PRODUCTION AND OPTIMIZATION OF PIGMENTS AND BINDER IN LOW-COST EMULSION HOUSE PAINT USING RESPONSE SURFACE METHODOLOGY

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Abstract

Paint is any liquid, liquefiable or mastic composition which after application to a substrate in a thin layer is converted to an opaque solid film. The objective of this research was to investigate the effects of some basic paint components i.e., prime pigment, extender pigment and binder on pH, viscosity and density on white emulsion paint formulation. The research was aimed at producing low-cost white emulsion house paint and optimizing the pigments and binder in other to obtain the optimal conditions for which the best paint formulation can be obtained using the Response Surface Methodology (RSM). The pH, density, viscosity and cost were chosen as the responses while the prime pigment, pigment extender and binder were selected as the factors. Twenty (20) different samples of white emulsion house paint were successfully formulated and produced using Central Composite Design. The physico-chemical properties of twelve (12) of the produced paints fell within the conventional specified range by Nigeria Industrial Standard. The results obtained revealed that the pH, viscosity and density were largely affected by the concentration of the prime pigment, pigment extender and binder. Also , the analysis of the variance (ANOVA) of the models showed that the developed models for viscosity, density, pH and cost were significant with F-values of 109.19, 99.55, 10.31 and 2278.53 respectively, and P-values less than 0.05 (P<0.05). In addition, the generated optimum conditions of the desired paint formulation were 66.85 g for the prime pigment, 440.56 g for the pigment extender and 0.108 L for the binder while the quality test optimum conditions were 5.819 poise for viscosity, 1352.836 g/L for density and 7.035 for pH at a cost of ₦336.465. Using the optimum conditions to validate the experiment, the obtained experimental values of 5.95 poise of viscosity, 1380 g/L of density and pH of 7.30 were close to the predicted values and it confirmed the validity and adequacy of the predicted models. While Percentage error deviation of 2.26%, 2.0%, 2.64% and 2.5% were calculated for the viscosity, density, pH and cost, respectively.

Keywords: *Emulsion; Prime pigment; Pigment extender; Binder; Optimum condition; Quality Parameters*

1.0 Introduction

Paint can be defined as any liquid, liquefiable or mastic composition which after application to a substrate in a thin layer is converted to an opaque solid film (1). Paint can also be defined as a liquid solution of pigment and solvent, which is applied on different surfaces for decorative and protective reasons. Emulsion house paint is a water-based paint principally used for internal and external surface coatings, mostly in buildings for appearance and protection. The processes involved in paint production, quality and performance of emulsion paint depends largely on the properties of its constituents and the ratios of these constituent (2). The constituents generally used for the

production of emulsion house paints include prime pigments, solvents, extenders pigments, binders and additives.
In the constituents for the production of emulsion house paints, prime pigment (titanium dioxide) and binder (PVA) usually takes between 50 to 70% total cost of production depending on paint type and this raw material are not available locally, therefore imported. Hence, high cost of this materials (PVA and Titanium dioxide) and consequently high cost of production due to the use of expensive binder and prime pigment (3). Another challenge with emulsion paint production is the challenge of selecting suitable composition of pigments and binder (ratio of prime pigment, extender pigment to binder) for standard house paint formulations. A large percentage of commercial paint in the market compromise quality for low cost. Hence, the need to investigate into standard house paint at relatively low cost (4). Due to the aforementioned problems, the aim of this research was to produce high quality household white emulsion paint by optimizing the pigments and binder using response surface methodology. The objectives were to produce White Emulsion House Paints using standard formulation via Response Surface Methodology (RSM) design of experiment, optimizing the three main process variables (prime pigment, extender pigment and binder) and carrying out quality parameter test on the produced paints.

Optimization of various process factors affecting paint production is a complex process with a number of interactive controlling parameters. At industrial level, even a small improvement in the process, gives a better yield which may be beneficial commercially, making process optimization a major area of research in the field of industrial biotechnology. Therefore, there is a need for optimization of accurate process parameters which, improves the quality of the paint significantly. In this study, optimization of process controlling factors was done by Response Surface Methodology (RSM). RSM is a statistical technique for analysing the effects of several independent variables on the responses. RSM is important in process design and optimization as well as for improving the performance of the system. This study will definitely alleviate the problems of high cost of commercial paints, improve the quality of produced paints by obtaining the best formulation ratio or formula, and on a long run serves as means of creating employment. The study is limited to laboratory formulation and production of emulsion house paint and to carry out the quality parameters tests on the produced paints. These parameters include viscosity, pH, density and cost.

