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RÉSUMÉ

Les auteurs ont déterminé le processus de formation de l'adhérence de l'acier à la pâte de ciment modifiée au latex grâce à une nouvelle technique, soit la méthode de la poutre en porte-à-faux. Ils ont étudié six types de latex. En général, l'ajout de latex améliore l'adhérence, qui dépend des facteurs suivants : la teneur en eau non évaporable, la chaleur d'hydratation et la teneur en CH. Ils ont également évalué le rôle que joue la zone d'interface sur l'adhérence.
BOND STRENGTH DEVELOPMENT BETWEEN LATEX-MODIFIED CEMENT PASTE AND STEEL

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ABSTRACT
Bond strength development between latex-modified cement paste and steel was determined using a new technique, the overhanging beam method. Six latexes were studied. In general bond strength is improved with latex addition. Factors affecting bond strength include non-evaporable water content, heat of hydration, and CH content. The role of the interface zone on bond strength has been evaluated.

Introduction

Active interest in cement systems containing water dispersible polymers on latexes began more than 50 years ago (1). The most commonly used polymer latexes include: styrene-butadiene rubber, poly(acrylic ester), poly(vinylidene chloride-vinyl chloride), poly(ethylene-vinyl acetate) and poly(vinyl acetate). Latex-modified portland cement mortar and concrete have increased tensile strength, reduced drying shrinkage, increased durability, and improved adhesion or bond strength over conventional mortar and concrete (1).

There is a substantial amount of published data on the bond strength of latex-modified cements applied to mortar and concrete substrates. However, only a limited amount of data is available on the bond strength between polymer-modified cements and steel surfaces. Increased bond strength between cementitious materials and steel is of practical importance in the application of repair materials to damaged surfaces, and in the design and use of steel fiber-reinforced cement composites and grouts for structural purposes.

This paper discusses the development of bond strength between

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latex-modified cement paste and steel employing a new procedure (overhanging beam method) developed by the authors (2). The technique permits estimates of both internal stress (due to hydration of cement) and bond strength at early ages. It is applicable to thin coatings encountered in repair situations, does not involve application of external load, and is easily adaptable to testing in various controlled environments. Results obtained with this method are presented for cement paste modified with six different latexes. Factors affecting the development of bond strength are discussed using SEM and EDX analyses of selected debonded surfaces.

**Experimental**

**Materials**

The Portland cement used had the following composition: SiO₂ (20.78%); Al₂O₃ (6.20%); Fe₂O₃ (2.23%); CaO (64.83%); MgO (1.84%); SO₃ (3.17%); Na₂O (0.02%); K₂O (0.4%). Blaine fineness was 300 m²/kg.

The following six latexes were used: two poly(ethylene-vinyl acetate) copolymers {EVA(I) and EVA(II)}; styrene butadiene rubber (SBR); poly(vinyl acetate) (PVA); poly(acrylic-vinyl acetate) copolymer (AVA); and poly(vinylidene chloride-vinyl chloride) copolymer (V-V). Some properties of these latexes are given in Table 1. A defoaming agent was added to the AVA before its use with cement paste.

The steel substrate consisted of standard steel feeler gauge pieces of dimensions 12.70 mm × 304.80 mm × 0.25 mm.

**Cement Paste Mixes**

Latex-modified cement pastes were produced with each of the six latexes. The pastes were made at three water/cement ratios: w/c = 0.25, 0.30, and 0.35. Three latex/cement ratios, l/c = 0.04, 0.08, and 0.15 were used at each w/c ratio. Water-cement ratio = 0.40 was adopted for V-V modified cement paste because of the paste's low workability at w/c = 0.25. Cement pastes containing no latex were also made.

**Specimen Preparation**

Composite beam specimens for use with the overhanging beam method were made by applying latex-modified cement paste to the surface of steel feeler gauge with a spatula between guides set adjacent to the feeler gauge. Four test specimens were made for each test condition. The thickness of the paste (approximately 0.75 mm) was controlled by the thickness of the guides. The surface of the feeler gauge was cleaned with acetone prior to the application of the paste. All specimens were prepared at a temperature of 20±2°C.

