The Effect of Process Parameters on Zinc Coating Layer Thickness in Hot-Dip Galvanizing

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Introduction

Hot dip galvanizing is the process of forming a protective coating against corrosion on metal surfaces by immersing it in a molten zinc bath [1, 2]. The resistance of zinc coating against mechanical stresses and damages is largely due to the metallurgical bond between the steel surface and the zinc coating. Galvanization is a diffusion controlled process. When the material is immersed into the molten zinc bath, firstly the material reaches the temperature of the molten zinc and then zinc layer on the surface of the pipe is formed by diffusion reaction. In this reaction, a series of intermetallic iron-zinc phases is formed as zinc diffuses inward and iron diffuses outward. The finished product consist of four layers on the steel; the outer layer is free zinc, and the inner three layers are separate intermetallic layers that are metallurgically bonded each other and steel. The coating produced consists of a series of zinc-iron alloys, with increasing zinc content occuring toward the coating [3]. Zn/Fe alloy layers are rather hard and brittle and their mechanical formability (bending, flexing, etc.) is quite limited. On the other hand, the pure zinc layer is softer and more resistant to such kind of mechanical stresses [3]. Effective corrosion resistance performance of hot-dip galvanized coatings depends on the composition of intermetallic layers and layer-wise morphology and topography.

The protection performance is proportional to thickness of the zinc coating layer [4]. The presence, thickness and distribution of Zn-Fe alloy layers determine the characteristics of the product and depend on the chemical composition of the zinc bath [5, 6-8], the chemical composition of steel [3], the temperature of zinc bath and sample [9, 10], immersion time [5, 6, 10], cooling rate [11], the surface roughness of specimen [12], the withdrawal velocity and angle from the bath [13, 14], and whether or not any procedure has been applied before [15]. As a result, the thickness of coating applied by hot dipping is primarily a function of;

- The duration of immersion, which controls the zinc of alloy layer;
- the speed of withdrawal from the bath, which controls the amount of unalloyed zinc adhering and
- The temperature of the bath, which affects both the alloy and free zinc layers.
- Coating weight is affected also by the amount of zinc removed by wiping. Each of these considerations has a distinct effect and may be used control the galvanizing process [16].

The zinc coating is mainly a function of dipping time, molten zinc temperature, withdrawal angle and speed, outside wiping pressure, inside blowing media, time and pressure. Therefore, process parameters, given above on inside zinc coating thickness of the steel pipe were investigated by some practical experiments.

2. Experimental Study

In this study, different size of pipes, manufactured by electric resistance welding (ERW) method conforming to EN 10255-M, were used. The material quality of pipes conforms to S195 T, of which chemical composition is as given in Table 1, being classified as Class 1 according to EN 10025-2 in terms of suitability for hot dip galvanizing. The pipes were galvanized according to European Standard EN 10240 [23]. The pipes were degreased in an acidic degreasing bath at ambient temperature for 10 min and subsequently rinsed in water bath and then pickled in HCI solution at a concentration of 15 wt.-% for 15 min to remove impurities such as scales and rust. After pickling, rinsing was performed in water bath and then fluxing was carried out in $ZnCl_2 \times NH^4Cl$ solution at 30 °Be at ambient temperature. The chemical treatment parameters are summarized in Table 2. Chemical composition of molten zinc bath is given in Table 3. Getting sample from each pipe was done according to Fig. 1. In order to see the effect of variable parameters on the coating layer thickness, samples were taken before internal cleaning. In other words, it is taken 10 cm long samples out of each meter of pipe length. From each pipe, 7 samples are taken and numbered 0 to 6. Number 6 represents the front edge of the pipe which exit out of zinc bath firstly and in the same manner number 0 represents the rear edge. Samples were taken to measure zinc coating weight in gr/m^2 by chemical way according to EN ISO 1460.

Table 1. Chemical composition of pipe material (wt.%).

C	Si	Mn	P	S	Cu	Ni	Cr	Mo	Al
0.034	0.011	0.25	0.01	0.01	0.042	0.045	0.028	0.005	0.058

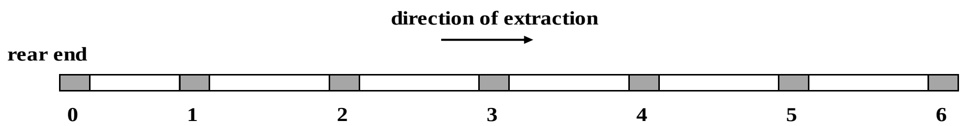
Table 2. Application parameters of chemical treatment.

