BLISTERING OF EPOXY COATING ON CONCRETE FLOOR

H. Saricimen, K. Attar, M. Maslehuddin, I. Asfaha, M. Shameem, and M.S. Barry

Center for Engineering Research, Research Institute, King Fahd University of Petroleum and Minerals, P. O. Box: 1686, Dhahran 31261, Saudi Arabia.

ABSTRACT

The objective of this study was to determine the cause of partial debonding of anti-static epoxy coated floors in several rooms at a Precision Measurement Equipment Laboratory building in Dhahran. Analysis of concrete core samples retrieved from the floor slab showed carbonation and salt concentration in the surface layer of the concrete slab. Analysis also showed the presence of high concentration of cations, silicate and sulfate ions in the blister fluids. Moisture studies demonstrated the migration of moisture in the floor slab originating most probably from the groundwater. The results indicated that the number one cause of the blistering of epoxy coating on the floor slab was the upward migration of moisture in the concrete slab by osmotic and capillary forces.

KEYWORDS

Blistering; capillary rise; concrete; debonding; epoxy coating; osmotic pressure.

INTRODUCTION

Concrete floor slabs in a Precision Measurement Equipment Laboratory (PMEL) in Dhahran were covered twice in the past ten years with vinyl sheeting bonded by two-component polyurethane adhesive. On each occasion and specifically after the later, approximately 15 months prior to the time of this investigation, November 1995, some of the vinyl sheeting developed pinholes and cracks at several locations, small bubbles of brown liquid material were observed on the top surface of the sheeting, and swelling and debonding occurred in others. The vinyl sheeting was removed and replaced one year ago, by an anti-static epoxy floor after thorough cleaning and preparation of the concrete surface. Approximately four weeks after installation, bubbles or blisters appeared on the flooring and they continue to appear up to the day this investigation was made. The diameter of a single blister seldom exceeds 15 mm and thickness (height) is virtually undetected to the touch, as reported by the staff of PMEL. The recurrence of blistering has caused a delay in achieving full utilization of the role of PMEL. The objective of the study was to determine the cause of partial debonding of anti-static epoxy coated floors in several rooms at PMEL, so that mitigative measures can be undertaken.

EXPERIMENTAL WORK

The investigation consisted of the following tasks:

    (1)   Visual inspection
    (2)   Collection of concrete specimens and samples of blister fluid,
    (3)   Analysis of water-soluble components in concrete
           specimens,
    (4)   Analysis of inorganic species in the blister fluid,
    (5)   Monitoring of humidity and temperature in the rooms,
    (6)   Evaluation of engineering drawings, and
    (7)   Data evaluation and reporting.

Visual Inspection

The facility was inspected before the initiation of the study. The visual inspection revealed the following:

    (1)   Blistering was observed to varying degrees in three rooms designated as R-1, R-2 and R-3 (the calibration and repair section), while no visible blistering was noted in Room-4 (the store room of the equipment cleaning section).
    (2)   Rooms R-1 to 3 were warmer than the other rooms, Room-4 being the coldest.
    (3)   The surface layer above the big bubbles indicated loss in rigidity as noted by pushing down the bubble surface with fingers using minimum force.
    (4)   Pinholes drilled near some of the bubbles indicated that they contained a light-yellow or brownish and slightly viscous liquid.
    (5)   According to the information provided by the client, the epoxy floor system comprised of an epoxy primer, a highly conductive undercoat and a 2 mm thick epoxy resin based floor topping.

Sample Collection

The client designated R-1 where the coating on the floor is badly blistered for this study. Samples were collected from the floor concrete slab and from fluid inside the blisters for chemical analysis of inorganic constituents in the laboratory.

Collection of Concrete Specimens. Concrete core specimens, 75 mm ϕ, were drilled from three locations in R-1 using a diamond tipped core barrel. Two of the cores were taken from blistered locations on the floor. The third core was taken from a location where no blisters were observed. The cores were used for analysis of water soluble inorganic constituents of concrete.

