The use of gypsum as an additive in waterborne base coats for automotive coating.

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ABSTRACT

The Automobile industry focuses interest in waterborne base coat lately due to low production output, long flash off time and adhesion before curing. Efforts are been made in researching for base coat formulation to ensure reduction of flash off time while maintaining and improving their properties. The paper investigates the effect of gypsum in waterborne base coats for automotive coating, formulation, preparation and drying of the waterborne base coat samples at various temperatures. The samples of deposited coat were weighed and analysed in Minitab software using Taguchi design. The waterborne base coat was observed when sprayed, having no splashing and droplet on the sample surface. The gypsum facilitated the evaporation of moisture from the film in a shorter time at low temperature of $50^{\circ}C$ which is called the flash off time before curing with clear coat. The flash off time of 7-8minutes was determined at $50^{\circ}C$ for the formulation. Adhesion test was carried out on the films and good adhesion between the primer, base coat and clear coat were achieved. Drying at $50^{\circ}C$ helps in the reduction of energy consumption in the automobile industry.

Keywords: Automobile, Base coat, Gypsum, Flash off time, Adhesion

1.0 Introduction

The incredible changes in the automotive coating industries occurs largely due to changes in process development and chemical nature of the film former for mechanical and adhesion properties. Research continues to formulate to improve the topcoats performance and the reduction in flash off time in automotive coating industries to maximise production output. The topcoat which is the final coat in an automotive, consist of two layers which are the base coats and clear coats. The base coats gives colour to the automotive (Streitberger, 2008). They are applied over the primer surfacer and covered by the clear coats layer to protect it from the environment. The main requirements for base coat formulation are high opacity and good intercoat adhesion to primer and to clear coats (Poth, 1995). The thickness of prime surfacer, base coat and clear coat differs from each other and the base coat thickness which is approximately $15\mu m$ is shown in Figure 1 (Nelson et al, 2016).

The base coats which are of two type's solventborne base coats and waterborne base coats. The waterborne base coats are preferred over solventborne base coats due to heavy emission of solvent and in compliance with the legislative law (Poth, 1995). Waterborne base coats have become the main base coat technology for all new automotive coating industries built after the year 2000 and have captured most of the market in the world. This success is driven partly by the environmental benefits, and partly by their superior performance and robust application properties (D¨ossel, 2008). The waterborne base coat consists of water and the dry coating layer is form by a solid

fraction of resin, pigments and additives. The drying process of these materials has to be optimised to achieve a desired quality of the final coating and to reduce flash off time. The time when an automobile is placed in the oven and begins to dry is called the flash off time (Stephan, 2005). The research described is part of the development of an innovative technology for waterborne base coats with a superior mix of material and application properties of drying and curing of films.

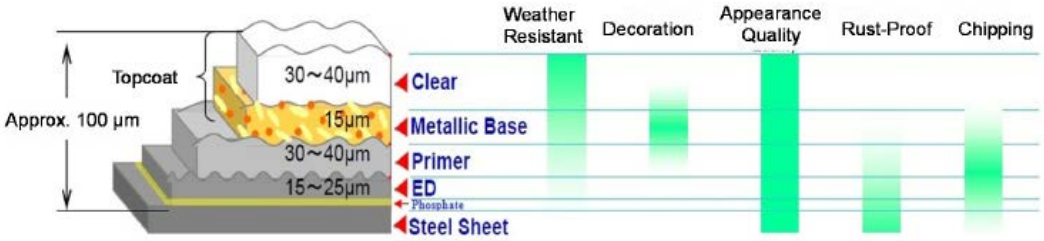

Figure 1: Automotive Coating Layers, thicknesses and purposes for An exterior Surface (Nelson et al, 2016).

2.0 Materials and Methods

2.1 Materials

The materials for formulation of automotive coating were resin (Polyvinyl formal), pigments (Titanium oxide), additive (Gypsum), distilled Water, automotive paint (clear coat), and primer surfacer. The sample surface used for the research experiment was Mild steel plates of size 160mm x 160mm × 0.8mm.

