



Comparison of Coatings Adhesion Performances for Buried SS316L Pipelines

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ABSTRACT

Six polymeric coatings viz. 3LPE, 3p/2p CAT, PU, VE, LE and HSS have been selected and each type of coating has been applied separately on SS316L pipe external surface. The test samples have been subjected to adhesion tests by the peel strength and pull-off methods, before and after the hot water Immersion (HWI) test for 100 days. After completing the test, the peel strength values of 3LPE, 3p/2p CAT, VE, HSS are found about 10.9, 1.8, 0.35, 6.8 N/mm respectively, whereas the pull-off adhesion values of PU, LE are found about 10.77, 22.7 N/mm² respectively. The experimental results are analysed, and the adhesion performances of the coatings are compared based on the dimensionless number termed as the "Coating Bond Index" in this study. The relative rankings of the adhesion performances for these coatings are evaluated in determining coatings suitability for buried SS316L pipelines in the petroleum, petrochemical and natural gas industries.

Keywords: SS316L pipes, Polymeric coatings, HWI test, Peel strength and Pull-off methods, Adhesion performances.

INTRODUCTION

The petrochemical products are transported via buried SS316L pipelines. These pipelines are installed mostly buried and marginally overground. Overground SS316L pipelines do not need corrosion protection due to 1-3 nm (nanometre) thick chromium oxide (Cr₂O₃) passive layer^{1,2}. In buried condition, uncoated SS316L pipeline is excellent in high resistivity soil with a good drainage system³, but uncoated SS316L pipeline is uncertain in low resistivity soil with chlorides ions (Cl⁻) and a

deficient in oxygen⁴. Cl⁻ ions destroy passive layer and lead to form corrosion pits on SS316L surface⁵. Pits are covered by the porous corrosion products and remain undetectable until leaks⁶. Localized pitting corrosion penetrates rapidly to the thickness of SS316L pipeline and perforates pipeline within a few days⁷. The mechanism of pitting corrosion on buried uncoated SS316L pipeline is shown in Fig. 1 a), b). It is, therefore, imperative to apply external polymeric (organic) protective coatings on buried SS316L pipelines for safe transportation of the petrochemical products.



A thorough literature survey including analysis of research papers reveals that so far, most of the studies done on polymeric coatings focused primarily on the analysis of coatings performances and suitability for buried carbon steel pipelines. Many researchers have been devoted research efforts to the study of metallic coatings and organic coatings on SS316L substrates for marine environments or to the study of coatings to prevent pitting corrosion of SS316L under thermal insulation. But, to date, less attention has been paid to the research on coatings performances and suitability for buried SS316L pipelines and there is a research gap, which remains unanswered in this field. To address this outstanding gap, in the present study, six external polymeric coatings commonly used for buried carbon steel pipelines have been chosen for buried SS316L pipelines and are applied separately on SS316L pipes. The test samples have been subjected to the adhesion tests, before and after the HWI test.

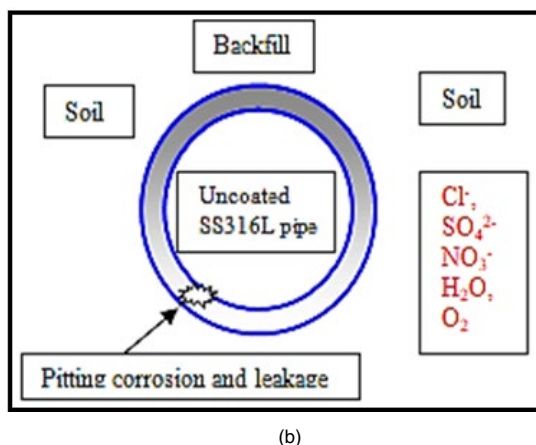
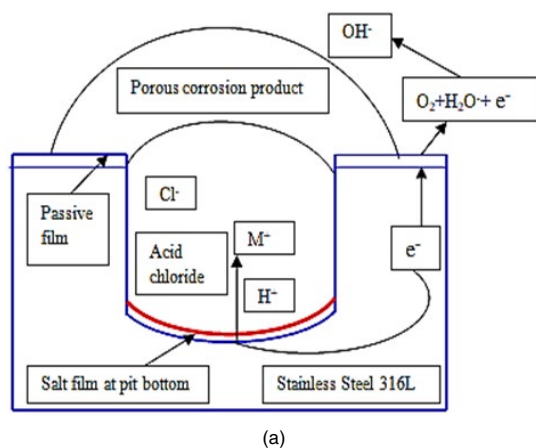


Fig.1 a), b). Schematic diagram of pitting corrosion in buried SS316L pipeline

MATERIALS AND METHODS

Chemical composition analysis and corrosion tests of SS316L

The test samples are drawn from 4-inch SS316L pipes for chemical composition analysis and corrosion tests. Cyclic potentiodynamic polarization test⁸ has been conducted at 25°C using three-electrode system within a glass cell filled up with 3.5% sodium chloride (NaCl) solution according to ASTM Standard Test Method G61⁹. Critical Pitting Temperature (CPT) test has been conducted at 22-24°C in reagent grade 6% ferric chloride (FeCl₃) solution as per ASTM Standard Test Method G48¹⁰ to determine minimum temperature to cause initiation of pitting corrosion.

