



“TO ENHANCE PHYSICAL PROPERTIES OF PIGMENT YELLOW 74 AND PIGMENT RED 3 BY MEANS OF SUITABLE SURFACE TREATMENT IN WATERBASED APPLICATIONS”

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1. ABSTRACT

In this project the author has selected two different colour index pigments like Pigment Yellow 74 and Pigment Red 3 Based on their colour chemistry and hue or chroma these pigments are widely used in waterbased paints, emulsions and ink applications. Also, solvents being harmful to human health, it's likely to be the trend in future that most of the ink emulsions and paint manufacturers will slowly switch over their production from solvent based to water based colorants as its more safe to human health as well as its eco friendly.

To study this, the author has carried out lot of laboratory work by varying dosages and different types of surfactants for every colour index number or for every pigment family, total six reactions for each Colour Index were carried out at different conditions and their results are evaluated, monitored and tabulated for study purpose and for selecting a right surfactant type for every product.

The need for this is, though a particular surfactant shows remarkable or outstanding results in one product family, it needs not to be that much helpful for other product families. The applied surface treatment modifies the pigment crystals and improves its dispersion which is main physical property for pigments especially for water-based applications. The applied surface treatments also give desired tonal properties and it also improves other physical properties like oil absorption, bulk density of pigment which helps the end user to load the pigment in a user friendly manner.

The author has set up a small laboratory set up in his present organization and carried out detailed laboratory work as per the plan. The main motto behind selecting the topic of the thesis “To enhance physical properties of pigments by means of suitable surface treatment in water based applications”, is preliminary to study the history of how pigments were being manufactured and used for making inks, paints and secondly most important is increasing market demands and competitions for stable water based colorants.

Earlier pigments were made by simple techniques and without many surface treatments as there were no technologies available as well as demands from customers or end users were limited. Earlier pigments were being supplied after basic evaluation using offset ink testing on Muller or paint testing's using short or long oil alkyd resin and dispersed on vibroshakers or skandex machines. Now, there are several new instruments came in operations like high speed mixers, Dispermat, bead mills, Dyno mills which grinds the pigments usually at higher rpms like 3000, 5000, 12000. Hence, as the normal non treated pigments fail to disperse in these applications, there is an increased demand in the world of paints for pigments with higher loading and suitable surface treatments.

In conclusion; the author has selected prepared most demanding two pigment families through a drastic surface modification in the urge to get desired results. The overall output of the entire detailed work mentioned in the thesis will surely be the useful information for research and development of water based colorants.

Another, most demanding types of pigments is classified azo pigments or organic pigment which is widely being used worldwide due to their dispersibility and environmental friendly natures after being surface treated. Azo or organic pigments are manufactured by diazotization reactions of the primary aromatic amines followed by precipitation of diazotized organic compound with alkaline arylides or its various derivatives followed by a desired surface treatment.

Day by day, customer requirements in terms of pigments coloristic behaviors' like its shade, strength, hue or chroma and physical behaviors' like increase in bulk density, oil absorption, hiding power, loading and compatibility in water based resin or mediums have been increased a lot. Though the basic idea or pigment chemistry is similar, it is necessary to develop a pigment by selecting suitable surface active agent which is also called as a surfactant in short. Based on the charges on surface of the pigment particles, suitable cationic, anionic and non ionic surfactants are added at various stages of pigment making.

Surfactants can be added at various levels like in the beginning or at the time of crystal formation or secondly at the time of crystal growth during heating or lastly to modify surface of pigment particles once the particles are fully developed at each level, surfactant have their different roles.

Here we have selected two organic pigments to enhance their physical properties with suitable surfactant in water based applications.

Most of the surface active agents do not actually stay in the pigment particle but they modify the crystal, improve pigments dispersibility and other physical properties and get washed away during filtration activity as well.

Introduction

Pigments are basically Colorants which are used to impart color to objects which are colorless modify the perceived color of objects. They are mainly of two types i.e. Pigments & Dyes. Pigments being insoluble in the medium do not dissolve in their medium. They are dispersed in the medium used but retained their crystal size & structure.

Pigments are further divided into Organic as well as Inorganic Pigments. Organic Pigments are further classified as Lakes & Toners

Traditionally, pigments were simply used to impart colours in various objects & articles. That time the requirements were very basic like shade, strength & overall Hue or Chroma of the pigment once applied on the substrates. The acceptance criteria's were also not very stringent. So, surface treatments were not given that much importance earlier.

To throw more light on this, we can discuss pigments behavior based on different end applications. Pigments are main raw materials for Inks, Paints, plastics & Dispersions.

For example, if a pigment is to be tested for Off-set Ink application, it's to be tested on traditional Muller & only shade, tinting strength & dispersion used to given preference. The flow properties are seen at various angles on glass plates. If a particular product or grade passes all these basic tests then earlier it's being called as a good quality pigment & pigment manufacturers were used to supply the material for Ink applications. Similarly for paint applications, shade, tinting strength & hiding power is being tested earlier & if a pigment behaves well in the paint system then they used to supply it to the Paint Industries.