2.2 Methods

The process of coating was carried out following the automobile coating industry procedure (Prendi, 2005). The samples surfaces were cleaned and prepared for deposition of prime surfacer and was dried. The base coat was deposited after drying of the prime surfacer. The samples were inspected for application of a clear coat to determine adhesion between both coats.

2.2.1 Formulation and preparation of waterborne base coat

The mixture was in proportion by volume percentage between pigment, resin, water, and additives (gypsum). The coat was prepared following a standard ratio 25:30:40:5 (Center for Industry Education Collaboration, 2013). The mixture which consists of 25% Titanium oxide, 30% Polyvinyl formal, 40% water and 5% Gypsum was prepared for coating shown in Figure 2.

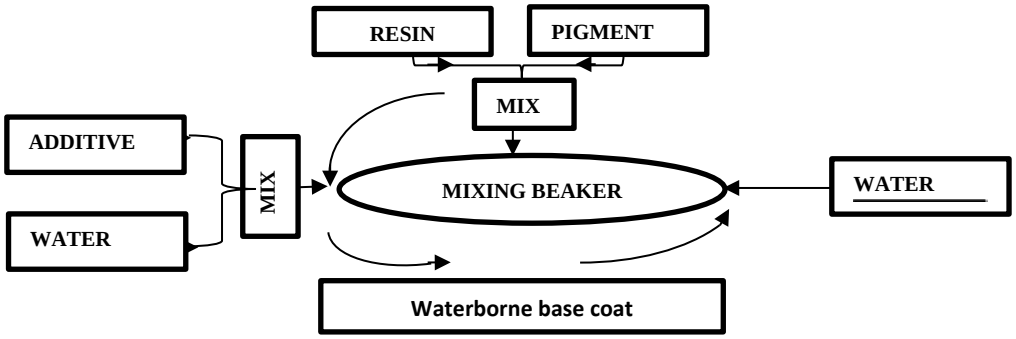

Figure 2: Preparation of waterborne base coat.

2.2.2 Drying of Samples

The waterborne base coat was applied with the gun spray vertically and horizontally. After deposition the steel plates were weighed before transferred into the oven for drying. The oven temperature was adjusted to achieve the desired flash off time for each steel plate. The temperature consider are 30˚C 35˚C 40˚C 45˚C 50˚C and the flash off time are 1-5 minutes these values correspond to conditions used in the automotive industry for forced flash-off of base coats before the application of clear coat(Domnick et al, 2011). The moisture content left was determine using the drying percentage expressed mathematically in equation (1)

$$D = \frac{w_2 - w_3}{w_2 - w_1} \times 100 \quad (1)$$

Where, D is drying percentage, w_1 is weight before spraying (g), w_2 is weight after spraying (g), and w_3 is weight after drying (g).

2.2.3 Factors and Drying Parameters Specification

The waterborne base coat was used with desired flash-off time and temperatures on the experimental samples using Taguchi Design. The experimental samples were analysed in three aspects by the design of experiments (DOE) which are factors, levels and responses. After editing the data with five levels and two factors each, there are 25 experimental runs taken into account in total and were two experiments were carried out manually to determine the outcome of drying.

Table 1: Factors and Level of drying parameters

Factors	*Levels*				
	1	2	3	4	5
Drying temperature (˚*C*)	30	35	40	45	50
Flash off time (*min*)	1	2	3	4	5

2.2.4 Drying rate experiment

The best parameter of drying analyzed in the taguchi design was used to determine the rate of drying for the steel plate, placed in the oven and under constant drying conditions, the loss in weight of moisture during the drying process is determined at constant time intervals of 1minute. The rate of drying "R" can be mathematically expressed in equation (2) (Muller and Poth 2011).

$$\mathbf{R} = \frac{S}{A}\frac{dX}{dt} \quad (2)$$

where, R is drying rate (gH_2O/sm^2), S is weight of dry solid (g), A is exposed surface area for drying (m^2), X is solid moisture content (g H_2O/g dry solid) and t is time (s).