Surface preparation of SS316L pipes

The external surfaces of 4-inch SS 316L pipes are prepared by fused alumina fine particles for a high level of adhesion¹¹. Surfaces are made dry and free from contamination prior to coating. Fused alumina particles, original pipe surface and pipe surface after blasting are shown in Fig. 2 a), b), and c) respectively.

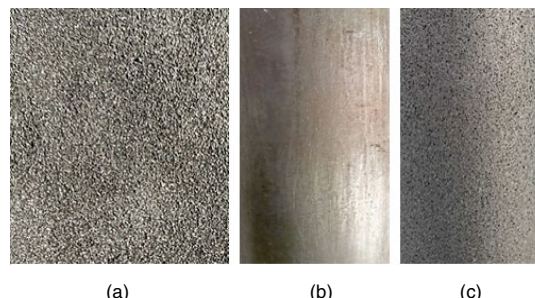


Fig. 2 (a). Fused alumina fine particles, (b) original pipe surface, (c) pipe surface after blasting

Types of polymeric coatings

Six types of generic polymeric coatings have been selected and each type of coating has been applied separately on each 4-inch SS316L pipe external surface after surface preparation. The types of coatings are as follows:

- (1) 3Layer Polyethylene (3LPE)
- (2) 3ply/2ply Cold Applied Tape (3p/2p CAT)
- (3) Solvent free liquid applied Polyurethane (PU)
- (4) Non-crystalline Visco-Elastic polyolefin with polymeric tape outer wrap (VE)
- (5) Solvent free Liquid applied Epoxy (LE)
- (6) Polyethylene based Heat-Shrinkable Sleeve (HSS).

A brief description of each coating system is given below:

3Layer Polyethylene (3LPE)

This anti-corrosion system consists of a three-layer coating system. First layer is a high-performance fusion bonded epoxy primer (FBE, minimum 200 microns) followed by a copolymer adhesive second layer (a maleic anhydride grafted polyethylene compound, about 200-250 microns) and third layer is an outer layer of high-density polyethylene, HDPE (about 2400-2600 microns) with UV stabilizers, which provides tough, durable protection. This coating system is applied in the coating plant only. This coating system provides corrosion protection of buried pipelines at operating temperatures from -40°C to 80°C.

3ply/2ply Cold Applied Tape (3p/2p CAT)

This is a cold applied multilayer coating system consisting of a liquid adhesive primer (butyl rubber, about 80-100 microns), 3ply anti-corrosion inner layer (a combination of HDPE and LLDPE co-extruded coating, tape thickness about 800 microns) and a mechanical outer layer (MDPE with UV stabilizers, tape thickness about 500 microns). Applied thickness of 3p/2p CAT is about 2500 microns or more. This coating system is applied in the coating plant and in the field. This coating system provides corrosion protection of buried pipelines at maximum operating temperature of 60°C.

Liquid applied Polyurethane (PU)

This is a thermoset aromatic polyurethane, which is a reaction product between a polyol (resin) and a diphenylmethane diisocyanate (MDI, activator). Liquid applied PU coating is solvent free (100% volume solid). Applied thickness of PU is about 1000-1500 microns in a single coat. This coating system is applied in the coating plant and in the field. This coating system provides corrosion protection of buried pipelines at operating temperatures from -20°C to 80°C.

Non-crystalline Visco-Elastic Polyolefin with polymeric tape outer wrap (VE)

This is a cold applied non-crystalline, non-cross-linked, non-reactive poly-isobutene wrap (about 2000-2500 microns thick) with a direct bond to the substrate. Over poly-isobutene wrap, an ultraviolet resistant flexible polyvinyl chloride (PVC, about

500 microns thick) tape coated with a rubber resin adhesive is applied. This coating system is applied in the coating plant and in the field. This system provides corrosion protection for buried pipelines at maximum operating temperature of 60°C.

Liquid applied Epoxy (LE)

This is a high-build solvent free (100% volume solid) two-component (part-A and part-B) novalac based epoxy. Applied thickness of LE is about 800-1500 microns in a single coat. This coating system is applied in the coating plant and in the field. This coating system provides corrosion protection of buried pipelines at maximum operating temperature from -20°C to 80°C.