Some specimens were also prepared for a direct-tension bond test. Two procedures were used. In the first, pastes were cast against a 25 mm square steel feeler-gauge surface epoxied to a brass test block. After a predetermined curing period at 100% relative humidity (RH) another block was epoxied to the free cement paste surface. In the second, paste was cast between the two brass blocks. Some specimens were allowed to dry at 0% and 55% RH before testing.
M. Nakayama and J.J. Beaudoin

### TABLE 1
Properties of Latexes

<table>
<thead>
<tr>
<th>Properties</th>
<th>EVA (I)</th>
<th>EVA (II)</th>
<th>SBR</th>
<th>PVA</th>
<th>AVA</th>
<th>V-V</th>
</tr>
</thead>
<tbody>
<tr>
<td>Non-volatiles (%)</td>
<td>55.5</td>
<td>46.0</td>
<td>48.4</td>
<td>55.1</td>
<td>55.4</td>
<td>56.2</td>
</tr>
<tr>
<td>PH</td>
<td>4.5</td>
<td>6.0±1.0</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Viscosity (cps)</td>
<td>1700</td>
<td>1000±500</td>
<td>1050±150</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Brookfield LVT 60 rpm</td>
<td>2300</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>RVT 20 rpm</td>
<td></td>
<td></td>
<td></td>
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<td></td>
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</tr>
<tr>
<td>RVT 25 rpm</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specific gravity</td>
<td>1.07</td>
<td>1.06</td>
<td>1.10±0.01</td>
<td>1.07±0.01</td>
<td>1.26</td>
<td></td>
</tr>
<tr>
<td>Particle size-range (μm)</td>
<td>2.5±1.5</td>
<td>0.35±0.15</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Particle Charge</td>
<td>Non-ionic</td>
<td>Anionic</td>
<td>Anionic</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

*measured by authors; other values of properties were provided by manufacturers.

### Bond Strength Determination

A novel technique employing an overhanging beam method (OBM) was used to estimate the bond strength between the latex-modified cement paste and steel. The method has been described in detail in previous papers (2-4). The procedure is briefly as follows. Specimens are prepared by applying a cement coating to a steel substrate. The geometry of the beam (overhang = 0.46 x midspan) is chosen to null out the effect of any uniformly distributed weight changes in the cement coating. Stresses developed in the cement paste coating can then be calculated from measurements of midspan deflection (2). Measurements are taken in two stages. In the first stage specimens are placed in a chamber conditioned at 100% RH. Deflections of the overhanging beam due to hydration of cement paste are continuously monitored. In the second stage the specimens are removed from the 100% RH environment and placed in a chamber at 0% RH. Shrinkage stresses in the cement paste due to drying result in additional deflection of the beam. The bond strength at a particular time is the resultant of the internal stress due to hydration at 100% RH and the additional shrinkage stress required to debond the sample at 0% RH. Bond strengths were determined for periods up to 168 h.

Bond specimens were also tested with a direct tension method using a Tinius Olsen testing machine. The loading rate was 0.50 mm/min.

### Non-evaporable Water-content Determination

Non-evaporable water content was determined for cement paste and latex-modified paste specimens at w/c = 0.30 and t/d = 0.08. The paste samples were removed from the steel substrate at various times, and hydration was stopped by pumping specimens in a vacuum desiccator until ready for testing. Non-evaporable water contents were determined by TGA in a nitrogen atmosphere using a 1090 Dupont Thermal Analyzer with a 951 TGA accessory. The heating rate was 20°C/min, and the maximum temperature was 1000°C. Non-evaporable water calculations included a correction for weight
loss due to decomposition of the latex. It is assumed that no chemically bound water is associated with the formation of new complexes due to any latex-cement mineral interaction. No such complexes have been identified.