$$X = \frac{m_1}{m_2} \quad (3)$$

Where, X is moisture content, m_1 is mass of water (g) and m_2 mass of dry solid (g)

2.2.5 Testing of composition

The tests were carried on the composition of mixture. The tests carried out for liquid base coat are viscosity, pH, volume solid (VS) and pigment volume concentration (PVC) expressed in equation (4) and equation (5).

$$VS = \frac{(V_p + V_R)}{V_T} x\ 100 \quad (4)$$

Where, VS is volume solid, V_p is volume of pigment, V_R is volume of resin and V_T is total volume of paint

$$PVC = \frac{V_p}{V_p + V_R} x\ 100 \quad (5)$$

The volumes (V) are calculated as the quotient of mass (m) and density (ρ): $V = \frac{m}{\rho}$ (Muller and Poth 2011).

3.0 Results and Discussion

3.1 Formulation test results of waterborne base coat

The resulting viscosity of the formulation measured at DIN4/23˚*C* is 15seconds and the test result for pH, volume solid and pigment volume concentration carried out are shown in Table 2. The pH 0f 9.45 is considered an alkaline having a colouration of a baking soda. The pigment volume concentration of 35.04% resulted into increases in drying of coat film, adhesion, cleanability and scrubbability.

Table 2: Results of test of formulation

Viscosity (seconds)	PH	Volume solid (%)	Pigment volume concentration (%)
15	9.45	52	35.04

3.2 Drying

The resulting amount of spray coat for each samples ranging from 1.4g – 3.3g due to the spraying machine output and recoating for samples not properly coated shown in Table 4. At 50˚*C* the moisture content was less with a drying percentage of 52% which was the best outcome to carry out the drying rate.

At 50˚*C* gypsum as a drying additive enables the coat release moisture at a flash off time of approximately from 7 minutes 5 seconds to 7 minutes 15 seconds and averagely 7minutes 10 seconds without interruption giving a good quality shown in Table 3. These results validated with the data obtain by (D¨ossel, 2008).

Table 3: Result of average total drying time

Temperature(˚*C*)	30	35	40	45	50
Total average of drying time (seconds)	920	860	691	580	430

Table 4: The experiments were carried out manually and the measured values were added in the design

Input Parameter			Output				
S/N	Temperature(˚C)	Flash off time(min)	Amount of spray coat(g)	Amount after drying(g)	Drying (%)	SNRA1	MEAN1
1	30	1	2.2	1.5	31.820	-27.0437	15.92
2	30	2	1.9	1.2	36.840	-28.3161	18.4275
3	30	3	2.1	1.5	26.571	-25.4779	13.29125
4	30	4	1.9	1.2	36.840	-28.3161	18.4275
5	30	5	2.0	1.2	40.000	-29.0309	20.0125
6	35	1	2.7	1.8	33.333	-27.4472	16.674
7	35	2	2.7	1.8	33.333	-27.4472	16.6765
8	35	3	2.7	1.8	33.333	-27.4472	16.6765
9	35	4	2.8	1.7	39.290	-28.8753	19.6525
10	35	5	2.5	1.5	40.000	-29.0309	20.0125
11	40	1	1.5	1.0	33.333	-27.4472	16.669
12	40	2	1.4	0.8	42.850	-29.6287	21.43
13	40	3	1.4	1.0	28.570	-26.1079	14.29
14	40	4	1.6	0.8	50.000	-30.9691	25.0075
15	40	5	1.6	0.8	50.000	-30.9691	25.0075
16	45	1	1.6	0.9	43.750	-29.8093	21.8825
17	45	2	1.7	0.9	47.060	-30.4427	23.5375
18	45	3	1.7	0.9	47.060	-30.4427	23.54
19	45	4	1.7	0.9	47.060	-30.4427	23.5375
20	45	5	1.4	0.7	50.000	-30.9691	25.005
21	50	1	2.7	1.5	44.400	-29.9374	22.21
22	50	2	2.5	1.2	52.000	-31.3098	26.0075
23	50	3	3.1	1.5	51.600	-31.2427	25.8075
24	50	4	3.3	1.6	51.500	-31.2258	25.76
25	50	5	2.5	1.2	52.000	-31.3098	26.01

3.2.1 Main Effects Plots for S/N ratios for drying percentage

The result of the main effects plot for the signal-to-noise ratio was is shown in Figure 3. The best outcome based on Figure 3 using smaller is better for the analysis was 50˚*C* and flash off time of 5minute.