Heat-Shrinkable Sleeve (HSS)

This is a wrap-around heat-shrinkable sleeve, which replicates the plant applied 3LPE coating. First layer is solvent free two component liquid epoxy primer (about 200 microns), second layer is co-polymer hot melt adhesive (about 200 microns), and third layer is radiation cross-linked HDPE with UV stabilizers (about 2000-2500 microns thick). This coating system is mostly applied in the field for pipe-to-pipe field-joints and is sometimes applied in the coating plant for pipe bend joints. This coating system provides corrosion protection of buried pipelines at maximum operating temperature of 80°C.

Preparation of test samples-thickness measurements and holiday tests

The test samples are prepared after cold cutting pipe segments from main 4-inch coated pipes for adhesion tests. The thickness measurement of each coating has been performed by digital thickness gauge. The entire surface of each coating has been subjected to holiday test applying a range of voltages based on the type of coating and thickness as per NACE Standard SP0274¹² to ensure each coating surface free from holidays (pinholes or defects).

HWI test of coatings

The purpose of HWI test is to subject coated test samples to the effects of long-term water exposure at a defined temperature and for a specified duration. After completion of the test, the samples are tested on their physical properties like adhesion by the peel strength or pull-off method. HWI test method measures the loss of adhesion strengths of the coatings due to water immersion and the results are compared to the coatings adhesion performances. Two sets of coated test samples are prepared because the adhesion tests are destructive in nature.

For the first set of test samples and prior to the HWI test, the adhesion strengths of 3LPE, 3p/2p CAT, HSS, VE have been measured by the peel strength methods in accordance with ISO 21809-3¹³ and the adhesion strengths of LE, PU have been measured by the pull-off methods in accordance with ISO 4624¹⁴.

The second set of test samples have been subjected to the HWI test. The test samples cut edges are sealed with silicone sealant to prevent water ingress from the sides of the samples. After curing the sealants, the test samples are fully immersed under the water in a vessel. The HWI test has been performed placing the vessel in a thermostatically controlled oven to maintain test temperatures about $55 \pm 5^\circ\text{C}$ for a period of 100 days as per ISO 21809-3. Fig. 3 a), b), c) show the thermostatically controlled oven and test samples. After completion of 100 days, these test samples are removed from the heated vessel and allowed them to cool to $23\text{-}25^\circ\text{C}$. The adhesion strengths of 3LPE, 3p/2p CAT, HSS, VE and LE, PU have been measured by the peel strength and pull-off methods respectively.

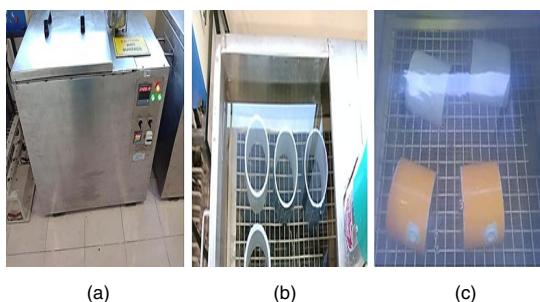


Fig. 3 (a). A thermostatically controlled oven, (b) & (c). Test samples of coating materials

Adhesion test by peel strength method

This test method measures the peel strength (peeling force per unit width) between the coating and the rigid metal substrate. The peel strength tests of 3LPE, 3p/2p CAT, HSS, VE coatings on SS316L substrates have been performed with a universal tensile testing machine, Tinius Olsen (Model No. H10KT)¹⁵. A schematic diagram of peel strength method and a universal tensile testing machine are shown in Fig. 4 a) and b) respectively.

A sample coating strip is cut in the circumferential direction of test sample, measuring 160 mm long and 25 mm to 50 mm wide. The cut end of the strip is secured to the gripping jaw of the tensile testing machine. The strip is peeled with a peel rate of 10 mm/min perpendicular to the

test sample axis. Each test has been performed at $23\text{-}25^\circ\text{C}$. During testing, the peel-force (vertical y-axis) versus length (horizontal x-axis) graph is generated for each coating of the three samples. From this graph, the peel strength of a sample has been evaluated from the average force and the arithmetic mean over 100 mm length using the Equation [1]. The peel strength of each coating has been computed from the arithmetic mean value of the three samples.

$$P_{\text{peel}} = F/w \quad (1)$$

Where,

F=Average force to peel the coating in Newton, N (or Pound, lb).

w=Width of the coating in millimeter, mm (or inches, in).

P_{peel} =Peel strength achieved at failure in N/mm (or lb/in).