2.0 Materials and Methods

2.1. Raw Materials

Raw materials used for the production of white emulsion house paint were purchased from Panteka Market Kaduna, Kaduna State-Nigeria and Muda- Lawal Market Bauchi, Bauchi State-Nigeria. List of materials, their brands and a typical “Emulsion House Paint” formulation are presented in Table 1.

2.2 Procedure for paint production

Zero-point four litre (0.4 L) of water was measured into a mixer, the stirrer (Model 50111, Type RZR1), was then switched on and set at 20 rpm. Measured amount of titanium dioxide as indicated in Table 2 was gradually dispersed into the mixing tank and allowed to grind for 5 min. Measured amount of calcium carbonate as indicated also in Table 2 was then dispersed and allowed to grind for another 10 min and other ingredients were added as itemized in Table 2 in their measured quantities. It should be noted that the thickener was added gradually, after which the mixture was allowed to grind properly before packaging.

Table 1. Raw materials and formulation of a typical emulsion house paint

Raw Material	Brand	Quantity
Water (L)	-	0.40000
Polyvinyl acetate (L)	Pexi Chem Private Ltd. New Delhi	0.10000 - 0.60000
Calcium carbonate (kg)	Calco, Freedom Groups	0.40000 – 0.60000
Titanium dioxide (kg)	Du Pont Ti Pure	0.06000 – 0.08000
Antifoam (kg)	SAF 130	0.00400
Texanol (kg)	East Man Texanol™	0.00600
Genepor (kg)	Drugbrand DB00305	0.00160
Formalin (kg)	Bamboo Charcoal	0.00400
Ammonia, (28%) (L)	Bird Brand Ammonia	0.00600
Thickener (Natrosol)	Natrosol Performax ™	0.00600

Table 2: A Typical Formulation of Emulsion House Paint

S/N	Chemical	Quantity
1	Water (L)	0.400
2	Antifoam (kg)	0.004
3	Texanol (kg)	0.006
4	Genepor (kg)	0.0016
5	Formalin (kg)	0.004
6	Ammonia (28%/soln)L	0.006
7	Nitrosol (kg)	0.006
8	Brightener	2 drops
9	$CaCO_3$ (kg)	0.4 – 0.6
10	PVA (L)	0.1 – 0.6
11	TiO_2 (kg)	0.06 – 0.08

2.3 Design of Experiments with Response Surface Methodology (RSM)

Design of experiment (DOE) is a well-accepted statistical technique able to design and optimize the experimental process that involves choosing the optimal experimental design and estimate the effect of the several variables independently and also the interactions simultaneously (4). Response surface methodology (RSM) is a statistical method used for experimental modelling and analysing the relationship between the input and response variables (5). In this study three process variables viz. Prime pigment, Extender pigment and Binder were selected as independent factors while pH, Viscosity, Density and cost were chosen as responses.

Table 3: Coded Values of Independent Factors and Experimental Ranges

Factor	Name	Units	Levels	
			Low	High
A	Prime Pigment	g	60.00	80.00
B	Extender Pigment	g	400.00	600.00
C	Binder	L	0.1000	0.1400

The basis of the experimental design was 1 L, and the levels were chosen based on the paint formulations by (5). Analysis of variance (ANOVA) was used for analysis of regression coefficient, prediction equations, and case statistics. Diagnostics plots and model graphs were obtained using the Design expert V.12 to analyse the effects of variables individually and their interactions to determine their optimum level.

2.4 Quality parameters Test of paints produced.

Quality parameters test were carried out on the twenty paint samples produced using the following methods, viscosity, Density, pH (digital pH meter).

3.0 Results and Discussions

3.1 Optimization of Paint production

The objective of this study was to investigate the effects of some of the basic paint components i.e prime pigment, extender pigment and binder on pH, viscosity and density on white emulsion paint. The optimal levels for the independent variables and the effect of their interaction on paint production were further explored using the central composite design of RSM. The three factors involved in this study were prime pigment, pigment extender and binder. By using CCD, a total of 20 runs were generated with different set up condition. The responses were viscosity, density, pH and cost of the produced paints as listed in Table 4 below. It should be noted that these values were generated by the design expert software. The response values in Table 5 were obtained from the laboratory experimental run. With CCD, the goodness of fit was able to be determined by coefficient of determination, R-squared while the statistical significance of the regression model was checked by the Fisher statistical test (F-test) in analysis of variance (ANOVA).

