



Production of Paint from Locally Sourced Raw Materials

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Abstract- The use of locally available materials has been successfully formulated with other ingredients for the production of textured and emulsion paints. In the formulation, titanium dioxide was completely replaced with talc. Also, the conventional marble dust was replaced with treated river sand in textured paints, white natrosol (hydroxyl ethyl cellulose – imported thickener) was substituted with local starch. The properties (specific gravity, viscosity, pH, opacity, spreading rate, good dispersion, and drying time) of the formulated paints were compared with PMAN, specification and standard, and were within acceptable range. The formulated paints stayed for over two (2) months without loss of stability.

Keywords- Paint, Raw Materials, Resin.

I. INTRODUCTION

Paint can be traced historically from cave men, through Egyptian civilization to present day industrialization. Primitive men were credited with making the first paints about 25,000 years ago (school science series[1]). They were hunters and cave dwellers and were probably inspired by the dark formations of their cave walls to outline and form the shapes of animals they hunted. Chemical analysis of cave paintings discovered at Altamira (Spain) and Lascaux (France), showed that the main pigments used by Paleolithic artists were based on iron and manganese oxides dug out from local soil, possibly from cave floors (Harward[2]).

Paints can be defined as a fluid, with viscosity, drying time and flowing properties dictated by formulation, normally consisting of a vehicle or binder, a pigment, a solvent or thinner and a drier which may be applied in relatively thin layers and which changes to a thin opaque continuous layer on surfaces. The opaque film is mainly used for decorative and/or protective purposes. The main chemical constituents of a standard paint are solvent/vehicle, binder, pigment, extender, thinner, plasticizer, dispersant and surfactant, while the minor ingredients include biocide, anti-form, rust inhibitor tinker, pH adjuster, wetting agent, perfume, anti-skinning agent, and anti-floating agent. These constituents play an important role in the formulation of quality paint. Vehicle is that part of paint in which the pigments are suspended and is referred as a carrier medium for dispersion. It is made up of binding medium which is the non – volatile matter and the thinner, which is the

volatile matter (Michael and Irene[3]), volatile vehicle are used to reduce the viscosity, affect formulation, consistency, leveling, drying/adhesion and durability (Hercules[4];Matthew[5];Lamboune[6]). Good quality paint must possess the following properties ranging from gloss, flow, adhesion, opacity, blocking short drying time and weather resistance; which are described in literature (Ohanyere[7], Tiflo[8], Hoechst[9], shamrock[10], Michael and Irene[3], Kirk[11]).

Paint can be classified based on the type of solvent used. Water based paints (emulsion), oil based paint (gloss). Classification could also be based on the substrate part of application (exterior paints, interior paint, automotive paints, marine paints and industrial paints). Emulsion paints are the most widely used surface coatings in Nigeria because of their numerous industrial applications. Nigerians, today; depends mainly on imported finished products because of low industrial development. Majority of the raw materials used in paint making technology are also imported, which led to the high cost of paints in the market. For example, white titanium oxide pigment remains the costliest raw material in paint manufacture, also analysis of cost of production of emulsion paints from manufacturing industries has shown that thickeners count much among other constituents cost (Oyoh and Nnamchi[12]). The focus of this work is to source the locally available raw materials for the production of emulsion paint, substitute titanium oxide with local talc, marble dust with river sand and imported thickener (Hydroxyl ethyl Cellulose with locally available starch.

II. EXPERIMENTAL

A. Paint Formulation

The amount of materials in the formulation is determined by the type of paint required and batch size of the mixer. Formulation of mill bases for water based paints can be derived from specific gravity, viscosity, water demand data, dispersant demand data and pigment volume concentration at flow point for pigment and extenders being used. Paints Manufacturers Association of Nigeria (PMAN) has the following under listed percentage constituents as standard for water based paints.

