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Stoichiometric Optimisation of Vat Dyeing for Sustainable Colour Fastness in Cotton Fabrics

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Abstract

Purpose: Vat dyes are widely recognised for their superior colour fastness on cotton substrates; however, the efficiency of their reduction–oxidation cycle is highly dependent on the stoichiometric balance among the dye, sodium hydroxide (NaOH), and sodium hydrosulphite ($Na_2S_2O_4$). This study investigates how controlled variations in reagent ratios influence colour fastness, to develop a stoichiometrically grounded optimisation framework applicable to artisanal and semi-industrial dyeing systems in Ghana.

Methodology/Design: An experimental factorial design was employed using mercerised cotton fabric as the substrate. All reagent ratios were expressed in a fixed order (dye: NaOH: $Na_2S_2O_4$), while the mixing sequence was treated as a separate variable. The commonly used 1:2:2 ratio was adopted as a comparative baseline rather than a universal standard. A total of 27 dyeing conditions were tested across three vat dye systems (golden yellow, red, and violet). Colour fastness to rubbing (dry and wet) and washing was evaluated using ISO-based grey scale assessments. Each experiment was replicated three times, and results are reported as mean ± standard deviation.

Findings: The results demonstrate that stoichiometric balance between NaOH and $Na_2S_2O_4$ governs the efficiency of dye reduction, fibre penetration, and subsequent fixation. Ratios approximating equilibrium, particularly 5:1:1 and 2:1:1 (dye: NaOH: $Na_2S_2O_4$), consistently produced superior fastness grades. In contrast, reagent excess or deficiency disrupted the redox balance, resulting in reduced dye stability and increased colour loss. While no clear sequence-dependent trend was observed, this outcome is interpreted cautiously due to confounding between dye type and mixing sequence.

Practical and Social Implications: The study provides an empirically validated, ratio-based guideline that can improve process consistency, reduce chemical excess, and enhance product durability in resource-constrained dyeing environments. The framework supports a transition from intuition-based practice to controlled, evidence-informed dyeing processes.

Originality: This research advances the application of stoichiometric theory within textile dyeing by linking quantitative reagent balance to measurable performance outcomes under practical conditions. It contributes a context-sensitive optimisation model that integrates chemical precision with artisanal production systems, extending beyond conventional laboratory-based studies.

Keywords
Stoichiometry, vat dyes, colourfastness, sodium hydrosulphite, caustic soda, Ghana/artisanal dyeing.

1 | INTRODUCTION

Vat dyes occupy a central position in textile colouration due to their exceptional resistance to washing, rubbing, and light exposure (Kim & Jang, 2010; Zhang & Xiao, 2005; Wakida, 2002; Broadbent, 2001). Their durability arises from a distinctive dyeing mechanism based on a reduction–oxidation (redox) cycle, in which the insoluble dye pigment is reduced to a soluble leuco form that diffuses into the fibre structure and is subsequently re-oxidised to its insoluble state within the fibre matrix (Rippon et al., 2011; Kim & Jang, 2010). This reversible transformation underpins the high performance of vat dyes, but it is also chemically sensitive to factors such as reagent concentration, temperature, and the redox conditions of

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the dyebath (Santhi, 2016). In practical dyeing contexts, particularly in small-scale and artisanal systems, dyebath preparation is often guided by empirically derived ratios rather than chemically optimised formulations (Ericksson et al., 2021). For instance, the commonly used working ratio of 1:2:2 (dye: NaOH: $Na_2S_2O_4$) serves as a practice-based baseline rather than a universally validated standard. This formulation is widely applied for its operational simplicity in workshop settings, where precise stoichiometric calculations are rarely employed. As Saikhao et al. (2022) and Saikhao et al. (2018) note, no studies identify a 1:2:2 (dye: NaOH: $Na_2S_2O_4$) ratio as a theoretically derived standard. Instead, these recipes serve as experiential starting points, adjusted according to colour yield, fibre type, and environmental considerations, including efforts to replace $Na_2S_2O_4$ with greener reducers such as sugars. Within this system, sodium hydrosulphite acts as the reducing agent (Wang et al., 2024), sodium hydroxide provides the alkaline medium necessary for dye solubilisation (Abebe & Gelebo, 2019), and atmospheric oxygen facilitates re-oxidation after fibre penetration (Shuvo, 2018). However, the efficiency of this process depends on maintaining a balanced stoichiometric relationship among these reagents. Deviations may lead to incomplete reduction, inefficient dye fixation, or fibre degradation due to excessive alkalinity (Abebe & Gelebo, 2019).