Nigerian Journal of Tropical Engineering
Vol. 16 No. 1 |ISSN: 1595-5397| Dec. 2022 **DOI: 10.59081/njte.16.1.003**

Table 4: Experimental Set-up for 2-level-3-factor by Central Composite Design

Run	Factors			Responses	
	Prime Pigment (g)	Pigment Extender (g)	Binder (L)	Cost	Quality
1	80	500	0.12		
2	70	500	0.12		
3	64	559	0.11		
4	64	559	0.13		
5	70	500	0.14		
6	70	500	0.10		
7	76	559	0.11		
8	64	441	0.13		
9	76	441	0.13		
10	70	400	0.12		
11	64	441	0,11		
12	76	559	0.13		
13	70	500	0.12		
14	70	500	0.12		
15	70	500	0.12		
16	70	500	0.12		
17	70	600	0.12		
18	76	441	0.11		
19	60	500	0.12		
20	70	500	0.12		

Table 5: Experimental Set-up for 2-level-3-factor by Central Composite Design

Run	Factors			Responses			
	Prime Pigment (g)	Pigment Extender (g)	Binder (L)	Viscosity poise	Density g/L	pH	Cost ₦
1	80.00	500.00	0.12	5.90	1287	6.31	385.00
2	70.00	500.00	0.12	5.91	1410	7.45	367.50
3	64.00	559.00	0.11	5.79	1260	7.21	362.80
4	64.00	559.00	0.13	5.71	1371	8.77	381.70
5	70.00	500.00	0.14	5.73	1418	8.60	387.50
6	70.00	500.00	0.10	5.70	1300	7.35	347.50
7	76.00	559.00	0.11	5.82	1260	6.55	382.80
8	64.00	441.00	0.13	5.71	1335	6.35	352.20
9	76.00	441.00	0.13	5.89	1283	6.32	373.20
10	70.00	400.00	0.12	5.91	1250	6.59	342.50
11	64.00	441.00	0.11	5.79	1360	6.74	332.20
12	76.00	559.00	0.13	5.88	1410	7.35	402.80
13	70.00	500.00	0.12	5.92	1400	7.75	367.50
14	70.00	500.00	0.12	5.92	1408	7.90	367.50
15	70.00	500.00	0.12	5.93	1400	7.71	367.50
16	70.00	500.00	0.12	5.91	1404	8.00	367.50
17	70.00	600.00	0.12	5.93	1300	7.99	392.50
18	76.00	441.00	0.11	5.82	1261	6.90	353.20
19	60.00	500.00	0.12	5.69	1376	6.41	350.00
20	70.00	500.00	0.12	5.93	1400	7.01	367.50

3.2 Statistical analysis of the responses

3.2.1 Viscosity

The optimal levels for the independent variables and effect of their interaction on paint production were explored using the central composite design of RSM. By applying multiple regression analysis on the experimental data, the second order polynomial equation (1) was generated to explain the viscosity of the produced paints.

$$\text{Viscosity} = 5.92 + 0.0559A + 0.0017B + 0.0015C - 0.0013AB + 0.00362AC - 0.0013BC - 0.0446A^2 - 0.0004B^2 - 0.0729C \quad \ldots (1)$$

Where A, B, C are the coded values for prime pigment, pigment extender and binder respectively. The statistical significance of the second order polynomial equation was checked by an F-test (ANOVA). All the corresponding data are shown in Table 6.

Table 6: Viscosity Regression Analysis for Quadratic Response Surface Model Fitting.

