

Comparative Spectrophotometric Analysis and Physicochemical Characterization of Indigo and Sulphur Dyes at Different Concentrations

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Abstract

Dyes are extensively used in textile and other industrial processes, and their accurate quantification is important for quality control, process optimization, and environmental monitoring. The present study investigated the applicability of UV-Visible spectrophotometry for the quantitative estimation of two commercially important vat dyes, indigo and sulphur dye, together with selected physicochemical parameters. Standard dye solutions were analyzed using a UV-Visible spectrophotometer, and calibration relationships between dye concentration and absorbance were established according to the Beer-Lambert principle. Indigo was investigated over a concentration range of 0.5–10.0 g L⁻¹, whereas sulphur dye was studied over 1.0–12.0 g L⁻¹ for calibration. Unknown samples corresponding approximately to 5–25 g L⁻¹ were subsequently estimated from the calibration slopes. The calculated concentrations for indigo ranged from 5.07 to 24.85 g L⁻¹, while those for sulphur dye ranged from 4.91 to 24.90 g L⁻¹. The dye solutions exhibited strongly alkaline pH values, ranging from 11.78–11.88 for indigo and 12.86–12.95 for sulphur dye. Negative ORP values were observed for both dye systems, consistent with reducing chemical conditions associated with vat-dye processing. Conductivity and total dissolved solids increased with increasing dye concentration. The results demonstrate that UV-Visible spectrophotometry, complemented by pH, ORP, conductivity, and TDS measurements, provides a simple and rapid analytical approach for the routine characterization of vat-dye solutions. The method has potential applications in textile process control and preliminary monitoring of dye-containing aqueous systems.

Keywords: Indigo dye; sulphur dye; vat dyes; UV-Visible spectrophotometry; Beer-Lambert law; calibration curve; oxidation-reduction potential; conductivity; TDS; textile dyes

Introduction

Dyes constitute an important class of chemical compounds that are extensively used in a wide range of industrial applications, including textiles, leather, paper, plastics, food, cosmetics, and related manufacturing processes. Their ability to produce intense and relatively persistent coloration makes them indispensable in industrial production. However, the chemical stability and resistance to degradation that are desirable for achieving good colour fastness can also make dye-containing effluents difficult to treat and analytically characterize. The discharge of coloured wastewater from textile-processing industries is therefore an important environmental concern because residual dyes and associated chemicals can affect water quality, aquatic ecosystems, and other environmental receptors [1–3].

Textile dyes can be classified according to their chemical structures and methods of application into several major groups, including reactive, vat, sulphur, direct, acid, basic, disperse, and other dye classes [4]. Among these, vat dyes are particularly important because of their excellent fastness properties, especially resistance to washing and light. Indigo is one of the most historically significant vat dyes and remains extensively associated with denim manufacture. In their oxidized state, vat dyes are generally insoluble in water and therefore cannot be applied directly to textile fibres. The dye must first undergo reduction under alkaline conditions to produce a water-soluble leuco form. The reduced dye can then penetrate and associate with the textile fibre, after which oxidation regenerates the original insoluble coloured form within or on the fibre [5,6]. This reduction-oxidation chemistry is a fundamental feature of vat-dye application.

Sulphur dyes constitute another important class of textile dye, particularly because of their relatively low cost and their suitability for producing deep shades such as black, blue, green, and khaki. Similar to vat dyes, sulphur dyes are applied through reduction and solubilization followed by oxidation after the dye has been transferred to the fibre. Sodium sulphide and other reducing systems have traditionally been used during sulphur-dye application. However, these processes may generate sulphide-containing effluents and other chemical residues, thereby contributing to the environmental burden associated with textile wastewater [5,7]. The reduction potential and extent of reduction are important process variables because insufficient or excessive reduction can influence dye uptake, colour yield, and final shade development [5].

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Reactive dyes represent another major category of textile colorant and are widely used for dyeing cellulosic fibres because of their broad colour range and good wash fastness. Unlike vat and sulphur dyes, reactive dyes are designed to form covalent bonds with suitable functional groups on textile fibres. In the case of cellulose, reactive dye molecules can react with hydroxyl groups under alkaline conditions, producing covalent dye–fibre linkages [8,9]. However, competing hydrolysis reactions can convert reactive dye molecules into non-reactive forms. Consequently, not all of the applied dye is fixed to the fibre, and unfixed or hydrolysed dye may remain in the dye bath and ultimately contribute to coloured wastewater [8]. Thus, the chemical characteristics of individual dye classes have a direct influence on both dyeing performance and wastewater composition.

