

Research Article

Study of Qualification of Coating Procedure Specifications as Per Din 30670 for 3lpe Coating of 8 Inch Diameter Erw Pipe

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Abstract

In present study qualification of 3 layer polyethylene coating procedure is discussed which is adopted for the corrosion protection of API 8 Inch ERW line pipes. API monogrammed bare pipes were supplied by the local manufacturer while coating was performed as per DIN 30670 standard. Raw materials including surface preparation and coating raw materials steel grit, acid and chromate, epoxy, adhesive, HDPE and bare pipes were tested and compared with the manufacturer testing certificate for specification compliance check, results are presented in tables 1-4. Physical inspections of bare pipes were carried out before assigning unique identification number to each pipe. Surface preparation including pre-heating of pipes at least 3°C above dew point temperature, steel grit blasting of pipes to get the desired Sa 2.5 surface with 50 to 80 microns profile, acid washing of pipes to remove excess chloride ions, Rinsing of pipes after acid washing with RO water, drying of pipes through blowers, induction heating of pipes up to the epoxy manufacturer recommended temperature which is 200 to 220 degree celsius, first layer epoxy powder application through electrostatic spray gun technique, second layer of adhesive and third layer of Polyethylene film application through side extrusion process, quenching of coated pipes, full length coating holiday inspection and in the end Final inspection of coated pipes. Four test pipes were selected for coating procedure qualification. Out of these 4 test pipes, First pipe coated with adhesive layer for adhesive layer thickness measurement, Second pipe coated with epoxy layer for epoxy layer thickness measurement, third pipe coated with Polyethylene layer for polyethylene layer thickness measurement along with sampling for indentation hardness test, fourth pipe coated with all three layers and tested for thickness measurement of combined three layers, holiday detection, peel ad-

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Citation: Qazi HAA (2019) Study of Qualification of Coating Procedure Specifications as Per Din 30670 for 3lpe Coating of 8 Inch Diameter Erw Pipe, Pakistan. J Environ Sci Curr Res 2: 008.

Received: March 15, 2019; Accepted: April 17, 2019; Published: April 25, 2019

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hesion strength test, impact test and cathodic disbondment test. All testing were evaluated to check compliance against the standard DIN 30670 requirements and coating procedure specifications.

Keywords: Cathodic disbondment test; Chloride content test; Coating procedure qualification; Coating procedure specifications; Corrosion protection; Dew point; DIN german institute for standardization; Elongation test; Holiday test; Impact test; Indentation Test; Peel Adhesion; Surface preparation; Thickness measurement; Three layer polyethylene coating (3LPE).

Introduction

Since the advancements in piping technology for oil and gas transportation, three layer polyethylene coating have been in use for not only giving corrosion protection to the pipelines but also enhancing the service life of it. 3LPE (Three Layer Polyethylene) systems consists of first functional layer: a high performance Fusion Bonded Epoxy (FBE) an anticorrosion layer, followed by second functional layer a copolymer adhesive which makes bond between epoxy and polyethylene and third functional layer an outer layer of high density polyethylene which provides tough and durable protection. 3LPE coating systems provide the most efficient pipeline protection for small and large diameter pipelines with moderately operating temperatures from -40°C up to 80°C which is comparatively higher range.

Prior to start of regular coating production of each order, a procedure qualification trial is adopted for each pipe diameter range, Inch, for each combination of coating material, and for each plant, to set up that plant, materials and coating procedures resulting quality of finish product confirming to the properties of the material, relevant standards, specifications and material manufacturer's recommendations. In current study, coating procedure qualification is performed for 8 inch diameter ERW pipe, four test pipes are selected for coating procedure qualification. Out of four test pipes, one pipe coated with adhesive, one pipe coated with epoxy, one pipe coated with polyethylene and one pipe coated with all three layers. Properties of the starting raw materials as mentioned in test certificates supplied by the respective manufacturers is compared with the standard specifications raw materials include client supplied bare pipes which are manufactured as per API 5L 45th edition. Surface preparation raw materials which include steel grit, phosphoric acid and chromate. Coating raw materials which include epoxy powder, copolymer adhesive and high density polyethylene.

Surface Preparation Materials Properties

Seen from tables 1-3.

