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Assessment of Adhesives Performance of 3-Layered Polyethylene Coatings in the Oil and Gas Industry

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Abstract- **Corrosion possesses a major threat to long (several thousands of kilometers) pipeline infrastructure in Pakistan and is a key engineering concern. Construction of pipeline involves field wielded joint, wherein 3 layered poly-ethylene (3-LPE) protective layer is applied in a non-controlled environment involving many complexities. The risk factor is amplified by surface degradation (corrosion) and gas leakage, which is a major economic concern for various gas transmission and distribution industries. This research work is aimed at addressing the behavior of 3-LPE on the gas transmission line to avoid the pipe (substrate) surface from harsh environmental conditions and longevity in service life. There is well-published and known technique in the lab for carrying out the application of 3-LPE on pipelines, but in field welded joints, it is quite challenging to achieve the equivalent bond strength of 3-LPE applied on the wielded jointly due to certain factors. The current research aims to apply 3-LPE followed by on-site testing using peel test and other techniques. The field application of 3-LPE starts with surface preparation using cleaning and sandblasting, followed by FBE primer application onto substrate surface at elevated temperature, and finally, PE layer is applied. The strength and durability of the applied layer are measured using the peel test. Regression analysis is performed using ANOVA to assess the durability, strength, and service life of 3-LPE on steel substrate, wherein three key factors were, i.e., surface preparation, pre-heat, and film thickness, were selected for investigation. Surface preparation is significantly important among the three, showing a value of 38.41%, followed by film thickness then pre-heat.**

Index Terms-- **Adhesive layer, ANOVA, Coatings, Corrosion, Polyethylene, Peel test, Field wielded joint, Primer**

I. INTRODUCTION

Coatings have been used to avoid corrosion in the transmission, oil and gas storage, and distributions network for many years. Pakistan's hundred thousand kilometers of transmission and distribution pipeline network is under corrosion threat. This is a serious predicament in terms of labor, resources, and infrastructure loss of the piping network. The cost of adhesive coating is only a small portion of the piping infrastructure cost, yet it could perform a lead role in the protection of steel pipe (substrate) from the harsh environment polyethylene-based coatings have promising applications in protecting against corrosion about recent oil and gas industry [2, 3]. Adhesives are often modified to bring enhancement in interfacial bonding among the three components when these components congregate in the molten state [4, 5]. A wide range of functional groups are available that could be grafted onto Polyethylene via free radical reactions, for instance, MAH (Maleic Anhydride) has proven effective in enhancing the adhesion process between epoxy and polyethylene topcoat, in both solutions and melt process [6-9].

The oil and gas industry usually uses three types of coatings in their piping networks.

- CTE (Coal Tar Enamel) is the first type of protective layer and is used for many years, nevertheless, its manufacturing poses serious threats to human health.
- The second type which is used for almost 2 decades, is FBE (Fusion Bonded Epoxy) coating system a three-layered protective system (see Figure 1). The first layer is a primer (Fusion Bonded Epoxy), a hot melt adhesive layer, and a Polyethylene top layer. Its service life is almost 60 years. FBE protective coatings are contemplated as the best protective coats for underground piping networks application and it offers the best adhesion properties, low permeability to oxygen, excellent thermal stability, high chemical resistance, and better flexibility [10]. Even though FBE protective coatings have shown excellent results against corrosion, their key shortcomings have been rectified by CP (Cathodic Protection) in conjunction with existing FBE systems.

A protective layer of coating serves as a physical barrier between the steel substrate and an external hostile environment. Ideally, the protective coat should avoid the corrosion 100 percent, nonetheless, no such coating media exists in reality. The purpose of the coating is to bring the corrosion to a minimal level possible and extend the service life of the substrate (steel pipeline and storage tanks). Proper adhesion of the protective layer to the surface of the substrate, additionally, low permeation to ions and water is key to the proper functioning of any protective layer [11, 12].