Nowadays, technology has been changed a lot & all the end users of pigments have their own specific requirements about the products which they are using & respective of the systems they are using i.e. Solvent base as well as Water base systems. Since solvents itself acts as a vehicle, though the pigment has slight inferior dispersion the solvent in the resin which is usually a long or short oil Alkyl with Mineral Turpentine as a solvent, it helps the pigment to get disperse with resin being used with the help of grinding media.

On the other hand, there are so many regulations for pigments used globally by most of the Paint manufacturers like eco friendly paints or VOC free systems, most of the Paint industries are in a state to convert from manufacturing Solvent base paints than water base stainer or machine colorants.

Since the solvents act as a carrier & help the pigment to grind well with the help of grinding media in solvent based paints, the pigments gets well dispersed but as the name indicates water based paints do not contain any grinding media as well as carrier medium. Hence to get good dispersion of pigments used in water base medium, it is necessary to apply surface treatment to the pigments.

II: Objective of the Study:

The main objectives of this study are as follows:

- To enhance physical properties of Pigments for water based paints or colorants.
- To develop surface treated pigment with better dispersibility in water based colorants.
- To apply suitable surface active agent to pigments to improve dispersibility in water based colorants.

III: Methodology of the Research work

- Following methodology is decided to carry out:
- Study Design: The study will be done in laboratory. Required reactions will be carried out based on work experience & literature support to meet the objective of the study.
- Duration of the Study: The detailed study will take approximately 8-9 months to carry out the experiments & validate the results with repeatability and reproducibility.
- Sample selection: It is decided to develop Pigment Yellow 1, Pigment Yellow 12, Pigment Yellow 14, Pigment Yellow 42, Pigment Yellow 74, Pigment Yellow 83, Pigment Red 2, Pigment Red 3, Pigment Red 4, pigment Red 57:1, Pigment Red 101 & Pigment Red 112 with improved physical properties like dispersibility & viscosity in water based application.

- Technique to be used: All the twelve pigments mentioned above will be synthesized in laboratory & then they will be given proper surface treatment based on their nature & behaviour in water based applications.

Research Methodology

Since all the pigments are formed by chemical synthesis i.e. simple precipitation techniques in Inorganic pigments precipitation of acidic metals salts and alkali whereas organic pigments are formed by means of diazotization of aromatic amines at lower temperatures and pH conditions and precipitate it with alkaline solutions of arylides followed by crystal growth by means of heating, resination and suitable surface treatment. The author has decided to conduct six numbers of reactions for both the pigment families and apply suitable surface treatments. The results will be studied on shade, strength i.e. coloristic as well as physical properties to finalize and optimize the desired surface treatment for the selected product.

So overall twelve reactions will be carried out in laboratory for two different products. Let us discuss the surface treatment study one by one for all the selected colour indices.

3.1 Experimental work for the 2 CI's:-

3.1.1 Pigment Yellow 74:

In today's times, the most widely used pigment in water-based colorant application due to their greener to redder Chroma & high tinting strength is Pigment yellow 74 or PY 74 in which the popular grades commercially available in the market are 2GX & 5GX depending upon their colour chemistries. It is also known as Monoazo pigment families.

It is being manufactured with the synthesis between Meta-nitro-ortho anisidine commercially known as Fast Red B Base & coupling component Aceto acet-ortho anisidine.

Meta nitro ortho anisidine undergoes diazotization at acidic conditions & lower temperatures for comparatively more time as it is a slow reacting amine. Further alkaline coupling component of Aceto acet ortho anisidine was being made & coupled with diazo component to get precipitated slurry of Pigment Yellow 74.

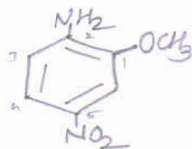
Since PY 74 has inferior stability in water based applications & poor weather fastness, it is necessary to apply suitable surface treatments to PY 74 so that it will exhibit better results in water based application.

Pigment Yellow 74

1. MNOA :- Meta Nitro Ortho Anisidine (FRBB)

CAS :- 97-52-9

MW :- 168



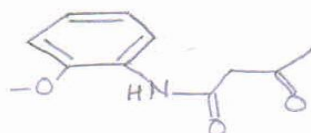
2-Amino-5 Nitro Anisole
2-Methoxy-4 Nitro aniline



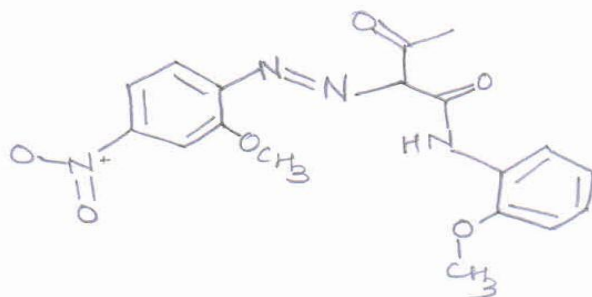
2. AAOA :- Acetoacetyl-O-Anisidine

CAS :- 92-15-9

MW :- 207.23 (C₁₁H₁₃NO₃)



3.