Model	Sum of squares	df	Mean square	F-value	P-value	
Model	0.1518	9	0.0169	109.19	<0.0001	significant
A Prime pigment	0.0426	1	0.0426	276.19	<0.0001	
B Pigment extender	0.0000	1	0.0000	0.2649	0.6180	
C Binder	0.0000	1	0.0000	0.1984	0.6655	
AB	0.000	1	0.0000	0.0809	0.7818	
AC	0.0105	1	0.0105	68.08	<0.0001	
BC	0.0000	1	0.0000	0.0809	0.7818	
A^2	0.0287	1	0.0287	185.91	<0.0001	
B^2	2.769E-06	1	2.769E-06	0.0176	0.8961	
C^2	0.0766	1	0.0766	496.20	<0.0001	
Residual	0.0015	10	0.0002			
Lack of fit	0.0011	5	0.0002	2.86	0.1367	significant
Pure error	0.0004	5	0.0001			
Cor total	0.1533	19				

The model F-value of 109.9 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. The Lack of Fit F-value of 2.86 implies the Lack of Fit is not significant relative to the pure error (Sajeena et al., 2014). There is a 13.67% chance that a Lack of Fit F-value this large could occur due to noise. Non-significant lack of fit is good. In addition, the ANOVA of the quadratic regression model demonstrated that the model was highly significant. ($P<0.05$). The model in terms of prime pigment (A), interaction between A and C (AC), prime pigment (A^2) and binder (C^2) were significant ($P<0.05$). Indicating that these two variables had an individual effect on viscosity. However, model in terms of pigment extender (B), binder (C), interaction between (AB), (BC) and (B^2) were insignificant ($P>0.05$) suggesting that there was no linear and quadratic effect of this variables on viscosity. The predicted correlation coefficient (R^2) of 0.9394 is in reasonable agreement with adjusted (R^2) of 0.9809 i.e the difference is less than 0.2. The R^2 value indicated a measure of variability in the observed response values which could be described by the independent factors and their interactions over the range of the corresponding factor. This implied that the sample variation of 98.09% of the total variation could be explained by the model and only 0.91% of it was not explained by the model. So, quadratic model was chosen for this analytical work. The percentage of coefficient of variation (CV %) is a measure of residual variation of the data relative to the size of the mean. Usually, the higher the value of CV, the lower is the reliability of experiment (Sajeena and Jose, 2014). Here a lower value of CV (0.2128%) indicated a greater reliability of the experiment. The Predicted Residual Sum of Squares (PRESS) was a measure of how well the model fitted each point in the design. The smaller the PRESS statistics, better would be the model fitting the data points. Here the value of PRESS found as 0.0015. Moreover the "Lack of Fit F-value" of 2.86 implies the Lack of Fit is not significant. The mean value of 5.84 indicates the average experimental viscosity value obtained. The obtained value falls within the desired range of Nigeria Industrial Standard, there by indicating that the generated quadratic model is best fit for the experiment.

3.2.2 Density

A second order polynomial equation (2) was also generated to explain the density of the produced paints.

Density = 1403.65 – 19.16A + 10.70B + 33.42C +23.75AB + 10.75AC +33BC – 25.38A^2 – 45.36B^2 – 15.6 … (2)

The statistical significance of the second order polynomial equation was checked by an F-test (ANOVA). All the corresponding data are shown in Table 7. The model F-value of 99.5 implies the model is significant. There is only a 0.01% chance that an F-value this large could occur due to noise. P-values less than 0.0500 indicate model terms are significant. The Predicted R^2 of 0.9242 is also in reasonable agreement with the Adjusted R^2 of 0.9790; i.e., the difference is less than 0.2.

Table 7: Density Regression Analysis for Quadratic Response Surface Model Fitting.

Source	Sum of squares	df	Mean square	F-value	P-value	
Model	73235.18	9	8137.24	99.55	<0.0001	
A	5014.05	1	5014.05	61.34	<0.0001	
B	1562.75	1	1562.75	19.12	0.0014	
C	15255.93	1	15255.75	186.65	<0.0001	
AB	4512.20	1	4512.50	55.21	<0.0001	
AC	924.50	1	924.50	11.31	0.0072	
BC	8712.00	1	8712.00	106.59	<0.0001	
A^2	9284.14	1	9284.18	113.59	<0.0001	
B^2	29648.32	1	29648.32	362.73	<0.0001	
C^2	3533.69	1	3533.69	43.23	<0.0001	
Residual	817.37	10	81.74			
Lack of fit	718.04	5	143.61	7.23	0.0244	significant
Pure error	99.33	5	19.87			
Cor total	74052.55	19				

3.2.3 pH

For the pH, the model F-value of 10.31 implies the model is significant (Table 8). There is only a 0.06% chance that an F-value this large could occur due to noise. Also, the ANOVA of the quadratic regression model demonstrated that the model was highly significant. (P<0.05). In this case B, C, AB, BC, A^2 are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction

