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"TO ENHANCEPHYSICAL PROPERTIESOF SYNTHETICIRON YELLOWOXIDE AND SYNTHETIC IRON RED OXIDEBY MEANS OF SUITABLESURFACE TREATMENT IN WATERBASED APPLICATIONS"

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1.1ABSTRACT

Chemistry to develop Inorganic pigments is similar everywhere as the basic chemistry never changes. What makes a product different than others is its surface area which behaves drastically different after application of suitable surface treatment.In this project the author has selected twelve different colour index pigments like C.I. Pigment Yellow 42 (SIO), Pigment Red 101(SRO)

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 treatmentsalso 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 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 the history of how pigments were being manufactured and used for making inks, paints and secondary most important is increasing market demand and competition.Inorganic pigments are manufactured by precipitation reaction between metal salts with strong alkalis.They have very good weather fastness and light fastness and acid alkali resistance. Some metal based inorganic pigments like lead chromate pigments were also being used in paints lent now a days due to the increased concerns and health threats as well as environmental crisis, most of the countries started using lead free pigments or they have put a regulatory requirement to all the products having all such substances present within the specified limit.

Hence, the author has selected synthetic Iron oxide yellow and Red pigments for the study of surface chemistry and its use in water based application.

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 inorganic 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.

Let us see an example of Inorganic pigment. Forexample synthetic Iron Yellow and Red Oxide.

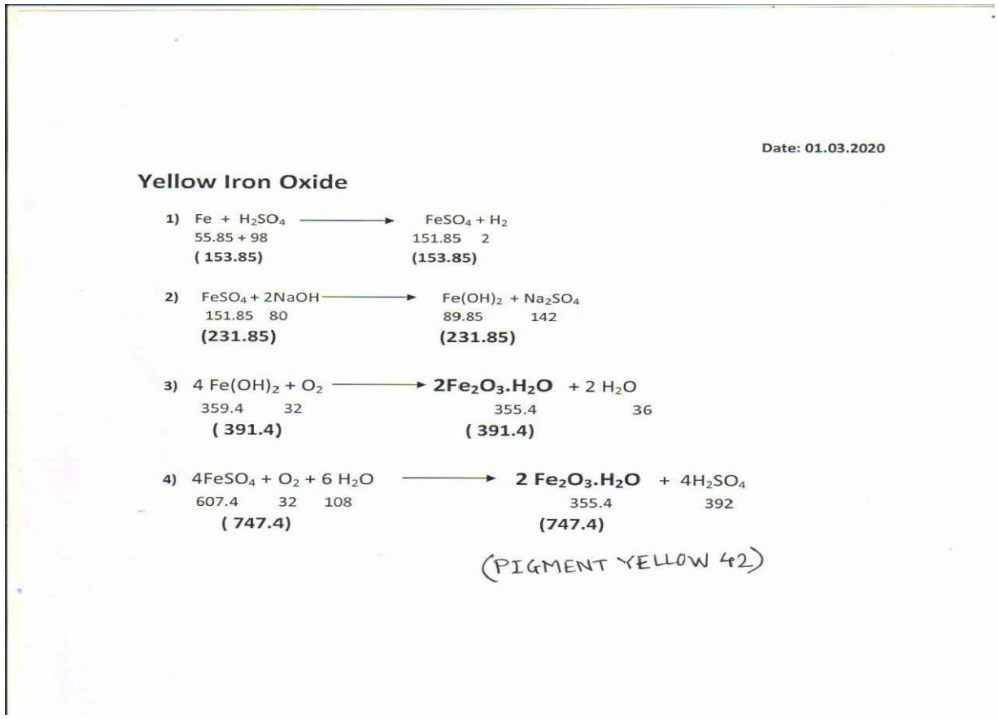
1.2 Introduction:

Pigment Yellow 42:-

Pigment Yellow 42 most commonly known as synthetic Iron yellow oxide is synthesized with precipitation reaction by most commonly known reaction called "Pennimans Zoph Process".In this reaction firstly a nucleous of ferric hydroxide called "seed" is formed by reacting diluted ferrous sulphate or ferric chloride with alkali solution of sodium hydroxide or sodium carbonates, calcium carbonates or calcium hydroxide. The nucleous thus formed is subjected to further cold aeration and continued till desired acidic mixture of greenish yellow colored is formed.

The final seed component is further transferred in another vat and there it is further diluted and hot aerated for 60-150 hours till the desired shade is achieved. Iron is the main raw material used as a catalyst or reaction inhibitor.

Being an acidic natured product, it sometimes does not behave properly in water based systems if it is not surface treated.