Collection of Fluid Samples from Blisters. Samples of blister fluid were extracted from several blisters in three locations on the floor using a hypodermic needle and stored in sample bottles for analysis in the laboratory.

Chemical Analysis

Analysis of Concrete for Water Soluble Cl⁻, SO₄⁻ and OH⁻ ions and Carbonation. The core specimens were cut into thin slices at 0, 10, 20, 50 and 100 mm. Each slice was pulverized and mixed with hot distilled deionized water. The filtrate was analyzed for hydroxide (OH^-), chloride (Cl^-) and sulfate (SO_4^{--}) concentration. The results are presented in Tables 1 and 2.

The OH^- concentration was determined by titration of the filtrate against 0.01 M nitric acid. The Cl^- content was determined using a spectrophotometric method [1]. The SO_4^{++} concentration was determined using the spectrophotometric method [2]. The carbonation depth was determined by phenolphthalein solution test. Carbonation of the concrete was also tested by dissolving 1.0 gram of each of the ground core slices in 25 ml of 6 N HCl in a tarred beaker. The amount of carbon dioxide released by the reaction between concrete powder and acid was determined to show relative degree of carbonation in the concrete core specimens. The acid soluble calcium was also determined by Inductively Coupled-Atomic Emission Spectrometry (ICP-AES) for comparison. Results are presented in Table 3.

Analysis of Concrete for Water Soluble Cations and Silicon. The filtrates from concrete core specimens were analyzed by ICP-AES for sodium (Na^+), potassium (K^+), calcium (Ca^{++}) and silicon (Si^{4+}), and by Ion Chromatography (IC) for hydroxide (OH^-). The results are presented in Table 4.

Analysis of Blister Fluid. Samples of blister fluid were analyzed by the standard addition method using ICP-AES for Na^+, K^+, Ca^{++}, and Si^{4+}. The silicon content was calculated as the silicate (SiO_3^{--}) ion. The anions SO_4^{--} and Cl^- were also determined by IC analysis. The hydroxide was determined by titration with a standard solution of hydrochloric acid (HCl). The results of the analyses are presented in Table 5.

RESULTS
Visual Inspection
Examination of the core holes indicated that the concrete slab was 130 mm deep which was followed by a gravel bed of 35 mm. The gravel bed was placed on a polyethylene plastic sheet used to reduce the migration of groundwater in the concrete. Examination of the cores showed that the epoxy coating had disbonded from the concrete in the core specimens taken from the blistered locations, while the coating on the cores from non-blistered location was easily removed by applying a small pressure. The brown colored primer was observed to be stuck on the underneath surface of epoxy coating rather than on the concrete.

Concrete Analysis
Hydroxide, Chloride and Sulfate. The OH^-, Cl^- and SO_4^{--} concentrations in the concrete specimens, from the sound and blistered locations, is shown in Tables 1 and 2, respectively. The OH^- concentration and the pH increased with depth in all the specimens. These values were less at the surface than at other depths of more than 10 mm. The pH values varied in the range of 12.84 to 13.14 and 12.59 to 13.11 in the concrete specimens from blistered and unblistered locations respectively. This reduction in the alkalinity at the surface may be attributed to carbonation of concrete, which reduces the pH of the concrete by converting the $Ca(OH)_2$ to $CaCO_3$.

The chloride concentration, expressed as percentage by weight and mg/Kg of concrete, generally decreased with depth in all concrete cores. The chloride values varied in the range of 0.05 to 0.08% and 0.03 to 0.08% by weight of concrete in the specimens from

blistered and unblistered locations respectively. These values were three times the allowable level (threshold) of water soluble chloride ion concentration, 0.03% by weight of concrete, suggested by ACI 318 [3] at the surface. The chloride concentration was more or less similar to the threshold values at a depth of about 100 mm in the cores from no-blister location (Table 1), while it was about 1.7 times the threshold value, at the depth, in the cores from blister location (Table 2).