The presence of dyes in industrial wastewater has attracted considerable attention because even relatively small concentrations of strongly coloured compounds can impart visible colour to water. In addition to colour, textile wastewater may contain dissolved and suspended solids, salts, auxiliaries, organic matter, and other processing chemicals. These constituents can contribute to variations in parameters such as chemical oxygen demand, total dissolved solids, conductivity, and pH [2,3]. Consequently, reliable analytical determination of dyes and associated physicochemical parameters is important for process optimization, quality control, wastewater characterization, and environmental monitoring.

Several analytical techniques are available for the qualitative and quantitative determination of dyes, including high-performance liquid chromatography, mass spectrometry, electrochemical methods, and spectroscopic techniques. Among these approaches, ultraviolet–visible (UV–Visible) spectrophotometry is particularly attractive for routine dye analysis because of its relative simplicity, rapidity, accessibility, and suitability for compounds containing chromophoric groups capable of absorbing radiation in the ultraviolet or visible regions. The colour of dye molecules is associated with selective absorption of electromagnetic radiation by their chromophoric systems, making spectrophotometric measurements useful for quantitative analysis [4,10].

The quantitative basis of UV–Visible spectrophotometry is the Beer–Lambert law, which relates absorbance to the concentration of an absorbing species and the optical path length. Under appropriate experimental conditions, the relationship is expressed as:

$$A = \epsilon lc$$

where A is the absorbance, ϵ is the molar absorption coefficient (molar absorptivity), l is the optical path length, and c is the concentration of the absorbing species [11]. A calibration curve prepared using solutions of known dye concentration can therefore be used to establish the relationship between absorbance and concentration and subsequently to determine the concentration of an unknown sample. The validity of this approach depends on appropriate experimental conditions, including suitable wavelength selection, concentration range, optical path length, and absence of significant spectral or chemical interferences [11].

In addition to spectrophotometric determination of dye concentration, measurements of physicochemical parameters can provide useful information about the chemical environment of dye solutions. Parameters such as pH, oxidation–reduction potential (ORP), electrical conductivity, and total dissolved solids (TDS) are particularly relevant to textile dyeing systems. The pH of a dye bath can influence dye stability, ionization, reduction reactions, and interactions between dye molecules and textile substrates. Conductivity and TDS provide information concerning the concentration of dissolved ionic and non-ionic substances and can therefore reflect changes in the chemical composition of the dyeing medium [2,3].

ORP is of particular significance for vat- and sulphur-dye systems because their application depends on controlled redox conditions. In vat dyeing, reduction of the insoluble oxidized dye to the soluble leuco form is essential for successful dye application, while subsequent oxidation restores the coloured, insoluble dye form [5]. Consequently, monitoring ORP together with pH can provide useful information about the chemical conditions under which reduction and oxidation processes occur. Such measurements may complement direct spectrophotometric determination and provide a more comprehensive description of the dye solution.

Although individual analytical techniques can provide information about dye concentration or solution chemistry, a simple combined approach incorporating UV–Visible spectrophotometry

with pH, ORP, conductivity, and TDS measurements may be particularly useful for routine laboratory and textile-process monitoring. Such an approach can provide both quantitative information about dye concentration and supporting information concerning the physicochemical conditions of the dye system.

Therefore, the present investigation was undertaken to develop a simple UV–Visible spectrophotometric method for the quantitative estimation of selected indigo and sulphur vat dyes and to characterize their corresponding solutions using pH, oxidation–reduction potential (ORP), conductivity, and total dissolved solids (TDS) measurements. The study further compares the observed physicochemical characteristics of the two dye systems and evaluates the applicability of the combined analytical approach for routine monitoring of textile dye solutions and processes.

Materials and Methods

Two commercially important vat-dye systems, indigo and sulphur dye, were selected for investigation. The dyes were used as received without additional purification. Aqueous solutions were prepared using distilled water under laboratory conditions.