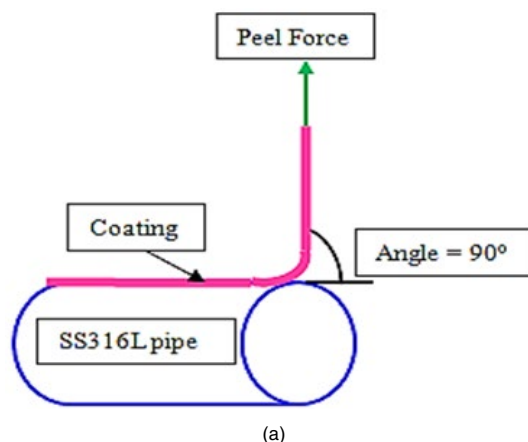


Fig. 4 a). Schematic diagram of peel strength method, b) A universal tensile testing machine

Adhesion test by pull-off method

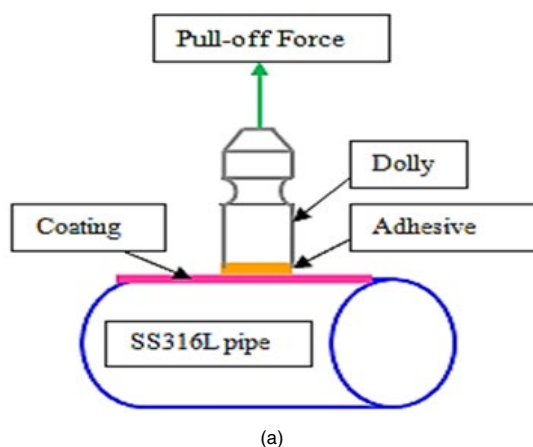
This test method covers a procedure for determining the pull-off adhesion strength of a coating on rigid metal substrate. The pull-off adhesion tests of LE, PU coatings on SS316L substrates have been performed with a portable adhesion tester, DeFelsko (Model No. PosiTest AT-A20)¹⁶. A schematic diagram of pull-off method and a portable adhesion tester are shown in Fig. 5 a) and b) respectively.

The rigid flat face of each steel dolly of 20 mm diameter is bonded directly to the surface of the test sample using epoxy adhesive at one end. The other end of steel dolly facilitates for connecting the portable adhesion tester. After curing of the adhesive, each dolly is pulled-off by the portable adhesion tester applying tensile stress in a direction perpendicular to the surface of the test sample. Each test has been performed at 23-25°C. The force required to detach each dolly from the coating is measured. The pull-off adhesion strength of a sample has been evaluated from the tensile stress using the Equation [2]. The pull-off adhesion strength of each coating has been computed from the arithmetic mean value of the three samples.

$$P_{\text{pull}} = 4F/\pi D^2 \quad (2)$$

Where,

F=Force required to detach the dollies in



Newton, N (or Pound, lb).

D=Diameter of the original surface area stressed in millimeter, mm (or inches, in).

P_{pull} =Pull-off adhesion strength achieved at failure in N/mm^2 (or Psi).

RESULTS AND DISCUSSION

Chemical composition analysis and corrosion tests of SS316L

The chemical composition analysis and corrosion tests results of SS316L are reported in Table 1. The chemical composition analysis conforms to ASTM Standard Specification A312¹⁷. The cyclic potentiodynamic polarization plots of test samples are shown in Fig. 6. From the polarization plots, E_{PIT} values are evaluated. It is observed from the polarization plots that from E_{PIT} , the current increases sharply indicating transition to transpassive corrosion, which is a state of rapid dissolution of metal resulting in significant pitting corrosion and the destruction of protective Cr_2O_3 passive film on SS316L surface in the presence of Cl^- ions. The significant pitting corrosion has been observed under Scanning Electron Microscopy (SEM) examination after cyclic potentiodynamic polarization test and is shown in Fig. 7. CPTs have been found to be 15-16°C, which indicate that SS316L is susceptible to localized pitting corrosion in chloride bearing environments much below room temperature.