Methodology

Client supplied bare pipes were visually inspected for surface defects dents, pits, bevel and root damage, straightness, surface contamination etc., QC cleared pipes sent for surface cleaning process while rejected pipes sent for re-working or replacement. Detailed coating process steps are presented in flow diagram below (Figure 1).

	Manufacturer	C%	Mn%	Si%	S%	P%	Hardness (HRC)	Microstructure	Density (g/cm³)
Steel Grit	Thomas Abrasive	0.97	0.91	0.88	0.018	0.02	47-52	Tempered Martensite	7.4
Phosphoric Acid	Manufacturer and Type		Density (g/cm³)			Color			
	Chemetall, Oakite 33		1.310			Light Yellow			
Chromate	Manufacturer and Type	Appearance	Specific Gavity @ 20°C	PH @ 20°C	Hexavalent Cr. As g/l CrO3	Hegmann Gauge Test			
	Chemetall, Accomet PC	Dark Brown Liquid	1.91	2.3	97	No Particle exceeding 12 microns			

Table 1: Properties of steel grit, phosphoric acid and chromate as per manufacturer test certificate.

Fusion Bonded Epoxy	Manufacturer and Type	Moisture Content %	Particle Size % 150 Microns	Particle Size % 250 Microns	Density (G/L)	Thermal Characteristics- Inflection Point (J/G)	Cathodic Disbondment Radius of Disbondment (-3.5 V, @ 65°C, 24 hours)	Impact Resistance	Water Soak	Flexibility Test at 3 Degree @ 30°C
	Jotun JotaPipe 1003-21S	0.19	2	0.2	1455	39.7	1 mm	>1.5 Joule	Rating 1	No Cracking

Table 2: Properties of fusion bonded epoxy as per epoxy manufacturer test certificate.

* Epoxy raw material tests done by the manufacturer as per CSA-Z245.20-10 standard.

Adhesive	Manufacturer and Type	Density @ 23°C (Kg/cm³)	Melt Flow Rate (g/10 min) @ 190 °C & 2.16 Kg	Ultimate Tensile Strength (MPa)	Hardness (Shore-D)	Vicat Softening Point (A50, 10N), Unit (°C)	Melting point (°C)	Flexural Modulus (MPa)	Tensile Impact @ 20 °C (J/cm²)	Water Absorption (%)
	Lushan, L-5R	929	1.81	19.6	60	101.9	125.4	280	12.6	0.06

Table 3: Properties of Copolymer Adhesive as per adhesive manufacturer test certificate.

* Adhesive raw material tests performed by the manufacturer as per ASTM relevant standards.

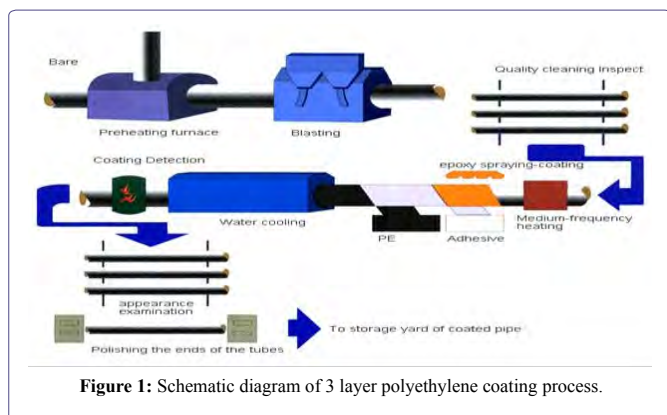
Polyethylene	Melt Index @ 190°C & 2.16 Kg load (g/10 min)	Density Compound (Kg/m³)	Carbon Black Content (%)	Oxidation Induction Time @ 210°C Al Pan (Minutes)	Moisture Content (ppm)	Total Volatiles (ppm)
	0.49	954	2.18	52	10	110

Table 4: Properties of Copolymer Adhesive as per adhesive manufacturer test certificate.

* Polyethylene raw material tests performed by the manufacturer as per ISO relevant standards.