Hence before reaction goes to final stage of filtration, we have decided to apply surface treatment to improve final products dispersibility. The final slurry of pigment yellow 42 or simple synthetic Iron

oxide pigment is taken and further it is divided in six equal parts. All the parts are labeled as experiment 1- experiment 6 where experiment is considered as reference STD or non treated sample against which all the treated samples will be evaluated and final optimized surface treatment will be finalized for this product.

II: Objective of the Study:

The main objectives of this study are as follows:

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

III: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.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 Synthetic Iron Yellow & Red Oxides. Let us discuss the surface treatment study one by one for both the colour indices.

3.1Experimental work for the 2 CI's:-

3.1.1 Synthetic Iron Yellow Oxide (PY 42)

The treatment carried out forsix experiments is as follows:

A) Experiment 1 :-

Synthetic iron yellow oxide slurry is taken and heated to 70-75°C. It is held at hot condition for one hour and then filtered till salt free. It is then dried at 85°C and powdered in laboratory pulverizer and labeled as reference STD sample.

B) Experiment 2 :-

Synthetic iron yellow oxide slurry is taken and heated to 70-75°C. Then a solution of
(2% of pigmentation) sodium salt of Naphthalene sulphonic acid derivative which is used as an Anionic surfactant is being added to the slurry and the slurry being held at 75°Cfor one hour. After one hour, the slurry was cooled, filtered, dried at 85°C and powdered in laboratory pulverizer and labeled as experiment 2 for further analysis and evaluation.

C) Experiment 3 :-

Synthetic iron yellow oxide slurry is taken and heated to 70-75°C. Then a solution of (2% of pigmentation) castor oil derivatives which is used as an emulsifier is being added to the slurry and the slurry being held at 75°C for one hour. After one hour, the slurry was cooled, filtered, dried at 85°C and powdered in laboratory pulverizer and labeled as experiment 3 for further analysis and evaluation.

D) Experiment 4 :-

Synthetic iron yellow oxide slurry is taken and heated to 70-75°C. Then a solution of specially treated disproportionate rosin in short DPR rosin (2% of pigmentation) which is used for surface coating is

being added to the slurry and the slurry being held at 75°C for one hour. After one hour, the slurry was cooled, filtered, dried at 85°C and powdered in laboratory pulverizer and labeled as experiment 4 for further analysis and evaluation.

E) Experiment 5 :-

Synthetic iron yellow oxide slurry is taken and heated to 70-75°C. An equal amount of emulsion of anionic surfactant sodium lauryl sulphate and soya bean derivative is added to the slurry (2% of pigmentation) which is used as an emulsifier is being added to the slurry and the slurry being held at 75°C for one hour. After one hour, the slurry was cooled, filtered, dried at 85°C and powdered in laboratory pulverizer and labeled as experiment 5 for further analysis and evaluation.