Conduction Calorimetry

The rate and amount of heat development in hydrating cement and latex-modified cement systems were measured using a conduction calorimeter. Only specimens prepared at \( w/c = 0.30 \) and \( l/c = 0.08 \) were investigated. For data acquisition and processing, the calorimeter was interfaced with an Apple IIe microcomputer using a Taurus One 12-bit analog-to-digital convertor and an Apple super-serial interface card.

SEM and EDX Analysis

A Cambridge Stereoscan 250 with a TN-5500 X-Ray Analyzer was used for examination of debonded surfaces of selected specimens hydrated for 24 and 72 h. Latex-modified cement specimens made at \( w/c = 0.30 \) and \( l/c = 0.08 \) were examined. Specimens were obtained by cutting a test piece from the overhanging beam.

Results and discussion

Hydration Parameters

Hydration parameters such as non-evaporable water content, rate of heat development, and CH content are useful in following the microstructural development of cement systems.

Non-evaporable water content \( (w_n) \) determinations for the latex-modified cement paste systems \( (w/c = 0.30, l/c = 0.08) \) are plotted in Fig. 1. In the first 7 h, \( w_n \) values for the AVA, PVA, V-V, and EVA(I) systems are significantly greater than those for the control, SBR and EVA(II). At 24 h, PVA and EVA(II), have lower \( w_n \) values than the control, and V-V has the highest \( w_n \) value. At 168 h the \( w_n \) values are similar for all systems except for V-V and PVA, which have higher and lower values respectively.

In general these findings are in agreement with those of other workers. Latexes are known to accelerate or retard hydration of cement depending on latex type. Those latexes known to accelerate the hydration of cement, at least initially, include V-V and acrylics (5). PVA has been reported to retard hydration (6). It is subject to hydrolysis and interacts with CH. High \( w_n \) values for V-V, AVA, and PVA at 3 h may involve accelerated hydration of the aluminate phases as a significant amount of \( C_3S \) hydration has not occurred. Dehydrochlorination of V-V is known to occur in a highly alkaline cement system (5). Presence of chloride ion is known to accelerate hydration of cement. Release of chloride ion in the V-V system may contribute to increased \( w_n \) values at later ages.

Rate of heat development versus time curves were obtained for these systems using conduction calorimetry (Fig. 2). The heat developed in the first 2 h and 3.5 h is greater for AVA and PVA respectively compared to that with the control. This is in concert with the higher \( w_n \) values observed during this period. The initial rate of heat development for V-V was much lower than expected. After the first few hours the total heat developed is lower than the control for all latex-cement systems. The ranking of these systems for total heat developed is as follows: cement paste > EVA(I) > EVA(II) > SBR > V-V > AVA > PVA.
Estimates of peak bond stress (bond strength) between latex-modified cement paste and steel were made at various hydration times from experimental results of internal stress development with time. Maximum values of peak bond stress and peak bond stress values at 5 h hydration for all mixes are presented in Table 2. The maximum values for these systems increase in the following order: V-V < AVA < control < PVA < EVA(I) < SBR < EVA(II). The increase in maximum peak bond stress compared to the control varies from 15.7% for PVA to 73.2% for EVA(II). Values of bond-strength increase for PVA-modified mortar and steel may vary, and up to 59% for 20% PVA has been reported (8,9). There is a paucity of data available for other latexes.

In all cases but one the maximum values of peak stress occurred at $\ell/c = 0.08$. For EVA(II), $w/c = 0.30$ the maximum value is the same at $\ell/c = 0.08$ (24 h) and $\ell/c = 0.15$ (72 h). Latex cements are not normally cured at 100% RH for periods longer than 24 h to facilitate film formation. Prolonged moist curing usually is deleterious. The stable value of bond strength for most latex cements would appear to be the 24 h value. At 72 h the bond strength for $\ell/c = 0.15$ is still increasing. EVA(II) appears to have superior properties warranting further investigation. The $w/c$ ratio at which the maximum peak stress occurs was dependent on latex type. Except for the SBR system the $w/c$ ratio values for which the largest value of maximum peak stress was obtained are less than for the unmodified cement.
pastes, i.e., $w/c = 0.35$. It was found that the non-evaporable water versus time curve for the SBR system is in close proximity to the control paste.

