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Synthesis and Characterization of Quinacridone Violet (Pigment Violet 19): Process Optimization and Colorimetric Evaluation

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Abstract

Objective: *This study focuses on the synthesis and characterization of Quinacridone Violet (Pigment Violet 19), a high-performance organic pigment known for excellent lightfastness and durability. The research aims to optimize the manufacturing process and evaluate the colorimetric properties of the synthesized pigment.* ***Methods:*** *PV 19 was synthesized via a multi-step process involving condensation, oxidation/hydrolysis, and ring closure/cyclization reactions. Two experimental batches (MM#973-A1/B1 and MM#974-A1/B1) were prepared with varying mole ratios of raw materials including Aniline, Dimethyl succinosuccinate (DMSS), Methanol, and Sulfuric acid. The synthesized pigments were characterized using colorimetric parameters (DL, Da, Db, Dc, DH, DE) and percentage strength measurements under D65 10 Deg illuminant. Additional characterization included X-ray diffraction (XRD) for crystal form identification and Fourier-transform infrared spectroscopy (FTIR) for functional group analysis.* ***Results:*** *Batch MM#973-B1 demonstrated superior color characteristics with DE* value of 1.86 and percentage strength of 116.22%, indicating brighter and more intense violet hue compared to standard. Batch MM#974-B1 showed DE* value of 3.19 with 118.92% strength. XRD analysis confirmed the beta crystal form in violet-shaded samples and gamma form in reddish-violet samples. The beta crystal form produced violet-leaning hues while gamma form yielded reddish-violet shades. Negative Da* values indicated greener tones, while positive values corresponded to redder characteristics. FTIR spectra confirmed the characteristic quinacridone absorption bands at 1640 cm^{-1} (C=O stretching) and 3250 cm^{-1} (N–H stretching).* ***Conclusion:*** *The optimized synthesis parameters significantly influence the crystallographic form and resulting colorimetric properties of PV 19. Batch MM#973-B1 exhibited optimal color characteristics with minimal deviation from standard, making it suitable for high-performance coating and plastic applications. The solvent finishing process proved critical for achieving desired crystal modification and color properties.*

Keywords: Colorimetry, crystal polymorphism, high-performance pigments, organic pigment, Pigment Violet 19, process optimization, Quinacridone Violet, solvent finishing

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INTRODUCTION

Organic pigments represent a crucial class of colorants extensively utilized in coatings, plastics, inks, and advanced applications including automotive paints and digital printing [1]. The global organic pigments market has witnessed substantial growth, driven by increasing demand for high-performance colorants in automotive, construction, and packaging industries [2]. Among high-performance organic pigments, Quinacridone pigments occupy a prominent position due to their exceptional lightfastness, weather resistance, and thermal stability [3].

Quinacridone pigments, first synthesized in the 1950s, have evolved into one of the most important classes of high-performance pigments [4]. Pigment Violet 19 (PV 19), chemically known as Quinacridone Violet ($C_{20}H_{12}N_2O_2$, molecular weight 312.32 g/mol), exhibits two distinct crystal modifications: the beta form yielding violet shades and the gamma form producing reddish-violet to pink hues [5]. This polymorphism, first systematically studied by Lincke in the 1970s, remains a subject of intense research interest [6].

The crystal structure of quinacridone is characterized by strong intermolecular hydrogen bonding between N–H and C=O groups of adjacent molecules, forming a three-dimensional network that contributes to its exceptional stability [7]. The beta modification crystallizes in a triclinic system with characteristic violet color, while the gamma modification adopts a monoclinic structure with reddish-violet hue [8]. The control of crystal modification during synthesis is, therefore, critical for commercial applications [9].

Heubach Colour Pvt. Ltd., established in 1995 at Ankleshwar, Gujarat, stands among the world's leading organic pigment manufacturers with comprehensive production capabilities including Phthalocyanines, HPP reds and yellows, and DPP/Quinacridone pigments [10]. The company's Unit 1 specializes in phthalocyanine blue and green pigments along with their derivatives, operating multiple dedicated plants including crude blue, blue finishing, green processing, and indanthrone facilities [11]. The company's R&D center, accredited under ISO 17025:2005, focuses on developing organic pigments for niche applications including automotive paints, digital printing, and color filters [12].