Indigo was selected because of its industrial importance and characteristic reduction–oxidation chemistry. Sulphur dye was selected because of its widespread use for dark textile shades and its dependence on reducing conditions during application.

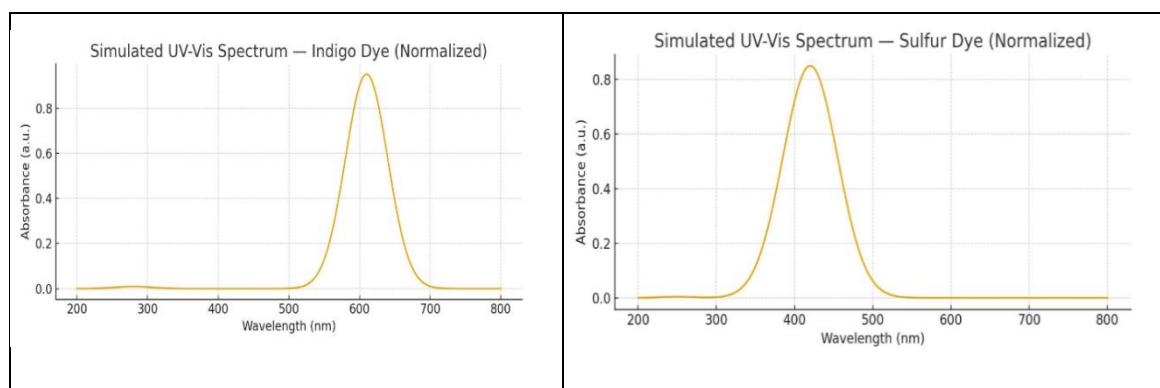
Preparation of Standard Solutions

Stock solutions were prepared by accurately weighing appropriate quantities of the dye and dissolving or dispersing them in distilled water. Working standards were subsequently prepared by serial dilution. For calibration studies, indigo standards covering 0.5–10.0 g L⁻¹ were investigated, while sulphur-dye standards covering 1.0–12.0 g L⁻¹ were used. All solutions were prepared immediately before measurement where appropriate and analysed under comparable laboratory conditions.

UV–Visible Spectrophotometric Analysis

Spectrophotometric measurements were carried out using a Shimadzu UV-1280 UV–Visible spectrophotometer. The absorption spectrum of each dye solution was examined over the UV–Visible region, and the wavelength corresponding to maximum absorbance (λ_{max}) was selected for quantitative measurements. Absorbance measurements were obtained using suitable cuvettes and appropriate blank correction. Calibration curves were constructed by plotting absorbance against dye concentration. The Beer–Lambert relationship was used as the basis for quantitative estimation: $A = \epsilon lc$

The pH of dye solutions was measured using a Thermo Orion Star A111 digital pH meter. The instrument was calibrated according to standard laboratory procedures before measurement. ORP measurements were performed using a Thermo Orion Star A324 ORP meter/electrode system. ORP values were recorded for the prepared dye solutions. Electrical conductivity was measured using a conductivity/TDS meter and expressed in mS cm⁻¹. TDS values were recorded in g L⁻¹. Calibration data were evaluated by examining the relationship between absorbance and concentration. Dye concentrations in test samples were calculated using the experimentally obtained slopes.



Results and Discussion

The experimental results demonstrate several similarities and differences between the two investigated vat-dye systems.

Both dyes exhibited increasing absorbance with increasing concentration, supporting the use of UV-Visible spectrophotometry for quantitative estimation. However, their calibration slopes differed, with indigo showing a calculated slope of 0.1203 and sulphur dye showing a slope of 0.0840 under the conditions employed.

Both systems also exhibited strongly alkaline pH and negative ORP values. The indigo samples showed pH values of 11.78–11.88 and ORP values of –720 to –729 mV, whereas sulphur dye showed pH values of 12.86–12.95 and ORP values of –582 to –599 mV.