	Degreasing	Pickling	Fluxing
Type of chemical	acidic base	hydrochloric acid (HCl)	double salt ($ZnCl_2$.2 NH_4Cl)
Temperature	ambient	ambient	ambient
Dipping time	10 min.	15 min.	4 min.
Concentration	-	%15	30 °Be
Fe content	-	50 gr/l	7 gr/l

Table 3. Chemical composition of molten zinc bath (wt.%).

Al	Cu	Pb	Sn	Cd	Fe	Mn	Sb	As	Zn
0.001	0.0051	0.006	0.003	0.001	0.018	0.0003	<0.002	<0.001	99.951

Fig 1. Measurements points along the pipe (m).



Dipping Time

To achieve satisfactory galvanizing, the pipes are dipped directly into the molten zinc bath after surface preparation and drying. Total dipping time which the pipe normally requires consist of two times, nominal time required for the pipe to attain temperature of the bath and time required for alloy formation. If the time is so short that the pipe does not attain the bath temperature, no alloy layer formation generally starts. Therefore, the time requried for the pipe to attain bath temperature becomes the function of the pipe thickness and time which affects the heat transfer between the molten zinc and the pipe itself. Based on this premises, it is assumed that pipe having the lower thickness will attain bath temperature much more earlier incomparison to the pipe having higher thickness [17]. On the other hand, too long dipping time results in higher alloy formation and higher percentange of zinc dross. The thickness of the coating does not increase substantially with longer immersion time [18]. The coating weigth increases at a rate less than linear with time [16]. Also, flaking which is one of the common faults in hot dip galvanizing can be seen because of higher coating thickness which comes from excessive dipping time. In the first experiment, the coating thicknesses obtained at different dipping times were investigated. For this purpose, immersion times were changed with the condition that all other parameters remained constant in two different pipe groups (Table 4). As can be seen from the test results (Table 5), the coating thickness increases with the increase of the dipping time (Fig 2).

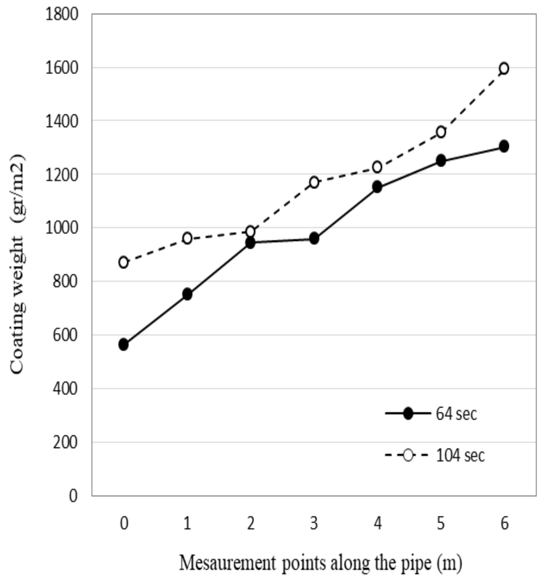
Table 4. Process paramaters for test I.

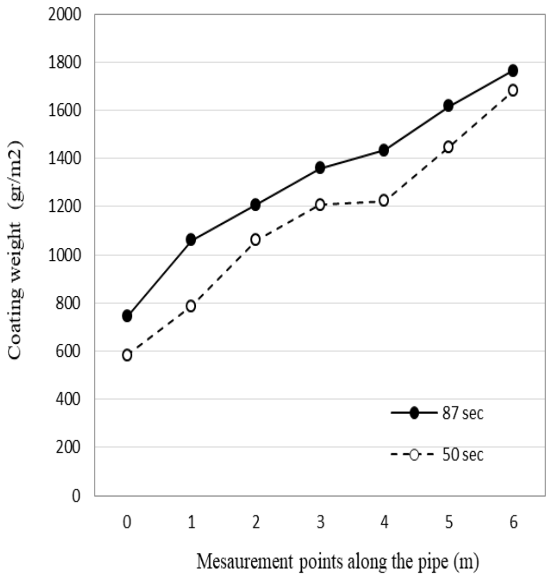
Test No	Group Code	Pipe Size (mm)	Dipping Time (s/piece)	Withdrawal Speed (m/s)	Zinc Bath Temperature (°C)	Inside Blowing Pressure (bar)
I	A	1"	64	1,00	450	0
	B		104			
	C	½"	87			
	D		50			

Table 5. Coating thickness along the pipe length (gr/m^2).