The sulfate concentration in the concrete specimens from locations with and without blisters also decreased with the depth (Tables 1 and 2). However, the gradient was not as steep as that observed in the chloride concentration. The sulfate values varied in the range of 0.04 to 0.06% and 0.047 to 0.060% by weight of concrete in the concrete specimens from blistered and unblistered locations respectively. The water soluble sulfate concentration was much less than the threshold value of 0.6% by weight of concrete, allowed by BS 8110 Part 124 [4].

The depth of carbonation in the concrete cores retrieved from both locations with and without blisters was determined to be 10 mm. This was supported by the phenolphthalein test, and low pH value and low level of soluble calcium in the surface layer of the concrete specimens, as shown in Tables 3 and 4.

Cations, Silicate and Hydroxide. The amounts of Na^+, K^+, Ca^{++}, Si^{4+} and OH^- in concrete specimens from blistered locations are presented in Table 4, in meq/Kg and mg/Kg of concrete, as a function of depth. The concrete cores from blister locations show similar depth profiles with respect to water soluble components. The sodium and potassium values are higher at the surface, while calcium and hydroxide values are lower. The sodium and potassium values varied with depth in the range of 1535 to 227 mg/Kg and 1920 to 507 mg/Kg of concrete respectively. The calcium and hydroxide values varied with depth in the range of 3180 to 8460 mg/Kg (159 to 423 meq/Kg) and 3916 to 7683 mg/Kg (230 to 452 meq/Kg) of concrete respectively. The calcium and hydroxide equivalent values (meq/Kg) are nearly identical indicative of calcium hydroxide.

The water soluble silicate concentrations were also high at the surface of the concrete specimens due most probably to silica filler in the primer used on the concrete slab before application of the epoxy coating. The silicate values varied in the range of 95 to 59 mg/Kg of concrete with the depth.

Blister Fluid
The results of inorganic analysis of fluid samples are presented in Table 5 in meq/L and mg/L. The results show that the fluids contained very high concentrations of Na^+ and K^+ in the ranges of 12,700 to 16700 and 16,000 to 25,900 mg/L respectively. The calcium content in the blister fluids was low. The calcium values varied in the range of 48 to 472 mg/L. The chloride concentration in the fluids was also low compared to sulfate values and ranged between 351 to 774 mg/L, whereas the sulfate content of the fluids was high in the range of 2400 to 6130 mg/L indicating that sulfate salts in the concrete were more soluble in the high pH fluid than chloride salts. The pH of the fluids was highly alkaline close to 14 as measured by IC technique.

Table 5 also shows the cation/anion balance of the blister fluids. It is evident from the table that the cation/anion balance of the fluid from blister No. 1 was satisfactory; while that of the other blisters was not. The imbalance indicates an excess of cations in the fluids from blisters 3 and 4. Screening of the samples did not show any other inorganic cations existing in solution. The difference can be attributed to the existence of organic salts in the blisters. A rough indicator for this assumption is the increase in the yellow color of the solution with the increase in cation/anion imbalance. The solution from blister No. 1 being colorless, becoming yellow in No. 3 and then darkens to yellow-orange in No. 4.

Moisture in Concrete. Following the drilling of cores, the core holes in the floor slab were dried for about two weeks and then they were covered and sealed to be air-tight. The core holes were covered for about a month, and then opened and checked for moisture build up inside. The examination showed that in two of the core holes, which were through the entire thickness of the slab at blistered locations, the humidity was 100 % since the walls of the holes as well as underneath of the covers were completely wet. The other core hole which was only halfway through the slab thickness at unblistered location, was dry and had no sign of moisture condensation inside the hole.

These results clearly demonstrated that there is definitely some kind of moisture movement in the floor slab. The moisture could be coming from the ground water under the floor slab. Another possibility for the source of moisture could be a leaking pipe underneath the building. It is not conceivable, however, to assume that the moisture condensation inside the core holes could be due to the original moisture entrapped in the concrete since under normal circumstances a concrete slab of six inches (150 mm) thickness should dry to an acceptable level in about six months if the slab is completely insulated from the ground water or floods [6].