First step is the surface preparation of pipes prior to coating application. To do so first pipes are passed through gas furnace for preheating of pipes above dew point temperature thereafter pipes are subjected to steel grit blasting using centrifugal blasting technique where all the rust on pipes surface is removed. In steel grit blasting, pipes are cleaned up to white metal surface appearance Sa2.5 with required surface profile. Using acid spraying technique, pipes are sprayed with phosphoric acid solution which reacts with chloride ions present on pipe surface and rinsing the pipes with RO water flash them out of the pipe surface. In the end of first step quality inspection is performed.

Second step is the coating application. Surface treated pipes are subjected to induction heating of pipes upto the epoxy manufacturer recommended temperature which 200-220°C. Epoxy powder is sprayed on pipes through electrostatic spray gun technique after some seconds of epoxy layer application second layer of copolymer adhesive and third layer of polyethylene is applied on pipes through side extrusion process there after pipes are passed through Quenching tank where cold water is sprayed on as coated pipes where soft coated pipes are converted to hard coated form. In the end of second stage coated pipes are subject to full length holiday detection where flaw in coating is detected.



Third step involves removal of coating up to the specific length from both ends through cutback brush rollers thereafter in the final stage coated pipes are inspected for thickness measurement, coating bevel, cutback length, marking, and other quality control testing. The testing results discussed in the results and discussion chapter.

Results and Discussion

In current study four pipes are selected as sample, Pipe Numbers 01 to 04 was selected from the batch all the measurements were recorded on these pipes. Dew point was calculated first to identify the temperature required to be set for preheaters. In current study dew point measured was 9.2°C as can be seen from the figure 2 hence the temperature of preheaters were set accordingly the temperature measured after preheaters was 39°C as can be seen from figure 3 which is quite higher than the dew point. Steel grit blasting of pipes carried out using centrifugal shot blasting technique, the surface profile measured 75 microns using testex tape and foil thickness gauge (replica method) refer figure 4 and satisfactory surface cleanliness level which is under the standard specifications as can be seen from figure 4, chloride content test using digital titration method performed before acid washing on blasted pipes which was measured 48 PPM which is higher than the client specifications (<20PPM) hence acid washing with 10% phosphoric acid +90% RO water solution was performed the chloride ions value after acid washing and RO rinsing was recorded 5 PPM which is under the client specified limit as can be seen from figure 5. As 200-240°C temperature before epoxy application is a requirement of first layer application, bare pipes passed through induction heating process the temperature measured using contact thermometer just before first layer epoxy application was 206 Degree Celsius which met the epoxy manufacturer recommendation as can be seen from figure 6. Application of all three layers were visually witnessed all production parameters including Pipe temperature, No. of epoxy guns, pressure of epoxy spray, adhesive and Polyethylene extruders melt temperature, pressure and RPM of driving screw and coating speed were recorded. For qualification testing, first adhesive layer is applied on bare pipe subsequent to cooling thereafter dry film thickness of adhesive layer measured which is 286-345 microns using dry film thickness calibrated gauge the adhesive layer sample reading was found under the client specifications (250 Microns Min. to 450 Microns Max.) as can be seen from figure 7. Thereafter epoxy layer qualification performed the thickness measured on epoxy cured film was 243-270 microns which met the client specifications (200 Microns Min.) as can be seen from figure 8. After both layer qualification performed normal production was started at the qualified parameters

one sample pipe No.4 was selected from the batch on which following tests were marked and performed: full length holiday inspection, 3 Layer Polyethylene thickness measurement as can be seen from figure 9, visual inspection, Cathodic disbondment test at 60°C as can be seen from figure 10, impact test figures 11 and 12, Peel adhesion test as can be seen from figures 13 and 14, indentation hardness test of PE sheet as can be seen from figures 15 and elongation test of polyethylene sheet as can be seen from figures 16 and 17.



Figure 2: Showing dry bulb and wet bulb temperature.



Figure 3: Temperature before shot blasting.



Figure 4: Shows shot blasted pipe surface profile.



Figure 5: Shows chloride content test value after acid wash.



Figure 6: Shows chloride content test value after acid wash.



Figure 7: Pipe temperature before epoxy application.



Figure 8: Shows epoxy cured film thickness 243 microns.



Figure 9: Shows 3 Layer thickness measured value 2.585 mm.



Figure 10: Cathodic disbondment test piece after test.



Figure 11: Shows impact test at fixed height of 1 meter.



Figure 12: Holiday detection at 25 KV after impact test.



Figure 13: Shows peel adhesion test.

