International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 04 Issue: 06 | June-2017 www.irjet.net p-ISSN: 2395-0072

TO INCREASE THE CONCENTRATION OF SPENT SULFURIC ACID IN ALPHA-BLUE PIGMENT PROCESS

Vishal U.Shah[1], Dharmendra Mandaliya[2]

[1] *Asst.Professor , Chemical Engineering Department, Sitarambhai Naranji Patel Institute of Technology & Research Center, Umrakh, Gujarat, India*

[2] *Asst. Professor, Chemical Engineering Department, Vishwakarma Government Engineering Collage, Gujarat, India*

Abstract - *The paper summarizes to increase the concentration in the α-Blue Manufacturing Process. There is a very big problem of sulfuric-acid in dye and pigment industries. They get the sulfuric acid in mother liquor around 25 to 30 %. So they can't use in anywhere directly. So if we try to increase the concentration of sulfuric acid then we can do something for this problem. These types of problem faced one company PIGMENT INDIA LIMITED, Ankleshwar. So we try to increase the concentration by process modification and achieving up to 50 % by process modification. The process description is presented in by taking into account the possible hazards in all the seven steps of reaction. A concentration is increasing in the steps of drowning section of the whole process. A hazard and operability study is presented. The evaluation of hazard offers the advantage of designing the processes for better health, safety, and environment.*

Key Words: *Sulfuric Acid, Copper Phthalocyanine blue (CpC Blue), Rosin, Caustic, Alpha-blue pigment powder.*

1. INTRODUCTION

A pigment is a material that changes the color of reflected or transmitted light as the result of wavelength-selective absorption. This physical process differs from fluorescence, phosphorescence, and other forms of luminescence, in which a material emits light. A pigment must have a high tinting strength relative to the materials it colors. It must be stable in solid form at ambient temperatures.

Pigments are used for coloring paint, ink, plastic, fabric, cosmetics, food and other materials. Most pigments used in manufacturing and the visual arts are dry colorants, usually ground into a fine powder. This powder is added to a vehicle (or binder), a relatively neutral or colorless material that suspends the pigment and gives the paint its adhesion. In some cases, a pigment can be manufactured from a dye by precipitating a soluble dye with a metallic salt

1.1 INTRODUCTION ABOUT REACTION

From this chapter about for reaction and reaction mechanism of Alpha-Blue Manufacturing process. In this Process mainly one reaction takes place and makes Alpha-Blue Pigment.

1 mole of the CPC (Copper Phthalocyanine) Blue is reacted with 1 mole of 70% H_2SO_4 and gives 1 mole of Alpha-Blue Pigment and 1 mole of water.

CPC blue reacted with Sulfuric Acid and Pigmentation takes place and we get Alpha-Blue Pigment. This reaction takes place at 20° C.

FIG-1 : Muller Mixer

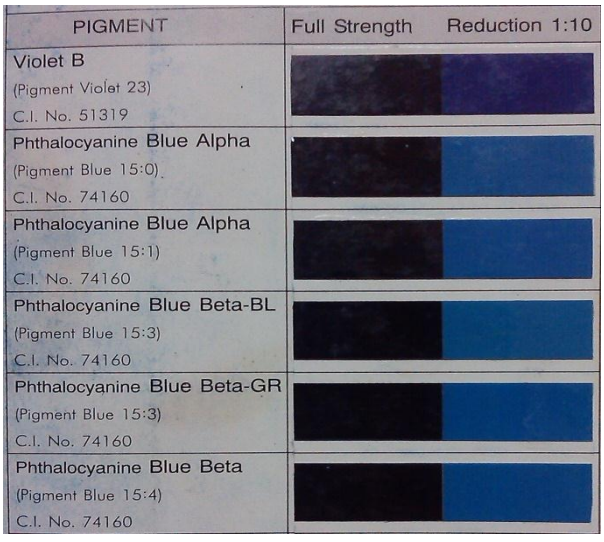

Fig -2: Color Strength Chart

Here we see the Structure of CPC Blue and Alpha-Blue, but from reaction only properties are change of CPC not change the Structure. After changing the properties, it partly soluble in water & it can be used to make water base paint.

3. LITERATURE RIVIEW

Gerson, H. and Sattar, invented that A Process for alpha-phase metal phthalocyanine pigments Process for Alpha-phase metal phthalocyanine pigment, US Patent 5534055,in this research paper they invented (a)acid pasting or acid swelling a crude metal phthalocyanine pigment(b) dry milling the acid-pasted or acid-swelled metal phthalocyanine pigment in the presence of 5 to 50 parts by weight of a stabilizer per 100 parts by weight of the crude metal phthalocyanine pigment until the average particle size is reduced to less than about 0.5 pm(C) finishing the milled metal phthalocyanine pigment by thoroughly mixing said milled metal phthalocyanine pigment with a finishing solvent mixture comprising 3 to 6 parts by weight, relative to the crude metal phthalocyanine pigment, of water and 0.4 to 1.0 parts by weight, relative to the crude metal phthalocyanine pigment, of an organic solvent, optionally in the presence of 0 to 45 parts by weight of a stabilizer per 100 parts by weight of the crude metal phthalocyanine pigment, with the proviso that the total amount of stabilizer used in steps (b) and (c) ranges from 5 to 50 parts by weight per 100 parts by weight of the crude metal Phthalocyanine pigment and(d) Isolating the alpha-phase metal phthalocyanine pigment.