Fig. 5 a). Schematic diagram of pull-off method, b) A portable adhesion tester

Table 1: Chemical composition analysis and corrosion test results of SS316L

%C	%Mn	%S	%P	%Si	%Cr	%Ni	%Mo	N(ppm)*	%Fe	E _{pit} (mV) vs. SCE **	CPT (°C)
0.021	1.24	0.005	0.042	0.26	16.32	10.05	2.03	380	Balance	361	16
0.022	1.25	0.004	0.043	0.27	16.08	10.06	2.03	395	Balance	363	15
0.018	1.27	0.004	0.043	0.20	16.29	10.05	2.04	440	Balance	380	16

(ppm)* - parts per million

SCE ** - Saturated Calomel Electrode as reference electrode

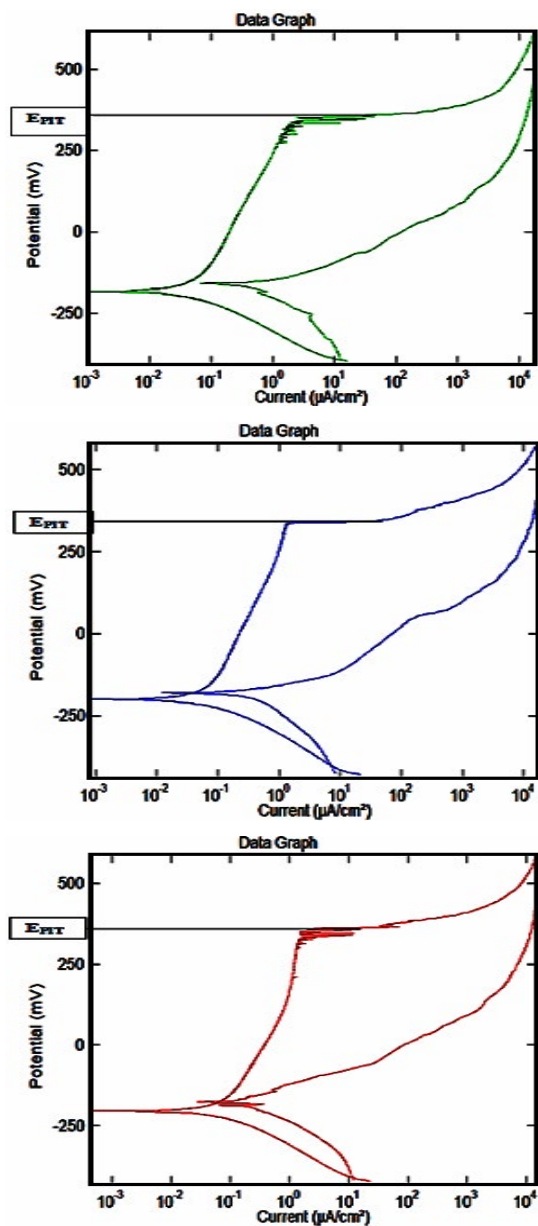


Fig. 6. Cyclic potentiodynamic polarization plots of SS316L test samples

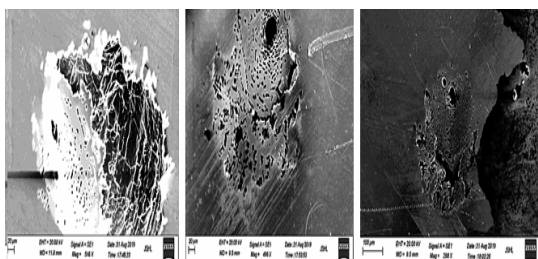


Fig. 7. SEM photographs on pitting corrosion of SS316L samples after polarization test

Thickness measurements and holiday tests results of coatings

The average thicknesses of the coatings in mm and a range of applied voltages in kV for holiday tests are shown in Table 2.

Adhesion test by peel strength method

Figure 8 and Fig. 9 show the peel strength graphs of 3LPE, 3p/2p CAT, VE, HSS, before and after the HWI test, respectively. The test samples, before and after the HWI test, are shown in Fig. 10 and Fig. 11 respectively. Prior to the HWI test, the peel strength of coating is considered as the initial peel strength and is represented as P_0 . Similarly, after the HWI test, the peel strength of coating is considered as the final peel strength and is represented as P_{100} . The P_0 and P_{100} values are shown in Table 3. It reveals from Table 3 that 3LPE has shown a substantial reduction in the adhesion strength after the HWI test, whereas HSS and VE have shown a slight reduction in their adhesion strengths.

Adhesion test by pull-off method

Figure 12 and Fig. 13 show the pull-off adhesion strengths of LE and PU in Psi unit, before and after the HWI test, respectively. Prior to the HWI test, the pull-off adhesion strength of coating is considered as the initial pull-off adhesion strength and is represented as P_0 . Similarly, after the HWI test, the pull-off adhesion strength of coating is considered as the final pull-off adhesion strength and is represented as P_{100} . The P_0 and P_{100} values are shown in Table 4. It is apparent from Table 4 that PU has shown a substantial reduction in the pull-off adhesion strength after the HWI test, whereas LE has shown marginally less reduction in the pull-off adhesion strength.

