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Influence of hydrochloric acid etching on bond strength between concrete substrate and repair materials

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Abstract

A comparison of experimental research into the influence of etching concrete substrate on bond strength between concrete substrate and repair materials was carried out. The test results showed that washing a concrete substrate with a hydrochloric acid solution with an appropriate consistency for a suitable period, resulted in a significant increase of the number of 10–50 μ m pores, micro-cracks and a reduction in fine sand particles, leading to a noticeable increase in micro- and fine roughness of the concrete substrate. As a result, the bond strength between concrete substrate and repair materials obtained a noticeable increase. © 2002 Elsevier Ltd. All rights reserved.

Keywords: Interface zone; Etching; Micro-roughness; Specific surface; Bond strength

1. Introduction

The bond strength between repair materials and onsite concrete is of vital importance [1–4]. Similar to the bond between the cement paste and the aggregate in a concrete [5–7], the main cause of adhesion between the hydration products of repair materials and the concrete substrate is the intermolecular force (Van der waals force); therefore, the specific surface of the concrete substrate has a significant influence on the bond strength. Unfortunately, many existing applications of concrete repairs were reported [1] not to be reliable although the necessary measures for roughing the old concrete surface (such as sand blasting, chipping with jack hammers, grinding, hydro-demolition and needle gunning [8]) were used to obtain a specific surface, as large as possible, based on previous investigations [1]. It should be noted that existing mechanical roughening methods can only increase the macro-roughness of the concrete substrate; the increase of the specific surface, therefore, is limited. Noticing that etching a surface might increase the micro- and fine roughness of the

concrete substrate, leading to an increase of the specific surface, the etching method was used in this research. It should be also noted that a badly carried out etching procedure may leave a thin layer of weakened concrete substrate [9]. The acid type, consistency as well as etching period, therefore, were chosen as variables based on careful considerations and experimental study.

2. Experimental

2.1. Type and consistency of acid

With reference to ACI committee 549 [9], a hydrochloric acid solution was chosen. HCl can primarily react with the Ca(OH)₂ of the hydrated cement paste to form CaCl₂ (which is easy to be flushed away), making the substrate more porous [9]. Because no adequate information concerning the influence of acid consistency on bond strength was available in current literature, an exploratory test to etch concrete substrate by using 1% and 2% hydrochloric acid solutions was carried out. The exploratory test results showed that 1% and 2% hydrochloric acid solutions had little influence on splitting bond strength between the concrete substrate and the repair material. Hydrochloric acid solutions of 5%, 7% and 10%, therefore, were chosen for formal testing.

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2.2. Materials

Ordinary Portland cement (Chinese Standard GB175-1999, analogy with ASTM C 150 Standard Specification) and a Class II fly ash (Chinese Standard GB15 96-91, analogy with ASTM C 618) were used in this research. The chemical analysis and physical properties of the cement and fly ash are presented in Table 1. Crushed stone with a maximum size less than 20 mm and medium sand (2.98 modulus fineness) were used for producing new and old concretes. Fine sand with a fineness modulus of 1.76 was used for making primers.

2.2.1. New and old concrete

The composition of the old and new concrete mixes was 0.4:1:1.57:2.55 (water:ordinary portland cement: sand:stone) by weight.

2.2.2. Primers

Three kinds of primers were made. The mix ratio for the cement mortar primer (CP) was 0.4:1:1 (water:cement:sand). The expansive paste primer (EP) was made by adding 10 wt.% of a U-type expansive agent in the cement mortar primer. The mix proportions of the fly ash modified mortar primer (FP) were 0.4:1:1:0.15 (water:cement:fine sand:fly ash).

2.3. Specimen preparation and repair procedure

2.3.1. Old specimen preparation

Two kinds of old concrete specimens were made. The first kind of specimen was made as shown in Fig. 1. A polyvinyl chloride partition panel without being coated with de-moulding agent was inserted in the middle of the mould so that two pieces of specimens were obtained. Noticing that macro-roughness has a significant influence on bond strength and that it is difficult to make all specimens having nearly the same macro-roughness, the smooth surface contacted with the polyvinyl chloride partition panel during casting was used to make the concrete substrate more consistent with one another and therefore more accurate.