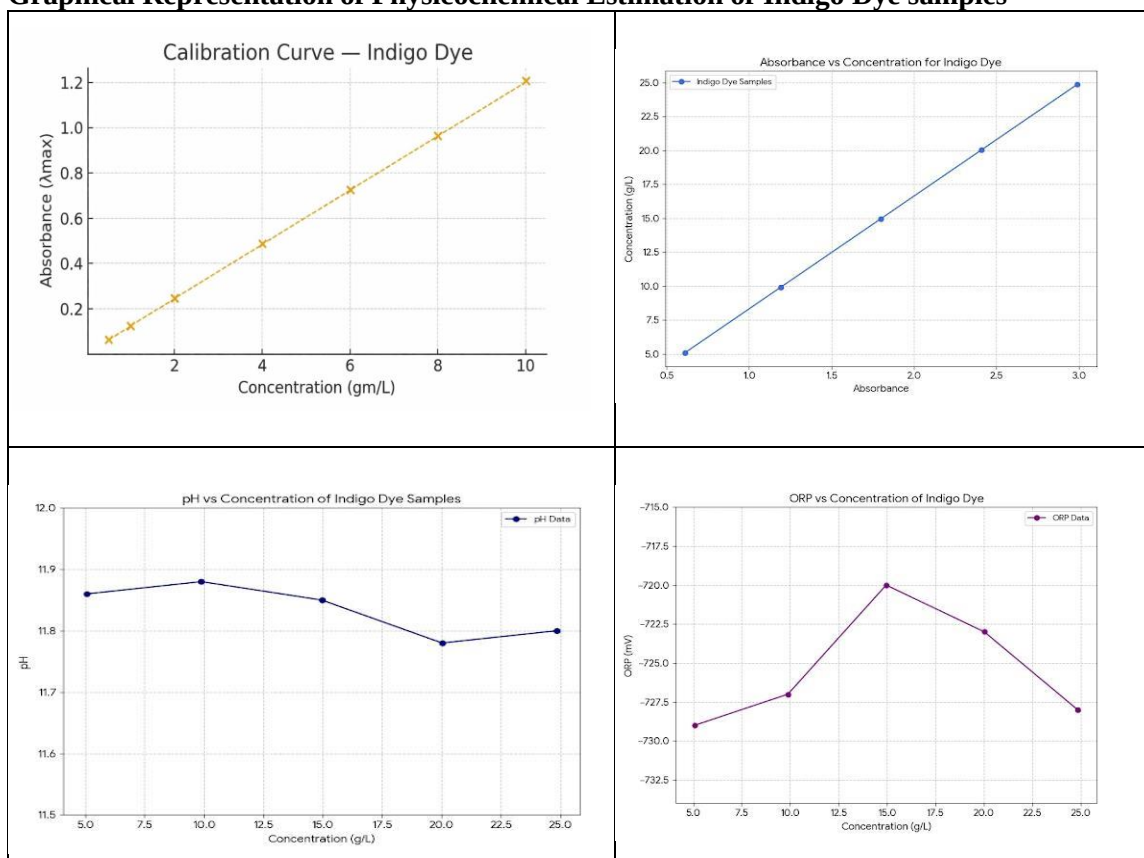
Conductivity increased with concentration for both dyes. Indigo showed conductivity values from 21.3 to 32.0 mS cm⁻¹, while sulphur dye showed values from 15.3 to 23.2 mS cm⁻¹. TDS similarly increased with concentration in both systems.

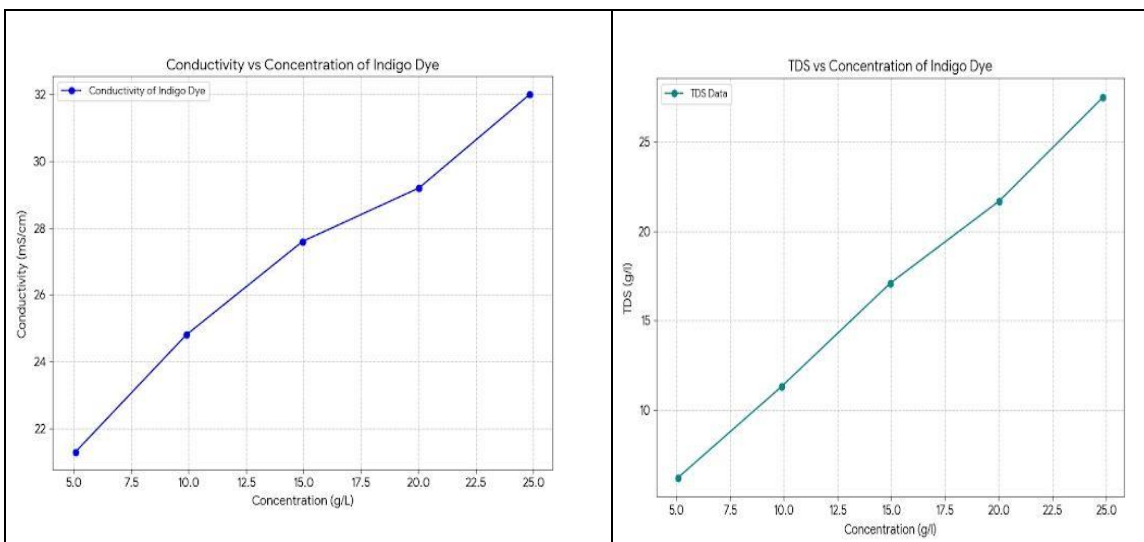
These observations demonstrate that spectrophotometric and physicochemical measurements provide complementary information. Absorbance is directly useful for quantitative estimation when an appropriate calibration model is established, whereas pH, ORP, conductivity, and TDS provide information concerning the chemical environment of the dye solution.