This also shows the ranking lists of the two factors from the most important to the least important.

Temperature is the most important factor that causes variation on drying of waterborne base coat followed by the flash off time shown in Figure 3. The temperature at 30˚*C* has the least effect on drying of the coat and 50˚*C* has more effect on drying of the coat. At 50˚*C* gypsum as a drying additive enables the coat release moisture at a flash off time of approximately from 7 minutes 5 seconds to 7 minutes 15 seconds and averagely

7minutes 10 seconds without interruption giving a good quality. These results validated with the data obtain by (D¨ossel, 2008).

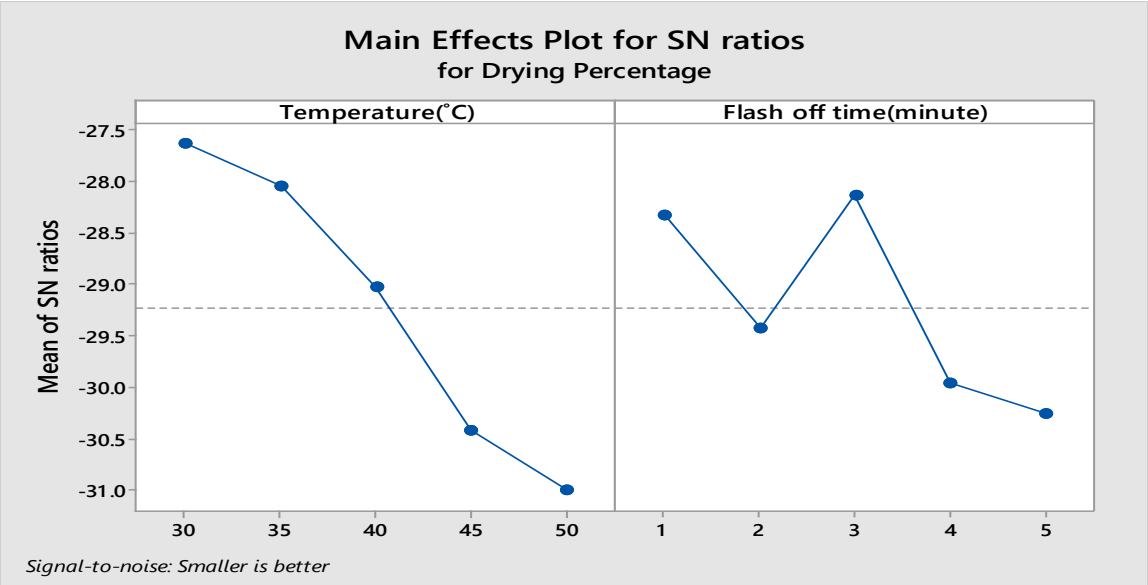

Figure 3: Main Effects Plots for Signal to Noise ratios for drying percentage

3.2.2 Analyse of variance method (ANOVA)

In this experiment, ANOVA was used to analyse the effects of temperature and flash off time on drying percentage. The ANOVA result for the drying percentage is shown in Table 5 This analysis was carried out a 5% significance level and a 95% confidence level. The significance of control factors in ANOVA is determined by comparing the F values of each control factor. The last column of the table shows the percentage value of each parameter contribution which indicates the degree of influence on the process performance. According to Table 5, the percentage contribution of the Temperature and flash off time factors on the drying percentage is found to be 58.13% and 21.59%. Thus, the most important factor affecting the drying percentage was Temperature at 58.13%. The percentage of error was considerably low at 20.28%.