The other kind of specimen was obtained by splitting a cube $(100 \times 100 \times 100 \text{ mm})$ into two pieces with a test machine. The split-broken surface was used as the old

 Table 1

 Chemical and physical properties of cement and fly ash

Chemical analysis (%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	LOI	Specific surface, Blaine (m ² /kg)	28 days Compressive strength (MPa)
Cement	19.5	4.4	6.22	65.9	1.5	1.09	0.30	1.43	462	45.9
Fly ash	52.5	19.1	8.2	14.7	1.98	0.35	0.48	3.6	565	-



Fig. 1. Production of old concrete specimens.

concrete surface for investigating the co-effect of both etching and different kinds of primers on bond strength (see Section 2.3.4).

2.3.2. Etching procedure

The etching of the surface was carried out in such a way that the hydrochloric acid solution was brushed on the surface of concrete substrate with a soft nylon brush at a rate of 20 times/min. The etched surface was then flushed under flowing tap water with another nylon brush up to 2 min and allowed to dry in the laboratory for 24 h.

2.3.3. Repair procedure

The old concrete specimens were placed in moulds with the substrate upwards. A layer (about 3 mm thick) of primer was brushed on the surface of the substrate, and the repair concrete was applied after no more than 30 min. The repaired portions for all of specimens were wet-cured by covering with wet burlap for 28 days before testing (Chinese Standard GBJ 81-85).

2.3.4. Specimen sets

A total of 88 cube $(100 \times 100 \times 100 \text{ mm})$ specimens (see Section 2.3.1) were made and they were divided into three sets as shown in Tables 2–4.

The first set had 20 specimens and was used to determine an optional consistency of hydrochloric acid solution, based on splitting tests (Chinese Standard GBJ 81-85, analogy with BS 1881: Part 117: 1983) (see Fig. 2). The cube or cylinder splitting test is simple to perform and give more uniform results than other tension test [10]. The failure takes place due to indirect tension

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fluence of acid consistency on 28 day splitting strength of concrete repaired interfaces (etching 5 min)	

Specimen group	%Hydrochloric acid solution	Concrete substrate preparation	Mean bond strength (MPa)	Coefficients of variation	Percentage of non-repaired concrete strength
1	None	Smooth	2.08	0.14	43.0
2	5%	Etching smooth	2.61	0.17	53.9
3	7%	Etching smooth	2.46	0.18	50.8
4	10%	Etching smooth	2.26	0.19	46.7

Table 3

Influence of etching period on 28 day splitting strength of concrete repaired interfaces

Specimen group	%Hydrochloric acid solution	Etching period (min)	Concrete substrate preparation	Mean bond strength (MPa)	Coefficients of variation	Percentage of non-repaired concrete strength
1	None	None	Smooth	2.11	0.08	42.0
2	5%	2	Etching smooth	2.22	0.08	44.2
3	5%	5	Etching smooth	2.74	0.05	54.6
4	5%	10	Etching smooth	1.13	0.07	22.5

Table 4

Influence of primer and etching on 28 day splitting strength of concrete repaired interface (etching 5 min with 5% consistency hydrochloric acid solution)

Specimen group	Primer	Concrete substrate preparation	Mean strength (MPa)	Coefficients of variation	Relative strength (MPa)	Percentage of non- repaired concrete strength
P_1	None	Split-broken	2.31	0.16	1	43.7
CP_1	Cement mortar	Split-broken	2.47	0.10	1.07	55.1
\mathbf{EP}_1	Expanded mortar	Split-broken	2.68	0.07	1.16	53.6
FP_1	Fly ash modified mortar	Split-broken	2.85	0.03	1.23	57.1
P_2	None	Etching split-broken	2.67	0.06	1.16	50.5
CP_2	Cement mortar	Etching split-broken	2.81	0.10	1.22	62.7
EP_2	Expanded mortar	Etching split-broken	2.91	0.04	1.26	58.2
FP ₂	Fly ash modified mortar	Etching split-broken	3.13	0.04	1.35	62.7



Fig. 2. Sketch of splitting test.

in the form of splitting along the interfacial layer. According to the different consistencies of acid solutions, the tests were divided into four groups as shown in Table 2, and each group had five specimens. The etching period was 5 min and cement mortar primer was used on all specimens. The age difference between new and old concrete was 3 months.

The second set had 28 specimens and was designed to determine a suitable acid washing period. They were divided into four groups as shown in Table 3. For each group, two specimens were used for micro-structure observation of the old concrete substrate after having been etched and flushed, and the other five specimens were repaired and used for splitting strength test. The consistency of the hydrochloric acid solution was 5% (see Table 2, the relatively best consistency based on the test results of the first set of specimens) and a cement mortar primer was used on all specimens. The age difference between new and old concrete was 3 months.