For qualification of coating procedure specifications some of the tests were performed on production floor and results evaluated at the same time while some of the tests were performed in laboratory under controlled temperature. Table 5 shows the qualification tests results for surface preparation, table 6 shows testing results conducted on shop floor whereas table 7 shows testing results conducted in laboratory.



Figure 14: Shows peel adhesion value which is 240 N/cm.



Figure 16: PE Sheet Elongation Test Specimen.



Figure 15: Indentation test of PE sheet @70°C.



Figure 17: PE sheet Elongation Measurement

Dew Point Measured	Preheating Temperature Requirement	Preheating Temperature Measured	Grit Blasting Profile Requirement	Grit Blasting Profile Measured	Chloride Content Test on grit blasted pipe surface Requirement	Chloride Content Test on grit blasted pipe surface Measured	Pipe Temperature Requirement Before Epoxy Application	Pipe Temperature Measured Before Epoxy Application
9.2°C	> 3°C of Dew Point	39°C	50- 80 Microns	75 Microns	<20 PPM	5 PPM	200-220°C	206°C

Table 5: Testing Results related to surface preparation prior to coating application.

*Chloride Content test as per standard ISO 8502-2

Thickness First Layer Epoxy Req.	Thickness Epoxy First Layer Measured	Thickness Second Layer Adhesive Req.	Thickness Adhesive Layer Measured	Thickness 3 Layer 3LPE Req.	Thickness 3Layer 3LPE Measured	Online Holiday Test at 25 KV Req.	Online Holiday Test at 25 KV Result	Peel Strength @ 25°C Req.	Peel Strength @ 25°C Measure	Impact Resistance at 23°C subsequent to Holiday Detection at 25 KV Req.	Impact Resistance at 23°C subsequent to Holiday Detection at 25 KV Result
>200 Microns	243-270 Microns	250-450 Microns	286-345 Microns	> 2.5 mm	2.5-3.1 mm	No Spark	No sparks	> 150 N/ Cm	240 N/ cm	No Spark	No spark

Table 6: Qualification testing results the testing was performed and evaluated on production floor.

*Thickness of all three layers measured as per Annex A of DIN 30670

* Holiday Detection as per Annex E of DIN 30670

* Peel adhesion strength as per Annex D of DIN 30670

* Impact Resistance test as per Annex H of DIN 30670

Cathodic Disbondment Test @ 60°C, -3.5 V, 24 hours Requirement	Cathodic Disbondment Test @ 60°C, -3.5 V, 24 hours Result	Indentation Test @ (i) 23°C @ (ii) 70°C Requirement	Indentation Test @ (i) 23°C @ (ii) 70°C Measured	Elongation Test @23°C Requirement	Elongation Test @23°C Measured
Radius of Disbondment < 7 mm	Radius of Disbondment 3 mm	(i) < 0.2 mm (ii) < 0.4 mm	(i) 0.07 mm (ii) 0.1 mm	> 400%	733%

Table 7: Qualification testing performed and evaluated in laboratory at controlled temperature.

* Cathodic disbondment test at 60°C as per ASTM G42

* Indentation hardness test as per Annex I of DIN 30670

* Elongation test as per Annex F of DIN 30670 and standard ISO 527-1 to 527-3

Conclusion

Coating application on line pipes is performed against corrosion protection and is considered as primary corrosion protection system for line pipes as there is a secondary corrosion protection technique usually cathodic protection which is adopted in field pipelines. Beside a collective purpose and function of 3LPE coating system, all three layers perform individual functions. First epoxy layer performs anti-corrosion function of the coating system which protects the line pipe against oxidation. The second layer of copolymer adhesive keeps the first and third layer of 3LPE coating system bonded while the third layer which is thickest of all three layers gives mechanical strength and protects 3LPE coating system from mechanical damage during handling and in-service mechanical damages of buried pipeline. As all three layer performs individual functions, all three layers raw materials properties verified individually prior to use in coating process to check compliance against DIN 30670, all three layers application were individually tested and as all three layers perform collective function some of the tests performed on 3LPE coating system. From the tables 1-7, it can be seen that all the results are complying with the manufacturing specifications and DIN 30670 hence the coating procedure specifications found satisfactory and approval is given for normal routine coating production.

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