Analysis of Effect of Protective Coatings on the Rate of Corrosion in Reinforced Concrete Sewers: Microscopic Study

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Abstract. Microbial induced corrosion (MIC) in reinforced concrete (RC) sewers are increasing in occurrence and severity which is leading to significant economic losses. Being able to prevent or significantly reduce the rate of MIC in RC sewers will be of great help in finding a suitable sustainable solution. This study has investigated the effect of MIC; sulphide corrosion, on RC sewers through analytical techniques; scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX). The aim was to create a sustainable solution; protective epoxy coating (PEC), that will be capable of preventing or significantly reducing the MIC in RC sewers. The PEC based solution was able to significantly reduce the MIC in RC sewers. Furthermore, the test results also outlined that even in the coated sample, crown corrosion was more as compared to the corrosion of the submerged portion of the sample.

1. Introduction

Due to microbial induced corrosion (MIC) in sewers, billions of dollars per annum losses have been incurred worldwide on sewerage system (Wei et al. 2013; Zhang et al. 2008a; E hewayde et al. 2007); more than \$50 billion for rehabilitation purpose in Germany (Hewayde et al. 2006), almost \$500 million for treatment of corroded and degraded pipes in Los Angeles (Apté et al. 2015) and approximately 10% of the total sewage treatment cost for treating MIC of sewers in Flanders, Belgium (Zhang et al. 2008). MIC is an immense problem in RC sewers infrastructure due to complexity of multistage processes (Sulikowski & Kozubal, 2016; Islander et al, 1992) that occurs externally (sulphate attack) and internally (sulphide corrosion) (Parande et al, 2006; Ling et al. 2014; Jiang et al. 2015, 2016; Dong, Shi, & Liu, 2017). Soil containing high sulphate content attacks the surface of concrete externally while sulphate-reducing bacteria, below the water line, acted on the sewage internally (Kuliczowska, 2016; Ling et al. 2014), produce hydrogen sulphide (H₂S) which enters into the moisture layer and converted into sulphuric acid (H₂SO₄) (Sun et al. 2015; Parker, 1951) which attack the concrete matrix and continuously deteriorated the sewer internally (Y. Liu et al. 2015; Parker, 1947). Below is summarized sulphide corrosion phenomena is (Sharma et al., 2008; Wells et al. 2012):

RC sewers are susceptible to heavy corrosion due to the sulphate content (Meyer & Ledbetter, 1970; Gemert, 2001; Little et al, 2009) present in the sewage which ultimately compromise the integrity of structure and shorten the service life of sewers. The economy of the sewerage system adversely affected by the service life of RC sewers (O 'connell et al, 2010).

Various investigations at multi-stage of MIC has been done to understand the origin and prevention (Jiang et al. 2015; Grengg et al. 2018; Eštokova et al. 2012); mitigating the penetration of carbonation (De Muynck, Cox, Belie, & Verstraete, 2008; Islander et al. 1992), interrupting the growth of *Thiobacillus* species (sulphate-reducing bacteria) (Okabe et al. 2007), controlling the production of H_2SO_4 (Santo et al. 2011), intervene the sulphide production by injecting oxygen (Gutierrez et al. 2008) and also by using oxygen and caustic simultaneously (Lin et al. 2017).