TABLE I. PMAN SPECIFICATIONS FOR PAINT CONSTITUENTS

Constituent	Textured Coating	Emulsion Paint
Aggregate	30 – 35%	-
Extender	20 – 30%	20 – 40%
Pigment	3 – 15%	3 – 15%
Binder	15 – 20%	10 – 20%
Thickener	0.70 – 1.0%	0.5 – 1.5%
Deformer	0.05 – 0.2%	0.05 – 0.2%
Plasticizer	0.30 – 1.0%	0.30 – 1.0%
Dispersant	0.02 – 1.0%	0.02 – 1.0%
Wetting Agent	0.02 – 1.0%	0.02 – 1.0%
Drier	0.02 – 1.0%	0.02 – 1.0%
Biocide	0.10 – 0.3%	0.10 – 0.4%
Solvent	8.0 – 1.5%	10.0 – 30.0%

TABLE II. PMAN SPECIFICATIONS FOR PAINT QUALITIES

Property	Textured Coatings	Emulsion Paint
Specific Gravity	1.45 – 1.70	1.30 – 1.40
Viscosity (25°C)	> 45 poises	1.30 – 1.40
pH valve	7.50 – 8.50	7.0 – 8.0 poises
Drying Time (25°C)	25 – 30mins	7.50 – 8.50
Hard Dry (25°C)	3 – 4hrs	25 – 30 mins
Opacity	One coat finish	Two coats finish
Good Dispersion (C. P. V. C. 181%)	Above 28%	Above 38%

TABLE III. PMAN SPECIFICATIONS FOR FORMULATIONS

Type of Emulsion	P. V. C.: C. P. V. C. Ratio
Exterior Material	0.8: 1
General purpose material	1:1
Medium quality	1.25:1

P. V. C. means pigment volume concentration. C. P. V.C means critical pigment volume concentration. Every paint has a recipe which is based on a 100% raw material (Tiflo System¹⁴). In this work, three different recipes for paint formulation 1, 2 and 3 are used as shown in Table 4, 5 and 6 respectively.

TABLE IV. RECIPE FOR PAINT FORMULATION

Constituent	Name	Percentage (%)	Mass (g)
Constituent	Marble Dust	33.00	198.00
Aggregate	Calcium Carbonate	30.00	180.00
Extender	Titanium Dioxide	5.00	30.00
Pigment	Polyvinyl Acetate	20.00	120.00
Binder	Natrosol	0.90	5.40
Thickener	Vinyl Isobuty/Ether	0.10	0.60
Deformer	Ethylene glycol	0.50	3.00
Plasticizer	Ammonia	0.50	0.30
Dispersant/Wetting Agent	Sodium Tripolyphosphate (STPP)	0.10	0.60
Drier	Kerosene	0.10	0.60
Biocide	Formaldehyde	0.25	1.50
Solvent	Water	10.00	60.00
	Total	100.00	600.00

B. Production Procedure

Eighteen (18) grams of water was introduced into a high shear impeller mixer operated at a medium speed (set point 2–3) and 0.60g STPP (dispersant) was added. Subsequently, 0.30g of vinyl isobutyl ether, 30g of titanium dioxide were added. Thereafter, another 18g of water and 180g of calcium carbonate were added and the content was stirred for 45mins to complete the first stage of dispersion. After 45mins of continuous stirring, 2.7g of natrosol, 12g of water and finally, the remaining 0.30g of vinyl isobutyl ether were added and the contents was again stirred for 15mins with the set point of the mixer at 3 – 5 rev/min to complete the second stage of dispersion. Then, 0.30g of ammonia, 3.0g of ethylene glycol, 0.60g thickener, 198.0g of marble dust and the remaining 12g of water were subsequently added and the contents was again stirred for 10mins at a set point of 5 rev/min. The procedure was repeated for paint 2 and paints 3 formulations (Tables 5 and 6).

