



# A study of the effects of filler particles on the degradation mechanisms of powder epoxy novolac coating systems under corrosion and erosion



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## ABSTRACT

Electrochemical impedance spectroscopy (EIS) tests were conducted on powder epoxy novolac (PEN) coating systems with different loadings and types of filler particles. The performance of the coatings was examined in an aqueous corrosive environment with a chloride concentration of 2000 ppm, pH of 3.5, temperature of 60 °C, and saturated with carbon dioxide (CO<sub>2</sub>). The coating's performance under the influence of grit blast erosion was also studied. Equivalent circuit models were proposed to study the degradation mechanisms of the coating systems under the synergic effect of corrosion and erosion. Coating systems with a low amount of filler particles provided the best protection against corrosion in the testing environment, while systems with an increased amount filler particles provided the best erosion resistance. Platy shaped filler particles were found to be the best in reinforcing coating's resistance against both corrosion and erosion.

## 1. Introduction

The protection of the internal surfaces of oil and gas pipelines in marine environments is of great importance. Pipelines are usually made of carbon steel because of its low cost and wide availability [1]. During oil and gas production, these pipelines are exposed to corrosive environment that include the presence of CO<sub>2</sub>, hydrogen sulfide (H<sub>2</sub>S), oxygen, and seawater [2–5]. In the Middle East, this corrosive environment is even more severe since the elevated ambient temperatures increase the rate of corrosion (the reaction rate doubles for every increase in 10° C [6]).

In addition to corrosive factors, abrasion and solid particle erosion should be considered in the protection of pipeline internal surfaces. When the formation strength of an oil/gas reservoir becomes lower, more sand is produced along with the oil and gas [7]. This mixture of sand, oil, and water flowing through the pipeline causes material degradation, especially in areas where there are changes in pipeline configurations such as: sudden expansions/contractions in pipe diameter, areas of cavitation, areas with pipe wall imperfections or protrusions, and joints [2,8,9]. Erosion and corrosion are often synergic processes leading to a very rapid failure of pipelines [2,10].

Organic coatings have been applied to internal pipeline surfaces for more than 60 years. In addition to preventing internal pipeline corrosion, the internal coating can also improve hydraulic efficiency of the pipelines and mitigate deposition of asphaltene. The base of the internal coating materials can be epoxy, phenolics, novolacs, nylon, or

urethanes. Field data have shown that the pipelines are well protected by epoxy coatings for more than 30 years under high temperature (more than 100° C), high pressure (100 psi), and presence of H<sub>2</sub>S (51 ppm) [3,4]. However, traditional organic coatings do not perform well against erosion due to their low erosion resistance. Abrasive wear and solid particle erosion caused mechanical damage to organic coatings, and a reduction in barrier properties of eroded coatings have been observed in a 0.3 wt% Na<sub>2</sub>SO<sub>4</sub> solution [11]. Experiments have shown that, by adding filler particles such as aluminum oxide, silica, and silicon carbide to polymer coatings, the hardness and wearing resistance of the coating systems increased [12–14]. Researchers had also found that, by adding platy shaped impermeable fillers such as talc and mica to polymer coatings, the permeability of the coating decreased and thus improved the corrosion protection of the coatings [15–17].

Despite of the widespread use of filler-reinforced polymer coating systems, not enough work has been done on the specific roles fillers play when the coatings were exposed to an erosive-corrosive environment. In this paper, electrochemical impedance spectroscopy (EIS) was applied to examine the degradation process of organic coating systems under solid particle erosion and constant immersion in a corrosive electrolyte. To study the role of the filler particles, two experimental coating systems with different amounts of filler particles were compared to a commercial coating system.

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**Table 1**  
Coating information.

Coating ID	Filler Area fraction (%) <sup>a</sup>	Filler volume fraction <sup>a</sup>	PLS Volume	SSH Volume
EL	2.5	2X <sup>b</sup>	X	X
CP	11	12X	6X	6X
EH	9	16X	15X	X

<sup>a</sup> Both filler PLS and SSH are included.

<sup>b</sup> X is an unknown constant.

## 2. Experiments

### 2.1. Materials and coatings

Organic coating systems composed of a powder epoxy novolac (PEN) polymer matrix and two types of fillers were applied to the steel panels by coating supplier NOV Tuboscope™. Information concerning the coatings is shown in Table 1. The approximate filler area fraction was calculated by an image processing software ImageJ [18]. Fig. 1 shows the SEM images of the surface of the three types of coatings. Filler particles can be observed in the SEM images marked with circles. The two types of fillers are different in size and material. The first type of filler is a platy-shaped, large-sized (15–20 μm in diameter) particles made from soft mineral (either a magnesium silicate, talc, type mineral or a phyllosilicate, mica, type mineral). This first type of filler will be labelled PLS (platy, large, soft), see Fig. 1. The second type of filler is a spherical-shaped, small-sized (5–10 μm in diameter) particles made from a hard mineral (a silica or an alumina). This second type of filler will be labelled SSH (spherical, small, hard), see Fig. 1. Experimental PEN coating systems with different amounts of filler (which will be called coating EL (for low) and coating EH (for high)) were tested to compare their performances with that of a commercial PEN coating system with a standard amount and type of fillers (which will be called coating CP) (see Fig. 1). In each coating systems, filler particles and polymer powder were mixed at high speeds in a shear mixer, which dispersed the particles uniformly in the polymer matrix. The powder coatings were applied by warm extrusion on steel panels that were preheated to 275°–375° F. After coating application, the coated panels were heated to 400°–500° F for period of 1–4 h for the crosslinking of the thermoset polymer coatings. The dry film thickness of the coating systems was 8–12 mils. Four replicas of each coating systems (12 specimens in total) were tested.