Despite the widespread use of vat dyes, particularly within artisanal textile production, empirical studies that systematically isolate and quantify the effects of reagent stoichiometry on measurable colour fastness outcomes remain limited. Existing research has largely focused on reaction kinetics under controlled laboratory conditions or general process optimisation without explicitly linking reagent proportionality to performance metrics such as rubbing and washing fastness. In artisanal contexts, dyeing practices are often guided by experiential knowledge rather than controlled formulation, which can result in variability in colour quality and inefficient chemical use (Appiah-Brempong et al., 2022). Consequently, there is a lack of experimentally validated, ratio-specific optimisation frameworks that are both scientifically rigorous and practically applicable to small-scale dyeing systems. This gap is particularly relevant in the Ghanaian context, where artisanal dyeing plays a critical role in cultural production and local economies. Persistent challenges, including inconsistent colour outcomes, inefficient chemical usage, and environmentally unsustainable effluent disposal (Gbadegbe et al., 2021; Ahiable, & Triki, 2021), highlight the need for a structured yet accessible optimisation model grounded in chemical principles.

The theoretical foundation of this study is rooted in stoichiometry, which describes the quantitative relationships among reactants in chemical reactions (Oriakhi, 2021). Within vat dyeing systems, stoichiometry governs the redox equilibrium between insoluble dye molecules and their soluble leuco forms, determining reduction efficiency, solubility, and subsequent re-oxidation behaviour (Maulik et al., 2022). Sodium hydrosulphite ($Na_2S_2O_4$) functions as an electron donor, while sodium hydroxide (NaOH) stabilises the leuco form by maintaining alkaline conditions conducive to electron transfer (Uddin, 2014). The reaction sequence is represented as:

Vat Dye (insoluble) $+Na_2S_2O_4+NaOH \rightarrow$ Leuco Dye (soluble)

Leuco Dye (soluble) $+ O_2 \rightarrow$ Vat Dye (oxidised, insoluble)

Equation (1). Stoichiometric Redox Conversion Process of Vat Dyes.

This equation is schematically illustrated in Figure 1.

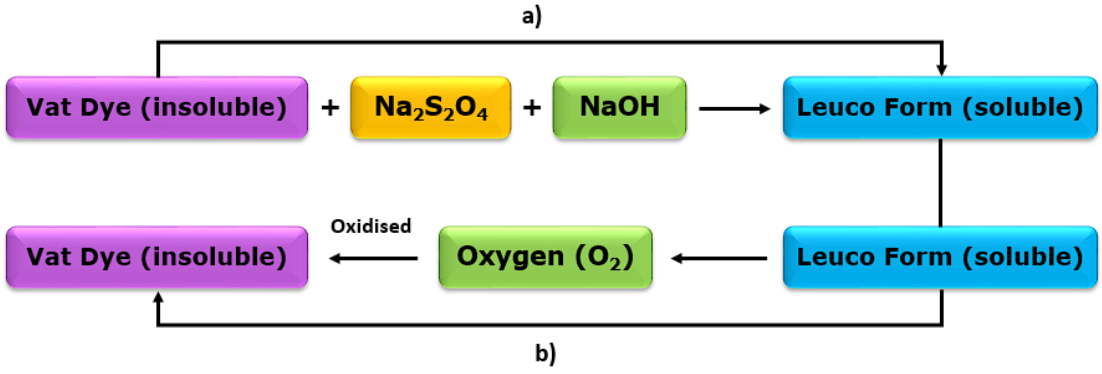


Figure 1. Stoichiometric Redox Conversion Process of Vat Dyes: (a) Reduction stage, (b) Oxidation stage