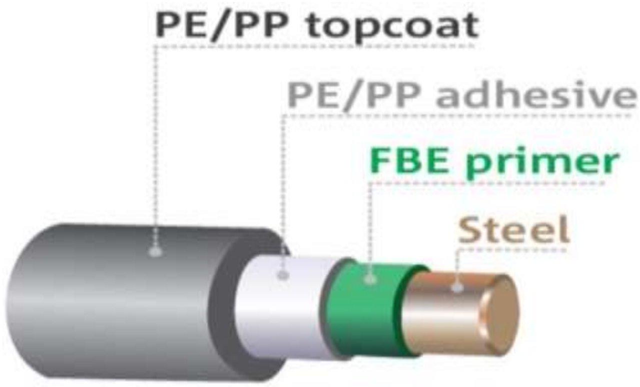

Figure 1: Layers of FBE Protective Coating

Since corrosion only occurs on wet surfaces, if oxygen penetration to the substrate surface is avoided by proper adhesion of the protective layer to the surface, corrosion will automatically subside.

With time coatings are degraded and their deterioration rages from complete stratification at the coating layer/substrate interface, yielding corrosion products to abatement at the steel surface, leading to peeling off of the protective coat from the steel surface [13]. Many factors affect the protective layer adhesion such as; contaminates, soluble salts, fat, dirt, wax, etc. at the layer-substrate interface. These mentioned factors are due to improper surface preparation before coating application. Due to their conductive nature, these contaminants provide a channel for a thin layer of water, which draws the ions resulting in corrosion [14]. The disbandment rate of any protective layer is a function of pre-existing contaminants (improper surface preparation), curing temperature, and the environment to which the pipeline is exposed [15].

Some premature protective coat deteriorates, in the case of the fuel storage tanks, due to exceeding the safe limit of soluble salts at the substrate-coating interface [16]. Various reports show some correlation between premature coating deterioration and chloride ion concentration [15]. It is also a fact, that the addition of a second contaminant in the presence of firs the t contaminant, expedites the deterioration rate e.g., sulfate or nitrate ions in the presence of already existing chloride ion [17-19].

Grit blasting is a well-known and most frequently used technique for pre-substrate surface preparation, ensuring a long-lasting protective layer, low or minimal corrosion and a smaller number of maintenance cycles required. FTIR (Fourier Transform Infrared Spectroscopy) and TOFSIM (Time-of-Flight Secondary Ion Mass Spectrometry) are enhanced analytical surface techniques regarding the chemical characterization of the protective layer[20]. Since contamination such as fat, wax etc., is a s,erious threat to the proper adhesion of the epoxy protective coating, the aforementioned techniques play a key role in overcoming its harmful impacts.

Proper adhesion is yet another important factor in determining the durability and qualitythe of the substrate-coating system. Adhesion is the measure of the energy that is liberated when the protective coating makes a firm bond at the substrate surface. In other words, adhesion could also be defined in terms of the energy required to disband a protective coating from a substrate surface. This could be a key parameter in determining the proper assessment of substrate-coat bonding.

The major problems associated fwith ield-applied coating are directly related to the actual prevailing working conditions, such as substrate preparation, and application techniques. Coating applied with poor surface preparation and uncontrolled application technique would result in a weak bond that ultimately would cause coating failures, making the pipeline at more risk of corrosion attack and decreasing pipeline useful life. It means that these factors are very important, and their values must be controlled during field application. Infield application of weld joint coating variations is observed in controlling these factors, a study is therefore required to analyze the impact of these varying factors on coating adhesion strength which ultimately effect pipeline useful life. This research proposes a technique of surface preparation using sandblasting with varying particle size jet speed and time of exposure and the effects of washing the surface with an acid solution before coating application is investigated.

II. GOALS AND OBJECTIVES

The key objectives of this research work are;

- To identify and assess the important factors affecting the response variable (protective coat adhesion, strength)
- To investigate the significance of the correlation between the factors affecting the response variable.
- To provide guidelines for the oil and gas industry (Users), so that they could optimize the coating application process in the field.

III. METHODOLOGY

The methodology adopted in this research work comprises field visits to transmission & distribution networks, and other plants coating application sites. The visits are thoroughly followed by an electrochemical and analytical analysis of FBE coatings and materials. These are then sampled and tested in the infield and laboratory.

Based on the literature review already discussed in detail, the relevant experiments are designed based on which the strength of adhesion is found.

Some of the steps involved in the proposed methodology are described below;

Step 1; Sand Blasting:

In this step, the surface is initially cleaned manually to remove the dirt, oil, grease, etc. that might pre-exist at the surface. The surface is thoroughly cleaned, and all the rust and corrosions are scrubbed off using sandblasting technique. The procedure is shown in the following diagram.