Nigerian Journal of Tropical Engineering
Vol. 16 No. 1 |ISSN: 1595-5397| Dec. 2022 **DOI: 10.59081/njte.16.1.003**

may improve the model. Adeq. Precision ratio of 11.914 indicates an adequate signal. pH =7.64 – 0.1551A + 0.4338B + 0.2557C – 0.2763AB – 0.1187AC + 0.4163BC – 0.4848A^2 – 0.1560B^2 + 0.0862C^2 …(3). The second order polynomial equation (3) was generated by the ANOVA to explain the pH quadratic model of the produced paints. This was obtained by applying multiple regression analysis on the experimental data.

Table 8: pH Regression Analysis for Quadratic Response Surface Model Fitting.

Source	Sum of squares	df	Mean square	F-value	P-value	
Model	9.7600	9	1.0800	10.31	0.0006	significant
A	0.3285	1	0.3285	3.12	0.1077	
B	2.5700	1	2.5700	24.42	0.6180	
C	0.8930	1	0.8930	8.48	0.0155	
AB	0.6105	1	0.6105	5.80	0.0368	
AC	0.1120	1	0.1128	1.07	0.3249	
BC	1.3900	1	1.3900	13.17	0.0046	
A^2	3.39	1	3.3900	32.18	0.0002	
B^2	0.3509	1	0.3509	3.33	0.0979	
C^2	0.1070	1	0.1070	1.02	xz0.3372	
Residual	1.05	10	0.1052			
Lack of fit significant	0.4055	5	0.0811	0.6266	0.6898	not
Pure error	0.6471	5	0.1294			
Cor total	10.82	19				

3.3 Numerical analysis of the optimization process

Table 9 shows the numerical optimization constraints that were put into consideration. The prime pigment and binder which are the two most expensive components of the paint formulation were minimized. This is aimed at having a good paint formulation at a relatively low cost without compromising the quality of the paint. Hence, the cost was also minimized in other to arrive at a very good optimum condition. Table 10 shows the different solutions that were generated by the design expert software, using the Central Composite Design. Sixty (60) different solutions were generated but the best three (3) are itemized in the above table while the other solutions will be listed in Appendix 1. As highlighted on the table, the numerical optimization result revealed that solution (48) gives the best optimum conditions for a very good quality paint formulation and cost effectiveness. At this optimum condition, the optimum values for the prime pigment, pigment extender and binder were 66.849 g, 440.556 g and 0.108 L respectively while the response optimum values of 7.035 and 5.819 poise for the pH and viscosity fell within the conventional specified range by Standard Organisation of Nigeria. Though the desirability may be less than one (1) but the response values which serves as basis for the paint quality were within the desired range. The optimum cost value of ₦336.47, which also is the objective of this research, happens to be the most minimal among other solutions, thereby justifying the cost effectiveness of the chosen optimum paint formulation. Graphical representations of the response surface are shown in the following three-dimensional surface graphs.

Table 9: Numerical Optimization Constraints

S/N	Name	Goal	Lower Limit	Upper Limit	Lower Weight	Upper Weight	Importance
1	Prime Pigment	Minimize	64.05	75.95	1	1	3
2	Pigment Extender	in range	440.54	559.46	1	1	3
3	Binder	Minimize	0.11	0.13	1	1	3
4	Viscosity	in range	5.69	5.93	1	1	3
5	Density	in range	1250.00	14.18	1	1	3
6	pH	in range	6.31	8.77	1	1	3
7	Cost	minimize	332.25	402.80	1	1	3

Table 10: Solutions for Combination of Category Factor Levels

RUN	Prime Pigment	Pigment extender	Binder	Viscosity	Density	pH	Cost	Desirability
48	**66.849**	**440.556**	**0.108**	**5.819**	**1352.836**	**7.035**	**336.465**	**0.896**
49	67.497	440.533	0.108	5.826	1349.614	7.112	337.598	0.869
52	64.654	440.540	0.112	5.808	1365.349	6.507	335.468	0.921

From Figure 3, an increase in the quantity of the extender pigment from 44 g to 559 g resulted to an insignificant increase in the pH value from 7.2 to 7.3 while an increase in binder from 0.11 L to 0.13 L resulted also to an insignificant increase in the pH value. Hence, it can be deduced that the binder and the pigment extender have no significant effect on the pH of the paint. An increase or decrease in the extender pigment and binder will not alter the pH of the paint (6).

