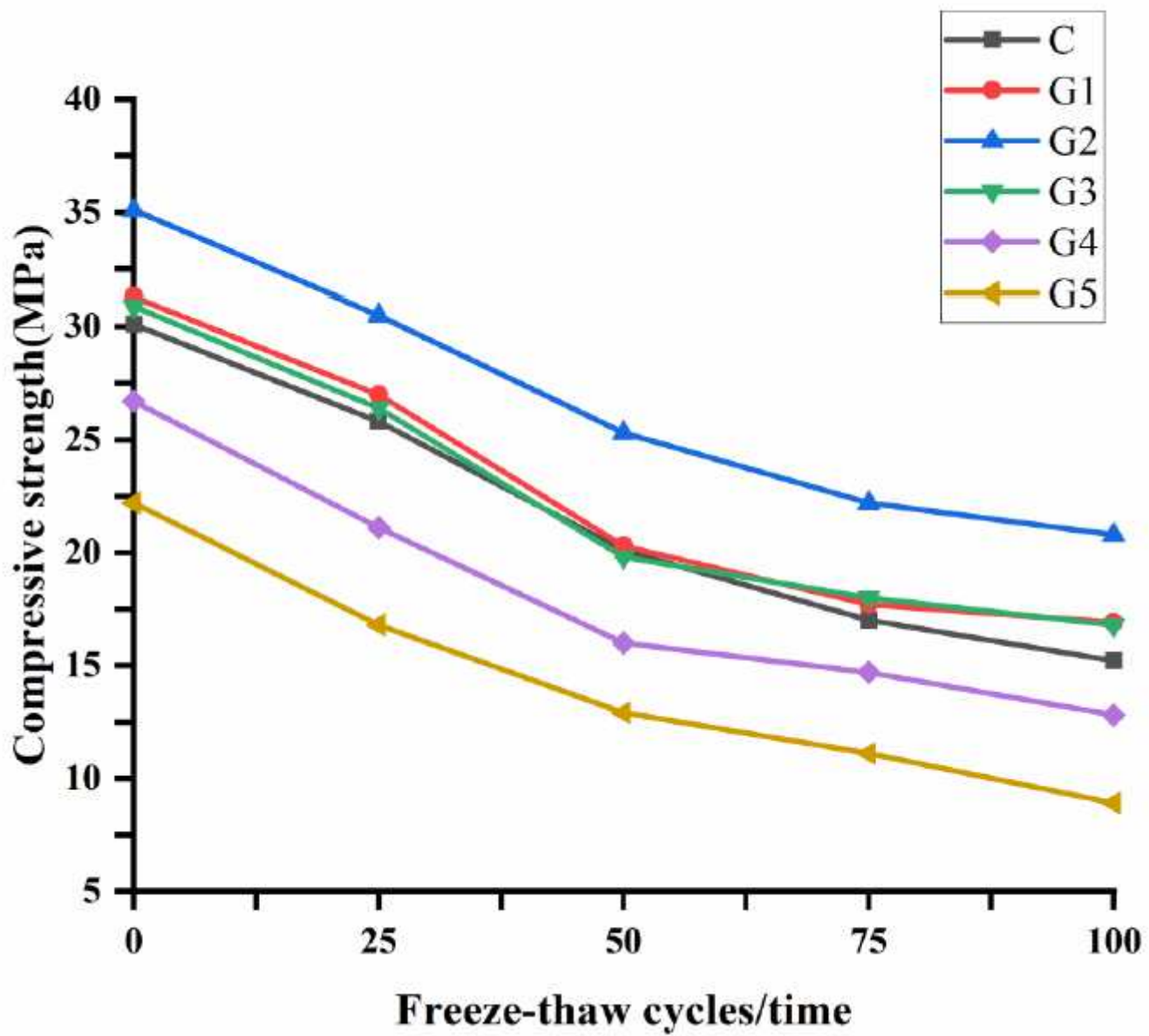


Figure 4

The compressive strength at different freeze-thaw cycles.