Figure 1: Sandblasting

Step no. 2: Preparation of 3LPE coating samples based on designed experiments

In this step, the steel substrate is initially preheated to the desired temperature and liquid epoxy is prepared. The liquid epoxy is evenly applied on the surface of the already prepared steel substrate, a coating layer is applied afterward and allowed to set for some time. The setting allows for proper bonding of the epoxy layer and coating. This process is depicted as follows.

ati rfa ing es heating

Step no. 3: Experiments performed at the field (peel-adhesion pull off tests)

This is the most important step in the coating application, where a known rectangular strip from the applied coating layer is peeled off using the apparatus called Peel Tester. The strength is measured and recorded for later analysis. The procedure is shown in the following figures;

Figure 2: Peeling off a layer of the applied coating layer

Figure 6: Peel-off Tester

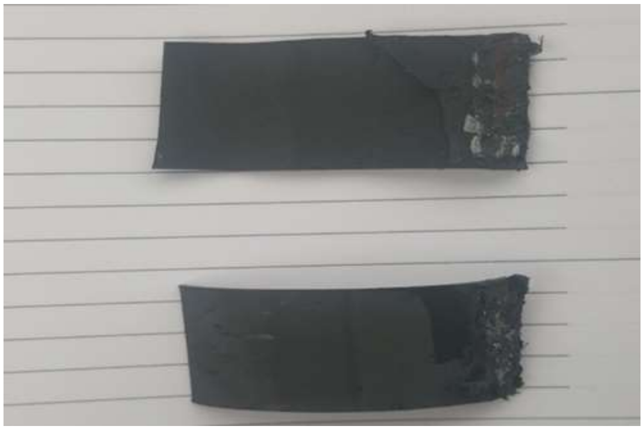

Figure 3: Portion of the coating that is being peeled off

Step no. 4: Data acquisition

Statistical data was obtained and analysis done based on the peel to adhesion strength was done in this step.

Step No. 5; Effects of wind speed, humidity, and Temperature:

Various parameters (e.g., substrate surface, coating materials, operator, and equipment) are set to assess the effects of temperature, wind speed, and humidity.

Step No 6: Peel Test Results

Obtaining Peel adhesion strength results. (lbs./inch)

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Obtaining Peel adhesion strength results. (lbs./inch)

Following points were taken care of during the experiments the following points were taken care of during the experiments

1. Care was taken in noticing the condition of the pipeline
2. Soil sampling was done and corrosively of soil was performed using LPR (Linear Polarization Resistance)
3. Peel off test was performed at different locations for consistent results
4. Potentials difference of soil was measured for conductive behavior and effects of leakage current
5. The coating samples were collected
6. Tests for surface contaminations were performed.

II. GENERAL FACTORIAL REGRESSION: PEEL STRENGTH

Factor Information

A generalized factorial regression is applied with Peel strength as the response variable. Three factors are selected as influencing variables i.e., K =3. The influencing variables each with three different levels are tabulated in the Table below

1. Surface preparation (microns)
2. Surface preheat (temperature)
3. Epoxy film thickness (microns)

Table 1: Peel strength vs response variable

Factor	Levels	Values
SR (microns)	3	25, 50, 100
PH (C)	3	40, 80, 140
FT (microns)	3	50, 100, 150

After constructing the full factorial design for three influencing variables on the Peel strength, the Analysis of Variance (ANOVA) was conducted. The Analysis of Variance results is tabulated in Table 2.

The ANOVA indicates that all the three-variable selected in the design study are significant. All the three variables i.e., SR

(Surface Preparation), PH (Pre-heat), and FT (Film Thickness) have p values equal to 0.00, 0.010, and 0.011 which is less than 0.05 (significance level of the design study). The F value for surface preparation is 16.89 higher than the values for surface pre-heat and film thickness which are 5.48 and 5.38, respectively. The contribution of surface preparation on peel strength is maximum with a contribution percentage value of 38.41. It means that among the three selected variables, the most significant variable is surface preparation followed by film thickness and surface preheat with percent contribution values of 12.56 and 12.22. The p-value for two-way interaction is 0.586, 0.800, and 0.999 which is greater than 0.05 (Significance level). It means that there is no interaction effect on the peel strength. Similarly, the p-value for three-way interaction is greater than 0.05 which indicates no three-ways interaction effect exists among the variables on the peel strength