F) Experiment 6 :-

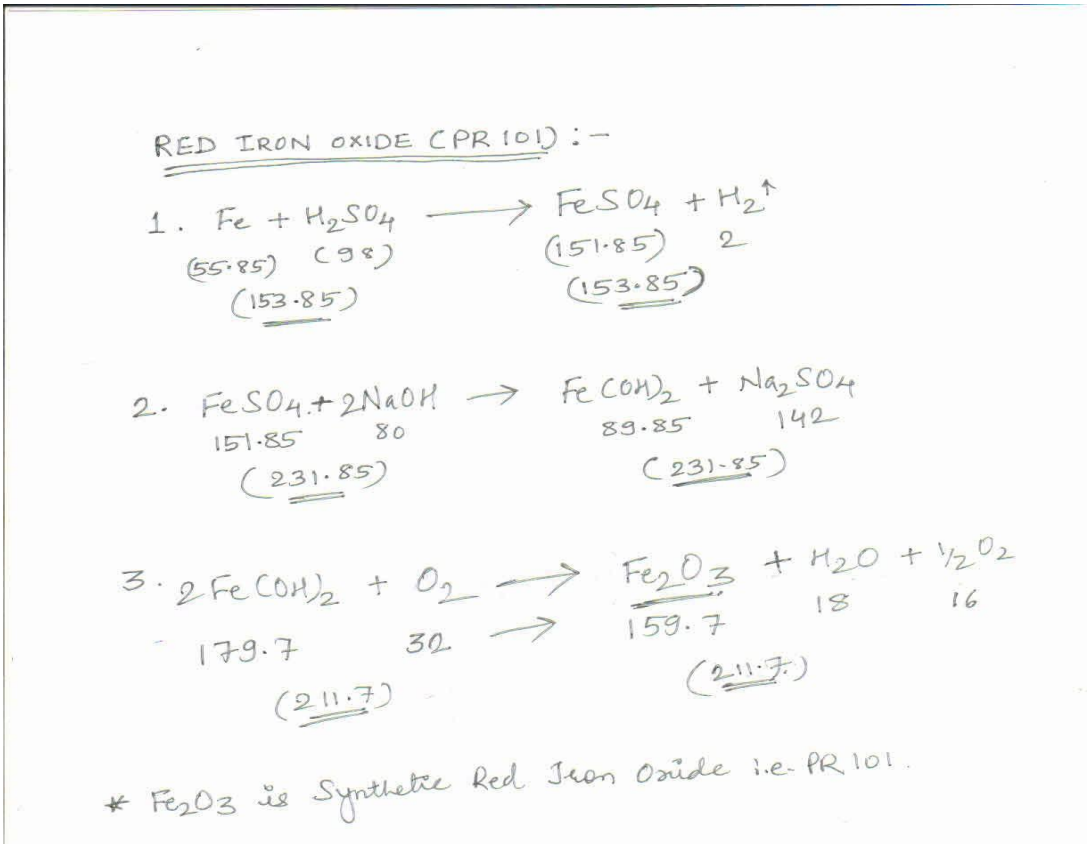
Synthetic iron yellow oxide slurry is taken and heated to 70-75°C. An equal amount of emulsion of anionic surfactant sodium lauryl sulphate and castor oil derivative is mixed properly to get uniform solution and is added to the slurry (2% of pigmentation) which is used as an emulsifier is being added to the slurry and the slurry being held at 75°C for one hour. After one hour, the slurry was cooled, filtered, dried at 85°C and powdered in laboratory pulverizer and labeled as experiment 6 for further analysis and evaluation.

All these six experiments are conducted in the laboratory 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.

3.1.2Synthetic red Oxide (PR101):-

Synthetic red iron oxide is chemically known as Pigment Red 101 or simplyPR 101. It is manufactured by precipitation reaction of dilute ferrous sulphate solution with dilute sodium hydroxide solution under controlled temperature pH conditions. The reaction mixture is then subjected to cold aeration till desired pH and desired rusty red colour is achieved. Like yellow oxide, red oxide has a different chemistry. The reaction mixture formed after precipitation and aeration is subjected to hot oxidation for 100 hours- 160 hours till the desired light red to maroon shade is achieved. Like synthetic yellow oxide, synthetic red oxide also has acidic nature. Hence, it is necessary to apply surface treatment.

Hence, decided to carry out one scale up reaction of synthetic red oxide and divide the slurry in six equal parts. Before the batch goes to final filtration stage, it is decided to give surface treatment with different category of surfactant and evaluate and analyze its effects in the form of coloristic as well as physical properties study.



The treatment is followed for the six experiments as follows:-

A) Experiment 1:-

As decided, slurry of synthetic iron red oxide is taken and heated to 90° C It is then held for one hour maintaining temperature 90°C. It is then cooled with water and filtered, washed till salt free. Further it is dried at 90°C, powdered in laboratory mixer and labeled as "Reference standard" for evaluation purpose.

B) Experiment 2:-

Slurry of synthetic iron red oxide is taken and heated to 90°C. Then asolution of sodium salt of Naphthalene benzene sulphonate condensate is added (2% of total pigmentation) to the reaction mixture. It is the anionic type surfactant. It is then held for one hour maintaining temperature 90°C. It is then cooled with water and filtered, washed till salt free. Further it is dried at 90°C, powdered in laboratory mixer and labeled as experiment 2 for further analysis and shade evaluation.

C) Experiment 3:-

Slurry of synthetic iron red oxide is taken and heated to 90°C. Then a solution of castor oil derivative(2% of total pigmentation) is added to the reaction mixture. It is the anionic type surfactant. It is then held for one hour maintaining temperature 90°C. It is then cooled with water and filtered, washed till salt free. Further it is dried at 90°C, powdered in laboratory mixer and labeled as experiment 3 for further analysis and shade evaluation

D) Experiment 4:-

Slurry of synthetic iron red oxide is taken and heated to 90° C. Then a solution of specially treated disproportionate rosin simply called as DPR rosin (2% of total pigmentation) is added to the reaction mixture. It is the anionic type surfactant. It is then held for one hour maintaining temperature 90°C. pH of the slurry was adjusted to 7.0 ± 0.2 with 10% Aluminum sulphate solution. It is then cooled

with water and filtered, washed till salt free. Further it is dried at 90°c, powdered in laboratory mixer and labeled as experiment 4 for further analysis and shade evaluation