DISCUSSIONS OF THE RESULTS
The results showed that the surface layer of the floor slab in R-1 carbonated to a depth of about 10 mm. This was confirmed by phenolphthalein and carbon dioxide tests, and supported by lower pH and water soluble calcium content in the surface layer compared to the bulk of the core specimens. Carbonation of concrete starts from the moment it is cast. Calcium hydroxide which is produced during hydration of cement with water reacts with carbon dioxide in the atmosphere to form calcium carbonate. It reduces the pH of the pore solution due to neutralization of calcium hydroxide in the concrete. Carbonation rate depends on the quality and mix design of the concrete as well as environmental parameters [10-11]. However concrete coatings is expected to slow down or completely stop the carbonation rate since they isolate the concrete surface from the environment. The salt concentration in the surface layer was higher by 73% for sodium, 172% for potassium, and 33% for chloride compared to the bulk of the concrete. Sulfate concentration was slightly higher (by 16%) in the surface layer compared to concrete at 100 mm depth. As mentioned before, the chloride contamination of the concrete slab was almost 3 times the allowable threshold value at the surface, whereas sulfate contamination was about 0.1 times the allowable threshold value. This was probably due to higher chloride contamination in the groundwater and higher migration of chloride ions in concrete compared to sulfate ions.

The results indicated moisture movement from the bottom of the concrete slab to the top due to most probably seepage of ground water. Thus, water soluble salts migrated and concentrated in the top layer of the concrete upon evaporation of moisture at the surface. Furthermore, the seeping water was mainly contaminated by chloride salts rather than sulfate.

The concentration of salt by evaporation at the surface might have started right after casting of concrete and continued until it dried completely. Drying of the original moisture of concrete is not expected to cause significant accumulation of salts at the surface. The main contribution to salt concentration in the surface layer could be due to migration of moisture supplied by a water source on the other side of the floor slab. The moisture observed in the core holes support this argument. After coating of the floor slab, the driving force for moisture movement in the concrete slab must have been provided by osmotic pressure. Concrete by virtue of being porous behaves as a semi-permeable membrane and allows osmosis to occur specially if differential of salt concentration finds time to develop in the concrete slab as in the present case [9].

The epoxy coating on the core specimen taken from the sound part of the floor could be easily removed by exerting very little force. This could be interpreted as inadequate surface preparation of the floor concrete slab before application of the epoxy coating system. However, even optimum surface preparation may not be enough to stop coating failure by blistering if there is entrapped water in the concrete either due to lack of drying in the first place, before application of the coating, or due to moisture originating from ground water on the other side of the concrete slab. Entrapped water in the concrete may eventually redeposit salts on the surface through capillary action within the concrete [9] and encourage development of osmotic pressure.

Poor surface preparation is one of the main causes of coating failure [5,9]. Surface preparation should eliminate all prior salt accumulation in the surface as well as it should provide enough roughness on the concrete surface for a strong bonding of the coating [5-7]. Dryness of concrete is also very important before application of coating. The actual drying rate of concrete depends on (a) moisture content, porosity, and temperature of concrete, (b) relative humidity in the surrounding air, and (c) the velocity of air flowing over the concrete [6,8]. Some of the primary coating properties that affect the compatibility with concrete surfaces include (a) tolerance to alkalinity, (b) moisture sensitivity, (c) cure shrinkage, (d) viscosity, and (e) permeability [6].

The high concentration of salts in the surface layer of the floor slab in the present investigation, due to prior evaporation or inadequate surface preparation or both whichever the cause may be, is an evidence of the presence of osmosis in the concrete. Osmotic pressure which is the cause of most of the blistering problems depends only on the quantity of dissolved substances not on their chemical nature. Osmotic blistering normally occurs on surfaces contaminated with salts. Added difficulty with concretes is that concrete is porous and contains numerous soluble salts by the nature of its constituents. When a concentrated solution forms at the interface between the coating and the concrete, the solution tend to draw moisture vapor through both the underlying concrete and the coating depending on their permeability [9].