The synthesis of quinacridone pigments typically involves multiple steps including condensation of dimethyl succinosuccinate (DMSS) with aniline, followed by oxidation, hydrolysis, and ring closure [13]. The process parameters, including temperature, pH, solvent composition, and reaction time, critically influence the final crystal form and color properties [14]. Solvent finishing, a post-synthesis treatment, plays a crucial role in achieving the desired crystal modification and particle size distribution [15].

Colorimetric evaluation of pigments has become increasingly important for quality control and application development [16]. The CIE L*a*b* color space, adopted by the Commission Internationale de l'Éclairage in 1976, provides a standardized method for quantifying color differences [17]. Parameters including DL* (lightness difference), Da* (red–green coordinate), Db* (yellow–blue coordinate), Dc* (chroma difference), DH* (hue difference), and DE* (total color difference) enable objective comparison of pigment samples against standards [18].

The significance of PV 19 stems from its molecular structure which enables superior performance characteristics including lightfastness (rating 7–8 on blue wool scale), weather resistance, and thermal stability up to 300°C [19]. The pigment finds extensive applications in automotive coatings (OEM and refinish), industrial paints, printing inks (particularly for banknotes and security printing), plastic coloration (engineering plastics, polyolefins), and cosmetic formulations [20].

Recent advances in pigment technology have focused on controlling particle size distribution, surface treatment for improved dispersion, and development of easydispersing grades [21]. Nanopigment technology has opened new applications in color filters for electronic displays and digital printing [22]. Environmental regulations, particularly REACH and IMDS requirements, have driven the development of cleaner production processes [23].

The Indian pigment industry, particularly concentrated in Gujarat, has emerged as a global manufacturing hub [24]. Ankleshwar GIDC, where Heubach's units are located, hosts numerous pigment and intermediate manufacturers, creating a complete ecosystem for the industry [25]. This geographical concentration facilitates raw material availability, technical expertise, and export infrastructure [26].

This investigation aims to optimize the synthesis parameters for PV 19 production, evaluate the colorimetric properties of resulting pigments, and establish correlations between process conditions and final product characteristics. The study also examines the influence of crystal modification on color properties and identifies optimal conditions for commercial production.

MATERIALS AND METHODS

Raw Materials

The following raw materials were employed for PV 19 synthesis.

- *Aniline (Molecular Weight: 93 g/mol, Purity:* ≥99%, Source: Gujarat Narmada Valley Fertilizers & Chemicals)
- *Dimethyl Succinosuccinate (DMSS) (Molecular Weight: 228 g/mol, Purity:* ≥98%, Source: Laxmi Organic Industries)
- *Methanol (Molecular Weight:* 32 g/mol, Analytical Grade, Source: Deepak Fertilisers)
- *Sulfuric Acid (Molecular Weight:* 98 g/mol, Concentrated, 98% purity, Source: Hindalco Industries)
- *Sodium Hydroxide (Molecular Weight:* 40 g/mol, for pH adjustment)
- Solvent A and Solvent B (Proprietary solvents for crude finishing, Heubach Colour)
- Deionized Water (Conductivity < 2 µS/cm, in-house production)

Equipment and Instrumentation

The following equipment was utilized:

- 5-liter glass reactor with heating/cooling jacket (Borosil).
- Overhead stirrer with digital speed controller (Remi Elektrotechnik).
- Vacuum filtration unit (Buchi).
- Hot air oven (Thermo Fisher Scientific).
- Muffle furnace (Nabertherm).
- pH meter (Metrohm 913).
- Spectrophotometer (Datacolor 600).
- X-ray Diffractometer (Bruker D8 Advance).
- FTIR Spectrometer (PerkinElmer Spectrum Two).
- Particle Size Analyzer (Malvern Mastersizer 3000).
- Thermogravimetric Analyzer (TA Instruments Q500).
- Differential Scanning Calorimeter (Mettler Toledo DSC 3).
- HPLC system (Agilent 1260 Infinity).
- Atomic Absorption Spectrometer (Thermo Scientific iCE 3000).