Table 5: Results of ANOVA for drying percentage.

Variance source	Degree of freedom (DOF)	Sum of squares (SS)	Mean square (MS)	F ratio	Contribution rate (%)
Temperature(˚C)	4	948.4	237.10	15.24	58.13
Flash off time(minute)	4	352.3	88.09	5.66	21.59
Error	16	248.9	15.56	-	20.28
Total	24	1549.7	-	-	100

3.2.3 Drying Rate

The best outcome gotten from the Taguchi design it was implemented in experimental determination of rate of drying, $50^{\circ}C \pm 5^{\circ}C$ and 7 minutes was used at an interval of 1minute to determine the rate of drying. The drying rate results are presented in Table 6

Table 6: Results of drying rate

Drying Time(minute)	0	1	2	3	4	5	6	7	8
Weight after dried(g)	272.0	270.7	270.1	269.4	268.7	268.2	267.5	267.1	267.1

The solid moisture content(X), slope of plotted moisture content with time and drying rate(R) were determine using equation (2) and Equation (3) shown in Table 7

The moisture content was plotted against time shown in Figure 4, the rate of drying curve was obtained by measuring the slopes of the tangents drawn in Figure 6, and the values of dX/dt was obtained at values of time. The rate of drying(R) was calculated for each point using equation (2). The rate of drying curve was obtained by plotting R against solid moisture content (X) as in Figure 5.

The rate of drying curve is majorly on two points, constant and falling rate period. At time zero the initial moisture content of the solid is shown at point A depending on the solid temperature. In Figure 5 the rate of drying curve for constant drying conditions is shown. At point A the solid is at normal room temperature and when inserted in the oven of its ultimate temperature the rate of evaporation was increased and the weight of the solid decreased to Point B. At point B the temperature of solid rises which started from point A. This state of adjustment temperature and time during weighing the solid are usually quite short and is often ignored in the analysis of times of drying.

From point C to D Figure 4 there was a little decrease in temperature which leads a decrease of evaporation. From point CDEF to G the line is straight and hence the slope and rate are constant during this period. This constant rate of drying period is shown as line CG in Figure 5 at both figures the rate of drying start to decrease in the falling rate period at point C to reach point G. The falling rate period is usually shown linear and is represented in Figure 5 form point CDEF to G.

At point B, the evaporation rate was high which lead to insufficient of water on the surface at Point C in Figure 5 to maintain a continuous film of water. The solid was no longer wet and there was continuous decreases in weight at each point in the falling rate period from CDEF to G were the solid was completely dried.

The final falling rate period which began from point G were the solid was dried and there was no detectable change until the equilibrium moisture content of the solid is reached, at point H.

The time required to remove an amount of moisture in the falling rate period at each point was small. This can be seen in Figure 5. The period AB for constant rate drying last for about 1minute and reduce X from 0.028 to 0. H_2O/g dry solid a reduction of

0.0057gH_2O/g dry solid. The falling rate period from B to H lasted for 6minutes and gave a reduction of X only from 0.0232 to 0.0095H_2O/g dry solid. The gypsum affected the drying with it capability to release water from the surface at 50℃ in a short period of time. At point A to B there was a drastic reduction of weight due to elevated temperature which occurs from room temperature to the oven temperature. The plotted rate of drying curve was validated with (Stephan, 2005) at 50℃ give gypsum an advantage to affect the drying rate curve.

Table 7: Results of Calculated solid moisture content and rate of drying

S/N	Drying Time(minute)	X (g H_2O/g dry solid)	dX/dt	R(g/s m^2)
1	0	0.0280	0.0050	51.68
2	1	0.0231	0.0050	51.68
3	2	0.0208	0.0045	46.51
4	3	0.0181	0.0040	41.34
5	4	0.0155	0.0035	36.18
6	5	0.0136	0.0030	31.01
7	6	0.0120	0.0025	25.84
8	7	0.0095	0.0010	10.34
9	8	0.0095	0.0000	00.00