The analysis showed that the fluids taken from the blisters on the floor in this investigation were very alkaline (pH = about 14) and highly concentrated in cations and anions. The fluids contained 8 to 10 times more sodium, 8 to 13 times more potassium, 43 to 206 times more silicate, and 4 to 10 times more sulfate than the surface layer of the concrete specimens. The chloride and calcium contents in the blister fluids were, however, less than in the concrete surface. The chloride and calcium contents were 0.43 to 0.96 times and 0.008 to 0.07 times those in the surface layer respectively. The high level of sodium and potassium in the blister fluids can be explained by osmotic and capillary migration of these cations to the surface of the concrete. The high silicate level in the fluids, however, can be attributed to leaching of water soluble silicates in the concrete and the silica based fillers in the epoxy coating and the primer. The cation/anion imbalance in the blister fluids is an indication of some of the cations dissolved from the coating. High level of sulfate in the blister fluids could be due to presence of water soluble sulfate compounds in the epoxy coating and primer. The results also confirm that most of the calcium in the concrete is insoluble to the blister fluid due to carbonation in the surface layer of the floor slab.

CONCLUSIONS

1. The floor concrete slab is carbonated to a depth of 10 mm due to the reaction between carbon dioxide of the air and calcium hydroxide in the concrete.

2. The top layer of the concrete slab had higher levels of sodium, potassium, silicate and chloride compared to the bulk of the concrete due most probably to the evaporation during drying of concrete before application of coating, and osmotic pressure and capillary action in the concrete after application of the coating.

3. The blister fluids were very alkaline and more concentrated in sodium, potassium, silicate and sulfate compared to the concrete slab.

4. The moisture tests in the core holes indicated water migration in the concrete slab due to probably ground water seepage or pipe leakage under the premises. The seepage water must have had chloride contamination rather than sulfate.

5. The blistering in the coating occurred most probably due to moisture movement from under side of the concrete slab to the top surface by evaporative, osmotic and capillary actions in the concrete slab. The osmotic pressure must have developed in the concrete slab due to build up of high concentrations of salts in the surface layer of the concrete slab and at the concrete/coating interface, carbonation and porous structure of the concrete.

6. The build up of high concentration of salts in the surface layer and at the interface could have happened due primarily to evaporation of seeping water, inadequate surface preparation, and inadequate drying of the concrete before coating application.

7. The moisture migration occurred under osmotic pressure and capillary action which most probably was caused by porous structure of the concrete, and high concentrations of salts at the top layer of the concrete slab and at the concrete/coating interface.

RECOMMENDATIONS

Several options can be considered for the repair of the damaged concrete slabs.

1. First thing to do is to identify the source of moisture and stop its contact with the concrete slab. Otherwise, as long as there is moisture available for migration in the concrete slab, there is no way of stopping the blistering of the coating no matter how appropriately the surface is prepared and how perfectly it is coated.

2. Live with the problem; cover the slab with a thick opaque plastic sheet, and anchor it at the corners of the room.

3. Remove the epoxy flooring and the carbonated and salt concentrated concrete and place a dense layer of epoxy mortar. The bond between the old concrete and the new mortar can be achieved by using an epoxy bonding agent.

4. Replace the entire old concrete in the floor slab and apply new floor system with proper insulation underneath to stop water seepage.

REFERENCES

1. Vogel, I. A. A. (1985), *Textbook of Quantative Inorganic Analysis*, 5th Edition, Revised by J. Basset, Longman, London, p. 754.
2. *Standard Methods for the Examination of Water and Waste Water*, 16th Edition, American Public Health Association, Washington, 1985.
3. ACI 318 (1993), Building code requirements for reinforced concrete, American Concrete Institute, Detroit.
4. BS 1881 Part 124 (1985), Testing of concrete, British Standards Institution, London.
5. Steele, J., (Nov. 1994), Testing adhesion of coating applied on concrete. Material Performance, pp. 33-36.
6. Harriman, L., (January 1995), Drying and measuring moisture in concrete, Part I. Material Performance, pp. 34-36.
7. Greenfield, T.K., (March 1994), Dehumidification equipment reduces moisture in concrete during coating application. Material Performance, pp.39-40.
8. Greenfield, T.K., (July 1994), Specifying coatings for concrete surfaces. Material performance, pp.3741.
9. Dively, R.W., (May 1994), Osmotic blistering of coatings and linings applied to concrete surfaces. Material Performance, pp.42-44.
10. Kudo, N. (1993). Corrosion and its prevention of reinforcement in concrete. *Corrosion Engineering*, vol. 42, pp. 565-581.
11. Hobbs, D.W. (1988). Carbonation of concrete containing pfa. *Magazine of Concrete Research*, vol. 40, No. 143, pp. 69-72.