From Figure 4, at a constant increase in pigment extender, there is a little or insignificant effect on the viscosity of the paint. But an increase in the prime pigment from 64 g to 73 g gave a significant increase in the viscosity from 5.82 poise to an optimum value of 5.93 poise. Hence, the prime pigment has more effect on the viscosity of the paint than the pigment extender (7). And the optimum point of viscosity corresponds to the desired value by Nigeria Industrial Standard (5.8 to 6.0 poise). The use of proper paint viscosity is critical for obtaining a quality finish; excessive viscosity can cause orange peel while a low viscosity can create a film that is too wet and creates runs (8).

From Figure 5, density increased with increase in pigment extender and prime pigment. Though the effect of pigment extender was not significant compared to that of prime pigment, but the optimum density condition of 1374.25 g/L corresponds to the desired value as stated by Nigeria Industrial Standard.

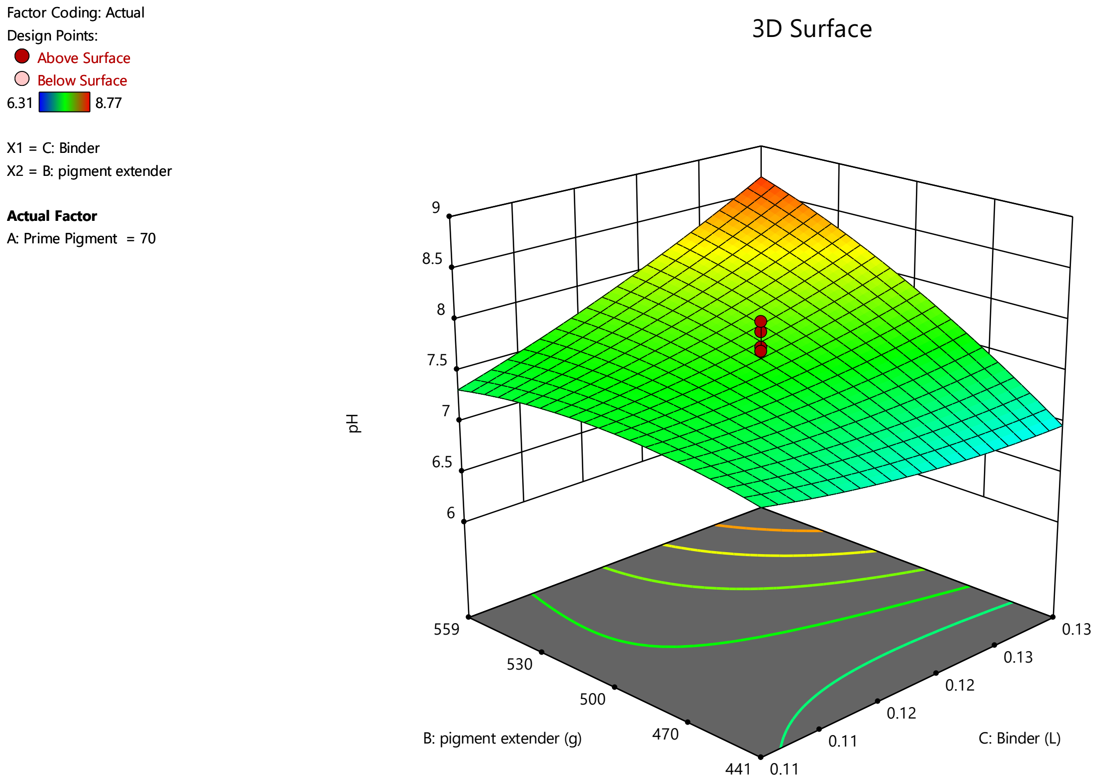

Figure 3: Effect of pigment extender and Binder on pH

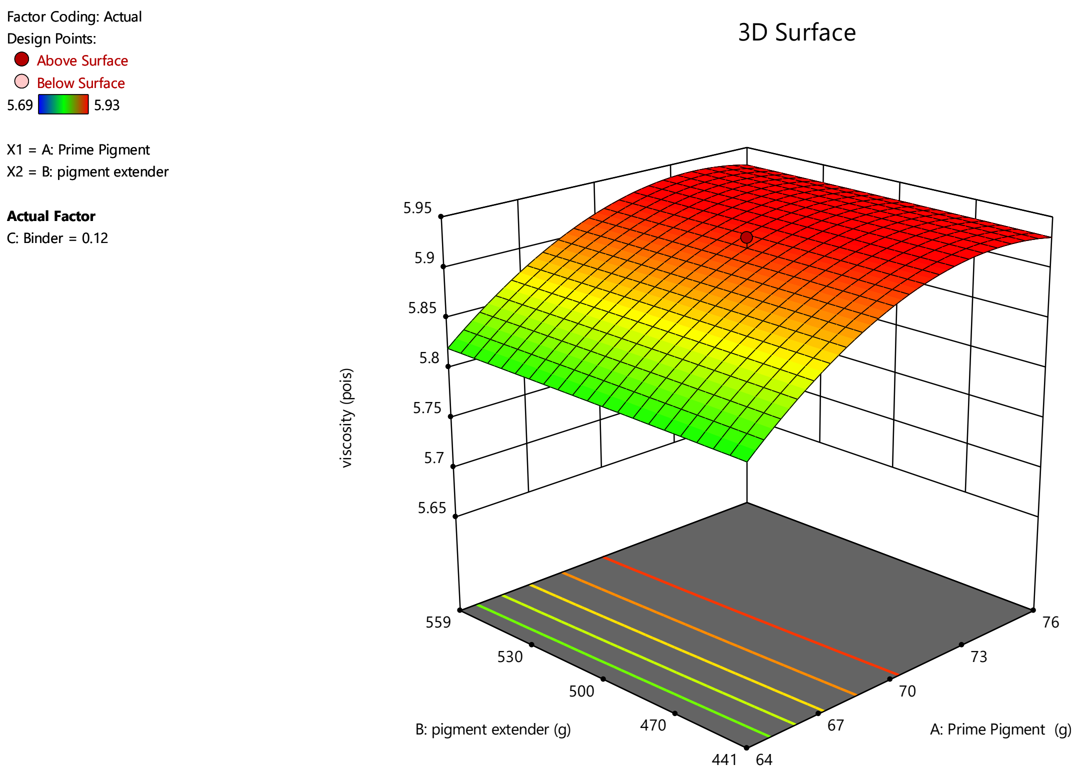

Figure 4: Effect of pigment extender and prime pigment on viscosity

Nigerian Journal of Tropical Engineering
Vol. 16 No. 1 |ISSN: 1595-5397| Dec. 2022 **DOI: 10.59081/njte.16.1.003**