The last set included 40 specimens and was used for studying the co-effect of etching and different kinds of primers on bond strength. According to different treatments of concrete substrates and primers, these tests were divided into eight groups (as shown in Table 4) and each group had five specimens. Noticing that in the field old concrete has a longer age than the test cubes and could be thought to have completed hydration and shrinkage, cubes $(100 \times 100 \times 100 \text{ mm})$ cast two years ago were split and used as old concrete (see Section 2.3.1) to simulate field conditions better. The split-broken surface was used as the old concrete substrate. The two pieces of a broken cube were used for making two different kinds of repair specimens with and without etching the concrete substrate (e.g. one piece for making FP₁ specimen and the other for making FP₂ specimen as shown in Table 4) so that the two halves of the cube might have a similar macro-roughness.

3. Results and discussion

3.1. Optional consistency of acid

The splitting test results are presented in Table 2. The splitting strength of the specimens etched with 5% hydrochloric acid solution was 46.1% lower than that of the non-repaired concrete. The substrates etched with 0%, 7% and 10% hydrochloric acid solutions showed a 25.5%, 6.1% and 15.5% drop respectively in the splitting strength compared to those etched with 5% hydrochloric acid solution. Evidence indicates that etching with 7%

and 10% hydrochloric acid solutions over-weakens the concrete substrate [9]. Thus, the 5% hydrochloric acid solution was chosen for further testing.

3.2. Suitable etching period

Some fine sand particles of the concrete substrate were removed as a result of the surface being etched and flushed. Fig. 3 shows the micro-structures of the etched and untreated concrete substrates observed with a H-1030 SEM. It can be seen that compared to the etched concrete substrates, the untreated substrate was more dense and uniform. The number of 10-50 µm pores and micro-cracks on the surface of the concrete substrate, after being etched 5 min and flushed for 2 min increased significantly; this led to a noticeable increase of the micro- and fine roughness of the concrete substrate. As shown in Table 3, the splitting strength of the specimens etched for 5 min was 45.4% lower than that of the nonrepaired concrete, 29.9%, 23.4% and 142.5% higher than those etched for 0, 2, and 10 min, respectively. Evidence indicates that 10 min etching over-weakens the concrete substrate. Thus, etching with 5% hydrochloric acid solution for 5 min was chosen for further testing.



Fig. 3. SEM micrographs of old concrete substrate: (a) microstructure of non-repaired old concrete substrate; (b) microstructure of old concrete substrate after 2 min etching; (c) microstructure of old concrete substrate after 5 min etching.

3.3. Co-effect of primers and etching on bond strength

As shown in Table 4, the bond strength of specimens using both FP primer and etching treatment was 62.7% as high as that of non-repaired concrete, 9.8% and 26.7% higher than those of using FP and CP primer only, respectively. The noticeable increase in bond strength had a twofold reasons; the fly ash reacted with the $Ca(OH)_2$ to form C-S-H [5,7], and the etching increased the micro- and fine roughness of the concrete substrate. Both measures led to a noticeable improvement in the micro-structure of the interface zone and a noticeable increase of the specific surface of the concrete substrate. Because the reaction of fly ash does not start until sometime after mixing [10], it can be inferred that the micro-structure of the interface zone can be improved further with time by a secondary reaction [10] between the Ca(OH)₂ present and pozzolana, thus leading to a even denser interface zone with better durability.

4. Conclusions

Within the indicated scope of this study, the particular conclusions may be summarized as follow:

- 1. The bond strength of the specimens etched with 5% consistency of hydrochloric acid solution was 25.5%, 6.1% and 15.5% higher than those etched with 0%, 7% and 10% hydrochloric acid solutions.
- 2. The bond strength of the specimens etched with 5% consistency of hydrochloric acid solution for 5 min was 29.9%, 23.4% and 142.5% higher than those etched for 0, 2 and 10 min.
- 3. Etching the concrete substrate with 5% consistency of hydrochloric acid solution for 5 min and using a cement mortar primer, led to a 13.8% increase in bond strength compared with using mortar primer only.

- 4. The bond strength of the specimens using both etching treatment and an expanded mortar primer was 8.6% higher than using expanded mortar primer only.
- 5. The bond strength of the specimens using both etching treatment and fly ash modified mortar primer was 9.8% higher than using a fly ash modified mortar primer only.

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