TABLE V. RECIPE FOR PAINT 2 FORMULATION

Constituent	Name	Percentage (%)	Mass (g)
Aggregate	Sand (inert)	33.00	198.00
Extender	Calcium Carbonate	23.00	138.00
Pigment	Talc	10.00	60.00
Binder	Polyvinyl acetate	22.00	132.00
Thickener	Local starch	1.00	6.00
Deformer	Vinyl Isobutyl Ether	0.10	0.60
Plasticizer	Ethylene glycol	0.40	2.40
Dispersant	ammonia	0.05	0.30
Wetting Agent	Liquid soap	0.10	0.60
Drier	Kerosene	0.10	0.60
Biocide	Formalin	0.25	1.50
Solvent	Water	10.00	60.00
	Total	100.00	600.00

TABLE VI. RECIPE FOR PAINT 3 FORMULATION

Constituent	Name	Percentage (%)	Mass (g)
Pigment	Talc	14.90	90.00
Extender	Calcium Carbonate	34.16	206.00
Binder	Polyvinyl acetate	15.90	95.88
Thickener	Local Starch	6.39	38.53
Defoamer	Vinyl Isobutyl ether	0.15	0.90
Plasticizer	Ethylene glycol	0.70	4.22
Stabilizer	Ammonia	0.075	0.45
Dispersant	Liquid Soap	0.15	0.90
Drier	Kerosene	0.15	0.90
Biocide	Formalin	0.37	2.25
Colour	Yellow paste	0.10	0.603
Solvent	Water	26.96	162.51
	Total	100.00	603.14

C. Experimental Analysis

1) Determination of Refractive Indexes of the Pigments and Extender

The refractive indexes of the pigments and extenders were determined with a high powered refractometer. The 20g powered TiO₂ pigment to be measured was mixed with 20ml de ionized water. Two drops was sand witched on a glass slide placed on the platform. A cover containing a reflecting mirror was then used to cover the sample beneath which is a right angle prism of high refractive glass. Light was directed from an electric bulb through an aperture to the mirror, which reflected the light into the lower polished surface of the illuminating prism. The rays were 90° to the mixture prism boundary. Two viewing holes with two adjusting knobs are provided on the equipment. One was used to read off the valve of index and the other used to adjust the boundary lines, until it touched the intersection of two lines when viewing through the telescopes. By the demarcating line set to the cross wire, telescope block on which the prism were mounted the refractive index was read off directly from a scale. In the same way, the refractive indexes of Talc, CalciumCarbonate, Kaolin and Zinc oxide were determined.

2) Purification of Otammiri River Sand

Two kilograms sample of the river sand was put in a container containing water; A OIMEDTA solution was added to precipitate the dissolved ions in the river water after which 10g of potash alum was introduced to coagulate the colloidal particles in the sample. Filter funnel was used to remove the precipitates and the colloids. The 21g sand sample was taken to tap where jets of water was used to wash out the remaining impurities and also as a means of dilution of the basic and acidic compounds. The sand was allowed to drain after which it was boiled for 30mins in a beaker to kill the pathogens. The sample was then drained filtered and dried.

3) Determination of Water Demand Test for the Pigment and Extenders [Ball Point and Flow Point]

The ball point of an extender or pigment is the amount of water required to form a stiff paste or ball for a given mass. Flow point of an extender or pigment is the amount of water required to form a free flowing paste from a spatula held at 45° for a given mass. Fifty grams of titanium dioxide pigment was weighed into a 200ml glass vessel and 5ml of water was added from a 50ml graduated burette and thoroughly mixed using stiff spatula. Another 5ml of water was added and again thoroughly mixed, and was repeated until 2ml and 1ml addition as the end-point was approached where all the pigments is wetted just sufficiently to form a stiff paste or ball. The total volume of water required to reach this stage was recorded as the ball point. More water was then added to the paste in 1ml quantities until it flowed smoothly from spatula inclined at 45°. The total volume of water required to reach this end point known as the flow point was recorded as the water demand of 50g titanium dioxide. The same procedure was repeated for all the pigments and extenders.