E) Experiment 5:-

Slurry of synthetic iron red oxide is taken and heated to 90° C. An equal amount of emulsion of anionic surfactant sodium lauryl sulphate and soya bean derivative is added to the slurry (2% of pigmentation) which is used as an emulsifier is being added to the reaction mixture. It is then held for one hour maintaining temperature 90°C. pH of the slurry was adjusted to 7.0 ± 0.2 with 10% Aluminum sulphate solution. It is then cooled with water and filtered, washed till salt free. Further it is dried at 90°C, powdered in laboratory mixer and labeled as experiment 5 for further analysis and shade evaluation.

F) Experiment 6:-

Slurry of synthetic iron red oxide is taken and heated to 90°C. An equal amount of emulsion of anionic surfactant sodium lauryl sulphate and castor oil derivative is added to the slurry (2% of pigmentation) which is used as an emulsifier is being added to the reaction mixture. It is then held for one hour maintaining temperature 90°C. pH of the slurry was adjusted to 7.0 ± 0.2 with 10% Aluminum sulphate solution. It is then cooled with water and filtered, washed till salt free. Further it is dried at 90°C, powdered in laboratory mixer and labeled as experiment 6 for further analysis and shade evaluation

All these six experiments are conducted in the same laboratory 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.

3.1.3Testing Important Parameters of all the six experiments of the 2 pigmentswith 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 which means 6 samples of 2 different pigments for reduced tone only.For reference purpose, all the results are attached separately in the Result Analysis & Interpretations topic.

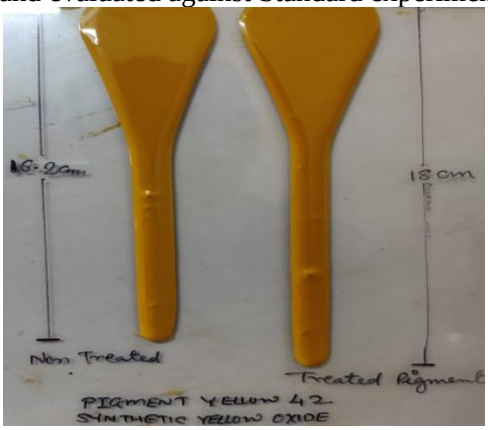
- **Flow Property:**

Flow is an important property of pigments as it is directly related to thedispersion of the pigments as well as its flow in required water-basecolorant or ink application**.**Flow is measured by making ink on Automatic Muller machine usingdoubly purified linseed oil as a medium. 10% ink is generally made andtransferred gently on smooth surface of Muller glass plates. It is then mixedproperly with the help of palette knife and grinded with 5Kg weight for 100rotations on Muller. The ink thus formed is subjected to one more round of100 further rotations & the ink is collected and labeled properly. The

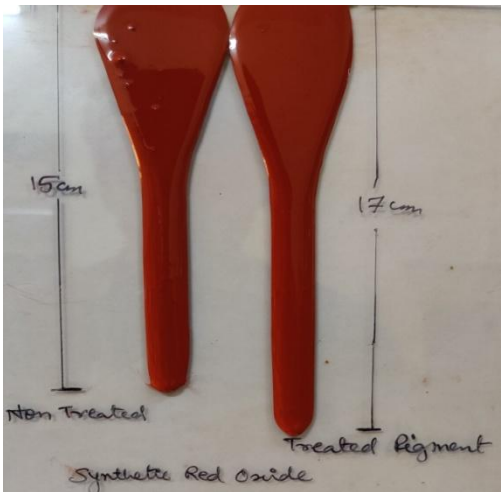
sameprocess is repeated for Standard sample & other than flow, one draw down ismade for preliminary evaluation of shade & strength which gives a rough ideaon the pigments colour trends.The flow is measured on a glass plate at 90 °C. Both the equallyweighed standard and sample inks prepared on Muller are placed near eachother on a horizontal plate. It is then placed at 90 °C and the flow ismeasured 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 ofboth the pigments.

1. **Pigment Yellow 42(Synthetic Yellow Oxide):**
Flow of 10% ink of Synthetic Iron Yellow Oxide pigment made in Linseed oil & tested on Muller and evaluated against Standard experiment.



2. **Pigment Red 101 (Synthetic Red Oxide):**
Flow of 10% ink of Synthetic Iron Red Oxide pigment made in Linseed oil & tested on Muller and evaluated against Standard experiment.