As the peel strengths of 3LPE, 3p/2p CAT, VE, HSS and the pull-off adhesion strengths of LE, PU are expressed in N/mm and N/mm² units respectively, it appears unfeasible to draw a comparison of the coatings adhesion performances. In this situation, it has been deduced that it is reasonable to compute the ratio (P_{100}/P_0) of each coating to obtain a dimensionless number, which is found to be useful in comparing the coatings adhesion performances after the HWI test and drawing a logical conclusion. This dimensionless number is termed as the "Coating Bond Index" in this study. This index represents as the bond between a coating and SS316L substrate and also the adhesion performance of a coating. The highest index number indicates the strongest bond and best adhesion performance of a coating that ensures the coating remains adhered to SS316L substrate surface for long.

Table 2: Thickness Measurement (in mm) and Holiday Test (in kV) of coatings

Type of coating	1	2	3	4	5	6	7	8	Av.*	Vol.**	Holidays
3LPE	2.96	2.93	2.89	2.93	2.98	2.86	2.85	2.80	2.9	20 - 22	Nil
3p/2p CAT	2.48	2.52	2.55	2.49	2.51	2.52	2.45	2.48	2.5	19 - 20	Nil
PU	1.7	1.65	1.49	1.5	1.48	1.57	1.58	1.43	1.55	8 - 12	Nil
VE	3.03	3.09	3.07	2.95	2.91	3.03	2.89	3.1	3.0	22 - 25	Nil
LE	0.96	1.06	0.92	0.97	0.92	0.9	0.92	0.95	0.95	5 - 10	Nil
HSS	2.31	2.22	2.45	2.3	2.46	2.24	2.2	2.22	2.3	16 - 18	Nil

Av.* - arithmetic mean of thickness

Vol.** - a range of applied voltages

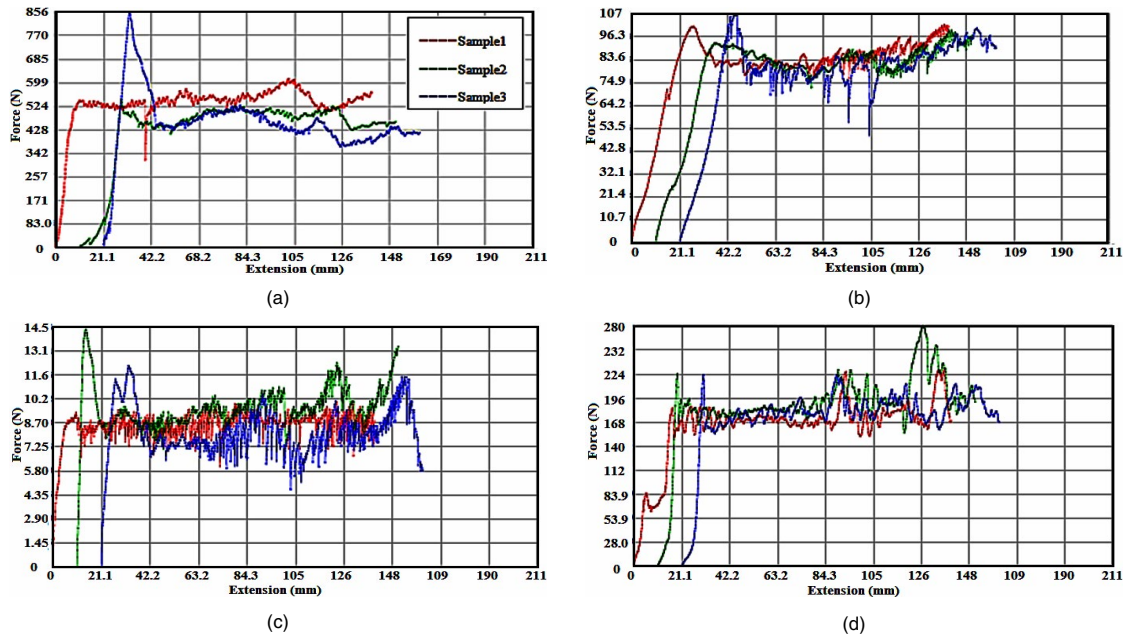


Fig. 8. Initial peel strength graphs before HWI test-a) 3LPE, b) 3p/2p CAT, c) VE, d) HSS