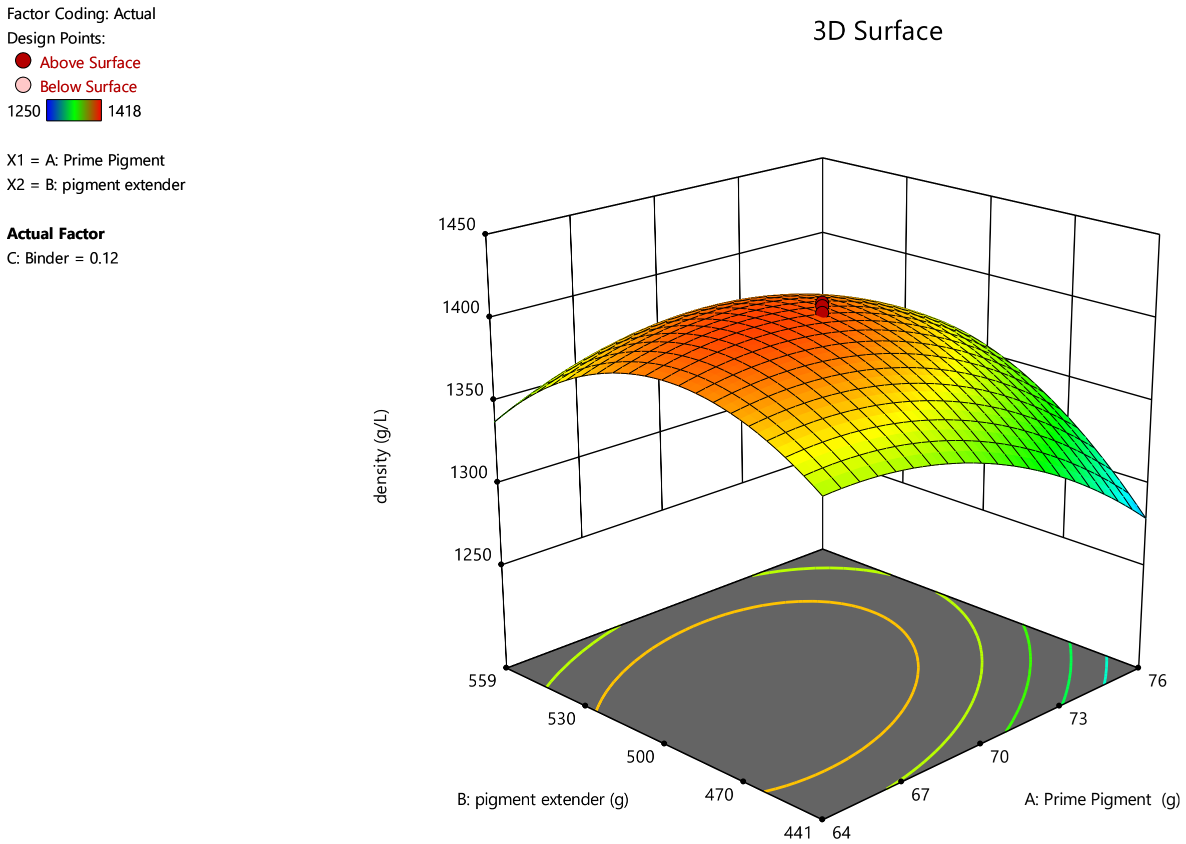

Figure 5: Effect of pigment extender and prime pigment on density.

3.4 Validation of Experiment

The suitability of the model equation for the prediction of the optimum response values was validated using the optimal conditions suggested by CCD. The following response values were obtained during the optimum condition validation in the laboratory. The percentage error of deviation was calculated using equation (6)

$$\%\text{error of deviation} = \frac{experimental\ value - predicted\ value}{predicted\ value} \times 100 \quad \ldots(6)$$

The error deviations lower than 30 % can be accepted in the experimental validation run (9). The obtained experimental values of 5.95 poise of viscosity, 1380 g/L of density and pH of 7.30 were close to the predicted values and it confirmed the validity and adequacy of the predicted models. Under the optimum conditions, the percentage errors of deviation were 2.64% for pH, 2.26% for viscosity, 2.0% for density and 2.5% for the cost (10). The result of the analysis proved that the response model was adequate for reflecting the expected optimization and the model of equations were satisfactory and accurate.

Nigerian Journal of Tropical Engineering
Vol. 16 No. 1 |ISSN: 1595-5397| Dec. 2022 **DOI: 10.59081/njte.16.1.003**

Table 11: Central Composite Design confirmation

Response	Predicted value	Experimental value	Std Dev	%Error
Viscosity	5.8185	5.950	0.0124	2.26
Density	1352.836	1380	9.0409	2.00
pH	7.112	7.30	0.3244	2.64
Cost	336.443	345.12	1.1736	2.50

4.0 CONCLUSION

From the results obtained in this study, the following conclusions were drawn.

- ✓ 20 different samples of white emulsion house paint were successfully formulated and produced using Central Composite Design. In addition, the physico-chemical properties of twelve (12) of the produced paints fell within the conventional specified range by Nigeria Industrial Standard.
- ✓ The prime pigment, pigment extender and binder of the produced paints were successfully optimized, and the numerical analysis carried out on the responses revealed that good quality paint was obtained at optimum conditions of 66.849 g of prime pigment, 440.556 g of extender pigment and 0.108 L of binder. The quadratic models also developed for the responses were found to be significant with P-values less than 0.05 (P 0.05).
- ✓ In addition, as revealed from the obtained optimum condition results on quality test of the produced paint, the pH value of 7.03, viscosity of 5.819 poise and density of 1352.836 g/L all fell within the conventional specified range by Standard Organisation of Nigeria (SON) and Nigeria Industrial Standard (NIS), thereby justifying the quality of the produced paint.
- ✓ The optimum conditions generated were validated by repeating the experiment and the obtained values of 7.30 for pH, 5.95 poise of viscosity and 1380 g/L of density were all close to the predicted values. The percentage error of deviation was calculated to be 2.26% for the viscosity, 2.0% for density, 2.64% for pH and 2.5% for the cost.

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