4) Determination of Wetting Time of Pigment/Extender in Different Resin Concentrations.

Twenty ml sample of resin solution of various concentrations (0%, 10%, 20%, 30%, 40%, 50%) was placed into 5 beakers. Steadily (taking half a minute) pour 20g of tiO₂ from a paper scoop into the centre of the liquid and the time taken for the binder solution to wet the whole pigment was recorded. The same experiment was performed using talc and caco₃.

5) Determination of Pigment Volume Concentration at Flow Point P. V. C. [f] using different Resin concentration.

Resin concentration of 10%, 20%, 30%, 40% and 50% was used. Twenty gram of titanium dioxide pigment was weighed into a beaker and was titrated with resin solution. The resin solution was added in 1ml proportions. Toward the first end point (known as the ball point or wet point) the entire mass began to coalesce and the ball point was the titre value when the added resin solution was just sufficient to form a coherent ball with the pigment. Titration was then continued to the flow point which is of more practical significance. This is the titre value at which the added resin solution was just sufficient to form a free flowing paste. The titre values at the ball point and flow point were recorded for all resin concentrations.

Other properties determined are (i) minimum Dispersant demand at flow point using 0.1% resin solution, (ii) Effect of extended ball milling on degree of pigment/extender dispersion as indicated by Brightness of dry films (iii) Specific gravity and viscosity and (iv) Effect of acids on river sand, and marble dust.

III. RESULTS

The results obtained from the experimental analysis are shown in following Tables.

TABLE VII. REFRACTIVE INDEXES OF PIGMENTS AND EXTENDERS

Pigment/Extender	Refractive Index
Titanium Dioxide (TiO ₂)	2.71
Talc (3mg04Si02 H20)	2.35
Calcium carbonate (CaC03)	1.67
Zinc Oxide (Zn0)	2.00
Kaolin (Al2O3.2Si02.2H0)	1.58

TABLE VIII. OTAMMIRI RIVER SAND SAMPLE AFTER PURIFICATION

Property	Result
Particle Sizes	0.1 – 0.05
Acidity	Nil
Basicity	Nil
pH	7.00
Colour	Off White
Chemical Nature	Inert

TABLE IX. WATER DEMAND OF PIGMENTS AND EXTENDER BALL POINT AND FLOW POINT

Pigment/Extender	Water Demand Ball Point (g/50g)	Water Demand Flow Point (g/50g)
Titanium Dioxide	11.0	13.0
Calcium Carbonate	13.0	14.5
Talc	15.0	17.0
Kaolin	13.5	14.0
Zinc Oxide	11.0	12.0

TABLE X. WETTING OF PIGMENT AND EXTENDER WITH RESIN CONCENTRATION

Resin Conc (%)	Wetting time (min) TiO2	Wetting time(min) Talc	Wetting time (mins) CaCO3
10.0	2.2	3.20	6.0
20.0	5.0	7.0	10.2
30.0	15.0	18.50	22.0
40.0	35.0	39.00	42.0
50.0	67.0	71.00	72.0

TABLE XI. PIGMENT VOLUME CONCENTRATIONS WITH RESIN CONCENTRATION

Resin Conc (%)	Titre value(ml) TiO2	Titre value (ml) Talc
10.0	11.0	12.50
20.0	11.5	13.30
30.0	12.0	14.50
40.0	12.5	15.80
50.0	13.2	17.00

TABLE XII. MINIMUM DISPERSANT DEMAND AT FLOW POINT USING 10% RESIN SOLUTION

Dispersant solution STPP (1%)	TiO2 Flow point titre value (ml)	Talc flow point titre value (ml)	CaCO3 flow point titre value (ml)
0.10	30.0	48.5	45.0
0.20	20.0	36.0	34.0
0.30	21.5	31.0	30.0
0.40	23.5	32.5	31.0
0.50	26.5	35.0	34.0