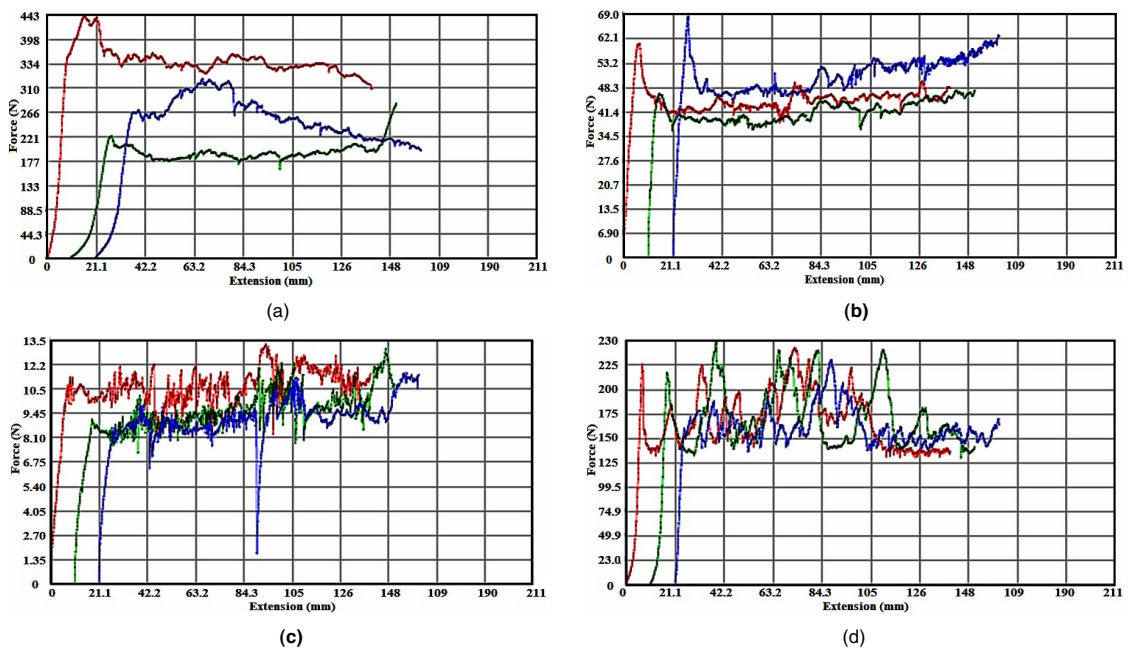


Fig. 9. Final peel strength graphs after HWI test-a) 3LPE, b) 3p/2p CAT, c) VE, d) HSS

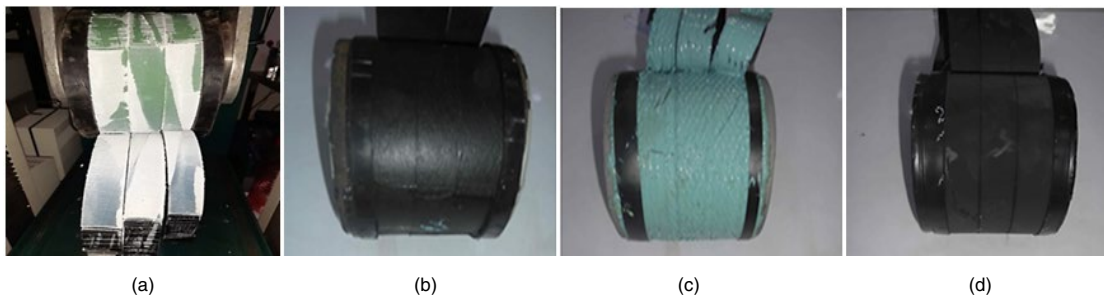


Fig. 10. Peel strength tested samples before HWI test-a) 3LPE, b) 3p/2p CAT, c) VE, d) HSS

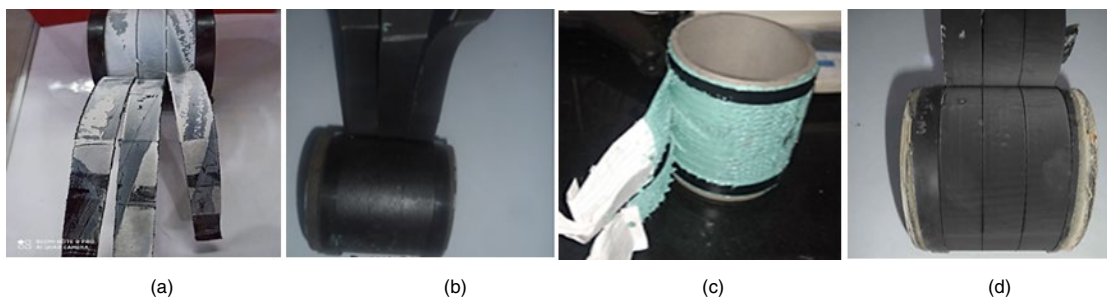


Fig. 11. Peel strength tested samples after HWI test-a) 3LPE, b) 3p/2p CAT, c) VE, d) HSS

Table 3: Peel strength results of coatings

Type of coating	P ₀ (N/mm)	P ₁₀₀ (N/mm)
3LPE	19.7	10.9
3p/2p CAT	3.4	1.8
VE	0.39	0.35
HSS	7.3	6.8