Analysis of Variance

Table 2: Results of Analysis of Variance

Source	**DF**	**Seq SS**	**Contribution**	**Adj SS**	**Adj MS**	**F-Value**
Model	26	3747.93	70.05%	3747.93	144.15	2.43
Linear	6	3381.24	63.20%	3430.54	571.76	9.64
SR (microns)	2	2055.15	38.41%	2004.02	1002.01	16.89
PH(C)	2	654.04	12.22%	650.10	325.05	5.48
FT (microns)	2	672.06	12.56%	638.60	319.30	5.38
2-Way Interactions	12	319.27	5.97%	318.41	26.53	0.45
SR (microns)*PH(C)	4	182.18	3.41%	178.01	44.50	0.75
SR (microns)*FT (microns)	4	97.97	1.83%	97.24	24.31	0.41
PH(C)*FT (microns)	4	39.12	0.73%	41.53	10.38	0.17
3-Way Interactions	8	47.42	0.89%	47.42	5.93	0.10
SR (microns)*PH(C)*FT (microns)	8	47.42	0.89%	47.42	5.93	0.10
Error	27	1602.17	29.95%	1602.17	59.34	
Total	53	5350.09	100.00%			

Table 3: Source and P-value

Source	P-Value
Model	0.013
Linear	0.000
SR (microns)	0.000
PH(C)	0.010
FT (microns)	0.011
2-Way Interactions	0.928
SR (microns)*PH(C)	0.567
SR(microns)*FT(microns)	0.800
PH(C)*FT (microns)	0.949
3-Way Interactions	0.999
SR (microns)*PH(C)*FT (microns)	0.999
Total	

Model Summary

The model summary gives detail about how well the predicted values fit the model. The results of the model are given in Table 4. The S values indicate how far the predicted values deviate from the model fitting line. The small value of S indicates that the model is not entirely deviated from the fitting line. Similarly, the R sq. value which is 90.05 %, which is in the region of very good fit and shows less variation in the response variable i.e., peel strength, similarly the R-square value shows good results (81.22).

Table 3: Root square and adjusted value of Strength

S	R-sq.	R-sq.(adj)
7.70321	90.05%	81.22%

Coefficients

T value as given in the below Table gives criteria for rejection of the null hypothesis. If the T values lie within the confidence interval values, it indicates that the influencing variable is significant. Similarly, the smaller VIF value will indicate that the variables are significant. As for all the three variables selected, it is evident from the T values that it lies in the 95 % CI values. The T value for surface preparation is -0.57 and lies between the range -11.67 and -05.54. For surface preheat, the T values also lie in the confidence interval. Similarly, for film thickness, the t value lies in the range of confidence interval levels. All the three variable T value indicates that the variables are significant. For two-way interaction between the variable, the T value is out of bound showing that the interaction effect between the variables is insignificant. Also, for the three ways of interaction, the T values are beyond the confidence interval.