When stoichiometric balance is achieved, dye molecules are fully reduced, enabling uniform diffusion into the amorphous regions of cotton fibres. Controlled oxidation subsequently restores the insoluble dye form, resulting in strong fibre–dye bonding. In contrast, imbalances in reagent proportions produce

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distinct deficiencies. Under-reduction, typically due to insufficient hydrosulphite, leads to poor solubility and uneven dye distribution (Patra et al., 2018; Chakraborty, 2011). Over-reduction, caused by excess hydrosulphite, may induce molecular cleavage and weaken dye fixation (Rippon et al., 2011). Similarly, excessive alkali enhances fibre swelling but can promote cellulose degradation, negatively affecting fastness properties (Paul et al., 2017). These interactions demonstrate that dyeing efficiency is governed by stoichiometric proportionality rather than reagent abundance (Santos-Silva, 2022).

This study extends stoichiometric theory into applied textile practice by systematically varying reagent ratios and evaluating their influence on colour fastness performance. By experimentally linking reagent proportionality to measurable outcomes, the study develops an empirical framework for optimising vat dyeing conditions within practical production contexts. This approach translates theoretical equilibrium principles into accessible guidelines that can be implemented without complex chemical calculations, thereby enhancing applicability within artisanal and semi-industrial settings. The primary aim of this study is to investigate how variations in the concentrations of sodium hydroxide (NaOH) and sodium hydrosulphite ($Na_2S_2O_4$) affect the colour fastness of vat-dyed cotton fabrics. Using an experimental factorial design, the study seeks to identify reagent ratios that maximise rubbing and washing fastness while promoting more efficient chemical utilisation. Given that unregulated chemical use in small-scale dyeing systems has been associated with environmental and health risks (Appiah-Brempong et al., 2022), the findings provide a basis for improving both process control and sustainability in textile dyeing practice.

By situating vat dyeing within a stoichiometric framework, the study contributes to textile chemistry by demonstrating how controlled reagent balance can be operationalised as a practical optimisation strategy. It further supports the integration of scientific principles into artisanal production systems, advancing more consistent, efficient, and environmentally responsible dyeing practices within Ghana and comparable contexts.

2 | METHODOLOGY

2.1| Research Design and Theoretical Framework

This study employed a full-factorial experimental design to systematically evaluate the effects of reagent stoichiometry (NaOH and $Na_2S_2O_4$ concentrations) and mixing sequence on the colour fastness of vat-dyed cotton fabrics. Factorial designs enable simultaneous examination of multiple variables and their interactions, thereby increasing experimental efficiency and analytical robustness (Singh & Chauhan, 2022). The design was grounded in stoichiometric optimisation theory, which posits that chemical reaction efficiency is maximised when reactants are present in quantitatively balanced proportions (Camosso, 2019). Within the vat dyeing system, this principle governs the reduction–oxidation equilibrium and, consequently, the efficiency of dye–fibre bonding. The experiment specifically examined:

- Main effects:
  - (i) sodium hydroxide (NaOH) concentration
  - (ii) sodium hydrosulphite ($Na_2S_2O_4$) concentration
- Interaction effects:
  - (iii) combined influence of NaOH and $Na_2S_2O_4$ ratios on fastness outcomes
  - (iv) interaction between reagent ratios and mixing sequence

A total of 27 experimental conditions were tested, comprising:

- 3 control samples using the 1:2:2 ratio (Dye: NaOH: $Na_2S_2O_4$), and
- 24 systematically varied ratios representing under-balanced, balanced, and over-balanced stoichiometric conditions.

The 1:2:2 ratio was used strictly as a controlled baseline and a comparative reference formulation, reflecting commonly reported operational practice in artisanal and semi-industrial vat dyeing contexts (Ericksson et al., 2021), rather than a universally optimised standard.