Langley, R. invented that Process for preparing an alpha or beta form copper phthalocyanine pigment US Patent 4104277, in this paper he invented a process for preparing an alpha or beta form copper phthalocyanine pigment which comprises dry milling a crude copper phthalocyanine followed by mixing with an aqueous emulsion comprising a liquid amine having from 3 to 20 carbon atoms which is insoluble in water or alkali and soluble in acid and a surfactant which is capable of emulsifying the liquid amine and subsequently acidifying the mixture to dissolve the amine.

Schiebler, S. and Spietschka, E. and Tronich, W. invented that Process for preparing copper phthalocynine pigments of the alpha- modification US Patent 3984433, in this paper they invented that , a process for preparing very pure copper phthalocyanine pigments of the alpha modification, wherein substituted or un substituted copper phthalocyanine having different degrees of purity are converted into copper phthalocyanine Salts Capable of being isolated' with the aid of suitable acids which dissolve the impurities contained in the dye stuff, wherein these salts are Separated from the acid, the copper phthalocyanine of the alpha-modification are Set free in 9 Pure form from the copper phthalocyanine Salts by the action of Water and wherein the isolated copper phthalocyanine is subject in an aqueous suspension to a mechanical fine division. This process yields copper phthalocyanine of alpha-modification in a very pure form without causing problems with respect to the waste water.

Raab, H. and Hornle, R. invented that alpha-copper phthalocyanine preparation US Patent 3137704, in this paper they invented that , a process for the production of the alpha modification of copper phthalocyanine. More particularly it concerns a process for the production of a strongly colored alpha modification of copper phthalocyanine. In a process for producing a strongly-colored alpha copper phthalocyanine by grinding at least one copper phthalocyanine pigment selected from the class consisting of beta-copper phthalocyanine and weakly-colored alpha-copper phthalocyanine, in the presence of a solid grinding adjutant, the improvement which. consists in grinding the copper phthalocyanine pigment in the presence of at least one organic additive selected from the class consisting of a halogen alcohol, a cyano alcohol and an ester thereof, said alcohol moiety having 2 to 6 carbon atoms with not more than one hydroxyl group 3-chloro-2-hydroxypropanol-l; effecting the grinding under dry milling conditions at a temperature not exceeding about 700.

Schiessler, S. and Spietschka, E. and Tronich, W. invented that Process for preparing copper phthalocyanine pigments of the alpha-modification US patent 4224222, in this paper they invented to a process for the preparation of a very pure copper phthalocyanine pigment of the a modification which comprises dissolving crude copper phthalocyanine in a 80 to 86% sulfuric acid isolating the copper phthalocyanine sulfate formed, hydrolyzing the sulfate and grinding the copper phthalocyanine thus obtained in an aqueous organic medium.

John W. Minnich, Wilmington, Deh, invented that Production of Phthalocynine Pigment US Patent 3051720, in this paper they invented that 18' parts of a substantially chlorine-free copper phthalocyanine pigment (obtained by the reaction at an elevated temperature of phthalic anhydride with urea and copper chloride in kerosene and in the presence of ammonium molybdenum as a catalyst) is charged to a ball mill containing about 1000 parts of \Cyl- pebs" (\Cyl- pebs" are steel rods approximately 542" x 1"). The size of the mill is such that the full charge of pigment and \Cyl- pebs" occupies approximately 60|65% of the total volume of the mill. The mill is rotated at about 70% of the critical speed (the critical speed is that at which the centrifugal force overcomes the force of gravity so that the grinding elements are retained against the outer wall of the mill) for about 6 hours. The dry powder is discharged from the mill through a suitable screen. l5 parts of this dry powder is then mixed with 55 parts of

70% sulfuric acid using only. Sufficient agitation to completely wet the dry powder. Some heat is developed during this operation, but no added heat is necessary nor is any cooling necessary under normal circumstances. The mixture of acid and pigment is allowed to stand for about 1 hour after which 300 parts of cold water is added. Agitation is then commenced and the slurry is heated to the boil and stirred for about 1/2 hour at the boil. It is then filtered, washed free of soluble salts and the pigment is dried at 800. To give a red shade copper phthalocyanine pigment of Alpha-Phase gives superior tectorial strength.