Table 4: criteria for rejection of the null hypothesis

Term	Coef	SE Coef	95% CI	T-Value	P-Value	VIF
Constant	55.10	1.06	(52.93, 57.28)	51.93	0.000	
SR (microns)						
25	-8.60	1.49	(-11.67, -5.54)	-5.77	0.000	1.35
50	3.23	1.49	(0.17, 6.29)	2.16	0.039	1.35
PH(C)						
40	-4.66	1.49	(-7.72, -1.60)	-3.12	0.004	1.35
80	3.67	1.49	(0.61, 6.73)	2.46	0.020	1.35
FT (microns)						
50	-4.77	1.53	(-7.91, -1.63)	-3.11	0.004	1.42
100	1.12	1.49	(-1.94, 4.18)	0.75	0.460	1.39
SR (microns)*PH(C)						
25 40	0.99	2.10	(-3.32, 5.31)	0.47	0.640	1.79
25 80	-2.34	2.10	(-6.65, 1.98)	-1.11	0.276	1.79
50 40	1.83	2.10	(-2.49, 6.14)	0.87	0.393	1.79
50 80	-0.51	2.10	(-4.82, 3.81)	-0.24	0.812	1.79
SR (microns)*FT (microns)						
25 50	1.94	2.13	(-2.44, 6.31)	0.91	0.371	1.83
25 100	-0.95	2.10	(-5.27, 3.36)	-0.45	0.655	1.86
50 50	-2.40	2.13	(-6.77, 1.98)	-1.12	0.271	1.83
50 100	0.55	2.10	(-3.77, 4.86)	0.26	0.796	1.86
PH(C)*FT (microns)						
40 50	-0.34	2.13	(-4.71, 4.03)	-0.16	0.875	1.83
40 100	-0.40	2.10	(-4.71, 3.92)	-0.19	0.852	1.86
80 50	-1.17	2.13	(-5.55, 3.20)	-0.55	0.587	1.83
80 100	0.44	2.10	(-3.88, 4.75)	0.21	0.836	1.86
SR (microns)*PH(C)* FT (microns)						
25 40 50	0.34	2.99	(-5.80, 6.47)	0.11	0.910	2.40
25 40 100	-0.10	2.97	(-6.20, 5.99)	-0.04	0.972	2.52
25 80 50	1.67	2.99	(-4.46, 7.81)	0.56	0.580	2.40
25 80 100	-0.94	2.97	(-7.03, 5.15)	-0.32	0.754	2.52
50 40 50	-0.49	2.99	(-6.63, 5.64)	-0.17	0.870	2.40

50 40 100	1.23	2.97	(-4.86, 7.32)	0.41	0.682	2.52
50 80 50	-0.16	2.99	(-6.30, 5.97)	-0.05	0.958	2.40
50 80 100	-0.10	2.97	(-6.20, 5.99)	-0.04	0.972	2.52

Regression Equation

The regression equation for peel strength is given below; The value of peel strength can be predicted for any value of the influencing variable. For any values of the three-influencing variables, the peel strength will be predicted with an R square value of 70 %.

$$PEEL\ STRENGTH\left(\frac{N}{cm}\right) = 55.10 - 8.60\,SR(microns)\_25 + 3.23\,SR(mic \\ + 3.67\,PH(C)\_80 + 0.99\,PH(C)\_140 - 4.77\,FT(\\ + 3.65\,FT(microns)\_150$$

Figure 7 depicts the Pareto chart of the standardized effect of the influencing variable on the peel strength. The standardized effect value is 2.052 shown by the red line in the figure. All those bars crossing the red lines indicate the significant variable. The bars that cross the red line indicate that the dividual effect of the influencing variables is significant. Similarly, the chart shows that the interaction effect is insignificant as the bars for the interaction effect are on the left side of the red line and have not crossed it. The graph shows that the most important and high significant variable that affects the peel strength is surface preparation with the highest value on Pareto chart.

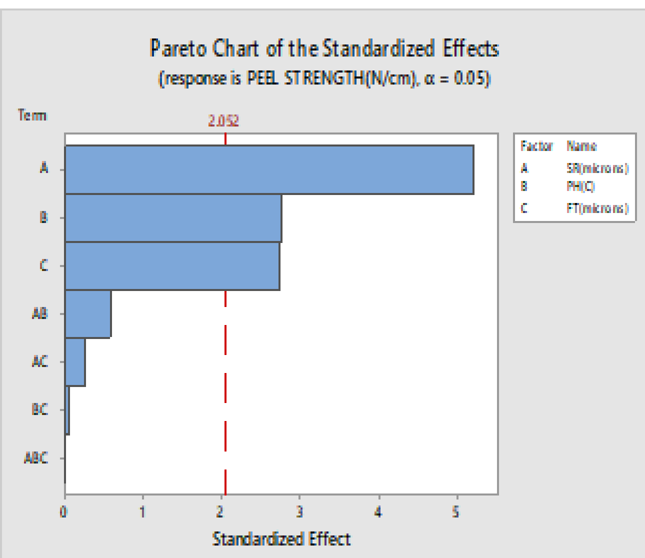

Figure 4: Pareto Analysis of Standardized Effects

Figure 8 indicates the normal probability plot of the standardized residuals which is the difference between actual values and fitted values. Anderson Darling test is used to check the data is normally distributed. If the p-value for the Anderson Darling test is greater than the significance level which is kept at 0.05 for this study design, the data will follow a normal distribution. The graph shows that the residuals are normally distributed. The p-value is 0.168 greater than the significance level of 0.05. The Anderson Darling value for the distribution is 0.530 with the mean value of 8.12 x 10-16 and a standard deviation of 1.104.