TABLE XIII. EXTENDED MILLING EFFECT OF TITANIUM DIOXIDE, TALC AND CALCIUM CARBONATE ON BRIGHTNESS OF DRIED FILM BRIGHTNESS (%)

Ball Milling Time	Titaniumdioxide	Talc	Calcium Carbonate
0.0	0.0	0.0	0.0
10.0	36.0	34.0	25.0
30.0	65.0	63.0	55.0
50.0	81.0	78.0	71.0
70.0	89.0	87.0	80.0
90.0	93.0	91.0	87.0
110.0	96.0	94.0	90.5
130.0	98.0	96.0	93.0
150.0	99.0	98.0	95.0

TABLE XIV. PHYSICAL PROPERTIES OF MATERIALS

Material	Property Specific Gravity	Viscosity
Titanium Oxide	4.00	-
Calcium Carbonate	2.60	-
Zinc Oxide	5.60	-
Talc	2.50	-
Polyvinyl Acetate	1.09	4.7
Natrosol	1.03	4.9
Ethylene Glycol	1.14	3.5
Water	1.00	-
Ammonia	0.88	-
Kerosene	0.79	-
Sand	2.65	-

TABLE XV. COMPARISON BETWEEN PMAN STANDARD, PAINT 1 AND PAINT 2 SAMPLES

Property	PMAN Standard	Texture Pain 1	Paint 2
Specific Gravity	1.45 – 1.70	1.50	Paint 2
Viscosity (250C)	Above 45 poises	45 poises	1.50
pH	7.50 – 8.50	8.0	50 poises
Drying Time 250C	25 – 30mins	28mins	8.0
Hard Dry (250C)	3 – 4hrs	3.10hrs	29mins
Opacity	One coat finish	One coat finish	3.15hrs
Spreading Rate	1.20 – 1.30square meter per liter	1.20 square meter per liter	1.15square meter per liter
Good Dispersion	Above 38%	55%	64.52%

TABLE XVI. COMPARISON BETWEEN PMAN STANDARD AND PAINT 3 SAMPLE

Property	PMAN Standard	Emulsion Paint Colour and Paint 3
Specific Gravity	1.30 – 1.40	1.34
Viscosity (250C)	7.0 – 8.0 poises	7.50 poises
pH	7.50 – 8.50	8.10
Drying Time 250C	25 – 30mins	26mins
Hard Dry (250C)	3 – 4hrs	3.0hrs
Opacity	Two coats finish	Two coat finish
Spreading Rate	2.50 – 3.50square meter per litre	3.0 square meter per litre
Good Dispersion	Above 38%	48%

TABLE XVII. COMPARISON BETWEEN PAINT 1 AND PAINT 2

Property	Paint 1	Paint 2
Colour	Brilliant White	Off White
Best Purpose	General Interior and Exterior	Interior
Stability	Biostable	Biostable
Durability	Good	Unknown
Effect of Acid Rain	Gradual Reaction	No visible reaction
Immediate Defect	None	Slight Chalking
Specific Gravity	1.50	1.50
Viscosity	45 poise	5 spoise
pH	8.0	
Opacity	One cost finish	8.0

Spreading rate	1.20 square meter per litre	1.15 square meter per litre
C. P. V. C. (f)%	55%	64.52%
Water Demand	Low	Moderate
Binder Demand	Low	Moderate
Thickener Demand	Low	Moderate
Effect of UV radiation	Gradual Degradation	Unknown
Hard Dry (250C)	3.10 hrs	3.15hrs
Drying Time (250C)	28mins	29mins
Economic Feasibility	Costly	Cost effective