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2.2| Materials and Apparatus

2.2.1| Fabric and Chemicals

- Substrate: Mercerised 100% cotton fabric samples (5.8” × 8.3”; A5-size).
- Dyes: Vat dye colourants; golden yellow, red, and violet - were selected to evaluate chromatic consistency across hues.
- Chemicals: Analytical-grade caustic soda (NaOH) and sodium hydrosulphite ($Na_2S_2O_4$)

These materials were selected to ensure chemical consistency and reproducibility across experimental conditions. Figure 2 presents the fabric, vat dye and its reagents used in the experiment.

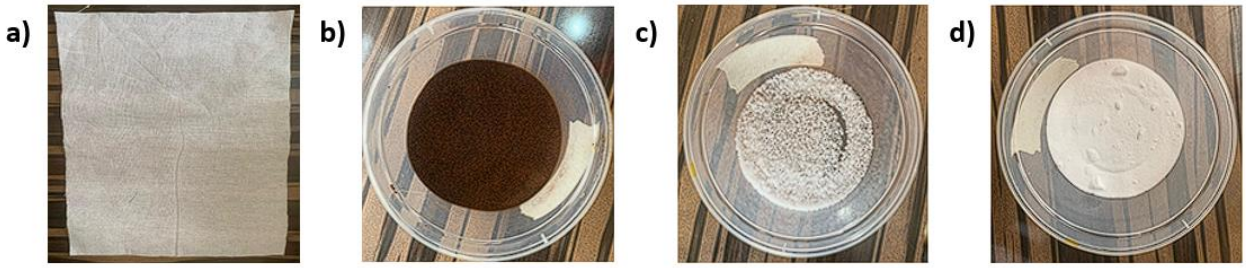


Figure 2. Fabric, Colourant and Reagents: (a) Mercerised cotton, (b) Vat dye, (c) Caustic soda (NaOH), and (d) Sodium hydrosulphite ($Na_2S_2O_4$)

2.2.2| Dyeing Tools

- Measuring tools: metric measuring cylinders, beakers, test tubes, and stirring rods.
- Dyeing setup: 300 mL capacity dyeing bowls used for each dyebath sample. Figure 3 shows examples of some tools used for the experiment.

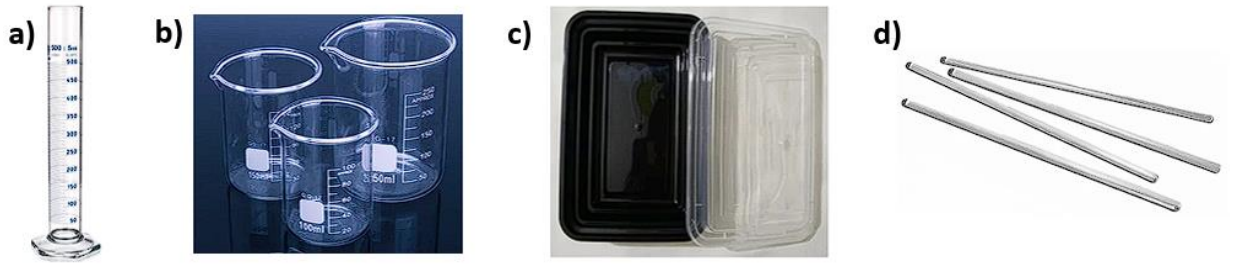


Figure 3. Dyeing Tools: (a) Measuring tube, (b) Beakers, (c) Dyeing bowl, and (d) Stirring rods

2.2.3| Testing equipment

The following equipment in Figure 4 was used for testing dyed samples in the study.