Physicochemical Estimation of Indigo Dye samples

Sr. No.	Concentration (gm/l)	Absorbance	pH	ORP (mV)	Conductance (mS/cm)	TDS (gm/l)
1	5.07	0.61	11.86	-729	21.3	6.2
2	9.89	1.19	11.88	-727	24.8	11.3
3	14.96	1.80	11.85	-720	27.6	17.1
4	20.03	2.41	11.78	-723	29.2	21.7
5	24.85	2.99	11.80	-728	32.0	27.5

Graphical Representation of Physicochemical Estimation of Indigo Dye samples

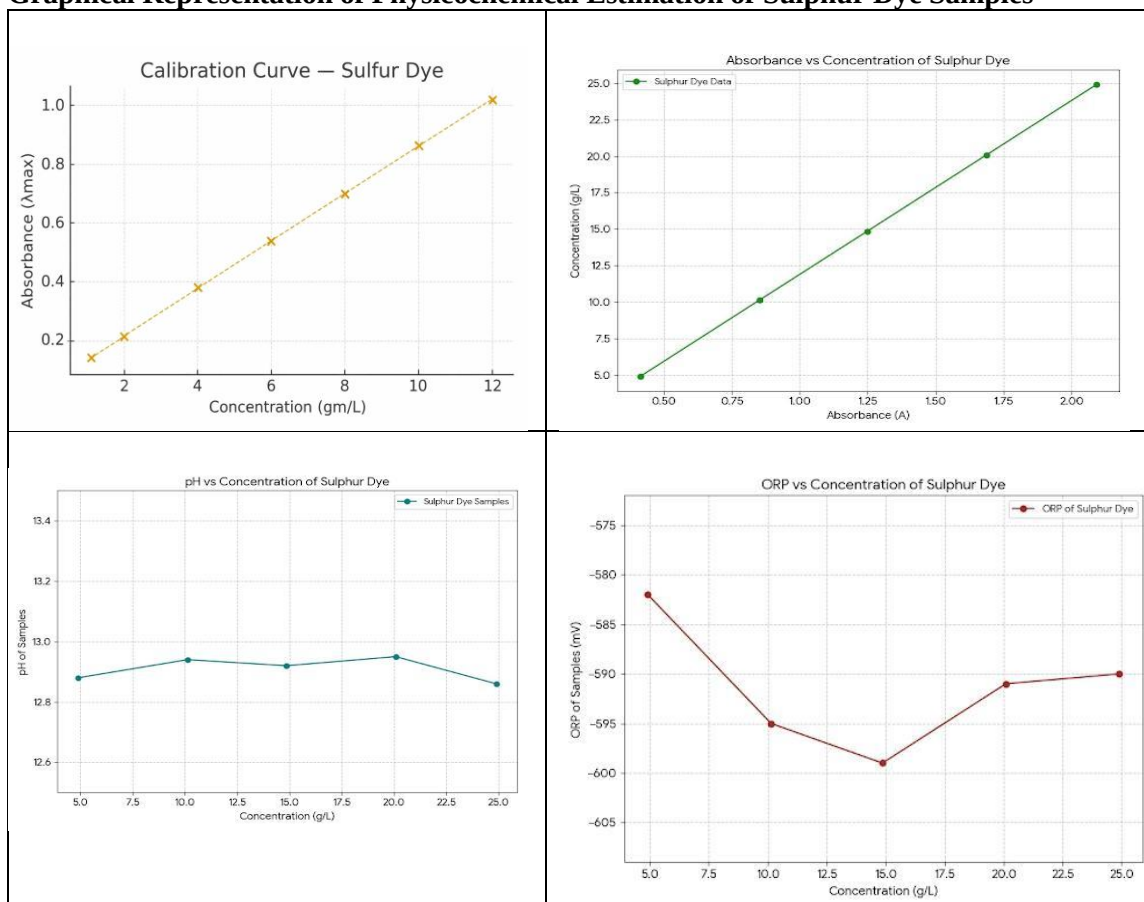


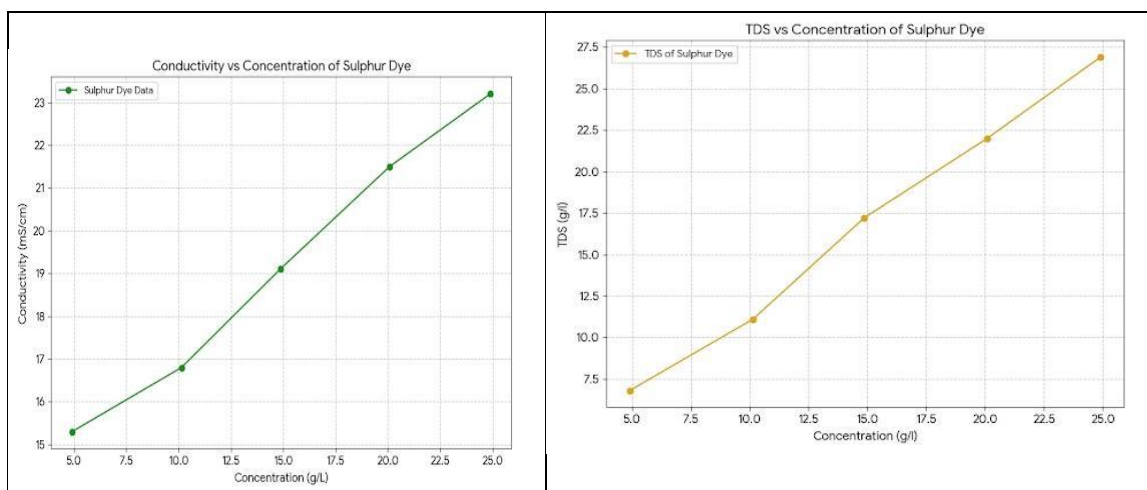


Physicochemical Estimation of Sulphur Dye Samples

Sr. No.	Concentration (gm/l)	Absorbance	pH	ORP (mV)	Conductance (mS/cm)	TDS (gm/l)
1	4.91	0.413	12.88	-582	15.3	6.8
2	10.14	0.852	12.94	-595	16.8	11.1
3	14.86	1.249	12.92	-599	19.1	17.2
4	20.09	1.688	12.95	-591	21.5	22.0
5	24.90	2.092	12.86	-590	23.2	26.9

Graphical Representation of Physicochemical Estimation of Sulphur Dye Samples





Conclusions

The present study demonstrated the applicability of UV–Visible spectrophotometry for quantitative estimation of indigo and sulphur vat dyes in aqueous systems. Both dye systems exhibited concentration-dependent increases in absorbance, enabling estimation of test-sample concentrations through calibration relationships. For indigo, the experimentally calculated calibration slope was 0.1203, while sulphur dye exhibited a slope of 0.0840 under the investigated conditions. The calculated concentrations of the test samples were approximately 5–25 g L⁻¹ for both dye systems, indicating consistent quantitative response across the investigated concentration range.

The physicochemical measurements provided complementary information. Both dyes exhibited strongly alkaline pH and negative ORP values, demonstrating that the investigated solutions were characterized by alkaline and reducing conditions. Conductivity and TDS increased with increasing dye concentration, indicating corresponding changes in the dissolved ionic and solids content of the solutions.

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