Pigment Yellow 74
MW - 386.23

Let us have a brief on experimental study:

A) Experiment 1:

Weighed quantity of MNOA or 2-methoxy-4-nitro aniline was taken in an acidic solution and stirred for one hour till smooth slurry was formed. It was then cooled to -5°C and further it was diazotized to get clear greenish yellow colored acidic solution called Diazo Component.

An alkaline solution of acetacet-orthoaniside was made and stirred to get clear solution called as the coupling component.

This diazo component was then added to alkaline solution of coupling component at desired pH and temperature within a time span of 90-120 minutes to get precipitated bright greenish slurry of pigment yellow 74. The slurry thus formed has been subjected to particular pH adjustments and given some special resination treatment.

The slurry was then heated to 80°C and held for 30 minutes at 80°C. It was then cooled to 65°C, filtered and washed till it was free from soluble salts. The wet cake thus obtained was dried at 70°C

till it dried completely. The dried lumps were powdered in a laboratory mixer and labeled as Experiment 1 for further physical as well as coloristic evaluation.

B) Experiment 2 :

Weighed quantity of MNOA or 2- methoxy -4 nitro aniline was taken in an acidic solution and stirred for one hour till smooth slurry was formed. It was then cooled to -5°C and further it was diazotized to get clear greenish yellow colored acidic solution called Diazo Component.

An alkaline solution of acetocet-orthoaniside was made and stirred to get clear solution called coupling component.

This Diazo component was then added to alkaline solution of coupling component at desired pH and temperature within a time span of 90-120 minutes to get precipitated bright greenish slurry of pigment yellow 74.

The slurry thus formed has been subjected to particular pH adjustments and an anionic surfactant i.e. castor oil derivative (4% of total pigmentation) was added to the slurry as a surfactant and stirred for 30 minutes.

The slurry was then heated to 80°C and held for 30 minutes at 80°C. It was then cooled to 65°C, filtered and washed till it was free from soluble salts. The wet cake thus obtained was dried at 70°C

till it dried completely. The dried lumps were powdered in a laboratory mixer and labeled as Experiment 2 for further physical as well as coloristic evaluation.

C) Experiment 3 :

Weighed quantity of MNOA or 2- methoxy -4 nitro aniline was taken in an acidic solution and stirred for one hour till smooth slurry was formed. It was then cooled to -5°C and further it was diazotized to get clear greenish yellow colored acidic solution called Diazo Component.

An alkaline solution of acetocet-orthoaniside was made and stirred to get clear solution called as the coupling component.

This diazo component was then added to alkaline solution of coupling component at desired pH and temperature within a time span of 90-120 minutes to get precipitated bright greenish slurry of pigment yellow 74. The slurry thus formed has been subjected to particular pH adjustments and polymer of ethylene glycol was (4% of total pigmentation) was added to the slurry as a surfactant and stirred for 30 minutes.

The slurry was then heated to 80°C and held for 30 minutes at 80°C. It was then cooled to 65°C, filtered and washed till it was free from soluble salts. The wet cake thus obtained was dried at 70°C

till it dried completely. The dried lumps were powdered in a laboratory mixer and labeled as Experiment 3 for further physical as well as coloristic evaluation.

D) Experiment 4 :

Weighed quantity of MNOA or 2- methoxy -4 nitro aniline was taken in an acidic solution and stirred for one hour till smooth slurry was formed. It was then cooled to -5°C and further it was diazotized to get clear greenish yellow colored acidic solution called Diazo Component.

An alkaline solution of acetocet-orthoaniside was made and stirred to get clear solution called as the coupling component.

This diazo component was then added to alkaline solution of coupling component at desired pH and temperature within a time span of 90-120 minutes to get precipitated bright greenish slurry of pigment yellow 74. The slurry thus formed has been subjected to particular pH adjustments and 4% quantity of non ionic surfactant based on primary alcohol was added to the slurry as a surfactant and stirred for 30 minutes.

The slurry was then heated to 80°C and held for 30 minutes at 80°C. It was then cooled to 65°C, filtered and washed till it was free from soluble salts. The wet cake thus obtained was dried at 70°C

till it dried completely. The dried lumps were powdered in a laboratory mixer and labeled as Experiment 4 for further physical as well as coloristic evaluation.

E) Experiment 5 :

Weighed quantity of MNOA or 2- methoxy -4 nitro aniline was taken in an acidic solution and stirred for one hour till smooth slurry was formed. It was then cooled to -5°C and further it was diazotized to get clear greenish yellow colored acidic solution called Diazo Component.

An alkaline solution of acetocet-orthoaniside was made and stirred to get clear solution called as the coupling component.