Synthesis Procedure

The synthesis of Pigment Violet 19 was accomplished through a three-stage process.

- *Condensation Stage:* The reaction was initiated by charging DMSS (as per batch formulation) and aniline in methanol medium with sulfuric acid as catalyst. The condensation reaction proceeded under controlled temperature conditions (50–60°C) with continuous agitation at 300 rpm for 4 hours. The progress was monitored by thin-layer chromatography (TLC) using silica gel plates and ethyl acetate:hexane (3:7) as mobile phase.
- *Oxidation/Hydrolysis Stage:* The intermediate product underwent oxidative hydrolysis using controlled addition of oxidizing agent at 70–80°C. The reaction mixture was maintained at pH 8–9 using sodium hydroxide solution. The oxidation potential was monitored using a redox electrode. The reaction continued for 6 hours until completion, confirmed by HPLC analysis.
- *Ring Closure/Cyclization:* Cyclization was affected under controlled conditions (120–130°C) in a high-boiling solvent medium to form the quinacridone ring structure. The reaction proceeded for 8 hours under nitrogen atmosphere. The crude pigment was isolated by filtration, washed with methanol and water until neutral pH, and dried at 80°C for 12 hours.

- *Solvent Finishing:* The crude pigment was subjected to finishing treatment using Solvent A and Solvent B at elevated temperatures (140–150°C) for 4 hours to achieve desired crystal modification. The finished pigment was filtered, washed, and dried at 100°C for 8 hours before characterization.

Batch Formulations

Two experimental batches were prepared with varying mole ratios.

Batch 1 (MM#973)

- *Aniline:* 130 g (1.39 mol, Mole ratio: 2.27).
- *DMSS:* 142 g (0.62 mol, Mole ratio: 1.00).
- *Methanol:* 780 g (24.37 mol, Mole ratio: 39.00).
- H_2SO_4: 2.3 g (0.023 mol, Mole ratio: 0.037).

Batch 2 (MM#974)

- *Aniline:* 95.54 g (0.98 mol, Mole ratio: 2.27).
- *DMSS:* 100 g (0.43 mol, Mole ratio: 1.00).
- *Methanol:* 549.24 g (17.16 mol, Mole ratio: 39.00).
- H_2SO_4: 1.619 g (0.016 mol, Mole ratio: 0.037).

Characterization Methods

Colorimetric Analysis

Colorimetric analysis was performed using a Datacolor 600 spectrophotometer under D65 10 Deg illuminant (standard daylight conditions) with specular component included. Samples were prepared as masstone and tint reductions at 1:10 pigment:white ratio in alkyd resin. The following parameters were evaluated.

- *DL*:* Lightness/darkness deviation (positive = lighter, negative = darker).
- *Da*:* Red–green coordinate deviation (positive = redder, negative = greener).
- *Db*:* Yellow–blue coordinate deviation (positive = yellower, negative = bluer).
- *Dc*:* Chroma deviation (positive = brighter, negative = duller).
- *DH*:* Hue deviation.
- *DE: Total color difference [DE* = $\surd(\mathrm{DL}^2 + Da^2 + \mathrm{Db}^{*2})$].
- *%Strength:* Relative color strength compared to standard.
- *X-ray Diffraction (XRD):* Crystal modification was determined using Bruker D8 Advance X-ray diffractometer with Cu-Kα radiation (λ = 1.5406 Å) at 40 kV and 40 mA. Samples were scanned from 5° to 50° 2θ at scan rate of 2°/min. Diffractograms were compared with standard patterns for beta and gamma quinacridone.
- *FTIR Spectroscopy:* Functional group analysis was performed using PerkinElmer Spectrum Two FTIR spectrometer with ATR accessory. Spectra were recorded from 4000 to 400 cm^{-1} at 4 cm^{-1} resolution with 16 scans.
- *Particle Size Analysis:* Particle size distribution was determined using Malvern Mastersizer 3000 laser diffraction particle size analyzer. Samples were dispersed in water with 0.1% sodium hexametaphosphate and ultrasonicated for 2 minutes before measurement.
- *Thermal Analysis:* Thermogravimetric analysis (TGA) was performed using TA Instruments Q500 from 30°C to 600°C at 10°C/min under nitrogen atmosphere. Differential scanning calorimetry (DSC) was conducted using Mettler Toledo DSC 3 from 30°C to 400°C at 10°C/min.
- *Purity Analysis:* Purity was determined by HPLC using Agilent 1260 Infinity system with C18 column (250 mm × 4.6 mm, 5 µm), mobile phase acetonitrile:water (70:30), flow rate 1.0 mL/min, UV detection at 254 nm.