- Crockmeter (Model XHF-04B) – for rubbing fastness (ISO 105-X12)
- Colour Matching Cabinet (ISO 105-A05) – visual assessment under D65 illumination
- Colour Precision Reader (Amstat; CIELAB system) – supplementary instrumental verification

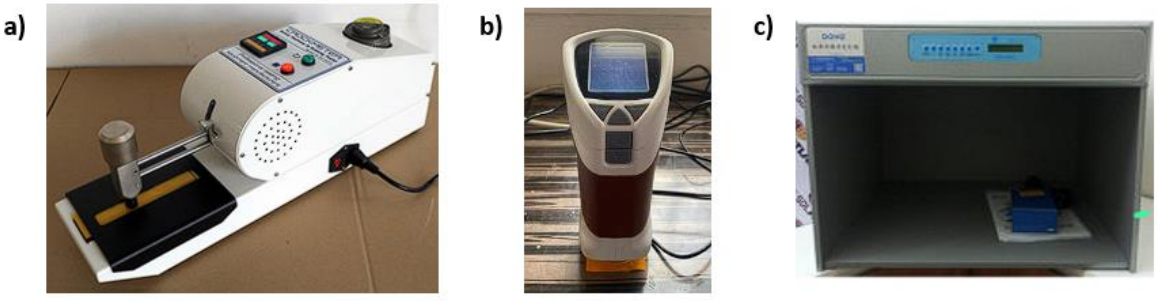


Figure 4. Testing Equipment: (a) (Model XHF-04B), (b) Colour Precision Reader (Amstat), (c) Colour Matching Cabinet (ISO 105-A05)

2.2.4| Conceptual Framework

A conceptual framework guided the study to understand the relationships among the variables in the experiment. The experimental framework integrates stoichiometric theory with textile process variables. It illustrates the interrelationships among the three key variable categories: independent, controlled, and

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dependent variables within the dyeing process. Figure 5 shows the conceptual framework for the experimental design.

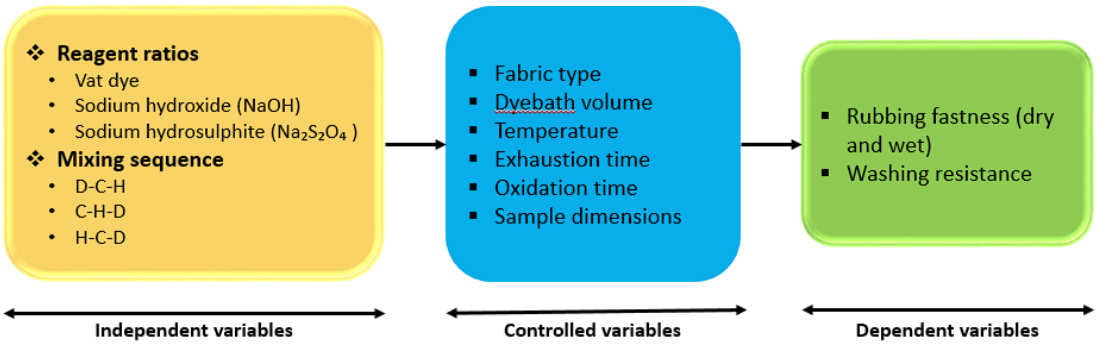


Figure 5. Conceptual Framework of the Experiment (Adapted from Oriakhi, 2021; Camosso, 2019)

The independent variables comprise the quantitative ratios of dyes, sodium hydroxide (NaOH) and sodium hydrosulphite ($Na_2S_2O_4$), together with the mixing sequence of reagents as secondary variables introduced in dyebath preparation. These variables represent the stoichiometric factors expected to influence the reduction–oxidation efficiency of the dyeing system. The dependent variables are the performance outcomes, specifically, rubbing fastness (dry and wet) and washing resistance, which indicate the degree of colour fixation and fibre–dye bonding achieved under each ratio condition. Several controlled variables were maintained to ensure validity and comparability across trials. These included the fabric type (mercerised cotton), dye bath volume (300 mL), exhaustion and oxidation times (3 and 5 minutes respectively), temperature (ambient, ~25–28°C), and sample dimensions (A5 size). The framework assumes that stoichiometric equilibrium between NaOH and $Na_2S_2O_4$ governs redox efficiency, thereby determining dye penetration and fixation. This conceptual link guided the formulation of experimental ratios and informed the selection of variables throughout the study.