This diazo component was then added to alkaline solution of coupling component at desired pH and temperature within a time span of 90-120 minutes to get precipitated bright greenish slurry of pigment yellow 74. The slurry thus formed has been subjected to particular pH adjustments and an equal amount of emulsion prepared with anionic surfactants like castor oil derivative and ethoxylate lauryl alcohol which is mainly a coconut oil derivative. The slurry was then stirred for 30 minutes.

The slurry was then heated to 80°C and held for 30 minutes at 80°C. It was then cooled to 65°C, filtered and washed till it was free from soluble salts. The wet cake thus obtained was dried at 70°C

till it dried completely. The dried lumps were powdered in a laboratory mixer and labeled as Experiment 5 for further physical as well as coloristic evaluation.

F) Experiment 6 :

Weighed quantity of MNOA or 2- methoxy -4 nitro aniline was taken in an acidic solution and stirred for one hour till smooth slurry was formed. It was then cooled to -5°C and further it was diazotized to get clear greenish yellow colored acidic solution called Diazo Component.

An alkaline solution of acetocet-orthoaniside was made and stirred to get clear solution called coupling component. This Diazo component was then added to alkaline solution of coupling component at desired pH and temperature within a time span of 90-120 minutes to get precipitated bright greenish slurry of pigment yellow 74. The slurry thus formed has been subjected to particular pH adjustments and 4% of total pigmentation, a solution of Naphthalene sulphate condensate was added as an anionic surfactant. The slurry was then stirred for 30 minutes.

The slurry was then heated to 80°C and held for 30 minutes at 80°C. It was then cooled to 65°C, filtered and washed till it was free from soluble salts. The wet cake thus obtained was dried at 70°C

till it dried completely. The dried lumps were powdered in a laboratory mixer and labeled as Experiment 6 for further physical as well as coloristic evaluation.

All these six experiments are conducted in the same laboratory of Raveshia Group where the author works and also are performed by him personally to eliminate any errors. The finding of the surface treatments will be discussed at the time of result verification by using below mentioned Water-based Colorant made from all the six experiments



3.1.2 Pigment Red 3:-

Pigment Red 3 or most commonly known Toluidine Red Pigment is a well known pigment for its redder, brighter shade and good light fastness for all water-based inks and colorants application.

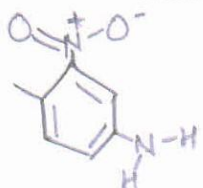
Pigment Red 3 is synthesized by coupling of acidic diazo formed by diazotization of Meta Nitro ParaToluidine and alkaline coupling component formed by dissolution of Beta Naphthol.

The precipitated slurry of Pigment Red 3 generally being surface treated with suitable surfactant so that the pigment behaves best in water based application without any gelling or flocculation issues.

Pigment Red 3

1. Fast Scarlet GG Base (MNPT):-

Meta Nitro Para Toluidine ($C_7H_7NO_2$)



CAS - 119-32-4

MW - 152.15 gm/mole

Orange Red/Yellow crystalline

5-Nitro-4 Toluidine



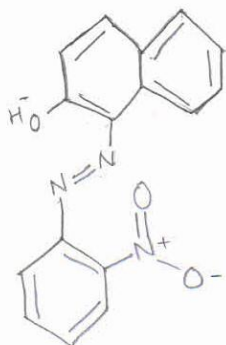
2. BNL :- Beta Naphthol

CAS Number - 135-19-3

MW - 144.17 ($C_{10}H_8O$)



3.



Pigment Red 3

MW:- 307.32

Let us discuss the experimental study for surface treatments as follows:-

A) Experiment 1 :-

Weighed quantity of Meta Nitro Para Toluidine was added to highly acidic solution and stirred to get uniform slurry. It was cooled to $-5^{\circ}C$ to $-7^{\circ}C$ and further diazotized to get clear solution called "Diazo component".

An alkaline solution of Beta Naphthol was made and stirred in warm condition to get clear solution called "coupling component".

Both the diazo and coupling component were coupled together at desired pH and temperature in 150-180 minutes to get slight bluer reddish precipitated slurry of Pigment Red 3.

The slurry was then subjected to pH adjustment and it was then heated to 80°C and stirred for 30 minutes. It was then cooled down to 65°C, filtered and washed till salt free. The wet cake thus obtained was dried completely at 70°C. The dried lumps were powdered in laboratory mixer and labeled as Experiment 1 and used as reference STD sample for further physical and coloristic evaluation because it is without any surface treatment and effect of other treatments can be verified after evaluation against STD sample.

B) Experiment 2 :-

Weighed quantity of Meta Nitro Para Toludine was added to highly acidic solution and stirred to get

uniform slurry. It was cooled to -5°C to -7°C and further diazotized to get clear solution called “Diazo component”.

An alkaline solution of Beta Naphthol was made and stirred in warm condition to get clear solution called “coupling component”.