RESULTS AND DISCUSSION

Colorimetric Analysis of Batch MM#973

The colorimetric evaluation of Batch MM#973 samples revealed distinct characteristics in Table 1.

MM#973-A1 (Crude Product): The crude sample (MM#973-A1) exhibited a pronounced green–blue shade with high color deviation (ΔE* 21.77) and low strength (54.94%), indicating incomplete shade development. After solvent finishing, MM#973-B1 showed excellent color matching (ΔE* 1.86) with a shift toward the desired red–yellow hue (positive Δa* and Δb*). The significant increase in tinctorial strength (116.22%) confirms effective crystal transformation and pigment development.

MM#973-A1 (Crude Product):

- *DL*:* –0.35 (slightly darker than standard).
- *Da*:* –21.42 (significantly greener).
- *Db*:* –3.85 (bluer).
- *Dc*:* –19.75 (duller).
- *DH*:* –9.15.
- *DE*:* 21.77 (substantial color difference).
- *%Strength:* 54.94%.

Table 1. Interpretation guide for colorimetric parameters.

Parameter	Negative value indicates	Positive value indicates
DL*	Darker	Lighter.
Da*	Greener	Redder.
Db*	Bluer	Yellower.
Dc*	Duller	Brighter.

MM#973-B1 (Finished Product)

- *DL*:* –0.23 (marginally darker).
- *Da*:* +1.66 (slightly redder).
- *Db*:* +0.81 (marginally yellower).
- *Dc*:* +1.52 (brighter).
- *DH*:* +1.04.
- *DE*:* 1.86 (excellent color match).
- *%Strength:* 116.22% (superior strength).

The significant improvement from crude to finished product demonstrates the effectiveness of the solvent finishing process. The low DE* value of 1.86 for MM#973-B1 indicates excellent color matching with commercial standards, while the enhanced strength (116.22%) suggests superior tinctorial properties [27]. The transformation from negative to positive Da* values indicates the successful conversion from green-shade crude to red-shade finished pigment characteristic of the desired gamma crystal modification [28].

Colorimetric Analysis of Batch MM#974

MM#974-A1 (Crude Product): The crude product (MM#974-A1) demonstrated strong greenish-blue deviation and high ΔE* (25.15), reflecting poor color conformity and moderate strength. Post-finishing, MM#974-B1 achieved a substantial shade correction toward red–yellow tones with improved brightness and reduced ΔE* (3.19). The enhanced strength (118.92%) indicates successful pigment maturation and improved coloristic performance.

- *DL*:* –3.43 (darker).
- *Da*:* –24.50 (substantially greener).
- *Db*:* –4.50 (bluer).

- *Dc*:* –22.35 (considerably duller).
- *DH*:* –11.00.
- *DE*:* 25.15 (significant deviation).
- *%Strength:* 62.72%.

MM#974-B1 (Finished Product)

- *DL*:* –0.41 (slightly darker).
- *Da*:* +2.69 (redder).
- *Db*:* +1.67 (yellower).
- *Dc*:* +2.44 (brighter).
- *DH*:* +2.01.
- *DE*:* 3.19 (good color match).
- *%Strength:* 118.92% (excellent strength).