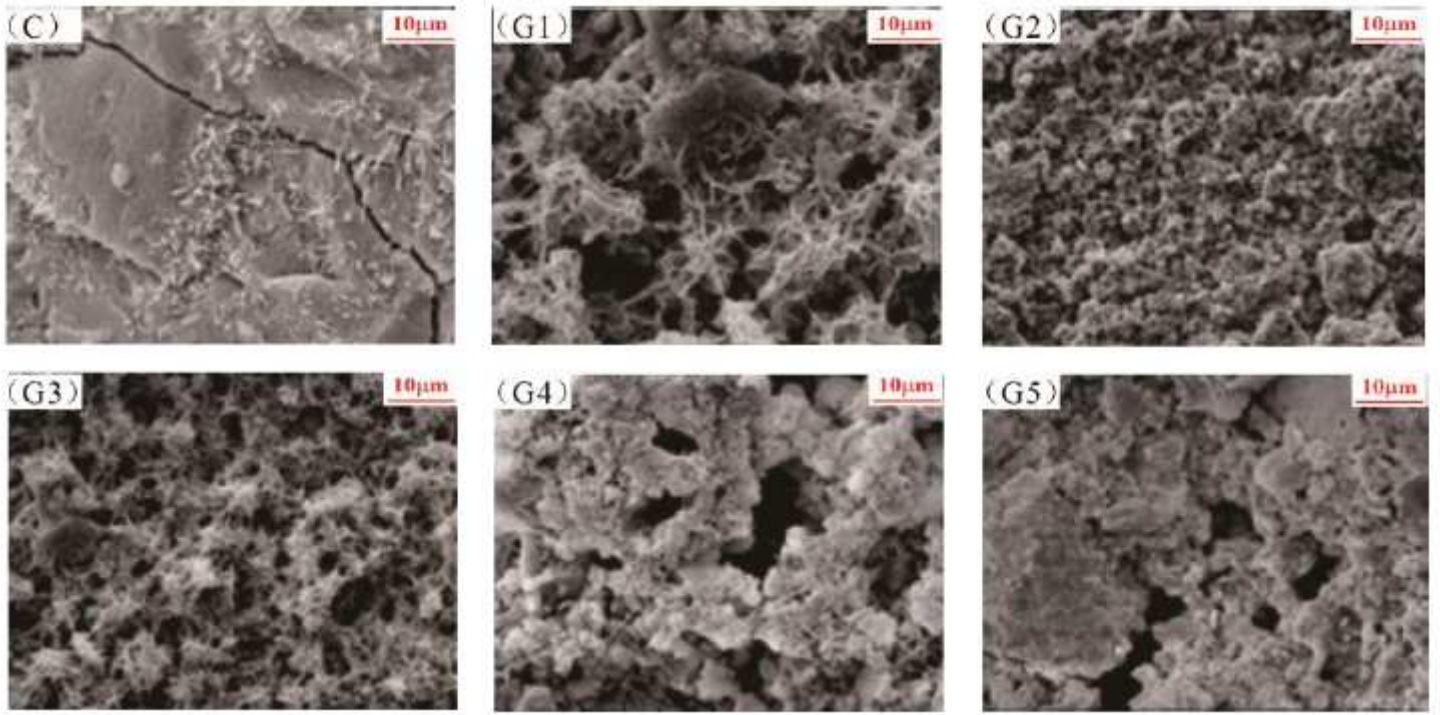


Figure 5

SEM scanning of concrete after 100 freeze-thaw cycles.