Group Code	Pipe No	Measurement points along to pipe length							
		0	**1**	**2**	**3**	**4**	**5**	**6**	**Ort.**
A	1	590	786	967	921	1081	1239	1328	**987**
	2	619	818	952	1022	1128	1313	1405	**1037**
	3	511	699	945	977	1183	1249	1245	**973**
	4	541	705	921	914	1213	1193	1231	**960**
		565	**752**	**946**	**959**	**1151**	**1249**	**1302**	**989**
B	1	937	1003	1027	1178	1210	1374	1678	**1201**
	2	839	908	1014	1277	1237	1282	1484	**1149**
	3	927	940	915	1086	1154	1456	1594	**1153**
	4	783	987	987	1138	1301	1317	1612	**1161**
		872	**960**	**986**	**1170**	**1226**	**1357**	**1592**	**1166**
C	1	704	1027	1202	1339	1467	1576	1753	**1295**
	2	804	1056	1121	1395	1440	1522	1763	**1300**
	3	728	1033	1208	1316	1466	1727	1709	**1312**
	4	741	1128	1296	1391	1363	1643	1833	**1342**
		744	**1061**	**1207**	**1360**	**1434**	**1617**	**1765**	**1313**
D	1	601	707	918	1101	1252	1401	1753	**1105**
	2	613	756	1071	1186	1321	1502	1581	**1147**
	3	598	733	1154	1254	1211	1489	1631	**1153**
	4	519	948	1096	1287	1106	1397	1754	**1158**
		583	**786**	**1060**	**1207**	**1223**	**1447**	**1680**	**1141**

Fig 2. Dipping time effect on coating thickness.





Bath Temperature

Bath Temperature is also an important factor which generally controls of reaction and also pure zinc layer carried over on the pipes. Temperature higher than 450 °C increase the rate of rection considerably between the pipe and zinc and also between walls of the kettle zinc kettle and zinc. Thus higher bath temperature obviously produces higher alloy layer formation on the pipe with a given dipping time and higher dross formation. Temperatures lower than 445 °C tend to decrease viscosity of the molten zinc and thus at the time of withdrawal, result in higher carry over of zinc from the bath. In addition to this it also results into the lower temperature of zinc at the time of steam blowing because by the time the pipe reaches the steam blowing station zinc is sufficiently solidified and thereby cannot be blown by the force of steam/air [17].

The molten zinc bath is operated at temperatures usually in the range of 445 to 465 °C. At 480 °C and above, the effect of these temperatures on both workpiece and galvanizing tanks are generally harmful [16]. Within the conventional galvanizing temperature range, an increase in temperature;

- increase the fluidity of molten zinc;
- accelerates the formation of oxides on the bath surface;
- heats the part to a higher temperature, thus increasing the time required for the zinc to solidify when the part is withdrawn;
- reduces immersion time, thereby incresing the capacity. The coating thickness for silicon-containing steels is very temperature dependant [18].

In second group of experimental tests, zinc bath temperature effect on zinc coating weight is investigated. For this purpose, the zinc bath temperature were changed to 450°C and 460°C, provided that all other parameters remained constant (Table 6). It was determined that the coating thickness increased by around 2 percent (Table 7) with an increase in the molten zinc bath temperature by 10 °C (Fig 3).

Table 6. Process paramaters for test II.

Test No	Group Code	Pipe Size (mm)	Zinc Bath Temperature (°C)	Dipping Time (s/piece)	Withdrawal Speed (m/s)	Inside Blowing Pressure (bar)
II	E	11/2"	460	98	0,60	0
	F		450			

Table 7. Coating thickness along the pipe length (gr/m^2).