TABLE XVIII. EFFECT OF ACIDS ON MARBLE DUST AND SAND

Sample	Reaction	Observation
Marble dust	$\text{CaCO}_3 + \text{H}_2\text{CO}_2 \rightarrow \text{Ca}(\text{HCO}_3)_2$	Dissolves and forms soluble carbonate
Sand	$\text{SiO}_2 + \text{H}_2\text{CO}_3 \rightleftharpoons \text{SiO}_2 + \text{H}_2\text{CO}_3$	No visible reaction
Marble dust	$\text{CaCO}_3 + 2\text{HNO}_3 \rightarrow \text{Ca}(\text{NO}_3)_2 + \text{H}_2\text{O} + \text{CO}_2$	Forms a white precipitate of calcium nitrate
Sand	$\text{SiO}_2 + \text{HNO}_3 \rightleftharpoons \text{SiO}_2 + \text{HNO}_3$	No visible reaction
Marble dust	$\text{CaCO}_3 + \text{H}_2\text{SO}_4 \rightarrow \text{CaSO}_4 + \text{H}_2\text{O} + \text{CO}_2$	Forms a white precipitate of calcium sulphate
sand	$\text{SiO}_2 + \text{H}_2\text{SO}_4 \rightleftharpoons \text{SiO}_2 + \text{H}_2\text{SO}_4$	No visible reaction
Marble dust	$\text{CaCO}_3 + 2\text{HF} \rightarrow \text{CaF}_2 + \text{H}_2\text{O} + \text{CO}_2$	Forms flou spar (fluorite)
sand	$\text{SiO}_2 + 6\text{HF} \rightarrow \text{H}_2\text{SiF}_6(\text{ag}) + 2\text{H}_2\text{O}$	Forms a complex silicon salt.

IV. DISCUSSION

The refractive indexes of the pigment and extender are shown in Table 7 with titanium dioxide having the highest value of 2.7, which was followed by talc zinc oxide, calcium carbonate and kaolin with the least value of 1.58. The result of the purification of sand shown that it is suitable for paint formulation as it is chemically inert, neutral and particle range (Table 8). It was observed that zinc oxide has the lowest demand for water (12ml), followed by titanium dioxide, while talc has the highest demand for water (17ml) as represented in Table 9. The implication of this is that the finer the average particle sizes of pigments/extenders the higher water demand property. High water demand gives a good opacifying power and flow characteristics to paint formulation. Water demand tests also showed a relationship between volume of paint and sizes of particles volume of paint formulated. They would suggest that talc will be more economical for industrial uses however; this work did not consider economic analysis of paint.

Figure 1 shows the wetting time curve. It was observed that titanium dioxide possess the least wetting time at 10% resin concentration. Talc gave a wetting time of 3.3mins at 10% resin concentration, while calcium carbonate gave a wetting time of 5.3mins at 10% resin concentration. This experiment however measured in a semi quantitative way the combined rates of wetting and penetration in the absence of

any mechanical shearing forces. It also lowers operation time by using a good disperse medium of 10% resin concentration.

In Figure 2, titre value of pigment volume concentration was plotted against resin concentration, for each percentage concentration, talc has a higher titre value, but with a slight difference of 10% concentration. The resin solution of 10% concentrations are the most suitable for determining the titre value for pigments/extenders for given masses. The minimum titre values showed good dispersion. Fifty (50) grams of titanium oxide required 11.0ml of resin solution while talc required 12.50ml resin solution.

The dispersant demand curve in Figure 3 showed that titanium dioxide required a minimum of 20ml resin solution at 0.2% dispersant concentration while the minimum titre value for both talc and calcium carbonate occurred at 0.3% dispersant concentration with 31.0ml and 30.0ml respectively. However, with these differences in value, the quality of paint formulation from each met PMAN specification.

Figure 4 showed that titanium dioxide has the greatest value of percentage brightness of 99% in I. C. I. reflectance chart. This test showed the extent of dispersion by considering the effect of extended ball milling on brightness of dried film. For routine control of production, this test and visual assessment of fine grind gauge of dried paint film are sufficient to establish the effective operation time.