The steel substrates were 4" by 6" panels made of ABS DH36/AH36 steel. The elemental composition of the steel is presented in Table 2. The steel substrate was blasted with aluminum oxide according to NACE No. 1/SSPC-SP5 standard [19] to obtain a white metal blast surface before the application of the coating systems.

### 2.2. Electrochemical testing and coating characterization

The testing environment was designed to mimic the anticipated

exposure in the field (in the absence of H<sub>2</sub>S). The field conditions were provided by Qatar Gas Company from the natural gas extracted from their Ras Laffan site in Qatar. The 12 coated specimens were immersed in an aqueous electrolyte with chloride concentration of 2000 ppm, pH of 3.5, temperature of 60 °C, and CO<sub>2</sub> saturated. HCl and NaCl were used to adjust chloride concentration and pH of the electrolyte. CO<sub>2</sub> was industrial grade 99.5% purified UN1013. A 50L water bath was used to maintain constant immersion temperature. The specimens underwent 50 weeks of constant immersion. Electrochemical tests were conducted periodically once a week during the long-time immersion to monitor the corrosion resistance of the coating systems. After 50 weeks of constant immersion, all the coated panels were eroded to an average thickness of 3 mils by Clemco® Pulsar III suction blast cabinet using Ballotini® Impact glass beads at 90° impinge angle. The thickness to which coatings were eroded was chosen because previous research has shown that electrochemical response of epoxy coatings only changed when the coating thickness decreased to 3 mils [20]. After erosion, the specimens were immersed again in electrolyte for 4 more weeks, and electrochemical tests were conducted periodically during immersion to monitor the coating degradation.

Gamry® Reference 600 Potentiostats were used to conduct electrochemical experiments. The EIS tests were carried out at open circuit potential; the applied frequency range was 10<sup>5</sup>–10<sup>−2</sup> Hz with a signal amplitude of 10 mV. The coated specimens served as working electrodes with immersion surface area of 173 cm<sup>2</sup>. A graphite rod was employed as a counter electrode. Saturated calomel reference electrodes (SCE) were used in all the electrochemical tests. For electrochemical experiments carried out in the heated water bath, a salt bridge (filled with saturated potassium chloride) was attached to the reference electrode so that SCE could work in ambient lab temperature. The EIS measurements on eroded specimens were carried out only on eroded part of the coating surfaces, which was 2.0 cm<sup>2</sup>. The eroded specimens were tested in a smaller electrochemical cell, in which the electrolyte used was the same as in immersion tests; however, the electrolyte was at ambient lab temperature during the measurements. Since the tests lasted about 2 h, we believe that the lower temperature did not have a significant impact on EIS response. The presented EIS results were area normalized for analysis. All measured values of potential are reported vs. SCE. The EIS data were analyzed by Gamry Echem Analyst™ and Scribner Associates Inc. Zview® software.

FEI Quanta 200 Environmental SEM, and Zygo NexView™ 3D optical profilometer were used to monitor and characterize the coating surface throughout the testing regime. Cross sections of the coatings before and after erosion were prepared in the following way: the PPN coatings and 1 mm thickness of steel substrate were sliced off the coated specimens by a precision saw. The coated slices were immersed in liquid nitrogen for 1 min and then bended by pliers. The coatings would crack thus creating cross sections which were then captured by SEM.

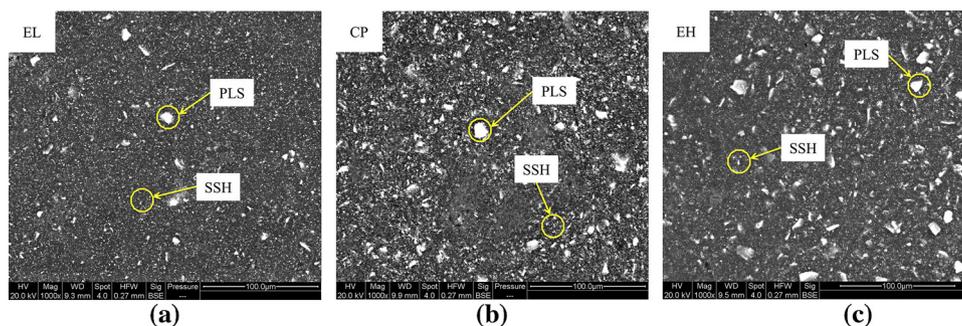


Fig. 1. SEM images showing representative distribution of filler particles PLS and SSH in coating systems EL, CP, and EH (electron accelerating voltage 20 keV).

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