Both the diazo and coupling component were coupled together at desired pH and temperature in 150-180 minutes to get slight bluer reddish precipitated slurry of Pigment Red 3.

The slurry was then subjected to pH adjustment and stirred for 30 minutes. A known quantity of anionic surfactant which is derivative of castor oil was added 4% of the total yield and stirred for 30 minutes. It was then heated to 80°C and stirred for 30 minutes. It was then cooled down to 65°C, filtered and washed till salt free. The wet cake thus obtained was dried completely at 70°C. The dried lumps were powdered in laboratory mixer and labeled as Experiment 2 for further physical and coloristic evaluation.

C) Experiment 3 :-

Weighed quantity of Meta Nitro Para Toludine was added to highly acidic solution and stirred to get uniform slurry. It was cooled to -5°C to -7°C and further diazotized to get clear solution called “Diazocomponent”.

An alkaline solution of Beta Naphthol was made and stirred in warm condition to get clear solution called “coupling component”.

Both the diazo and coupling component were coupled together at desired pH and temperature in 150-180 minutes to get slight bluer reddish precipitated slurry of Pigment Red 3.

The slurry was then subjected to pH adjustment and stirred for 30 minutes. A known quantity of non ionic surfactant which is a polymer of ethylene glycol derivative was added to the slurry 4% of the total yield and stirred for 30 minutes. It was then heated to 80°C and stirred for 30 minutes. It was then cooled down to 65°C, filtered and washed till salt free. The wet cake thus obtained was dried completely at 70°C. The dried lumps were powdered in laboratory mixer and labeled as Experiment 3 for further physical and coloristic evaluation.

D) Experiment 4 :-

Weighed quantity of Meta Nitro Para Toludine was added to highly acidic solution and stirred to get uniform slurry. It was cooled to -5°C to -7°C and further diazotized to get clear solution called “Diazo component”.

An alkaline solution of Beta Naphthol was made and stirred in warm condition to get clear solution called “coupling component”.

Both the diazo and coupling component were coupled together at desired pH and temperature in 150-180 minutes to get slight bluer reddish precipitated slurry of Pigment Red 3.

The slurry was then subjected to pH adjustment and stirred for 30 minutes. A known quantity of non ionic surfactant which is a derivative of primary alcohol was added to the slurry 4% of the total yield and stirred for 30 minutes. It was then heated to 80°C and stirred for 30 minutes. It was then cooled down to 65°C, filtered and washed till salt free. The wet cake thus

obtained was dried completely at 70°C. The dried lumps were powdered in laboratory mixer and labeled as Experiment 4 for further physical and coloristic evaluation.

E) Experiment 5 :-

Weighed quantity of Meta Nitro Para Toludine was added to highly acidic solution and stirred to get uniform slurry. It was cooled to -5°C to -7°C and further diazotized to get clear solution called “Diazo component”.

An alkaline solution of Beta Naphthol was made and stirred in warm condition to get clear solution called “coupling component”.

Both the diazo and coupling component were coupled together at desired pH and temperature in 150-180 minutes to get slight bluer reddish precipitated slurry of Pigment Red 3.

The slurry was then subjected to pH adjustment and stirred for 30 minutes. A known quantity of an emulsion made by equal proportion of two anionic surfactant was added. Generally 4% i.e. 2% of castor oil derivative and 2% of lauryl alcohol ethoxylate which is generally a coconut oil derivative was added and stirred for 30 minutes. It was then heated to 80°C and stirred for 30 minutes. It was then cooled down to 65°C, filtered and washed till salt free. The wet cake thus obtained was dried completely at 70°C. The dried lumps were powdered in laboratory mixer and labeled as Experiment 5 for further physical and coloristic evaluation.

F) Experiment 6 :-

Weighed quantity of Meta Nitro Para Toludine was added to highly acidic solution and stirred to get uniform slurry. It was cooled to -5°C to -7°C and further diazotized to get clear solution called “Diazo component”. An alkaline solution of Beta Naphthol was made and stirred in warm condition to get clear solution called “coupling component”. Both the diazo and coupling component were coupled together at desired pH and temperature in 150-180 minutes to get slight bluer reddish precipitated slurry of Pigment Red 3.

The slurry was then subjected to pH adjustment and stirred for 30 minutes. A known quantity of an anionic surfactant which is a derivative of Naphthalene benzene sulphonate condensate was added. Generally 4% of pigment and stirred for 30 minutes. It was then heated to 80°C and stirred for 30 minutes. It was then cooled down to 65°C, filtered and washed till salt free. The wet cake thus obtained was dried completely at 70°C. The dried lumps were powdered in laboratory mixer and labeled as Experiment 6 for further physical and coloristic evaluation.