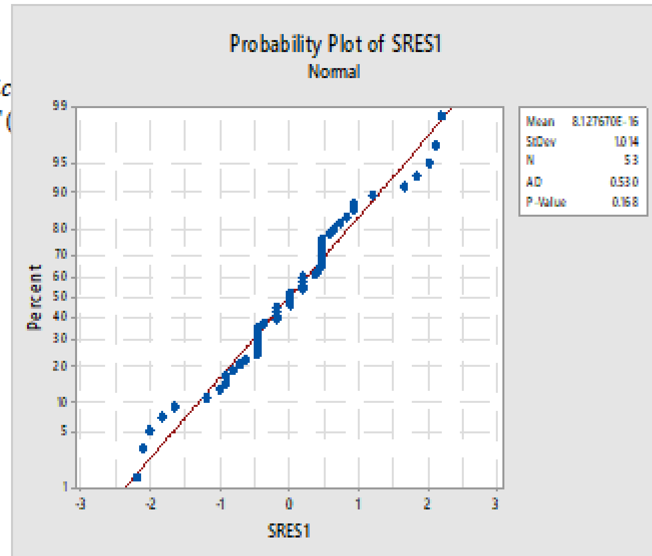

Figure 5: Probability Plot

Figures 4-5 exhibit the main effect plot of influencing variables on the peel strength. It is evident from the graph on the left of the main effect plot that the peel strength increases sharply increases as the value of surface preparation increases from 25 to 50. However, after the surface preparation value increases from 50 to 100, the peel strength is only slightly improved.

In the central chart of the main effect plot between peel strength and surface preheat, it can be observed that at first, the peel strength increases with an increase in the surface preheat value. However, after a certain value, it has shown a decrease in peel strength as the value of surface preheat is increased. The peel strength increases as the value of preheating increases from 40 to 80. However, it shows a sudden drop in peel strength as the surface preheat value increases from 80 and above.

In the main effect plot, the chart on the right shows the effect of film thickness on the peel strength. It is evident from the graph that the peel strength improves with film thickness. The relationship between the peel strength and film thickness is almost. However, at higher values of film thickness, the peel strength gradually increases as compared to the initial value of film thickness.

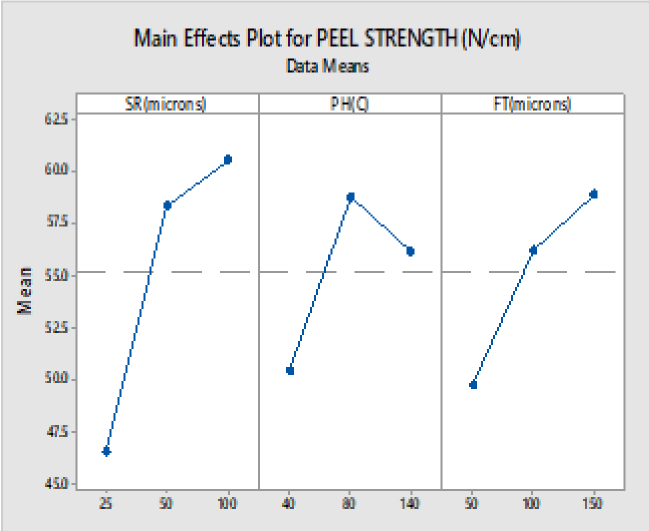

Figure 6: Plot of Peel Strength

The interaction effect plot for surface preheat and surface preparation on peel strength is given in Figure 12. It is evident from the graph that lines are almost parallel for both the variables except at a lower value of both surfaces preheat and surface preparation. The line crosses at lower values of the two influencing variables indicating that at lower values there is a slight interaction effect on the peel strength. However, at high values, there is no interaction effect at all on the peel strength. The interaction effect is insignificant as most of the lines of the value are parallel.