The 5 h peak bond stress values increase in the following order: AVA < SBR < control < V-V < EVA(II) < EVA(I) < PVA. The order varies from that of the maximum peak stress values. Stress values for EVA(I) and (II) and PVA remain significantly greater than the control. Most of the maximum 5 h peak bond stress values occur at the lowest $w/c$ ratio and $l/c$ ratio. Possibly the latex contiguity has not occurred.

An example of bond strength development is given by the curves for paste modified with EVA(II) in Fig. 3. Each data point is the average of four test results. Variation in results is dependent on hydration time and $w/c$ ratio. Variation from the mean ranges from about 4% at early ages to 18% at later ages. The bond strength for the EVA(II) system is significantly improved after 15 h for all latex contents. At $l/c = 0.08$ the maximum bond strength is reached in 24 h. Strength retrogression of latex cement with prolonged moist curing is well known! The retrogression for EVA(II) ($l/c = 0.04$ and 0.08 at 24 h) is, however, much less than for the other latex systems. The PVA-cement systems for example lose all strength at 72 h. Strength retrogression for unmodified cement paste has been attributed to CH crystal growth in a well defined transition zone at the cement paste-steel interface (2,10). This mechanism may also contribute to strength retrogression in the latex-modified cement systems.
<table>
<thead>
<tr>
<th>Latex</th>
<th>w/c</th>
<th>0.04</th>
<th>0.08</th>
<th>0.15</th>
<th>0.04</th>
<th>0.08</th>
<th>0.15</th>
</tr>
</thead>
<tbody>
<tr>
<td>EVA(I)</td>
<td>0.25</td>
<td>2.26</td>
<td>3.80</td>
<td>1.91</td>
<td>1.48</td>
<td>0.21</td>
<td>0.65</td>
</tr>
<tr>
<td></td>
<td>0.30</td>
<td>2.23</td>
<td>3.39</td>
<td>2.65</td>
<td>0.82</td>
<td>0.48</td>
<td>0.52</td>
</tr>
<tr>
<td></td>
<td>0.35</td>
<td>2.29</td>
<td>3.56</td>
<td>-</td>
<td>0.82</td>
<td>0.55</td>
<td>-</td>
</tr>
<tr>
<td>AVA</td>
<td>0.25</td>
<td>2.20</td>
<td>0.91</td>
<td>0.52</td>
<td>0.45</td>
<td>0.24</td>
<td>0.58</td>
</tr>
<tr>
<td></td>
<td>0.30</td>
<td>1.36</td>
<td>2.29</td>
<td>0.78</td>
<td>0.58</td>
<td>0.58</td>
<td>0.58</td>
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<tr>
<td></td>
<td>0.35</td>
<td>1.33</td>
<td>1.87</td>
<td>-</td>
<td>0.69</td>
<td>0.45</td>
<td>-</td>
</tr>
<tr>
<td>SBR</td>
<td>0.25</td>
<td>1.29</td>
<td>3.04</td>
<td>1.52</td>
<td>0.86</td>
<td>0.17</td>
<td>0.52</td>
</tr>
<tr>
<td></td>
<td>0.30</td>
<td>1.20</td>
<td>1.94</td>
<td>1.45</td>
<td>0.14</td>
<td>0.14</td>
<td>0.14</td>
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<tr>
<td></td>
<td>0.35</td>
<td>1.13</td>
<td>4.43</td>
<td>-</td>
<td>0.79</td>
<td>0.34</td>
<td>-</td>
</tr>
<tr>
<td>PVA</td>
<td>0.25</td>
<td>1.91</td>
<td>2.42</td>
<td>2.81</td>
<td>1.91</td>
<td>2.42</td>
<td>2.81</td>
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<tr>
<td></td>
<td>0.30</td>
<td>3.23</td>
<td>3.24</td>
<td>3.07</td>
<td>1.55</td>
<td>2.03</td>
<td>2.20</td>
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<tr>
<td></td>
<td>0.35</td>
<td>1.91</td>
<td>3.23</td>
<td>-</td>
<td>1.58</td>
<td>2.30</td>
<td>-</td>
</tr>
<tr>
<td>EVA(II)</td>
<td>0.25</td>
<td>3.23</td>
<td>4.52</td>
<td>4.07</td>
<td>1.34</td>
<td>0.96</td>
<td>1.48</td>
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<tr>
<td></td>
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<td>3.20</td>
<td>5.14</td>
<td>5.14</td>
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<td>0.86</td>
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<td>3.81</td>
<td>-</td>
<td>0.86</td>
<td>1.00</td>
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<tr>
<td>V-V</td>
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<td>1.58</td>
<td>1.84</td>
<td>-</td>
<td>1.17</td>
<td>0.21</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>0.35</td>
<td>1.84</td>
<td>1.29</td>
<td>0.97</td>
<td>0.48</td>
<td>0.38</td>
<td>0.34</td>
</tr>
<tr>
<td></td>
<td>0.40</td>
<td>1.26</td>
<td>1.45</td>
<td>1.33</td>
<td>0.38</td>
<td>0.27</td>
<td>0.38</td>
</tr>
<tr>
<td>CONTROL</td>
<td>0.25</td>
<td>1.24</td>
<td></td>
<td></td>
<td>1.03</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(no latex)</td>
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<td>2.06</td>
<td></td>
<td></td>
<td>1.10</td>
<td></td>
<td></td>
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<td>2.88</td>
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<td>0.72</td>
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<tr>
<td></td>
<td>0.40</td>
<td>1.82</td>
<td></td>
<td></td>
<td>0.58</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Bond strength values for the latex-modified pastes (w/c = 0.30) determined by direct tension tests were dependent on the test procedure used. The maximum values obtained in the test where the paste was cast between the two end blocks varied from 0.96 to 3.02 MPa at 72 h for PVA and SBR respectively (l/c = 0.08). The corresponding bond strength of unmodified cement paste was 2.74 MPa. SBR was the only latex for which the bond strength exceeded the control paste value. Optimum drying conditions for latex film formation were not provided in this type of test. It is not known why SBR latex performed better than other latexes in this test. Drying specimens at 0% and 55% RH for various times did not improve the bond strength, as moisture loss due to drying and subsequent film formation was greatly hindered by specimen geometry.