Along with investigations on MIC, an extensive research has been undertaken to monitor the sulphide production; by inspecting the up take activity of H_2S by ANN (Artificial neural network) (Jiang et al. 2016), by non-destructive method (X. Sun et al. 2014), creating a model for sewer corrosion through field and theoretical observations (Wells & Melchers, 2015), and inhibition techniques for MIC; dissolving the iron salts in sewage for sulfide control (J. Sun et al. 2015; Firer, Friedler, & Lahav, 2008), use of crown spray (i.e. magnesium hydroxide slurry) to deactivate the sulphate-reducing bacteria and neutralize the acid (Jiang et al. 2014; (Sydney, Esfandi, & Surapaneni, 1996), removal of dissolved organic carbon for sulphide reaction control (Guang-Hao, Ho-Wai, & Ju-Chang, 2001), adding Hydrogen peroxide (H_2O_2) for sulphide control (Joseph et al. 2012), use of nitrate to reduce the accumulation of sulphide (Auguet et al. 2015; Y. Liu et al. 2015; Zheng et al. 2017), use of sulphate free coagulants in drinking water instead of aluminium sulphate (Pikaar et al. 2014) to mitigate the sulphide activity, use of nitrous acid to control sewer corrosion (X. Sun et al. 2015).

In addition to the mitigation and prevention techniques, researches have also been done to modify the RC sewers material to cope with MIC (Grengg et al. 2018; Nielsen et al. 2008; Abraham & Ali Gillani, 1999); RC sewers made with alumina cement have better resistance to corrosion as compared to ordinary cement (Saucier & Lamberet, 2009); Alexander & Fourie, 2011), acid resistant RC sewer pipes (Fourie & Alexander, 2009), use of inert material (PVC, FRP and HDPE) (Vahidi et al. 2016) as well as pipes urban water management by dislodging the chloride ion by modified cement (Park et al. 2014).

Besides the MIC mitigation techniques; corrosion controlling agents, treatments of sewage and changing in material properties of RC sewers, protective polymer coating has been found to be a barrier between the sewage chemicals and sewer material (Ng & Kwan, 2015; William Guan, 2001), more proficient and even can influence at micro level on the composition of sewer material (Ling et al. 2014; De Muynck et al. 2008; Fenner, 2000; Ohama, 1995). These coatings reduce the permeability for seepage (Herisson et al. 2016; Valix et al. 2012), decrease ingress of chloride and sulphides (Noeiaghahi et al. 2017; Munger & Vincent, 1999; Irfan, 1998; Pletcher & Walsh, 1993), have good chemical and abrasion resistance, exceptional bonding on concrete surface thus contending the corrosion of sewers (Grengg et al. 2018).

Current research focused on the durability and analysis of polyurethane coating against MIC using ASTM C1723 (2010), scanning electron microscopy (SEM) and energy dispersive X-ray analysis (EDAX) (Stutzman, 2001; Jana, 2006). Polyurethane has been found to be effective against the corrosive environment; (Vipulanandan & Liu, 2005; Guan, 2003) integrates anti-microbial additives and weaken the corrosive environment (De Muynck, De Belie, & Verstraete, 2009), exceptional bonding with concrete, operative safety, economic, environmental friendly as well as good chemical and abrasion resistance. (Vera et al. 2013; Mobin et al. 2012).

2. Experimental Program

RC sewer Samples were used in this research as well as sewage samples were also obtained from existing waste water streams to present the in-situ conditions. Sewer samples were placed in the sewage whose sulphate concentration was known, and then SEM and EDAX were performed to determine the durability of protective coating against MIC.

2.1. Materials

Sewer samples were procured from Public Health Engineering Department (PHED) Khyber Pakhtunkhwa (KPK), Pakistan, which were made according to ASTM C-76 standard. These sewer samples, having thickness of 1.5in and length of 8ft, were cut down into small pieces approximately 2-3ft each, as shown in fig.1, for testing and analysis purpose then these samples were divided into two categories; sample A and sample B. They were washed and stored in a dry, clean place.

Figure 1. Cutting of Precast Reinforced Concrete Sewer using cutting tool

PEC **Sika Seal-105-PK**, polyurethane protective coating, comprises of two parts; the grey powder and the white liquid, was used for coating the RC sewer samples. For the preparation of PEC, matrix was kept 1:4 in which one part was white liquid and 4 parts were grey powder as per the preparation instruction of the vendor. Both components were continuously mixed for 2-3 minutes until a smooth thick paste was formed as shown in fig.2. Epoxy coating was applied at different stages with thickness of 2 μm and as per the instructions of a coating applicator (Mobin et al. 2012).