2.3| Experimental Procedure

2.3.1| Ratio Definition and Mixing Sequence

For clarity and consistency, all reagent ratios in this study are expressed in a fixed order of Dye: NaOH: Sodium Hydrosulphite (D: C: H), irrespective of the mixing sequence. Mixing sequence is treated as a categorical experimental variable and is denoted independently as:

- D–C–H: Dye → Caustic soda → Hydrosulphite
- C–H–D: Caustic soda → Hydrosulphite → Dye
- H–C–D: Hydrosulphite → Caustic soda → Dye

In the first sequence, the dye, caustic soda and hydrosulphite were introduced successively in 300 mL of water based on the selected ratio. In the second sequence, each reagent was first dissolved separately in 100 mL of water and then combined into a single bath to ensure homogeneous reduction. The last sequence reversed the first order to test the effect of reagent reversal on dye uptake efficiency. In the present design, each dye colour was tested under a single mixing sequence, which introduces a confounding effect between dye type and mixing order. Therefore, sequence-specific effects cannot be independently isolated and are interpreted cautiously.

2.3.2| Ratio Variations

These ratio variations were chosen to span under-reduction conditions (low $Na_2S_2O_4$), over-reduction conditions (excess $Na_2S_2O_4$), over-alkaline conditions (excess NaOH) and near-equilibrium conditions relative to typical stoichiometric requirements, exploring how the equilibrium between the alkali and reductant affects the reduction potential, dye solubility, and ultimate fibre fixation. Table 1 presents the ratio variations for the experiment.

Table 1. A Table showing the dyebath formulations expressed in fixed Dye: NaOH: $Na_2S_2O_4$ (D:C:H) ratios, with mixing sequence applied as an independent variable.

Golden Yellow (D–C–H) Sequence	Red (C–H–D) Sequence	Violet (H–C–D) Sequence
1 : 2 : 2	2 : 2 : 1	2 : 2 : 1

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2 : 1 : 2	1 : 2 : 3	3 : 4 : 1
1 : 2 : 1	2 : 3 : 1	3 : 5 : 1
2 : 1 : 1	3 : 1 : 3	2 : 3 : 3
3 : 1 : 2	2 : 2 : 4	4 : 2 : 3
2 : 2 : 3	2 : 3 : 2	5 : 2 : 1
5 : 1 : 1	1 : 1 : 5	1 : 1 : 5
1 : 5 : 1	1 : 5 : 1	5 : 1 : 1
1 : 1 : 5	5 : 1 : 1	1 : 5 : 1

The Golden Yellow series followed the D–C–H order, the Red series employed C–H–D, and the Violet series utilised H–C–D. These combinations enabled comparative analysis of how reagent sequence and ratio interact to determine the reduction–oxidation balance, dye–fibre bonding strength, and resulting colour fastness.

2.3.3| Preparation of Dye Solutions

Each dyebath was prepared by dissolving the vat dye in water with caustic soda and sodium hydrosulphite according to the specific ratio being tested. For standardisation, the base unit for all ratio calculations was defined by a Standard Mass Unit (SMU) of 2.5 grams. This SMU corresponds to the '1' unit for each reagent in the proportional ratios. Specifically, in the control ratio of 1:2:2 (dye: NaOH: $Na_2S_2O_4$), the actual masses used were: [2.5g (Dye)], [5.0g (NaOH)], and [5.0g ($Na_2S_2O_4$)]. All formulations were scaled proportionally to maintain stoichiometric integrity across treatments. Figure 6 shows a prepared dye solution.



Figure 6. Prepared Dye Solution

2.3.4| Dyeing Conditions

Each dyeing session utilised 300 mL of prepared dyebath volume for a 3-minute exhaustion time, followed by 5 minutes of air oxidation. An ambient temperature (25–28°C) was maintained to reflect artisanal dyeing conditions. The dyed samples were rinsed thoroughly to remove unfixed dye and residual chemicals. Figure 7 presents dyed samples with their colour (dye) and respective ratio variations from the experiment.