The maximum bond strength values in the alternate type of direct tension test, where the latex-cement surface was allowed to dry at 55% RH before attaching the brass end block, were obtained for the EVA(II) system. These values increased from 1.03 to 1.71 MPa as drying time increased to
FIG. 3

Bond strength versus time for poly(ethylene-vinylacetate) copolymer (EVA(II)). Specimens cured at 100% RH until tested by overhanging beam method

16 h. No appreciable bond strength was recorded for unmodified cement paste tested in this manner. Internal stress developed during drying was sufficient to weaken the bond in these specimens.

**Bond Strength and Hydration Parameters**

There is no single relationship between bond strength and $w_n$ for all the systems. If complexes form due to latex-cement mineral interaction the significance of $w_n$ values may be questioned. The general tendency is, however, that bond strength increases with $w_n$ for each system.

There is also no unique relationship between the amount of heat developed at a given hydration time and bond strength for all the systems. The significance of the total heat values would also depend on the possibility of complex formation due to latex-cement mineral interaction.

**Cement Paste-Steel Interface**

A unique zone - approximately 50 µm thick - at the interface between cement paste and various substrate materials has been identified by several workers (11). The zone is porous and rich in CH. A detailed description of this interface region is given elsewhere (10,12). It is generally felt that the properties of this zone influence the behaviour of cement composites.