Table 4: Pull-off adhesion strength results of coatings

Type of coating	P ₀ (N/mm ²)	P ₁₀₀ (N/mm ²)
PU	20.87	10.77
LE	28.97	22.7

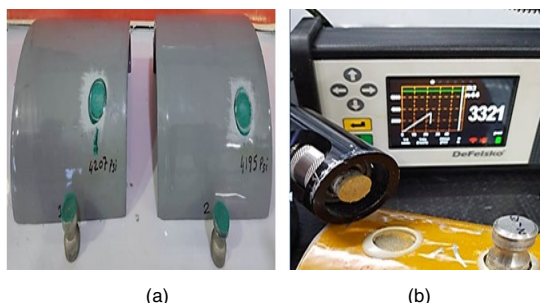


Fig. 12. Initial pull-off adhesion strength tested samples before HWI test-a) LE, b) PU

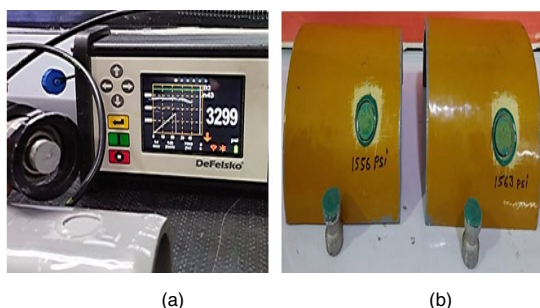


Fig. 13. Final pull-off adhesion strength tested samples after HWI test-a) LE, b) PU

The “Coating Bond Index” has been computed from the ratio (P_{100}/P_0) for each coating and is shown in Fig. 14. It is apparent from Fig. 14 that HSS has the strongest bond between HSS coating and SS316L substrate and has shown best adhesion performance among the coatings after the HWI test. Next to HSS, VE and LE have the stronger bonds and have shown better adhesion performances. PU, 3p/2p CAT and 3LPE have shown almost a relatively same level of the adhesion performances after the HWI test. Therefore, the relative rankings of adhesion performances for these coatings are found to be HSS-1, VE-2, LE-3, 3LPE/3p-2P CAT/PU-4, where ‘1’ represents the strongest bond and best adhesion performance.

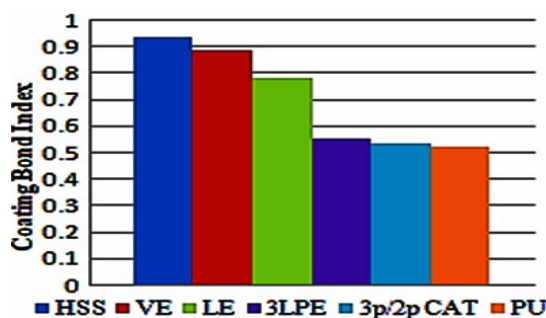


Fig. 14. Coating Bond Index of the coatings

CONCLUSION

The initial high adhesion strength of a coating before the HWI test cannot be ensured to be retained in terms of lifetime of a pipeline because a coating practically loses its original adhesion strength and the main problem encountered with coating failures has been loss of adhesion over time. In such situation, the HWI test has advantage as this is the most realistic method for determining expected long-term adhesion performance of a coating under actual operating conditions. The HWI test measures the loss of adhesion strength of a coating to in-service immersion conditions at a defined temperature and for a specified duration. It is, therefore, logical to conduct adhesion tests on coated pipes before and after the HWI test at temperatures about $55\pm 5^\circ\text{C}$ for a period of 100 days and compare the adhesion strength to the original (initial) value. A good coating would then be one that retained a substantial percentage of its original (initial) adhesion strength, or at least some level of adhesion strength after 100 days hot water immersion and is expected to perform a useful life of at least 25-30 years under actual operating conditions.

In this study, the adhesion strength of each coating, before and after the HWI test, has

been measured. A comparison of the coatings adhesion performances has been drawn based on the dimensionless number termed as the "Coating Bond Index". According to the "Coating Bond Index", HSS has shown the strongest bond and best adhesion performance among the coatings, whereas VE & LE have shown the stronger bond and better adhesion performances for buried SS316L pipelines in the petroleum, petrochemical and natural gas industries. PU, 3p/2p CAT and 3LPE have shown almost a relatively same level of the adhesion performances. The relative rankings of the adhesion performances for these coatings are HSS-1, VE-2, LE-3, 3LPE/3p-2P CAT/PU-4, where '1' represents the strongest bond and best adhesion performance.

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Conflicts of interest

The authors declare no conflict of interest.

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