All these six experiments are conducted in the same laboratory of Raveshia Group where the author works and also are performed by him personally to eliminate any errors. The finding of the surface treatments will be discussed at the time of result verification by using below mentioned Water-based Colorant made from all the six experiments



2.4 Testing Important Parameters of all the six experiments of the 2 Pigments with different CI's.

- **Colour Comparison:**

Since the surface treatment majorly impacts the dispersion of the pigment & if dispersion is superior, it directly reflects in more tinting strength of the pigment in reduced tone, we have decided to test all the 12 samples that are 6 samples of 2 different pigments in reduced tone only.

For reference purpose, all the draw downs are attached separately in the Result Analysis & Interpretations topic.

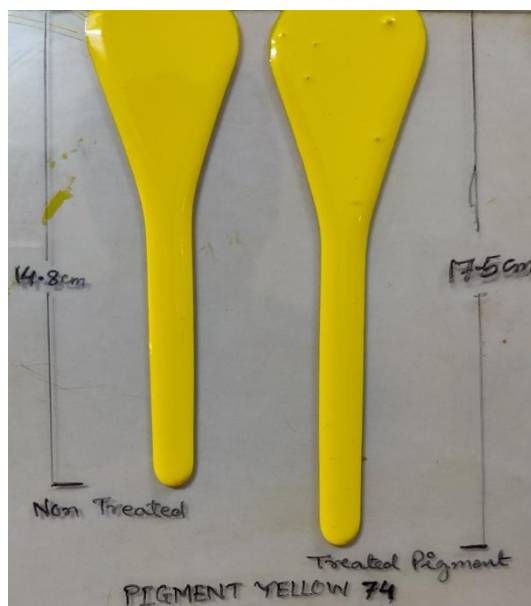
- **Flow Property:**

Flow is an important property of pigments as it is directly related to the dispersion of the pigments as well as its flow in required water-based colorant or ink application. Flow is measured by making ink on Automatic Muller machine using doubly purified linseed oil as a medium. 10% ink is generally made and transferred gently on smooth surface of Muller glass plates. It is then mixed properly with the help of palette knife and grinded with 5Kg weight for 100 rotations on Muller. The ink thus formed is subjected to one more round of 100 further rotations & the ink is collected and labeled properly. The same process is repeated for Standard sample & other than flow, one draw down is made for preliminary evaluation of shade & strength which gives a rough idea on the pigments colour trends. The flow is measured on a glass plate at 90 degrees. Both the equally weighed standard and sample inks prepared on Muller are placed near each other on a horizontal plate. It is then placed at 90 degrees and the flow is measured after minimum 10/15 minutes. It is generally expressed in "cm".

Let us have a look at some of the Flow testing done for random samples of both the pigments.

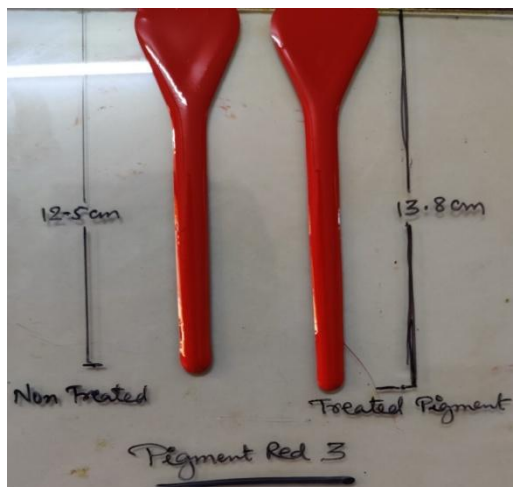
1. Pigment Yellow 74:

Flow of 10% ink of Organic pigment PY 74 made in Linseed oil & tested on Muller and evaluated against Standard experiment.



2. Pigment Red 3:

Flow of 10% ink of Organic pigment PR 3 made in Linseed oil & tested on Muller and evaluated against Standard experiment.



IV- Results and Interpretation:

As discussed earlier in research methodology topic, both the families of pigment were being given suitable surface treatment based on Literature survey & professional skills. Many physical properties like Oil absorption Value, Bulk density & water intake are tested. Also the physical nature of the colorant & its storage stability & settling properties are also studied. The coloristic effects of the surface treatments is also studied by applying draw downs of treated experiments against non treated standard experiments by using spectrophotometer in the Lab which Author used to perform all the PHD related research work.

4.1 Details of Testing Parameters for Physical Property Study:

A) **Oil Absorption value:** Oil absorption value is very basic but very important property of pigments. Pigments particle size, shape and particle type affects the Oil absorption value of the pigment. The lower the value the coarser the pigment particles & higher the value indicates the pigment particles are lighter, finer & have very usually good dispersibility.

It is evaluated using alkaline or double purified Linseed Oil as per ASTM D281-31 method & generally expressed as ml of oil per 100 gm of pigments or simply ml/100 gm.

B) **Bulk Density:** Bulk density is generally calculated for pigments to verify the nature of final pigments. Bulk density gives exact idea on the packing conditions of the material at the time of dispatches as well. Like Oil absorption this property also helps to understand whether the particle has become lighter or heavier or bulkier after surface treatment.