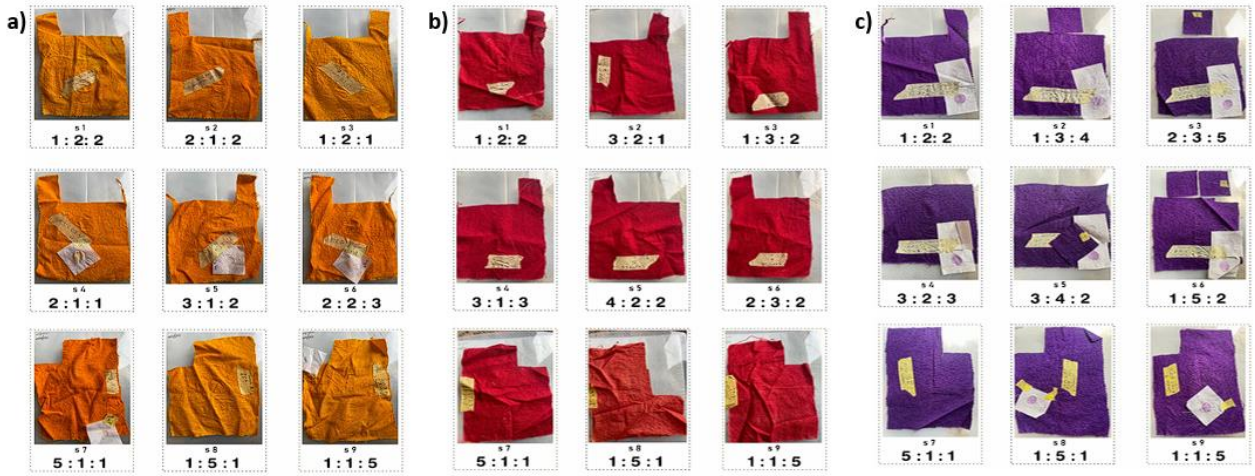


Figure 7. Dyed Fabric Samples: (a) Golden yellow samples with ratios, (b) Red samples with ratios, and (c) Violet samples with ratios

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2.4| Testing of Colour Fastness

Two key fastness tests were conducted to evaluate performance outcomes:

2.4.1| Rubbing Fastness

Both dry and wet rubbing fastness were measured using the Crockmeter in accordance with ISO 105-X12. Results were graded on the standard greyscale (1–5), where 1 represents poor fastness, and 5 denotes excellent resistance.

2.4.2| Washing Resistance (Simulated Domestic Laundering)

Washing performance was evaluated using a controlled hand-washing simulation involving 5 g/L ECE neutral detergent at 40°C for 5 minutes. This method was adopted to reflect typical artisanal and domestic laundering conditions rather than strict ISO 105-C06 protocols. Results were assessed using ISO grey-scale ratings (Grades 1–5). While this approach does not replicate standardised ISO washing procedures, it provides context-relevant insight into the durability of vat-dyed fabrics under realistic usage conditions.

2.5| Data Reliability and Evaluation

Comparative evaluation identified the ratios yielding optimal dry and wet rubbing fastness. Observed trends were interpreted through the stoichiometric framework, linking reagent concentration to redox completeness and dye–fibre bonding efficiency. Although the study focused on practical optimisation rather than formal statistical modelling, each dyeing trial was replicated three times under identical bath conditions to ensure consistency and reliability. Rubbing and washing fastness grades were evaluated using the ISO Grey Scale, and results were averaged across replicates. Variations among replicates remained within a ± 0.5 grade tolerance, confirming high reproducibility of the experimental procedure. Observed differences in colour fastness between reagent ratios were therefore interpreted as chemically significant rather than artefacts of experimental error. In addition to mean fastness grades, variability across replicates was quantified using standard deviation (SD). Given the ordinal nature of ISO grey scale ratings, variation remained within a narrow range (±0.5 grade). Mean ± SD values are therefore reported to provide a measure of dispersion, while recognising the semi-quantitative nature of the scale. One-way ANOVA was not applied due to the ordinal nature of the grading system; instead, comparative trend analysis was used, supported by consistent directional differences and replication agreement.

