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Preparation of New Pigments of Quinacridone Group and their Pigmentory Properties

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Abstract: As we know that from whatever commercially available pigments in the market is made from Derivatives of Aniline and its methyl and chloro derivatives. Here we are try to make new Pigment of Quinacridone series with very light shade like Azo pigments with rheological Properties of Quinacridone In terms of Viscosity, Gloss, Heat stability and weather fastness.
Keywords: New Pigments, Quinacridone Pigment, High Performance Pigment.

Introduction

Quinacridone pigment were first prepared[1,2,3] in 1935, their technical value initially went unrecognized. It was only when polymorphism was detected that quinacridone gained importance. The systematic synthesis of different crystal modification by appropriate after treatment of crude quinacridone made it possible to obtain variety of highly stable pigments which rapidly gained commercial interest as new class of pigments.

A considerable number of mono-to tetra- substituted quinacridone has been described so far. A publication which includes literature up to the year 1965 already lists more than 120 compounds, and many more than one crystal modification[4]. However this diversity of compounds does not reflect commercial variety. Only very few species have been offered, and even less command a major share of the pigments market.

Heading the list are two crystal modification of Pigment Violet 19 (the parent compound of quinacridone series). Both the red –violet beta and the red gamma modification are encountered in the market, while the pure alpha form and is not lightfast and durable enough to have any commercial value. However, trace of alpha modification is found in variety of types.

Of the substituted quinacridone systems, it is preliminary the 2,9-dimethyl derivative (Pigment Red 122) which enjoys appreciation for its excellent fastness properties and its pure bluish red shade. Besides, the list also includes 2,9-dichloro, 3, 10 dichloro, 4,11 dichloro, and 4,11 dimethyl quinacridones. Some of this are available as mixed phase with other quinacridone derivatives[5].

Other mixed phase are made from quinacridone quinine with substituted quinacridone or 4,11 – dichloroquinacridone as second component[6,7,8].

Quinacridone quinone itself is a tintorically relatively weak yellow compound with pour light fastness. Oxidation of dihydroquinacridone with less than molar amounts of chromate affords a quinacridone/ quinacridone quinone mixed phase which offers and interesting shade of gold.

Experimental Work

Step: 1: Preparation of 2, 5-dinapthalino-3, 6-dihydrotereph-thalate.

150.0 parts of succinylo-succinic acid dimethyl ester, 200.0 parts of 1-aminonaphthalene and 1500.0 parts of Methyl Alcohol are heated under nitrogen to 45° to 50° C., mixed while stirring with 15 parts of 33% hydrochloric acid and stirring is continued for 15 minutes. The mixture is heated within one hour to reflux temperature,. After heating for 4 hours to reflux temperature, Remove Methyl Alcohol by distillation. Then add 1500 parts water and stir for 1 hour at 50°. Than filtered it and washed it till neutralized. Dry it at 80°. Resulting product is 2, 5-dinapthalino-3, 6-dihydrotereph-thalate. Dry weight is 290 parts and yield is 92.5%

Step: 2: Preparation of 2, 5-dinapthalino-3, 6-dihydroterephalic acid

250 parts of diethyl 2, 5-diamlino-3, 6-dihydrotereph-thalate is added to 2000 parts of Methyl Alcohol in a jacketed vessel equipped with an agitator and a reflux condenser. There is then added, in turn, a solution of 200 parts of nitrobenzene-meta-sodium sulfonate in 100 parts of water and a solution of 200 parts sodium hydroxide in 200 parts of water. The mixture is then heated to the boil under good agitation and maintained at the boil under total reflux for about 12 Hours. The charge is then poured into about 2000 parts of cold water and slowly acidified with about 600 parts concentrated hydrochloric acid until there is a strong positive test on Congo red paper (pH less than about 3.0). After stirring about 15 minutes, the highly colored precipitate is isolated by filtering, washed free of soluble salts and dried at about 60° C. to give about 229 parts of a purple solid (97.2% yield). Resulting product is 2, 5-dinapthalino-3, 6-dihydroterephalic acid.