C) **Colour Values:** L^* , a^* , b^* values were calculated as per CIE 1976 method. The L^* , a^* , b^* values of the water based colorant were determined by using spectrophotometer of X-rite Ci4200 from AGS. L^* determines the lightness & darkness of the colorant. If L^* is positive the shade is lighter & if L^* is negative the shade is darker. Similarly, if a^* & b^* is

positive the shade is redder & yellower and if a* & b* is negative the shade is greener & bluer in nature.

D) Water Intake: Water intake is being tested with Atsopynt O 100 which is a surfactant most effective in Universal stainer applications or water-based colorant application. It's been filled in Burette and dry pigment is taken in a glass beaker. Drop by drop Atsopynt O-100 is added and the powder is mixed with palette knife. The moment it becomes flowable that burette reading is noted & recorded. The lower the value the better the wettability of the pigment.

Let us understand the physical testing table for all the labo surface treated experiments of the 2 pigments:

Physical Test Study of 2 Surface Treated Pigment Grades Selected For Study						
Sr.No.	Colour Index	Lab Experiment	Bulk Density		Oil Absorption	Water Intake
			As Is	Tapped		
1.	Pigment Yellow 74	Experiment 1	0.2647	0.4166	47.93	10.7
		Experiment 2	0.2783	0.4175	42.46	14.7
		Experiment 3	0.2647	0.4166	35.97	12
		Experiment 4	0.3571	0.5	36.78	11.4
		Experiment 5	0.2777	0.4545	33.3	12.8
		Experiment 6	0.3333	0.5	58.81	13.8
2.	Pigment Red 3	Experiment 1	0.1851	0.333	38.82	15.2
		Experiment 2	0.238	0.4166	45.86	15
		Experiment 3	0.2655	0.4204	46.07	12.4
		Experiment 4	0.238	0.3846	41.58	11.5
		Experiment 5	0.2272	0.3571	36.09	16.3
		Experiment 6	0.2566	0.3846	47.99	13.9

table 4.1.1: Physical property study and comparative chart.

From the above table, in almost all the families of pigments, the impact of effective surface treatments is clearly seen against Experiment No.1 for the 2 pigment families selected for the study.

Other than physical properties study, all the samples were tested in water based colorant application as follows:

In a stainless steel container 34 % pigment is loaded in waterbased resin & the mixture is premixed at lower rpm like 700-800 for 5-10 minutes. The same mixture is then grinded for 45 minutes at 4500 RPM on a high speed disperser to get uniform colorant.

The resultant colorants of all the pigment samples were evaluated by making their reduced tone with

4% pigmentation in water-based paint base. The results were evaluated by making draw down of all five experiments against Standard experiment no.1 for all the pigment families respectively.

4.2 Evaluation Results of Water-based Colorants against Ex.01 as STD:

Sr.No.	C.I. Name	Ex. No.	DE	DL	Da	Db	DC	DH	% Strength
01	Pigment Yellow 74	2	12.49	-0.12	-2.51	12.24	12.25	2.46	184.78
		3	5.43	-0.00	-0.87	5.37	5.36	0.92	130.83
		4	4.00	0.08	-1.46	3.72	3.72	1.48	121.27
		5	6.63	-0.14	-1.13	6.53	6.52	1.20	140.76
		6	14.36	0.11	-4.03	13.78	13.87	3.71	204.39
02	Pigment Red 3	2	2.37	-1.41	1.88	0.31	1.90	-0.17	113.58
		3	0.82	0.48	-0.56	-0.37	-0.63	-0.21	94.20
		4	4.32	-1.60	3.88	1.02	4.01	0.00	121.23
		5	7.78	-1.80	7.47	1.24	7.54	-0.62	138.59
		6	7.55	-0.59	7.46	0.93	7.47	-0.89	135.67

Table no.4.2.1 – Evaluation results of the 2 CI pigments in water based colorant application.

4.3 Interpretation:

Hence from all the above physical and coloristic evaluation, the interpretation on the results is as follows:

4.3.1 Pigment Yellow 74:

From the six experiments carried out to study the effectiveness of surface treatment, Experiment 3, Experiment 4 & Experiment 5 showed positive results and improvement in Oil absorption values by making the pigment more lighter in nature which ultimately improves its dispersion in water based colorant application and gives the final product with almost 20-40% higher tinting strength when evaluated in Water-based colorant application against non treated reference standard experiment 1.

Hence, in Pigment Yellow 74, the use of non ionic surfactant which is a polymer of Ethylene glycol, a non ionic surfactant which is primary alcohol ethoxylate and an equal proportion emulsion made up of two anionic surfactants improves the physical properties of Pigment Yellow 74 for water based application.