Group Code	Pipe No	Measurement points along to pipe length							
		0	**1**	**2**	**3**	**4**	**5**	**6**	**Ort.**
E	1	588	755	845	892	989	998	1086	**875**
	2	572	783	830	939	925	999	1190	**891**
	3	606	721	860	1025	1090	1137	1199	**945**
	4	648	735	880	995	1083	1146	1160	**950**
		604	**749**	**854**	**963**	**1022**	**1070**	**1134**	**917**
F	1	601	708	811	870	984	941	1179	**871**
	2	620	707	871	884	931	1035	1217	**895**
	3	597	750	846	953	1004	1002	1131	**898**
	4	623	762	901	989	1054	1102	1091	**932**
		610	**732**	**857**	**924**	**993**	**1020**	**1155**	**899**

Fig 3. Zinc bath temperature effect on coating thickness.

It is indicated that bath temperature and dipping time, as a result of line speed, are specific operating parameters that can be optimized to control efficient surface cleaning, pipe heating and minimization of alloy layer growth during the immersion step. The third group experiments were carried out to see the effect of withdrawal speed and time. It has been observed that the extraction speed has a great importance on the coating thickness.

Withdrawal Speed

After dipping, pipes are extracted from the zinc bath at a certain withdrawal speed. At the same time, zinc drainage from the inside surface of the pipe happens because of extraction angle. When the material is withdrawn from the kettle, a thin layer of free zinc remains on the zinc-iron alloys, resulting in characteristic bright, shiny, galvanized coating finish when allowed to cool quickly in air. The thickness of free zinc layer varies with the speed of withdrawal from the molten bath [18]. It may be pointed out that use of lower withdrawal speed results in higher extraction time. This obviously results in cooling down the pipes up to melting point of zinc. This will result in poor dust collection. The rate and level of drainage is indirectly proportional to the diameter of the pipe at a given withdrawal speed. This means that smaller pipes should be withdrawn at lower withdrawal speeds and bigger pipes may be withdrawn at higher withdrawal speed. Up to a point lower withdrawal speed will result in better drainage, hence the lower zinc carry over.

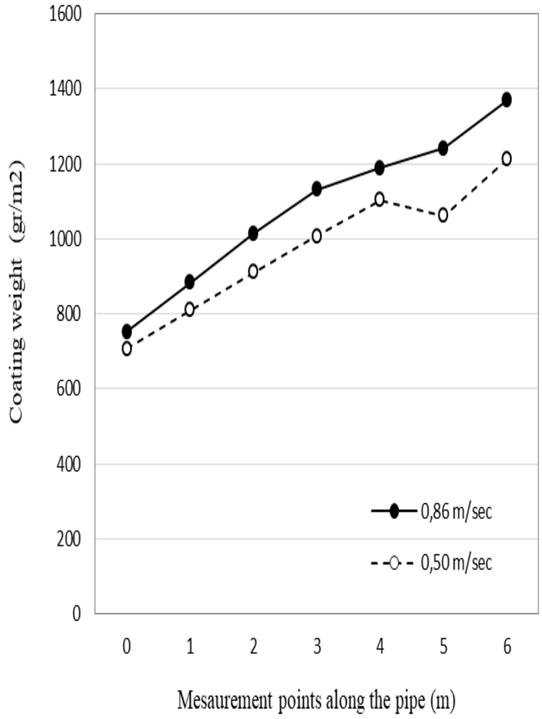
Table 8**.** Process paramaters for test III.

Test No	Group Code	Pipe Size (mm)	Withdrawal Speed (m/s)	Dipping Time (sec/piece)	Zinc Bath Temperature (°C)	Inside Blowing Pressure (bar)
III	G	½”	0,86	112	450	0
	H		0,50			
	J	¾”	0,67	72	455	
	K		0,87			
	L		1,20			

Table 9**.** Coating thickness along the pipe length (gr/m^2).