Figure 2. Preparation of epoxy coating

Sewage was required to chemically attack the samples as in real conditions. Hence, it was collected from two different places; one sample was collected from Khwar, referred as “X” (locally used name for waste water stream) and the other was collected from Hayyatabad Canal located in industrial area, referred as “Y” as shown in fig.3.

Figure 3. Sewage samples X and Y respectively

Sewage samples were filtered through the simple filter paper to remove large suspended particles and then the sulphate content of the sewage samples was calculated using a mass spectrophotometer. The sulphate concentration of sewage sample X came out to be 50mg/L and for sewage sample Y, it came out to be 98mg/L. Sewage sample Y was used to achieve the maximum amount of chemical attacks in minimum possible time.

2.2. Sample preparation

2.2.1. Coating and immersion in sewage

For the application of PEC on samples; initially RC sewer sample B was taken from the two prepared samples A and B, then sprayed with water to make the surface damp before PEC. The coating was done in two phases on sample B; in first phase, single layer of coating was applied all over the sample with the help of brush and allowed to dry for 6 hours. In second phase, after 6 hours, a second coat was applied all over the sample and allowed to dry as shown in fig.4. (Vera et al. 2013; Mobin et al. 2012).

Figure 4. Coating of sample B

Sample A was first placed in sewage sample “Y” for 30 days and after 30 days of chemical attack, it was washed and dried. A single layer of coating was applied in the same manner as for sample B (Mobin et al. 2012; Grengg et al. 2018).

2.2.3. In-situ conditions

The samples prepared were subjected to chemical attack in a specially designed arrangement of plastic containers, containing sewage sample “Y”, sealed with plastic covers so that an environment akin to the actual conditions can be provided. Plastic containers were used because of their inertness with the sewage. The duration of chemical attack was 30 and 60 days.

2.2.2. Sample formulation for microscopic study

After immersion in sewage sample “Y”, sample was prepared for SEM as per the specification of ASTM E2809 (2015) and ASTM C1723 (2010) standard.

The surface of samples was made smooth using grinding machine. To make the corners of the samples already placed on a conductive tape, a silver coating was applied all over the sides of the samples. To remove the excess charge, the samples were sputtered in a gold sputtering machine. After all these steps, the samples were ready to be tested under scanning electron microscope (Jana, 2006; Stutzman, 2001).

2.3. Testing of samples

Studying the concrete microstructure of RC sewer through microscopic techniques; SEM, XRD, can enable us to identify the durability problems of concrete and to estimate its service life by the analysis of precipitation; ettringite ($\text{Ca}_6\text{Al}_2(\text{SO}_4)_3(\text{OH})_{12}\cdot 26\text{H}_2\text{O}$) (Vera et al. 2013; Mobin et al. 2012). ASTM C1723 (2010) outlines the standard guidelines for examination of hardened concrete using SEM and EDAX.

The microscopic study; SEM AND EDAX, was conducted in two stages on both samples; A and B, simultaneously. Stage:1, sample A (uncoated) and sample B (coated) were placed in the plastic containers with half-filled sewage “Y” in it and chemically attacked for 30 days simultaneously.

After 30 days, the microscopic study was carried out on both samples. Stage:2, sample A₁ (uncoated sample A was coated after 30 days) and sample B (same sample as used in stage-1) were chemically attacked for more 30 days by placing in plastic container having sewage sample “Y”. After 60 days, again the microscopic study was carried out on samples.

3. Analysis and Results

3.1. SEM after 30 days of first chemical exposure

SEM analysis was carried out using scanning electron microscope (JEOL, JED-2300, Japan). It can be observed from fig.5, that the crown portion of the uncoated sample A and coated sample B shows more formation of ettringite (Jiahui, Jianxin, & Jindong, 2006) as compared to portion of sewer submerged in the sewage. This is because the formation of Sulphuric acid (H_2SO_4) near the crown portion of the sewer is more.