Step: 3: Preparation of crude Violet

110 Parts by weight of 2, 5-dinapthalino-terephthalic acid are introduced into 500 parts by weight of polyphos-phoric acid having a P_2O_5 content of 85.2%, at 110° to 120° with stirring. The batch is stirred for 3 to 4 hours at 120° to 130° C. The hot melt is hydrolyzed by pouring it into 90 parts by weight of water and the precipitated crude quinacridone is filtered off and washed until free of acid. Than Dry Crude Quinacridone at 80°and weigh. Dry weight is 96 parts and resulting yield is 95%.

Step: 4: Pigmentation

50 parts crude quinacridone and 500 parts N-methyl pyrolidone in a jacketed vessel equipped with an agitator and a reflux condenser. The quinacridone suspension is heated in a closed vessel to 140° to 150° C. and kept at this temperature for 4 hours with stirring. After cooling to 80° C., the NMP is distilled off and add equal amount of water, the pigment is filtered off, washed with water and dried. Dry weight is 48.2 parts and yield is 96.4 The resulting much yellower pigment is the linear quinacridone in the pure gamma crystal form. It is distinguished by grain softness, good dispersibility and a very pure shade.

Results and Discussion

From the **Table 1** we found following results.

Alkyd Paint Application :

Color Tone :

Color tone is defined from the value of mass tone. And we found that DA value is -5.45 seems that its greener against color tone of Pigment Red 122. And DB value is 20.93 seems that is much yellower against color tone of Pigment Red 122. DE value shows total color difference and its value is 22.76. So from that we conclude that New Pigment is greener and much yellower compared to Color tone of Pigment Red 122 in Alkyd Paint Application.

Color tone is defined from the value of mass tone. And we found that DA value is -12.06 seems that its much greener against color tone of Pigment Violet 19. And DB value is 11.37 seems that is yellower against color tone of Pigment Violet 19. DE value shows total color difference and its value is 16.89. So from that we conclude that New Pigment is much greener and yellower compared to Color tone of Pigment Violet 19 in Alkyd Paint Application.

Overall conclusion that New Pigment is greener and yellower compared to commercial available Pigment Red 122 and Violet 19 in alkyd paint application.

Hue: Hue is calculated from DC value. And Here DC is 5.78 against Pigment Red 122 and it shows that its brighter than Pigment Red 122. And DC is -3.74 against Pigment Violet 19 shows that its duller against Pigment Violet 19.

Color Strength:

Color Strength is always defined by value from tint tone. We found that product is greener and yellower compared to Pigment Red 122 and Pigment Violet 19 and due to very light shade it seems that less strength or weaker

against Pigment Red 122 and Pigment Violet 19. New Pigment is showing 63.45 % against pigment Red 122 and 64.87% against Pigment Violet 19.
We also observed that new pigment behave similarly in the presence Of white pigments in tint tone. From the value of DA, DB shows that it is greener and yellower against both pigments.

Gloss:

Gloss is defined from visual observation of mass tone. And we found that gloss is almost similar to Pigment Red 122 and Pigment Violet 19.

Waterbase Ink :

Color Tone :

Color tone is defined from the value of mass tone. And we found that DA value is -2.01 seems that its slight greener against color tone of Pigment Red 122. And DB value is 4.94 seems that is yellower against color tone of Pigment Red 122. DE value shows total color difference and its value is 0.64. So from that we conclude that New Pigment is Slight greener and yellower compared to Color tone of Pigment Red 122 in water base ink Application.
Color tone is defined from the value of mass tone. And we found that DA value is -14.15 seems that its much greener against color tone of Pigment Violet 19. And DB value is -4.04 seems that is slightly bluer against color tone of Pigment Violet 19. DE value shows total color difference and its value is 14.97. So from that we conclude that New Pigment is much greener and bluer compared to Color tone of Pigment Violet 19 in water base ink Application.
Overall conclusion that New Pigment is greener and yellower compared to commercial available Pigment Red 122 and greener and slightly bluer against Violet 19 in water base ink application.