Group Code	Pipe No	Measurement points along to pipe length							
		0	**1**	**2**	**3**	**4**	**5**	**6**	**Ort.**
G	1	745	859	998	1095	1176	1250	1325	**1064**
	2	720	813	941	1033	1108	1181	1390	**1027**
	3	757	923	1061	1164	1228	1250	1393	**1111**
	4	791	935	1059	1237	1244	1282	1368	**1131**
		753	**883**	**1015**	**1132**	**1189**	**1241**	**1369**	**1083**
H	1	661	804	942	1026	1040	1014	1250	**962**
	2	707	746	848	1029	1095	1015	1271	**959**
	3	725	850	913	1019	1083	1121	1190	**986**
	4	734	841	940	958	1197	1092	1131	**985**
		707	**810**	**911**	**1008**	**1104**	**1061**	**1211**	**973**
J	1	618	805	976	1120	1245	1286	1419	**1067**
	2	672	873	960	1090	1241	1206	1444	**1069**
	3	532	806	930	1060	1220	1244	1401	**1028**
	4	585	839	946	1076	1212	1226	1330	**1031**
		602	**831**	**953**	**1087**	**1230**	**1241**	**1399**	**1049**
K	1	552	819	964	1165	1231	1354	1438	**1075**
	2	642	854	999	1145	1226	1370	1428	**1095**
	3	617	809	989	1048	1205	1351	1402	**1060**
	4	647	852	1031	1123	1210	1429	1363	**1094**
		615	**834**	**996**	**1120**	**1218**	**1376**	**1408**	**1081**
L	1	643	937	1142	1209	1284	1592	1592	**1200**
	2	645	946	1177	1147	1431	1693	1482	**1217**
	3	586	848	1126	1205	1178	1410	1397	**1107**
	4	609	896	1143	1194	1147	1412	1588	**1141**
		621	**907**	**1147**	**1189**	**1260**	**1527**	**1515**	**1166**

Fig 4. Withdrawal speed effect on coating thickness.



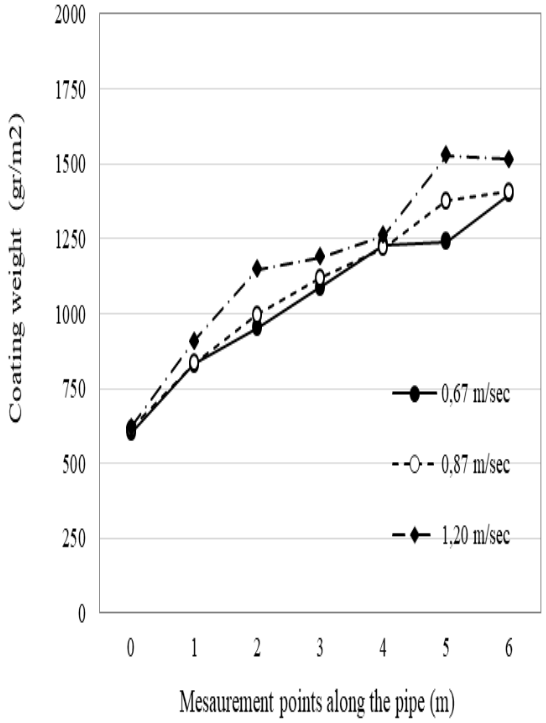


Table 9 gives the obtained coating thickness in grs/m^2 for ½" and ¾" pipes. It is noted that, the average thickness is 1510 grs/m^2 for 1,2 m/sec. withdrawal speed and 1428 grs/m^2 for 1,0 m/sec. withdrawal speed. There is around 5,5% decrease in the amount of zinc carry over in grs/m^2 inside surface area. The other diameter also show same tendency. For instance, 3/4" diameter pipes, average coating thickness is 1049 grs/m^2 for 0,67 m/sec withdrawal speed, while it is 1166 grs/m^2 for 1,20 m/sec.

a) It is obviously that zinc outlet, coming from the inside surface of the pipes during the extraction, is directly related to withdrawal speed. The slower withdrawal speed cause to having less zinc carry over on the pipe surface because of longer extraction time.

b) There is always a big risk to stay under the 400 gr/m^2 as coating thickness after blowing if the zinc layer temperature is still too high results from short extraction time. It is noted that, slower withdrawal speed allows longer time to zinc layer to solidify.

That will help to the galvanizers to perform standard requirements for inner coating thickness with the minimum usage of zinc. On the other hand, too long extraction time causes to solidify zinc layer before coming to inside blowing station. This results in poor dust collection and also unacceptable rough surface.

c) The lower withdrawal speed result in thinner zinc coating. On the other hand, too slow extraction causes both unacceptable surface and low production speed. Therefore, it is suggested that, each mill should make their own trials to establish a good balance between zinc usage and production speed by defining withdrawal speed and inside blowing pressure according to diameter of the pipe.