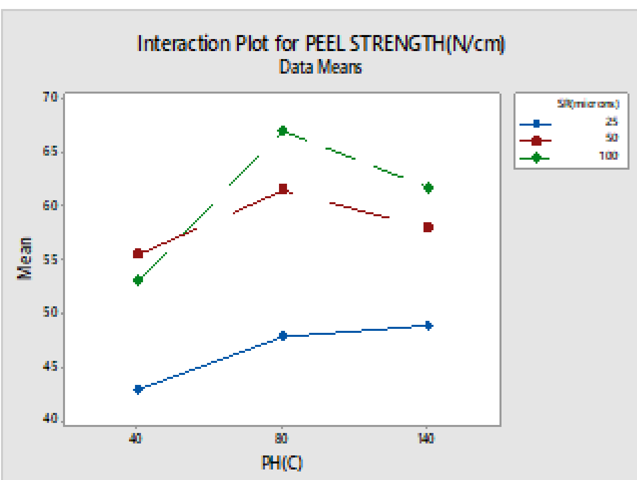

Figure 7: Interaction plot of Peel Strength

Figure 13 shows the interaction effect plot for surface preparation and film thickness on peel strength. The graph shows that the lines are almost parallel with intersection higher values of both surface preparation and film thickness. Parallel lines indicate the interaction effect is insignificant between the surface preparation and film thickness.

Figure 14 shows the interaction effect plot for surface preheat and film thickness on peel strength. The lines are almost parallel with no evidence of any interaction for any values of film thickness and surface preheat. The graph shows that the interaction effect is highly insignificant between the surface preheat and film thickness.

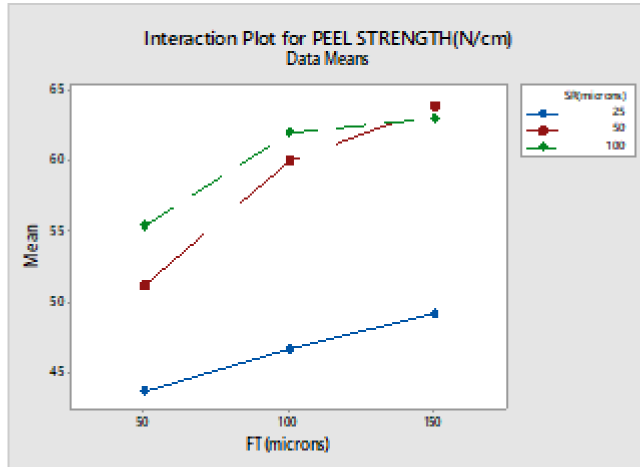

Figure 8: Interaction Plot for Peel Strength

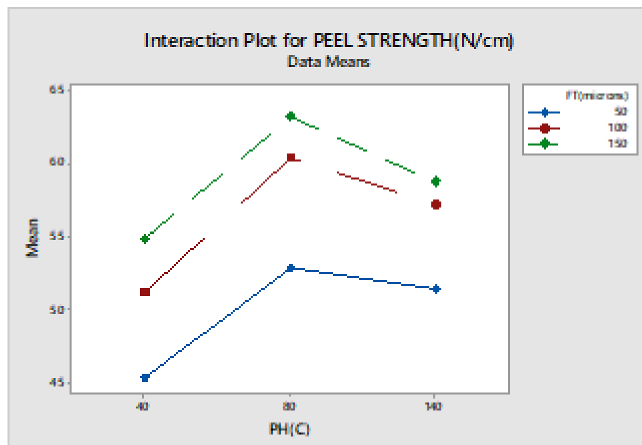

Figure 9: Interaction Plot of Peel Strength (Data Mean)

IV. CONCLUSIONS

Durability, strength, and service life of coatings (3-LPE) on steel substrate depend on the integrity of dimensional and interfacial stability of 3-LPE coatings to a higher degree. From a long service life and economic point of view (leakage avoidance), coatings play a significant role. Three factors were addressed in this work (i.e., K=3) in the analysis for generalized factorial regression as a function of peel strength. These factors were Surface preparation, pre-heat, and epoxy film thickness. Among the three factors surface preparation on the peel strength was of maximum importance, contributing a value of 38.41%, followed by epoxy film thickness and preheat. In light of the above conclusion, any industry pertaining to the coating process should bear in mind the importance of surface preparation.

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