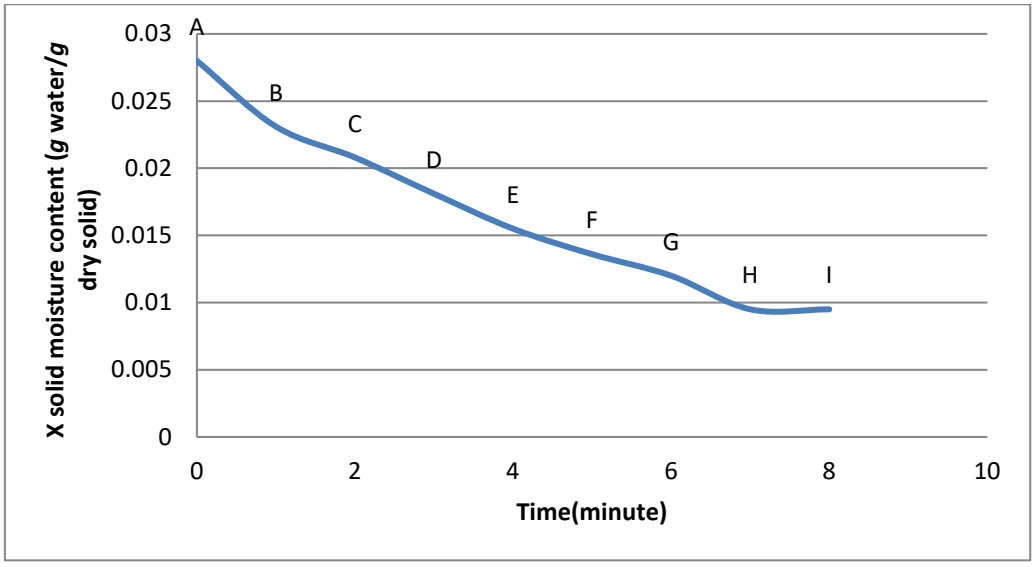

Figure 4: X solid moisture content versus time for constant drying conditions

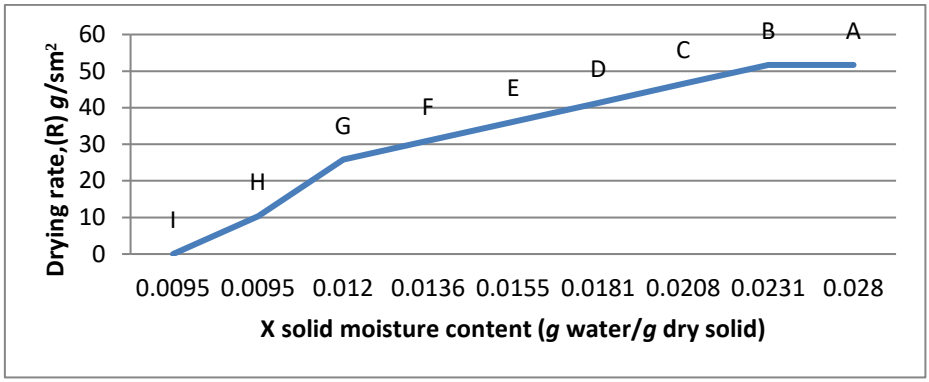

Figure 5: Rate of drying (R) versus X solid moisture content for constant drying conditions

4.0 Conclusions

The use of gypsum in waterborne base coats for automotive coating applications has been experimental investigated in formation and drying of the films.

In this research, waterborne base coat formulation process was successfully developed using 5% of Gypsum as an additive indicated it use. Gypsum can be used as a drying additive in coating, additives that increase coat viscosity immediately after a surface is sprayed and thereby prevent pigment movement and disorientation. Gypsum also gives good sealing properties and protects the sample from environmental damages and increases adhesion with the prime surface. The coat's cleanability and scrubbability increases lead to durability of the coat on the substrate.
Temperature has the strongest influence on gypsum on the total drying rate in the reduction of flash off time. The low temperature helps in the reduction of consumption of energy and reduces cost in the automobile industry.

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