Inside Blowing

The main subject of the steel pipe galvanizer's is to keep the inside coating thickness in a minimum level which already should match the requirement of the standards. Coating weight is mainly affected by the amount of zinc removed by inside blowing. Therefore, in the steel pipe galvanizing process, the most important factor is the internal blowing conditions. The purpose of inside blowing is to remove excess zinc from the inner surface area of the pipes while withdrawing the pipes from the bath [17].

In order to see the effect of inner cleaning, the experiments were carried out in two groups as steam blowing and air wiping. Firstly, experiments were made with three different pressures, 6, 8 and 10 bar, in steam blowing (Table 10). It was observed that the amount of zinc sprayed through the pipe increased with the increase in steam pressure (Table 11). However, it was observed that the zinc coating layer thickness was not homogeneous. Along the inner surface of the pipes, it was determined that the coating thickness in some areas fell below the standard values, while in some areas it was excessively thick (Fig 5).

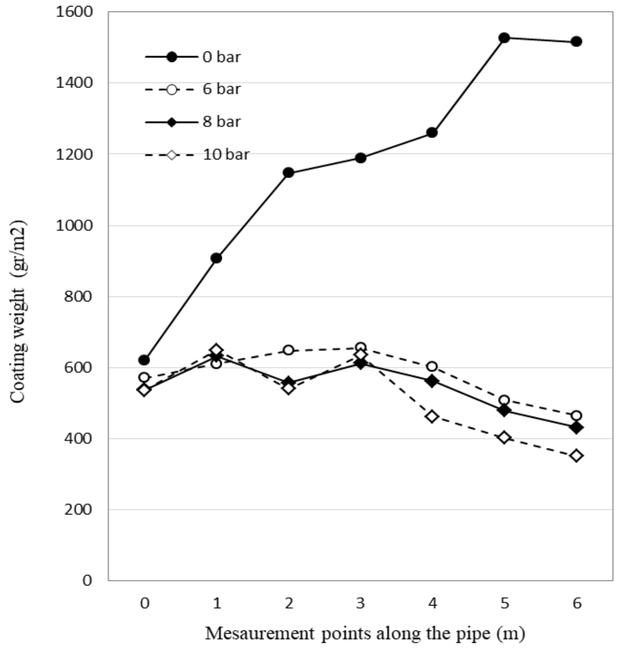
Table 10. Process paramaters for test IV.

Test No	Group Code	Pipe Size (mm)	Inside Blowing Pressure (bar)	Dipping Time (s/piece)	Withdrawal Speed (m/s)	Zinc Bath Temperature (°C)
IV	M	¾"	0	72	1,20	455
	N		6			
	P		8			
	R		10			

Table 11. Coating thickness along the pipe length (gr/m^2).

Group Code	Pipe No	Measurement points along to pipe length							
		0	**1**	**2**	**3**	**4**	**5**	**6**	**Ort.**
M	1	643	937	1142	1209	1284	1592	1592	**1200**
	2	645	946	1177	1147	1431	1693	1482	**1217**
	3	586	848	1126	1205	1178	1410	1397	**1107**
	4	609	896	1143	1194	1147	1412	1588	**1141**
		621	**907**	**1147**	**1189**	**1260**	**1527**	**1515**	**1166**
N	1	587	572	631	694	604	540	475	**586**
	2	620	594	692	721	598	523	501	**607**
	3	516	621	586	540	569	494	436	**537**
	4	559	657	682	669	638	475	446	**589**
		571	**611**	**648**	**656**	**602**	**508**	**465**	**580**
P	1	529	609	572	626	535	477	396	**535**
	2	566	637	561	597	520	433	420	**533**
	3	492	625	536	614	587	532	434	**546**
	4	564	656	558	610	607	472	473	**563**
		538	**632**	**557**	**612**	**562**	**479**	**431**	**544**
R	1	505	627	590	562	488	418	325	**502**
	2	572	659	507	709	383	369	355	**508**
	3	510	632	545	544	479	413	345	**495**
	4	557	677	516	726	498	408	380	**537**
		536	**649**	**540**	**635**	**462**	**402**	**351**	**511**

Fig 5. İnside blowing pressure effect on coating thickness.



Finally, with the help of a nozzle inserted into the pipe by means of a movable lance, pipe cleaning was carried out by using air. It has been observed that a much more homogeneous and thinner coating thickness is obtained in this method compared to steam blowing.