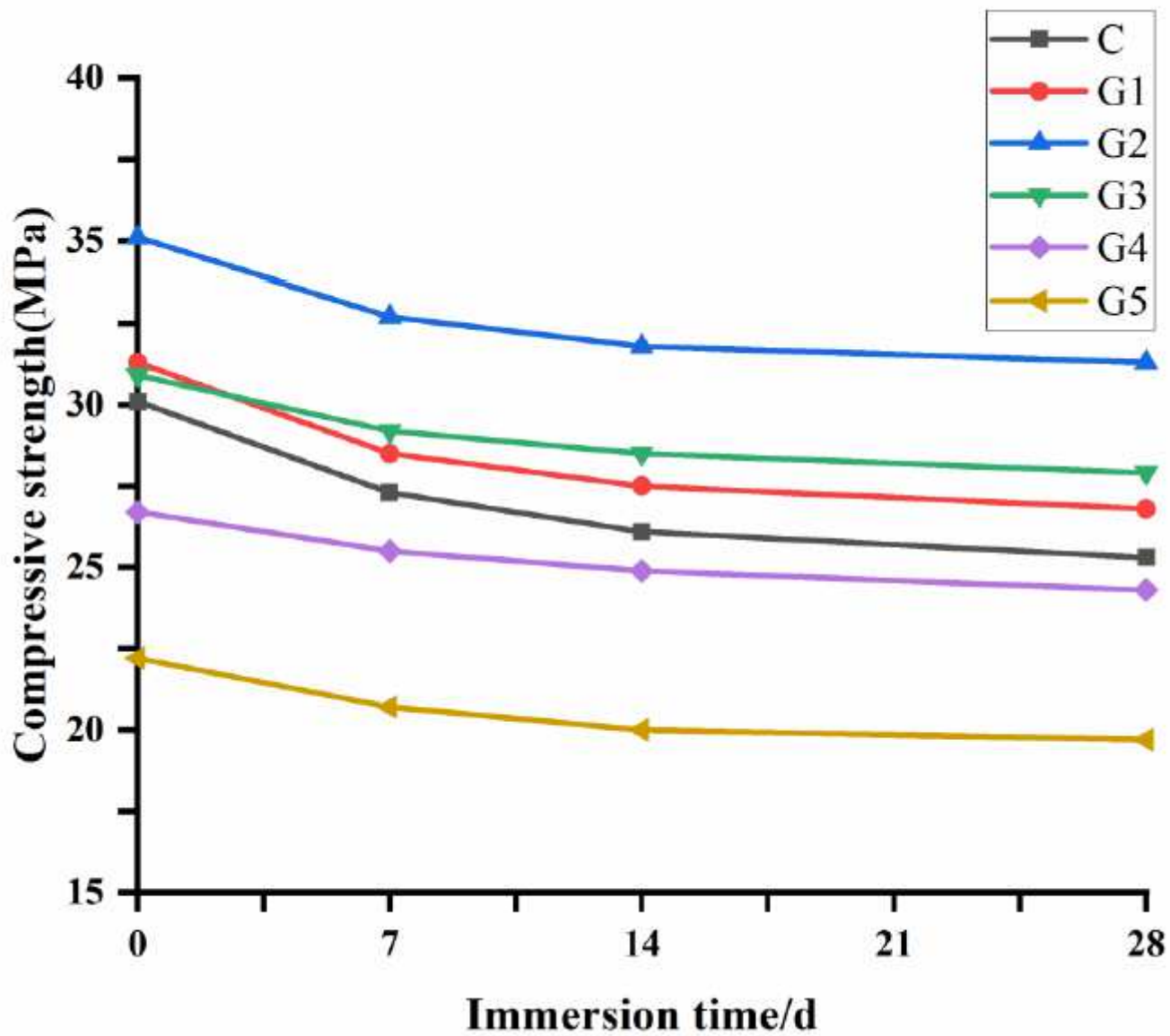


Figure 6

The compressive strength at different immersion times.

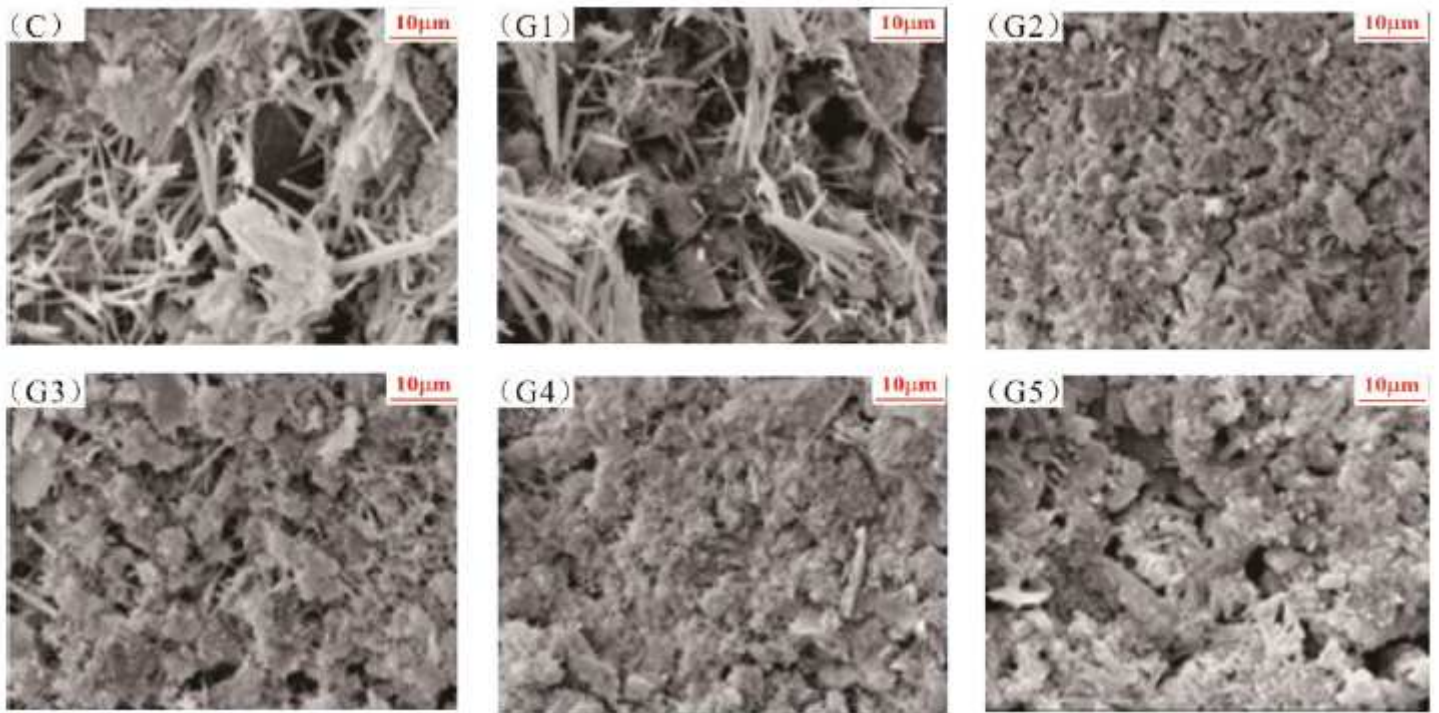


Figure 7

SEM scanning of 5% Na₂SO₄ solution soaked for 28d.

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- [Tables9.21.pdf](#)