Hue: Here DC is 5.49 against Pigment Red 122 and it shows that its brighter than Pigment Red 122. And DC is -14.22 against Pigment Violet 19 shows that its duller against Pigment Violet 19.

Color Strength:

Color Strength is always defined by value from tint tone. We found that product is greener and yellower compared to Pigment Red 122 and Pigment Violet 19 and due to very light shade it seems that less strength or weaker against Pigment Red 122 and Pigment Violet 19. New Pigment is showing 66.91 % against pigment Red 122 and 72.45% against Pigment Violet 19.
We also observed that new pigment behave similarly in the presence Of white pigments in tint tone against Pigment Red 122. And in case of Pigment Violet 19 it shoes some difference compare to mass tone. In mass tone its look slight bluer but in tint tone it is much yellower (DB 20.84). This is happen due to much color difference. From the value of DA, DB shows that it is greener and yellower against both pigments.

Gloss:

We observed that gloss of new pigment is slightly inferior than pigment Red 122 and Pigment Violet 19 in water base ink application.

NC base (Nitro Cellulose) Application :

Color Tone :

Color tone is defined from the value of mass tone. And we found that DA value is -6.83 seems that its greener against color tone of Pigment Red 122. And DB value is 26.08 seems that is much yellower against color tone of Pigment Red 122. DE value shows total color difference and its value is 28.39. So from that we conclude that New Pigment is greener and much yellower compared to Color tone of Pigment Red 122 in nitro cellulose (NC) base ink Application.
Color tone is defined from the value of mass tone. And we found that DA value is -10.15 seems that its greener against color tone of Pigment Violet 19. And DB value is 13.68 seems that is yellower against color tone of Pigment Violet 19. DE value shows total color difference and its value is 17.91. So from that we conclude that New Pigment is greener and yellower compared to Color tone of Pigment Violet 19 in NC base ink Application.
Overall conclusion that New Pigment is greener and yellower compared to commercial available Pigment Red 122 and Violet 19 in NC base ink application.

Hue: Here DC is 7.42 against Pigment Red 122 and it shows that its brighter than Pigment Red 122. And DC is -0.36 against Pigment Violet 19 shows that its duller against Pigment Violet 19.

Color Strength:

Color Strength is always defined by value from tint tone. We found that product is greener and yellower compared to Pigment Red 122 and Pigment Violet 19 and due to very light shade it seems that less strength or weaker against Pigment Red 122 and Pigment Violet 19. New Pigment is showing 65.30 % against pigment Red 122 and 67.08 % against Pigment Violet 19.

We also observed that new pigment behave similarly in the presence Of white pigments in tint tone. From the value of DA, DB shows that it is greener and yellower against both pigments.

Gloss:

We observed that gloss of new pigment is slightly inferior than pigment Red 122 and Pigment Violet 19 in NC base ink application.

Viscosity:

Viscosity is very important factor in solvent base application. And NC base ink is called as solvent base ink. Viscosity is measured by Zhan cup. We found that viscosity of new pigment is better than viscosity of Pigment Red 122. And Its very similar against viscosity of Pigment Violet 19.

Textile Application:

Color tone:

In textile application generally tone is defined from tint tone. We found that DA is -15.25 seems that its is greener against color tone of Pigment Red 122. And DB value is 45.10 seems that is very much yellower against color tone of Pigment Red 122. DE value shows total color difference and its value is 48.95. So from that we conclude that New Pigment is greener and very much yellower compared to Color tone of Pigment Red 122 in Textile Application.

We found that DA value is -16.60 seems that its greener against color tone of Pigment Violet 19. And DB value is 33.31 seems that is very much yellower against color tone of Pigment Violet 19. DE value shows total color difference and its value is 38.28. So from that we conclude that New Pigment is greener and very much yellower compared to Color tone of Pigment Violet 19 in Textile Application.

Overall conclusion that New Pigment is greener and very much yellower compared to commercial available Pigment Red 122 and Violet 19 in Textile application.