Steel and cement paste surfaces were examined in the SEM after the cement paste coating had released from the cement surface. Failure occurred within 5-10 µm of the steel surface in all cases, i.e., in the interface zone. The solids adhering to the steel surface at 24 h hydration appeared
very fine, with needle-like crystalline material consisting principally of C-S-H and CH. Photomicrographs of the SBR-cement system, (Fig. 4a) reveal needle-shaped crystals adhering to the steel surface. A small amount of ettringite was detected in some of the specimens. Latex film was found adhering to both the steel surface and hydration products attached to the steel in EVA(I) and (II), SBR, and PVA systems.

Latex-modified cement surfaces were generally covered by a thin layer of CH. This is shown in Fig. 4b for the EVA(I)-cement system. Large areas of fine material have been identified as CH. EDX analysis of these areas gives very high Ca/Si ratios. Clusters of needle-like C-S-H particles were observed in those areas where the CH layer was removed. These areas appear as a network of microcavities between large CH masses. Latex film was found around the C-S-H crystals, especially in EVA(I) and AVA-cements.

The solids adhering to the steel surface after 72 h hydration were generally C-S-H and CH, similar to the products present at 24 h. Large CH crystals (15-20 μm) were found in the PVA system (Fig. 4c).

Latex film was also found on the steel surface and around the solids attached to the steel in the EVA(II)-cement system (Fig. 4d). This may account for the relatively high bond strength in this system. It seems the latex film may act both as an adhesive and reinforcement of the porous interface layer.

Latex-cement surfaces were also covered by a layer containing CH and C-S-H. Numerous large CH crystals (5-10 μm) and plate-like CH crystals (10-100 μm) were found on PVA and AVA-cement surfaces respectively. Photomicrographs of the AVA-cement system (Fig. 4e and 4f), depict the growth of large CH crystals in the interface zone. CH crystal growth may be responsible for lower bond strength at 72 h.

Differences were observed in the morphology of the interface between unmodified cement paste and latex-modified cement paste. A large number of needle-like crystals (C-S-H and a small amount of ettringite) were observed in the interface zone in unmodified cement pastes at both 24 and 72 h. Needle-like crystals were sparse and indistinct at 24 h in the latex-modified systems (EVA(I) and (II), PVA, and AVA). Needle-like crystals (C-S-H and a small amount of ettringite) were abundant in the EVA(I) and (II) systems at 72 h (Fig. 4g) and not apparent in the PVA and AVA cement system. Needle-like morphology may be associated with higher bond strength of EVA(I) and (II). These crystals were also observed in the SBR and V-V modified systems at 24 h. The morphology changed, however, at 72 h.

Conclusions

1. A new technique - the overhanging beam method - for determining the development of bond strength at early ages between latex-modified cement and steel has been developed. The method accounts for the development of internal stress due to hydration of cement.
2. Latex addition generally increases the bond strength of cement paste to steel. All latexes tested except poly(vinylidene chloride - vinyl chloride) and poly(acrylic-vinyl acetate) copolymers increased the bond strength.
3. The maximum bond strength of latex modified cement was generally attained at about 24 h hydration. A substantial amount of CH formed due
FIG. 4
SEM micrographs of debonded surfaces:
(a) steel surface, SBR, 24 h hydration
(b) cement surface, EVA(I), 24 h hydration
(c) steel surface, PVA, 72 h hydration
(d) steel surface, EVA(II), 72 h hydration
(e) cement surface, AVA, 72 h hydration
(f) different magnification of (e)
(g) steel surface, EVA(II), 72 h hydration
to hydration of cement has been produced at this time.

4. Bond strength generally increases with non-evaporable water content in the first 24 h.

5. Bond failure of latex-modified cements generally occurs in a zone or interface layer 5-10 μm from the steel substrate surface.

6. The interface layer consists mainly of C-S-H and CH. Latex film in some latex cements can be detected in this layer. This film may contribute to increased bond strength.

7. There is a tendency for large amounts of CH to form in the interface zone rather than in the bulk of the cement system. Bond strength decreases at later ages, possibly due to growth of CH crystals present in the interface zone.

8. Caution should be exercised in using the estimation of total amount of CH as an indicator of the degree of hydration in latex-cement systems.

Acknowledgements

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References

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