

Improvisation in Pigmentory Properties of Quinacridone Pigment

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Abstract: Monosulphonation of Quinacridone pigment has been carried out at degree of sulphonation 0.9. A new additive has been than prepared by coupling sulpho derivative with long chain amine [Arquad 2HT-75 (Di (hydrogenated tallow alkyl) dimethyl ammonium chloride)]. Effect of addition of sulpho derivative and new additive on pigmentory properties in solvent base ink has been measured. Addition of sulpho derivative and additive in Quinacridone pigment (Pigment Red 122) improve the pigmentory properties like viscosity, gloss and transparency.

Keywords: Pigmentory Properties, Rehology, Viscosity, Gloss, Solvent base ink.

INTRODUCTION

This invention relates to materials for improving the dispersion properties of quinacridone pigments and to improved quinacridone pigments containing such materials. More specifically, it relates to sulfonated quinacridone compounds and the use there of as well as the use of free quinacridone sulfonic acid and other metal salts thereof both as agents to prevent crystal growth of quinacridone pigments during aqueous extraction steps and as agents for improving the shear strength uniformity of coating compositions containing a quinacridone pigment^{1,2,3}.

In spite of the apparent insolubility of quinacridone pigments in aqueous systems, it has been found that finely divided quinacridone products frequently undergo a substantial increase in particle size when subjected to an aqueous heat treatment.

Thus, it is common practice to reduce the particle size of quinacridone pigments by ball milling with a finely divided inorganic salt such as sodium chloride or hydrated aluminum sulfate, after which the salt is removed by a hot extraction in dilute aqueous acid. Similarly, particle size reduction may be accomplished by solution in concentrated sulfuric acid followed by dilution in water to precipitate the finely divided pigment. In each case the resulting particle size has been found to vary with changes in the extraction technique, particularly those of temperature. Thus, it is obvious that the crystallites may actually grow in size during the extraction. Since such crystal growth is reflected in deterioration of desirable pigment properties (e.g. tinting strength, transparency, etc.), a method of inhibiting this growth is sorely needed^{4,5,6,7}.

Furthermore, coating compositions containing quinacridone pigments may exhibit profound differences in color depending upon the method of application of the coating composition film, the amount of dispersive effort applied while depositing the coating upon the substrate, and the degree to which the film is disturbed during drying. Also, coating compositions pigmented with quinacridones may show objectionable flow properties so as to seriously limit the quantity of pigment which can be incorporated into

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the vehicle to produce a composition with adequate flow for practical use^{8,9,10}.

EXPERIMENTAL

All chemicals used in experimental work were AR grade.

Sulphonation Of Quinacridone Pigments:

100 grams quinacridone is stirred into 1000 grams of 98% sulfuric acid. The mixture is heated to 60-65°C for 12 hours and maintained temperature for 12 hours after which is cooled to 35°C. Add vigorous stirring to about 6000 ml ice water temperature <5°C during drowning maintained temperature 10-12°C stir 1 hour reaction mixture at 10-15°C. Filter it wash till Congo (-)ve collect w/c. and dry it at 80°C.

Degree of sulfonation :0.9.

Method for checking degree of sulphonation:

Take Accurate weight of sulphomass about 2.5 grams. In beaker add 200 ml distilled water. stir well for 10 to 15 minutes. Then titrate it against 1N NaOH solution up to pH 7.0 on pH meter

% Acidity :

$$\frac{\text{Burette Reading} * \text{Normality} * \text{Molecular Weight of H}_2\text{SO}_4}{\text{Weight Taken} * 10}$$

Then boiled it at 90°C for 10-15 minutes. Cool it down to room temperature. Then add more 200 ml distilled water and then titrated against Primary Amine (MW 185). Check run out. If water is complete comes out in form of run out than its called end point.

$$\text{A : } \frac{\text{Burette Reading} * \text{Normality Of Amine}}{\text{Molecular weight of Amine (MW 185)}}$$

$$\text{B : } \frac{\text{Weight of Sulphomass} * \text{Solid Content}}{\text{Molecular weight Of Compound} + 80}$$

Degree Of Sulphonation : A/B

Degree of sulfonation :0.9.

The degree of sulphonation is achieved by changing reaction time and temperature. By checking

the coloristic data of sulphonated pigments having different degree of sulphonation. It is found that the pigment having 0.9 degree of sulphonation should better results in polyamide and nitro cellulose base ink applications. There for we have used sulphonated pigment having 0.9 degree of sulphonation for preparation of additive.

Preparation of Additive:

The press cake containing about 50 grams (dry base) w/c 312 grams active content 16.2% of quinacridone sulfonic acid is reslurried in 1000 ml of water with vigorous agitation to stirred suspension of quinacridone sulfonic acid. Degree of sulfonation 0.9. Add 25ml 10% dilute caustic lye keeping pH 9. Heat to 80°C maintained temperature 1 hour. Add a solution of 75 grams 2 HT -75 Amine (active content 98%). The quinacridone sulfonated solution is attached 2HT-75 Amine. The suspension is isolated keeping pH 9 by dilute caustic lye and maintained temperature 80°C for 2 hours. The isolated product is filtration and washing the filter cake till free of alkali and sulfate ions and drying.