Table 1. Chemical analysis of concrete at the locations of no blisters.

Depth (mm)	OH⁻, mM/L	pH	Chloride		Sulfate	
			% wt.	mg/Kg of concrete	% wt.	mg/Kg of concrete
0	39	12.59	0.08	800	0.06	600
10	111	13.04	0.06	600	0.06	600
20	129	13.11	0.05	500	0.06	600
50	118	13.08	0.05	500	0.05	500
100	122	13.09	0.03	500	0.04	400

Table 2. Chemical analysis of concrete at the locations of blisters.

Depth (mm)	OH⁻, mM/L	pH	Chloride		Sulfate	
			% wt.	mg/Kg of concrete	% wt.	mg/Kg of concrete
0	70	12.84	0.08	800	0.060	600
10	126	13.1	0.07	700	0.059	590
20	137	13.14	0.07	700	0.047	470
50	118	13.08	0.07	700	0.055	550
100	105	13.01	0.05	500	0.059	590

Table 3. Chemical analysis of concrete for carbon dioxide and acid soluble calcium at location with blister (B) and no blister (NB).

Depth (mm)	CO_2 (B) % by wt	CO_2 (NB) % by wt	CaO (B) % by wt	CaO (NB) % by wt	$CaCO_3$ (B) % by wt	$CaCO_3$ (NB) % by wt
0	19.0	30.4	33.4	31.4	43.3	69.1
10	30.0	33.1	44.8	41.4	68.1	75.3
20	25.9	25.9	42.7	43.1	59.0	58.9
50	26.0	22.6	43.6	39.2	59.1	51.4
100	27.4	26.3	41.4	41.8	62.3	59.7

Table 4. Chemical analysis of concrete for cations at location with blister (B) and no blister (NB).

Depth mm	Sodium mg/kg (B)	(NB)	Potassium mg/kg (B)	(NB)	Calcium mg/kg (B)	(NB)	Silicate mg/kg (B)	(NB)	Hydroxide mg/kg (B)	(NB)	pH (B)	(NB)
0	1,484	1,587	1,782	2,079	5,074	1,290	76	110	5,154	2,678	13.03	12.70
10	398	458	628	741	7,978	8,314	68	65	7,084	7,354	13.16	12.95
20	407	389	737	679	7,988	8,290	68	61	7,681	7,201	13.19	13.05
50	299	340	612	636	8,138	8,772	68	68	7,342	8,024	13.22	13.11
100	398	78	835	187	8,044	7,428	61	57	6,892	6,622	13.25	13.07

Table 5. Chemical analysis of blister-fluids.

Ions	Fluid #1 meq/L	mg/L	Fluid #2 meq/L	mg/L	Fluid #3 meq/L	mg/L
Sodium	554	12,800	564	13,000	725	16,700
Potassium	412	16,100	542	21,200	664	26,000
Calcium	1.23	24.5	1.71	34.2	11.8	236
Silicate	517	19,700	248	9,400	106	4,040
Chloride	11.6	411	21.8	774	9.87	350
Sulfate	121	5,800	49.9	2,400	128	6,130
Hydroxide	265	4,500	355	6,040	208	3,540
pH	13.42		13.55		13.32	
Sum of cations	967		1108		1400.8	
Sum of anions	915		675		451.9	
% difference	5.4		39.1		67.7	
meq/L of organic salt	0.052		0.43		0.95	