Also, the ettringite formation in the crown portion of sample A was much more as compared to the crown portion of the coated sample B as shown in fig.6 because the sample A was uncoated, and the concrete was exposed to the reaction taking place in the sewer.

Figure 5. SEM images (after 30 days) of crown (a) and submerged portion (b) of uncoated sample A

Figure 6. SEM images (after 30 days) of crown (a) and submerged portion (b) of coated sample B

3.2. EDAX after 30 Days of First Chemical Exposure

EDAX was performed simultaneously with the SEM. The graphs clearly show the peaks of precipitations of corrosive components; sulphur peak is more in the crown portion of sample A (uncoated) as compared to submerged portion because the sulphuric acid formation is more in the crown portion as shown in fig.7.

The graphs of sample A show the highest peak of calcium (Ca) because sample is uncoated and calcium is the main component of concrete/cement. Also, the gold (Au) peaks were visible because the samples were spluttered in a gold sputtering machine.

The sample B (coated) show relatively low peaks or negligible peaks of sulphur both for crown and submerged portions because of the coating as shown in fig.8. Also, the silicon peak was observed because it is the main constituent of epoxy coating.

Figure 7. EDAX graphs (after 30 days) of (a) crown and (b) submerged portion of sample A

Figure 8. EDAX graphs (after 30 days) of (a) crown and (b) submerged portion of sample B

3.3. SEM after 60 days

The crown portion of sample A₁ showed more ettringite formation as compared to the submerged portion of the sewer as indicated by fig.9.

Also, the crown portion of coated sample A₁ showed more ettringite formation as compared to the crown portion of sample B (coated from the beginning; since 60 days) as shown in fig.10, because the sample A₁ was initially left uncoated and the reaction already took place in first 30 days of immersion in sewage sample “Y”.

Figure 9. SEM image (after 60 days) of crown (a) and submerged (b) portion of sample A₁

Figure 10. SEM (after 60 days) images of crown (a) and submerged (b) portion of sample B

3.4. EDAX after 60 days

After 60 days, the Sulphur peak of sample A₁ is lower than that of sample A, showing the decrease in corrosion rate because of the epoxy coating as illustrated in fig.12. Visible peaks of gold (Au) and silicon (Si) are because of gold sputtering and epoxy coatings of the sample. The crown and submerged portion of sample B show low peaks of the sulphur because it was epoxy coated as shown in fig.12. The silicon (Si) and gold (Au) peaks appeared because of the epoxy coating and gold sputtering of sample.

Figure 11. EDAX graphs (after 60 days) of crown (a) and submerged (b) portion of sample A₁

Figure 12. EDAX graphs (after 60 days) of crown (a) and submerged (b) portion of sample B

5. Conclusion

Based on the results from SEM and EDAX analysis of RC sewer, the following conclusions can be drawn:

- After 30 days of 1st chemical attack, the sample A (uncoated) shows high peaks of corrosive products as compared to sample B (coated).
- After 60 days of chemical attack, sample A₁ shows less surface precipitation; Sulphur content as compared to sample A because of application of epoxy coating but greater corrosive products than sample B, showing that corrosion had already started in this sample during the initial 30 days when it was left uncoated.
- The sample B has minimum rate of corrosion when compared to sample A and A₁ at both stages; stage:1 and stage:2, because of the application of epoxy coating over it.
- The epoxy coatings increase the service life of the sewers by enervating the corrosive environment moreover there is a likelihood that rehabilitated corroded sewers can be reused by applying epoxy coatings over it.
- Because of the limited resources, the experiment was carried out using a standing sewage sample, but It is expected that the gravity flowing sewage, as in actual field conditions, increase the rate of corrosion as compared to the samples placed in standing sewage.

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