RESULTS AND DISCUSSION

To find out the effect of sulpho derivatives and additive on the pigmentary properties of quinacridone pigment, different proportion of and additives varying from 2% to 10% were added to untreated pigment. The rheological properties like viscosity, gloss, color strength is measured in sulphoderivatives NC base ink and Polyamide ink application with untreated pigment. We have used 20% pigment loading in both applications and checked their rheological properties. We have measured the viscosity by Zahn Cup No 3, gloss By Gloss meter at 60°. The coloristic data were measured by Macbeth color matching machine and transparency by visual observation on transparent paper. The results are given in Table 1 And Table 2.

Table:1 Effect of Sulpho derivatives In Quinacridone Pigment (Pigment Red 122)

Sr.No		Degree of Sulphonation	Loading of Sulpho derivatives in untreated Pigment	Viscosity (in Sec.)		Gloss at 60°		Strength	Mass Tone				Transperency
				Untreated Pigment	Sample	Untreated Pigment	Sample		DA	DB	DC	DE	
1	NC APPLICATION	0.9	2.00%	E	E	12.2	11.1	94.76%	-2.117	-0.329	-2.102	2.183	+
2		0.9	4.00%	E	D	15.4	13	96.82%	-3.396	-0.491	-3.354	3.502	+++++
3		0.9	6.00%	E	C	15	13.3	93.73%	-6.414	-1.658	-0.59	6.772	++
4		0.9	8.00%	E	B	16.5	14.1	94.30%	-6.707	-1.454	-6.793	7.013	++
5		0.9	10.00%	E	A	16	13.8	91.46%	-7.242	-1.717	-7.393	7.599	+
6	POLYAMIDE APPLICATION	0.9	2.00%	24 SEC	9 SEC	43.8	50.2	111.60%	-0.087	-0.383	-0.148	0.409	++
7		0.9	4.00%	24 SEC	9 SEC	43.5	48.1	113.60%	-0.822	-0.645	-0.916	1.099	+++++
8		0.9	6.00%	24 SEC	9 SEC	42.9	45.8	109.03%	-2.021	-1.047	-2.163	2.385	+++
9		0.9	8.00%	24 SEC	9 SEC	40.7	41.1	106.43%	-2.957	-1.955	-3.213	3.712	+++
10		0.9	10.00%	24 SEC	10 SEC	40.1	39.9	103.25%	0.995	-0.691	1.196	1.212	++

A: BEST ; B:BETTER ; C:GOOD ; D:BAD ; E: WORST

Table:2 Effect of Additive In Quinacridone Pigment (Pigment Red 122)

Sr.No		Degree of Sulphonation	Loading Of additive in untreated Pigment	Viscosity (in Sec.)		Gloss at 60°		Strength	Mass Tone				Transperency
				Untreated Pigment	Sample	Untreated Pigment	Sample		DA	DB	DC	DE	
1	NC APPLICATION	0.9	2.00%	E	D	13.5	13.3	98.50%	-2.28	0.779	- 1.926	2.441	+
2		0.9	4.00%	E	C	13.2	14.7	96.90%	- 2.955	- 0.011	- 2.798	3.032	++
3		0.9	6.00%	E	B	13.3	13.1	100.54%	- 1.665	- 0.552	- 1.401	1.774	+++++
4		0.9	8.00%	E	A	13	13.1	96.60%	- 2.383	- 0.901	- 1.976	2.573	++++
5		0.9	10.00%	E	A	13.1	14.8	95.01%	-1.75	- 0.478	- 1.811	1.879	++
6	POLYAMIDE APPLICATION	0.9	2.00%	38 SEC	26 SEC	40.1	43.3	98.24%	- 0.995	0.238	- 0.935	1.044	++
7		0.9	4.00%	38 SEC	22 SEC	37.4	40.3	99.26%	- 0.696	0.4	- 0.615	0.813	+++
8		0.9	6.00%	38 SEC	18 SEC	38.9	41.5	99.92%	- 0.719	- 0.336	- 0.645	0.806	+++++
9		0.9	8.00%	38 SEC	15 SEC	36.6	42.3	100.24%	0.916	- 0.801	0.763	1.220	+++
10		0.9	10.00%	38 SEC	13 SEC	40.3	43.1	99.63%	1.053	- 0.603	0.93	1.229	++

A: BEST ; B:BETTER ; C:GOOD ; D:BAD ; E: WORST

Effect of Sulphoderivatives:

From the results given in Table 1, it is found that in NC base ink applications due to addition of sulpho derivative the viscosity is decreased while the gloss, color strength and transparency remain same with compare to untreated pigment. It is also observed that 4 % loading of sulpho derivative gave better results compare to other proportion of loading.

In Polyamide ink application we found that the effect of addition of sulpho derivative show lower viscosity, improved gloss, better transparency and higher color strength. For the internal comparison with different proportion we found that 4% loading of sulpho derivative in untreated pigment gives better rheological properties compare to 2%, 4%,8% and 10%.

From the results it may be generalized that the sulpho derivative shows better rheological properties in Polyamide Ink applications due to its non polar system.

Effect of Additives:

From the results given in Table 2, It is found that in both NC base and Polyamide base ink applications, the all rheological properties of pigment are increased with the addition of additive. The 6% loading of additive give excellent results in both applications in terms of viscosity, gloss, transparency and color strength with compare to other proportions.

From all results it is conclude that long chain quaternary amines coupled with sulpho derivative of pigment may be excellent additive for the improvisation of pigmentory properties.

ACKNOWLEDGEMENTS

The Authors are thankful to Uttar-Purva Gujarat Uchcha Kelavani Mandal, Pilvai and Alpanil Industries for laboratory facilities.

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