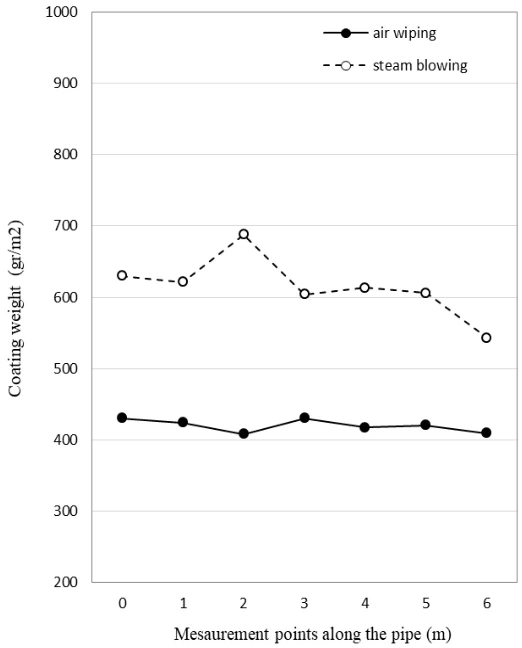
Table 12**.** Process paramaters for test V.

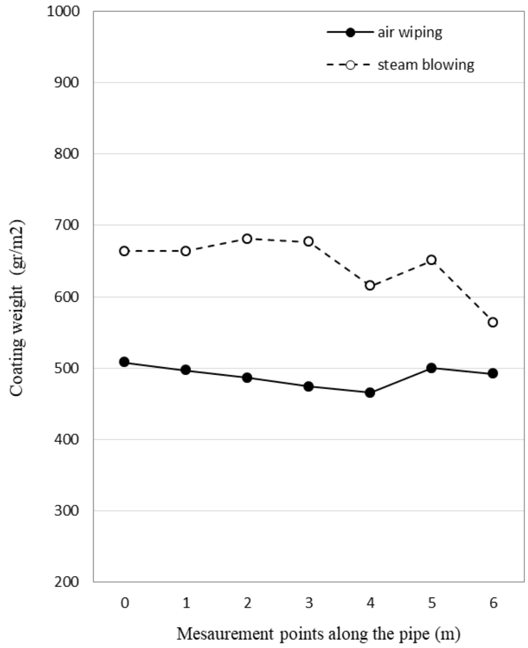
Test No	Group Code	Pipe Size (mm)	Zinc Bath Temperature (°C)	Inside Blowing Media	Inside Blowing Pressure (bar)
V	S	2"	451	air	2
	T			steam	9
	Z	4"	454	air	2
	Y			steam	9

Table 13**.** Coating thickness along the pipe length (gr/m^2).

Group Code	Pipe No	Measurement points along to pipe length							
		0	**1**	**2**	**3**	**4**	**5**	**6**	**Ort.**
S	1	417	428	404	432	410	409	413	**416**
	2	436	425	413	437	411	418	402	**420**
	3	437	419	406	421	429	432	413	**422**
		430	**424**	**408**	**430**	**417**	**420**	**409**	**420**
T	1	629	614	714	631	614	620	551	**625**
	2	638	619	683	578	638	600	584	**620**
	3	622	631	668	602	586	598	495	**600**
		630	**621**	**688**	**604**	**613**	**606**	**543**	**615**
Z	1	527	486	492	465	470	486	490	**488**
	2	486	494	478	477	459	510	497	**486**
	3	510	510	489	481	465	504	489	**493**
		508	**497**	**486**	**474**	**465**	**500**	**492**	**489**
Y	1	656	672	681	674	645	645	590	**652**
	2	662	659	692	674	662	649	582	**654**
	3	674	660	671	683	539	660	519	**629**
		664	**664**	**681**	**677**	**615**	**651**	**564**	**645**

Fig 6. Comparison of air wiping and steam blowing.





Advantages of blowing dry air with the lancet system are given below;

- Obtaining a very homogeneous zinc coating thickness on the inner surface,
- Absence of zinc deposits on the inner surface of the pipes, and having a glossy finish.
- A huge reduction in the amount of zinc dust collection generated during the process,
- Significant savings in zinc consumption because of returning the excess zinc sprayed from the inner surface of the pipe to the molten zinc bath as metallic zinc,
- Absence of noise and steam fumes in the working environment,
- Elimination of risky situation created by excessive humidity in dust collection units.

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