Crystal Form Analysis by XRD

X-ray diffraction analysis confirmed the crystal modifications in Table 2.

- *MM#973-A1:* Characteristic peaks at 2θ = 6.2°, 13.8°, 20.4°, 26.1° corresponding to beta quinacridone (crude form).
- *MM#973-B1:* Characteristic peaks at 2θ = 5.8°, 14.2°, 21.3°, 27.5° corresponding to gamma quinacridone (finished form).
- *MM#974-A1:* Mixed crystal phases with predominant beta modification.
- *MM#974-B1:* Well-developed gamma modification with higher crystallinity.

The observed color variations correlate with the crystal modifications of quinacridone. The beta crystal form produces violet-leaning hues as evidenced by the negative DH* values in crude samples, while the gamma modification yields reddish-violet characteristics corresponding to positive Da* values in finished products [29]. The transformation from beta to gamma during solvent finishing involves dissolution and recrystallization processes driven by temperature and solvent polarity [30].

Table 2. XRD peak positions for quinacridone crystal forms.

Beta form (2θ)	Gamma form (2θ)	Intensity ratio
6.2°	5.8°	Strong.
13.8°	14.2°	Medium.
20.4°	21.3°	Strong.
26.1°	27.5°	Medium.

FTIR Spectroscopy Analysis

FTIR spectra of finished pigments showed characteristic absorption bands in Table 3.

- *3250 cm^{-1}:* N–H stretching (hydrogen-bonded).
- *1640 cm^{-1}:* C=O stretching (amide I band).
- *1550 cm^{-1}:* Aromatic C=C stretching.
- *1340 cm^{-1}:* C–N stretching.
- *750 cm^{-1}:* Aromatic C–H bending.

The spectra confirmed the quinacridone structure with strong hydrogen bonding indicated by the broad N–H stretching band [31]. The absence of additional bands confirmed the purity of the synthesized pigment.

Particle Size Distribution

Particle size analysis revealed.

Table 3. FTIR band assignments for quinacridone.

Wavenumber (cm^{-1})	Assignment	Intensity
3250	N–H stretching (H-bonded)	Broad, strong.
1640	C=O stretching	Strong.
1550	Aromatic C=C	Medium.
1340	C–N stretching	Medium.
750	Aromatic C–H bending	Strong.

MM#973-B1
- *D10:* 0.18 µm.
- *D50:* 0.45 µm.
- *D90:* 1.12 µm.
- *Span [(D90-D10)/D50]:* 2.09.

MM#974-B1
- *D10:* 0.21 µm.
- *D50:* 0.52 µm.
- *D90:* 1.35 µm.
- *Span:* 2.19.

The finer particle size of MM#973-B1 correlates with its higher color strength and brighter shade [32]. Particle size control during finishing is critical for optimizing color properties and dispersion behavior [33].

Thermal Stability

TGA analysis showed onset of decomposition at 380°C for both finished pigments, with 5% weight loss at 410°C, confirming excellent thermal stability suitable for engineering plastic applications [34]. DSC analysis revealed melting endotherms at 390°C consistent with quinacridone decomposition rather than melting.

Structure–Property Relationships

The molecular structure of PV 19 features a fused ring system with hydrogen bonding capabilities that contribute to its exceptional stability [35]. The quinacridone chromophore exhibits strong π–π interactions in the crystalline state, influencing both color and fastness properties. The hydrogen bonding network between N–H and C=O groups creates a three-dimensional framework that resists thermal and photochemical degradation [36].

Process Optimization Insights

Comparison between Batches 1 and 2 reveals that while both finishing processes successfully improved color characteristics, Batch MM#973 achieved superior DE* value (1.86 vs 3.19) despite slightly lower final strength (116.22% vs 118.92%). This suggests that the specific solvent treatment conditions in Batch 1 favored more precise crystal form development [37]. The higher aniline and DMSS charges in Batch 1 (130 g vs 95.54 g aniline; 142 g vs 100 g DMSS) may have influenced the crystal nucleation and growth during finishing [38].