4.3.2 Pigment Red 3:

From the six experiments carried out to study the effectiveness of surface treatment, Experiment 3, Experiment 4 & Experiment 6 showed positive results and improvement in Oil absorption values by making the pigment more lighter in nature which ultimately improves its dispersion in water based colorant application and gives the final product with almost 10-35% higher tinting strength when evaluated in Water-based colorant application against non treated reference standard experiment 1.

Hence, in Pigment Red 3, the use of non ionic surfactant which is a polymer of Ethylene glycol, a non ionic surfactant which is primary alcohol ethoxylate and anionic surfactant which is a sodium salt of naphthalene sulphonate condensate improves the physical properties of Pigment Red 3 for water based application.

V- Conclusion & Recommendations:

5.1 Conclusion:

The main reason for studying the effectiveness of suitable surface treatments for pigments to be used in water based application is the existing market demands and environmental awareness approach developed by Pigment users for their variety of demanding applications like water based paints or inks.

The use of solvents has also reached to the extreme level which in turns affects the working environment as well as the surroundings very badly. It also has the chances of life threatening hazards like Fire or even explosions due to mishaps with solvent handling if all the required statutory and regulatory compliances are not followed at the time of Paint or Ink manufacturing.

The pigments cannot give a stable dispersion if it's not having a good dispersibility or wettability. The desired chroma will only be developed against the desired Standard Pigment if the targeted pigment is surface treated. With a suitable surface treated pigments it is easy to achieve improvements in certain physical properties of pigments like oil absorption which seems to be a basic but is an important property for pigments. Increase or decrease in Oil Absorption makes us understand the nature of the pigment particles. If the Oil absorption value is increased, it means the pigment becomes more lighter in nature and it gives good dispersibility in water based colorants where as if Oil absorption value of certain pigments is decreased, it means the pigment the pigment has become heavier and it gives us a pigment which can be loaded to more extent to make concentrated and cost effective Water based colorants.

Another important property is hydrophobic or hydrophilic nature of pigments in water based applications which is generally measured by water intake tests by using one surfactant which is a 3 in 1 combination of Non ionic and Anionic dispersant & a Polyglycol as a Humectant. It is a very helpful technique which helps us understand the basic nature of the pigment after effective surface treatment or even before treatment as well so that one can modify the route of surface treatments. If the water intake value for Non treated pigment is higher, it indicates that the resultant pigment is hydrophobic or water repellent which ultimately means the pigment cannot form a stable colorant or dispersion in water based application whereas if the water intake value is lower, it indicates that the surface treated pigment is hydrophilic or water loving in nature which ultimately means that the surface treated pigment can form a stable colorant or dispersion in Water based applications.

One more advantage of using Water-based colorants over solvent-based colorants is their eco friendly nature. The overall working environment is healthy and free of hazardous solvent fumes if the surface treated pigments shows similar colour trends in water based colorants like solvent based colorants and this is only possible with the help of effective surface treatment.

For all the Colorants other properties which are also of prime importance post production are their storage stability and their final viscosity. These properties are achieved by selecting suitable surface active agent or surfactant for particular pigments use in water based colorant application.

For Solvent based colorants required dispersion can be achieved as there are grinding media used like zirconium or steel balls or glass beads but most of the leading paint industries of today's generation do not use any grinding media for Water based colorants. Hence the dispersion property or fineness can only be achieved with the proper selection of surface active agent for Water based colorant application.

Apart from improvements in physical property, another main property or nature of the pigment which is drastically modified due to surface treatment is its colouristic behaviour. In our study also its clearly replicated that compared to non treated Standard reference experiments of the 2 different Colour Indices selected for surface treatment study, almost all products have shown outstanding improvements in tinting strength of pigments from minimum 5% to maximum 35% when tested in water based colorant application without any grinding media which clearly states the effectiveness of the applied surface treatments. Hence use of suitable surface treatment is very much essential in water based application of colorants as it not only improves the physical properties like Flow, viscosity, Oil absorption, storage stability, wettability of pigments but also helps improve coloristic behaviors of the resulting colorant by improving its % pigment loading as well as imparting higher tinting strengths which helps to make cost effective and stable Water based colorants.

5.2 Recommendations:

- In water based application to manufacture colorants like aqueous paints or inks, effective surface treatment must be selected and applied based on the nature of the non treated pigment particles which helps to improve or enhance all the required physical properties of the particular pigments to be used in Water based application. This article focuses mainly on the selection of the suitable surface treatments and its application in producing stable water based colorants.
- Selection of proper surfactant whether it is Cationic, Anionic or Non ionic in nature and its use in decided stages of pigment making like Coupling, Crystal growth, dosage and desired pH and temperature conditions where the treatment is being given is very important task while developing particular pigment for required water based application. This study will also be helpful for further studies on surface treatment to pigments for particular applications to future Technologists.
- There will be huge benefits to the Industries as well as to the Global environment if this study helps the colorant manufacturers can switch maximum of their pigment usage to Water based applications than the Solvent based applications in coming years. This will help to create a clean, user friendly, environment friendly and most importantly safest working atmosphere.

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