Hue: Here DC is -3.69 against Pigment Red 122 and it shows that its duller than Pigment Red 122. And DC is -3.16 against Pigment Violet 19 shows that its duller against Pigment Violet 19.

Color Strength:

Color Strength is always defined by value from tint tone. We found that product is greener and much yellower compared to Pigment Red 122 and Pigment Violet 19 and due to very light shade it seems that less strength or weaker against Pigment Red 122 and Pigment Violet 19. New Pigment is showing 77.85 % against pigment Red 122 and 74.77 % against Pigment Violet 19.

We also observed that new pigment behave similarly in the presence Of white pigments in tint tone. From the value of DA, DB shows that it is greener and much yellower against both pigments.

Gloss is not Defined in textile application

Plastic Application: (LDPE)

Color tone:

In plastic application we take LDPE as component because it test at higher temperature. In Plastic application generally tone is defined from tint tone. We found that DA is -13.73 seems that its is much greener against color tone of Pigment Red 122. And DB value is 36.25 seems that is very much yellower against color tone of Pigment Red 122. DE value shows total color difference and its value is 39.20. So from that we conclude that New Pigment is greener and very much yellower compared to Color tone of Pigment Red 122 in Plastic Application.

We found that DA value is -15.70 seems that its much greener against color tone of Pigment Violet 19. And DB value is 24.13 seems that is very much yellower against color tone of Pigment Violet 19. DE value shows total color difference and its value is 29.15. So from that we conclude that New Pigment is greener and very much yellower compared to Color tone of Pigment Violet 19 in Textile Application.

Overall conclusion that New Pigment is greener and very much yellower compared to commercial available Pigment Red 122 and Violet 19 in Textile application.

Hue: Here DC is -13.39 against Pigment Red 122 and it shows that its duller than Pigment Red 122. And DC is -12.17 against Pigment Violet 19 shows that its duller against Pigment Violet 19.

Color Strength:

Color Strength is always defined by value from tint tone. We found that product is greener and much yellower compared to Pigment Red 122 and Pigment Violet 19 and due to very light shade it seems that less strength or weaker against Pigment Red 122 and Pigment Violet 19. And generally for testing in Plastic application product required to be very fine in particle size. And our new pigment is research product hence it not in very fine particle compared to commercial well known product Pigment Red 122 and Pigment Violet 19. New Pigment is showing 45.08 % against pigment Red 122 and 41.98 % against Pigment Violet 19.

We also observed that new pigment behave similarly in the presence Of white pigments in tint tone. From the value of DA, DB shows that it is greener and much yellower against both pigments.

Gloss is not Defined in Plastic application.

Table:1

Sr.No	Application	Tested Against	Strength	Mass Tone				Tint Tone			
				DA	DB	DE	DC	DA	DB	DE	DC
1	Alkyd Paint	Pigment Red 122	63.45%	-5.45	20.93	22.76	5.78	-10.39	36.87	39.11	-5.97
		Pigment Violet 19	64.87%	-12.06	11.37	16.89	-3.74	-13.23	25.38	29.20	-6.51
2	Waterbase Paint	Pigment Red 122	66.91%	-2.01	4.94	0.64	5.49	-4.21	30.51	31.37	-5.10
		Pigment Violet 19	72.52%	-14.15	-4.04	14.97	-14.22	-7.66	20.84	22.62	-5.63
3	NC Base Ink	Pigment Red 122	65.30%	-6.83	26.08	28.39	7.42	-6.51	30.10	31.32	-8.06
		Pigment Violet 19	67.08%	-10.50	13.68	17.91	-0.36	-6.90	20.64	22.21	-5.51
4	Textile	Pigment Red 122	77.85%	-----	-----	-----	----	-15.25	45.16	48.95	-3.69
		Pigment Violet 19	74.77%	-----	-----	-----	----	-16.60	33.31	38.28	-3.16
5	Plastic	Pigment Red 122	45.08%	-----	-----	-----	----	-13.73	36.25	39.20	-13.39
		Pigment Violet 19	41.98%	-----	-----	-----	----	-15.76	24.13	29.15	-12.17

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