The properties of paint obtained from formulation 1 and 2 (texture coating) with PMAN standard (Table 14), it was observed that both paint comparable result and are within PMAN specification. On comparing emulsion paint (formulation 3) with PMAN standard, the result obtained met were within range. However, Table 16 showed the difference between paint 1 and paint 2. Paint 1 gave a brilliant white colour, suitable for both interior and exterior purposes with good durability while paint 2 samples gave fairly off white colour, suitable for both interior and exterior purposes with good durability while paint 2 samples gave fairly off white colour suitable for only interior purposes because of slight chalking and poor durability.

The effects of weak acids on marble dust and inert sand. It was however observed that marble dust containing CaCO_3 undergoes gradual reaction with weak acids to form white precipitate of calcium salts. On the other hand, the results showed that sand containing mainly SiO_2 , does not in any form show any visible reaction with weak acids except hydrofluoric acid. However, these weak acids are components of acid rain while hydrofluoric acid is only in trace amount in the atmosphere. This test showed that sand has more weather resistance than marble dust when used in texture paints.

V. CONCLUSION

The study showed that locally sourced raw materials can be used to formulate paints that met the specification established by PMAN. In the formulations, titanium dioxide the costliest pigment in paint making was substituted with talc, treated river sand was used to replace the conventional marble

dust in texture paints and the imported thickener natrosol was also completely replaced with locally available starch. According to Oyoh and Nnamchi 12, the overall product cost of paint shows that modified starch has the least cost compared to other thickener. It is cheaper affordable and available. The use of locally available raw materials can compare favourably

with imported material and more economical. However, titanium dioxide cannot be totally substituted but blending with talc will give a good material finishing was observed for over 60 days no degradation was noticed. However, they long term effects of weather adversely affect the paints.