4 Process Modification Process

Table -1: Process Modification Studies

Practical Number	Concentration of Sulfuric acid (taken)	Density of Sulfuric Acid (g/cc)	Quantity of Sulfuric Acid (ml)	Quantity of CPC (gm)	CPC Unmilled/Milled	Reaction Time (hr)	% H_2SO_4	Muller Sample Comparision	Water Quantity (ml)
1	70 %	1.60	200	100	Milled (22hr)	4	26%	60%	150
2	70 %	1.60	250	100	Milled (22hr)	4	38%	55%	300
3	98%	1.60	230	100	Milled (25hr)	4	47.7%	50%	200
4	70%	1.55	250	100	Milled (13hr)	4	38%	65%	500
6	70%	1.60	200	100	Unmilled	4	35%	40%	150water+100gm ice
7	98%	1.82	200	100	Unmilled	4	28%	45%	150water+100gm ice
8	98%	1.82	230	100	Unmilled	4	42.96%	48%	250water+70gm ice
9	70%	1.60	200	50	Milled (22hr)	1.5	49%	70%	150 ml water
10	70%	1.60	250	80	Milled (22hr)	1	47.8%	60%	150 ml water
11	70% Dilution (63%)	1.55	200	50	Milled (15hr)	1	51%	95%	150 ml water
12	70% Dilution (63%)	1.55	200	50	Milled (15hr)	1	53%	98%	120 ml water
13	**70% Dilution (63%)**	**1.55**	**200**	**50**	**Milled (15hr)**	**1**	**55%**	**100%**	**110 ml water**

First Set up all Equipment Properly, and ready all chemical for reaction. Now after set up all equipment and start process for making alpha-blue pigment. Take 70% H_2SO_4 (Dilution make 63%) 200 ml in batch reactor. Note down the temperature of that 70% H_2SO_4 (Dilution make 63%).Start Stirring. Then Start slowly adds CpC (Copper Pthalocyanine Blue) 50 gm. Here note down the temperature of mixture, and for maintaining temperature take water around the batch reactor in PP Bowl. Stirring this mixture for 1 hrs, and maintain temperature 200. After 1 hr that both mixture are completely mixed, then take all mixture in one Big Beaker, then add 110 ml fresh water and start manually stirring with glass road. Before starting filtration Start vacuum in Conical ask, Then Start to filter that mixture.

Fig-3 Muller Mixer Strength

After completion of filter we get in conical ask 55% H_2SO_4 (light yellow color).After completion of filtration washed wet cake with 1000 ml water for removing sulfuric from material. Further Take all wet cake in batch reactor and add sufficient amount of water ,then take 2.5 gm Caustic and set pH 9, also add 1.5 gm TLS Solution, all component are mixed in batch reactor and start stirring and heating. Heating up to 80 to 900.Then cool up to 500 and start filtration. Mother liquor sends to ETP (9 PH), and wet cake to further wash with water till 7 PH. Wet cake dried in Dryer.

List Item – Filtration Unit
List Item - Heating Mental
List Item - CSTR with stirring medium
List Item - Muller Mixer

5 CONCLUSIONS

This section gives the detail practical run and raw material detail. Here we take a different run with different concentration of sulfuric acid, but with different concentration of sulfuric acid we get different % of Spent Acid.

In first two run we take 70% sulfuric as a raw material and CpC we take (22hr) milled. and from reaction we get spent concentration but muller sample should not be matched with slandered sample.

In run 10, 11, 12 we take dilution sulfuric acid make 63% as a raw material and take (15 hr) milled CpC, after reaction we get 51-55 % spent sulfuric Acid and also this muller sample match with slandered sample, so this is our requirement for manufacturing of alpha-blue pigment.

In run 5, 6, 7 we take un-milled CpC but for proper reaction we require milled CpC, from un-milled CpC we can't get proper Alpha-Blue Product and also we can't achieve proper strength. So this process we can't use for making alpha-blue pigment.

REFERENCES

[1] Gerson, H. and Sattar, A.,Process for alpha-phase metal phthalocyanine pigments, Available at=http://www.google.co.in/patents/US5534055,year=(1996), month=jul9, publisher=Google Patents,note=US Patent 5,534,055.

[2] Langley, R.,Process for preparing an alpha or beta form copper phthalocyanine pigment,Available at=http://www.google.com/patents/US4104277,year=1978,month=aug 1, publisher=Google Patents,note=US Patent 4,104,277.

[3] Donegan, T.E. and Leary, T.G. and Patil, A.S., Process for making alpha-phase phthalocyanine blue pigment comprising substantially 100 percent isometric crystals, Available at=http://www.google.com/patents/US4859770,year=1989,month=aug 22 publisher=Google Patents, note=US Patent 4,859,770.

[4] Schiessler, S. and Spietschka, E. and Tronich, W.,Process for preparing copper phthalocyanine pigments of the Alpha modification, Available at =http://www.google.co.in/patents/US4224222,year =1980,month=sep 23, publisher=Google Patents, note=US Patent 4,224,222.

[5] Schiebler, S. and Spietschka, E. and Tronich, W.,Process for preparing copper phthalocyanine pigments of the Alpha modification, Available at =http://www.google.co.in/patents/US3984433,year =1976,month=oct 5, publisher=Google Patents, note=US Patent 3,984,433.

[6] Raab, H. and Hornle, R., Alpha-copper-phthalocyanine preparation, Available at =http://www.google.com/patents/US3137704,year=1964,month=jun16, publisher=Google Patents note=US Patent 3,137,704.

[7] John W. Minnich, Wilmington, Deh Production of phthalocyanine pigments, Available at =http://www.google.com/patents/US3051720,year=1962,month=aug28, publisher=Google Patents, note=US Patent 3,051,720.