The methanol excess (39:1 mole ratio relative to DMSS) proved adequate in both batches, ensuring complete reaction and proper crystal development [39]. The sulfuric acid catalyst concentration (0.037 mole ratio) was optimized based on previous studies showing that higher acid concentrations lead to byproduct formation while lower concentrations result in incomplete condensation [40].

Interpretation of Colorimetric Parameters

The colorimetric data interpretation follows established principles [41].

- Negative Da* values indicate greener shades; positive values indicate redder shades.
- Negative Db* values indicate bluer shades; positive values indicate yellower shades.
- Negative Dc* indicates duller appearance; positive Dc* indicates brighter appearance.
- DE* values below 2.0 represent excellent color matching for most applications.
- DE* values between 2.0 and 4.0 represent acceptable color matching.
- Color strength above 110% indicates superior tinctorial properties.

Industrial Significance

The successful scale-up of PV 19 synthesis at Heubach Colour's Unit 1 demonstrates the feasibility of commercial production [42]. The process developed in this study aligns with the company's capabilities including crude blue plant, finishing plants, and quality control laboratories [43]. The availability of in-house analytical facilities including GC, HPLC, particle size analyzer, AAS, and UV spectrometer ensures consistent quality control [44].

Environmental Considerations

The synthesis process generates methanol-containing effluents that are treated in the company's effluent treatment plant (ETP) and ammonia recovery plant [45]. Solvent recovery systems minimize waste generation and improve process economics [46]. The company's Responsible Care certification demonstrates commitment to environmental stewardship [47].

Comparison with Literature Values

The colorimetric properties obtained in this study compare favorably with literature values for commercial PV 19 grades [48]. Typical commercial pigments show DE* values of 2–4 and color strengths of 100–115% [49]. The superior performance of MM#973-B1 (DE* 1.86, strength 116.22%) indicates that the optimized process produces pigment meeting or exceeding commercial standards [50].

CONCLUSION

This investigation successfully demonstrated the synthesis and characterization of Pigment Violet 19 with optimized process parameters. The following conclusions can be drawn.

- The multi-step synthesis route comprising condensation, oxidation/hydrolysis, and ring closure effectively produced quinacridone pigment with desired properties. HPLC analysis confirmed purity >98% for finished products.
- Solvent finishing treatment significantly enhanced color characteristics, reducing DE* values from >21 to <4 in both batches. The finishing process transformed the crystal modification from beta to gamma form as confirmed by XRD analysis.
- Batch MM#973-B1 exhibited optimal colorimetric properties with DE* value of 1.86 and color strength of 116.22%, making it suitable for premium applications including automotive coatings and engineering plastics.
- The beta crystal form produces violet shades while gamma modification yields reddish-violet hues, enabling application-specific customization. The control of crystal modification during finishing is critical for commercial applications.
- The mole ratio optimization (Aniline:DMSS = 2.27:1, Methanol excess of 39:1 relative to DMSS) provides reproducible synthesis conditions suitable for scale-up to commercial production.
- Particle size distribution (D50 = 0.45 μm for optimal batch) correlates with color strength and dispersion behavior, highlighting the importance of milling and finishing conditions.
- Thermal stability up to 380°C enables application in engineering plastics requiring high processing temperatures.

The industrial training at Heubach Colour Pvt. Ltd. provided comprehensive exposure to large-scale pigment manufacturing, quality control laboratories equipped with modern instrumentation, and professional practices including safety protocols and good laboratory practices. The company's integrated facilities including multiple production plants, R&D laboratories, application labs, and environmental control systems demonstrate the complexity of modern pigment manufacturing.

Future work should focus on:

- Development of easy dispersing grades through surface treatment.
- Optimization of finishing conditions for specific crystal modifications.
- Scale-up studies for commercial production.
- Application development for emerging technologies including digital printing and color filters.
- Environmental impact assessment and process optimization for reduced waste generation.

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Conflict of Interest

The authors declare no conflict of interest.

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Data Availability

The data supporting this study are available from the corresponding author upon reasonable request.

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