IV: Results and Interpretation:

As discussed earlier in research methodology topic, the two 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 alsostudied.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 atRaveshia Company's 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/100gm.

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. 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 the 2 surface treated pigments:

Physical Test Study of 12 Surface Treated Pigment Grades Selected For Study						
Sr.No.	**Colour Index**	**Lab Experiment**	**Bulk Density**		**Oil Absorption**	**Water Intake**
			As Is	**Tapped**		
1	**Synthetic Yellow Oxide Pigment Yellow 42**	**Experiment 1**	**0.1785**	**0.2941**	**48.1**	**24.4**
		Experiment 2	**0.1851**	**0.3571**	**38.85**	**22.5**
		Experiment 3	**0.1785**	**0.3125**	**38.85**	**15.7**
		Experiment 4	**0.1724**	**0.3125**	**40.24**	**17.5**
		Experiment 5	**0.1666**	**0.2941**	**42.77**	**20.4**
		Experiment 6	**0.1923**	**0.3333**	**40.07**	**20.6**
2	**Synthetic Red Oxide Pigment Red 101**	**Experiment 1**	**0.62**	**1.00**	**20.35**	**12.8**
		Experiment 2	**0.66**	**1.00**	**18.5**	**3.4**
		Experiment 3	**1.00**	**1.428**	**18.5**	**3.3**
		Experiment 4	**0.66**	**1.00**	**18.5**	**3.4**
		Experiment 5	**0.90**	**1.25**	**17.57**	**4.0**
		Experiment 6	**0.76**	**1.11**	**16.65**	**4.1**

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 both thepigment 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 Inorganic pigments that are Synthetic Iron Yellow Oxide and Synthetic Iron Red Oxide were 65% loaded in water-based resin & premixed for 5 minutes on High speed mixing Dispermat Machine. It was further stirred at 4500rpm for 45 minutes and the resultant colorants were packed properly in plastic containers.

The resultant colorants of all the pigment samples were evaluated by making their reduced tone with 4% pigmentation in water-based paintbase.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
1	Pigment Yellow 42 Synthetic Iron Yellow Oxide	2	0.39	0.08	0.12	0.36	0.38	-0.05	100.80
		3	0.89	-0.32	0.13	0.82	0.83	0.04	106.19
		4	0.84	-0.30	0.25	0.74	0.78	-0.09	105.70
		5	0.48	-0.36	0.14	0.29	0.31	-0.07	104.24
		6	0.48	-0.02	0.03	-0.47	-0.46	-0.12	98.12
2	Pigment Red 101 Synthetic Iron Red Oxide	2	0.38	0.20	-0.21	-0.24	-0.30	-0.12	97.76
		3	0.95	0.13	0.34	0.88	0.71	0.61	101.28
		4	0.61	-0.20	0.34	0.46	0.52	0.24	102.94
		5	0.24	0.20	-0.00	0.13	0.06	0.12	98.73
		6	0.45	-0.03	0.19	0.41	0.36	0.27	101.36

Table no.4.2.1 – Evaluation results of both the 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 asfollows:

4.3.1 Pigment Yellow 42:

From the six experiments of Pigment Yellow 42 or Synthetic Iron Yellow Oxide, carried out to study the effectiveness of surface treatment, Experiment 3,Experiment 4 & Experiment 5 showed positive results and reduction in Oil absorption values by making the pigment heavier in nature which ultimately improves its wettability by increasing its loading in water based colorant application gives the final product with at least4-6 % higher tinting strength when evaluated in Water-based colorant application against non treated reference standard experiment 1.

Hence, in Pigment Yellow 42, the use of an anionic surfactant which is a castor oil derivative and an equal proportion emulsion made up of two anionic surfactants improves the physical properties of Pigment Yellow 42 for water based application.

4.3.2 Pigment Red 101:

From the six experiments of Pigment Red 101 or Synthetic Iron Red Oxide, carried out to study the effectiveness of surface treatment, Experiment 3 & Experiment 4 showed positive results and reduction in Oil absorption values by making the pigment heavier in nature which ultimately improves its wettability by increasing its loading in water based colorant application and help the product to disperse properly & maintain at least the strength of 101-102% when evaluated in Water-based colorant application against non treated reference standard experiment 1.

Hence, in Pigment Red 101, the use of an anionic surfactant which is a castor oil derivative and an equal proportion emulsion made up of two anionic surfactants improves the physical properties of Pigment Red 101 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 Humectants. 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 repellant 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 ballsor 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 bevaviour.
In our study also it's clearly replicated that compared to non treated Standard reference experiments of all
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 forparticular 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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