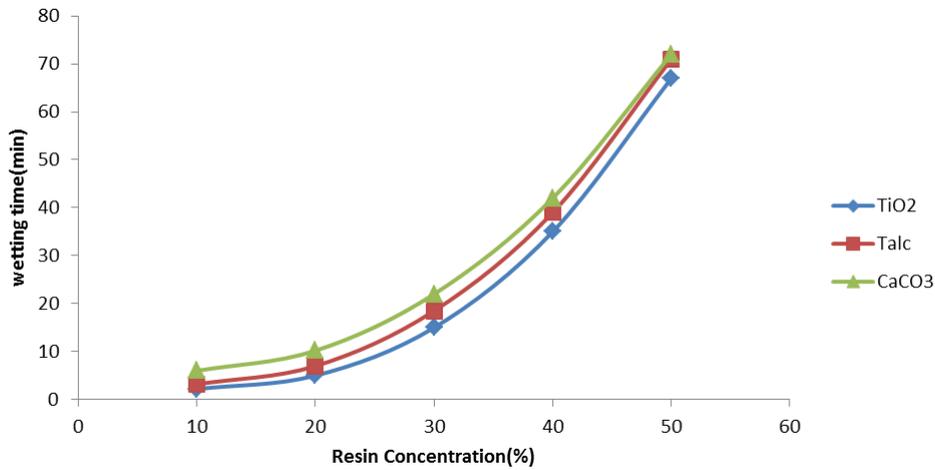


Figure 1. Wetting time versus Resin concentration

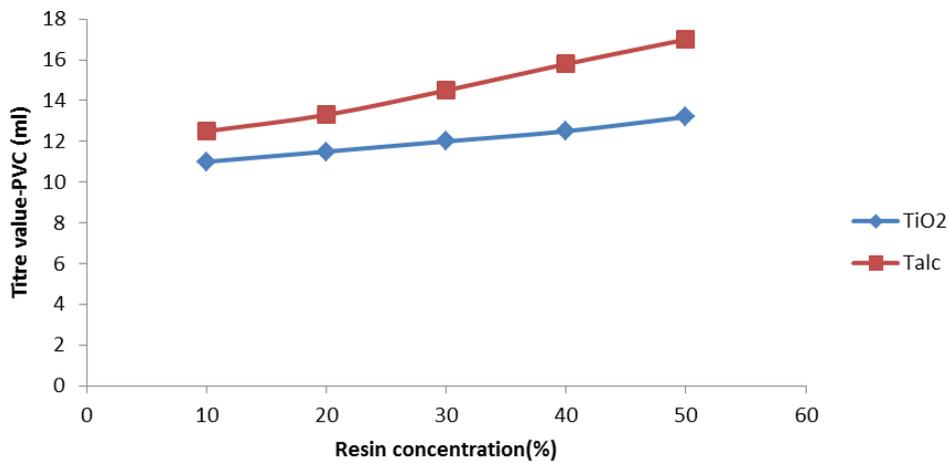


Figure 2. Pigment volume versus resin concentration

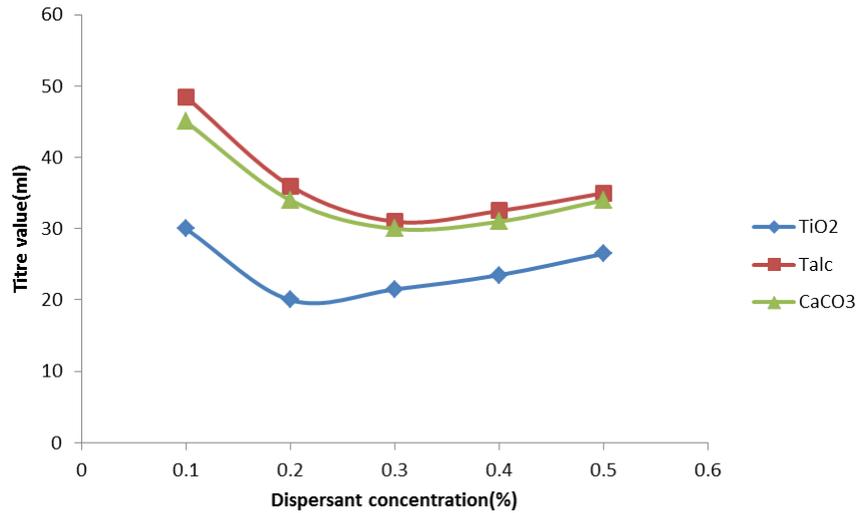


Figure 3. Dispersant demand curve (titre value Vs dispersant conc.)

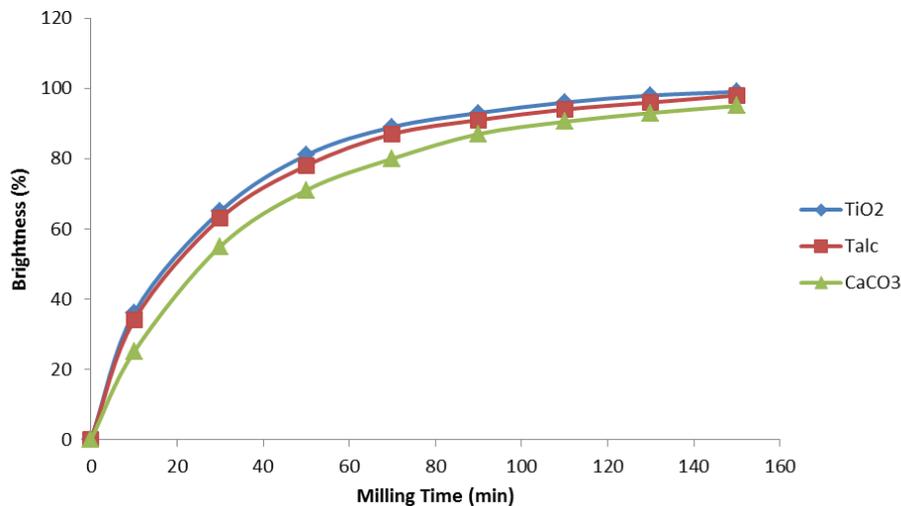


Figure 4. Brightness versus Milling time

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