3 | RESULTS

3.1| Rubbing Fastness of Vat-Dyed Cotton Fabrics

The results of the experimental trials are presented according to measured fastness properties across the defined reagent ratios (Dye: NaOH: $Na_2S_2O_4$; D: C: H). Rubbing fastness performance for the three dye systems - Golden Yellow, Red, and Violet - is illustrated in Figures 8–10. Dry and wet rubbing fastness were evaluated using the ISO greyscale (Grades 1–5), where higher values indicate greater resistance to colour transfer. Results are reported as mean values across three replicates, with observed variability remaining within ±0.5 grade. Across all samples:

- Dry rubbing fastness ranged from 4.0 to 5.0
- Wet rubbing fastness ranged from 1.0 to 4.5

This distribution indicates relatively stable dry fastness performance, with greater variability observed under wet rubbing conditions.

3.1.1| Golden Yellow (D–C–H Sequence)

As presented in Figure 8, dry rubbing fastness for the Golden Yellow series remained within a narrow range of 4.0–5.0, with multiple samples (Samples 3–8) attaining the maximum grade of 5.0. Wet rubbing fastness values ranged from 1.5 to 3.5, with the highest mean value (3.5 ± 0.5) recorded for Sample 6 (2:2:3, D: C: H). Lower wet fastness grades were observed in samples with disproportionate reagent ratios, particularly those deviating from balanced NaOH and $Na_2S_2O_4$ proportions.

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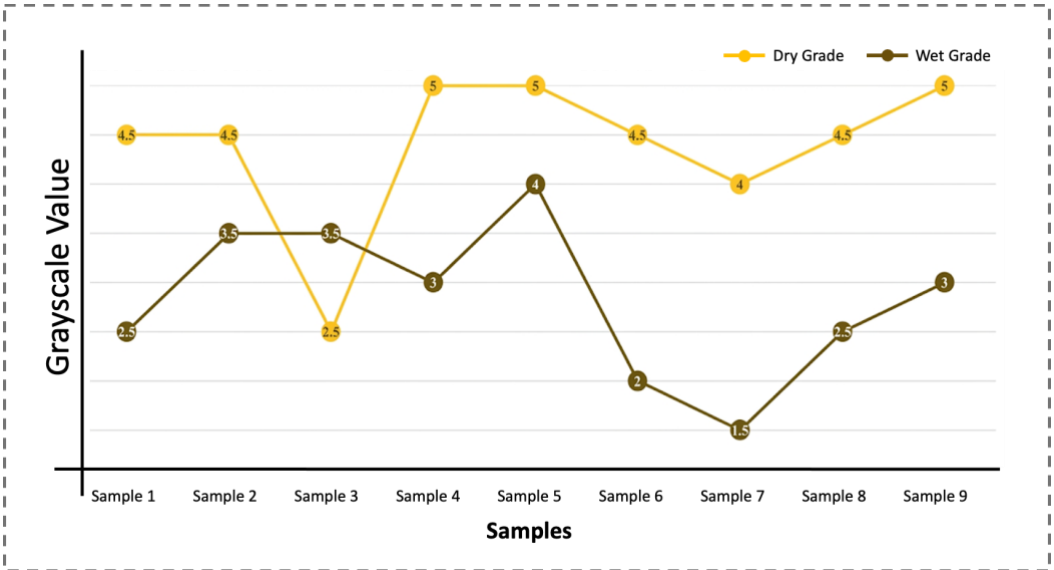


Figure 8. Rubbing fastness (dry and wet) of Golden Yellow vat-dyed samples under varying reagent ratios (D:C:H).

3.1.2| Red (C–H–D Sequence)

The Red dye system exhibited a broader range of rubbing fastness values (Figure 9). Dry fastness grades ranged from 1.5 to 5.0, while wet fastness grades ranged from 1.0 to 4.5. The highest dry fastness (5.0 ± 0.0) was observed in Sample 6 (2:3:2, D:C:H), while the highest wet fastness (4.5 ± 0.5) was recorded in Sample 7 (1:1:5, D:C:H). Samples with lower performance (Samples 1–3) corresponded to conditions with marked imbalance in reagent proportions.

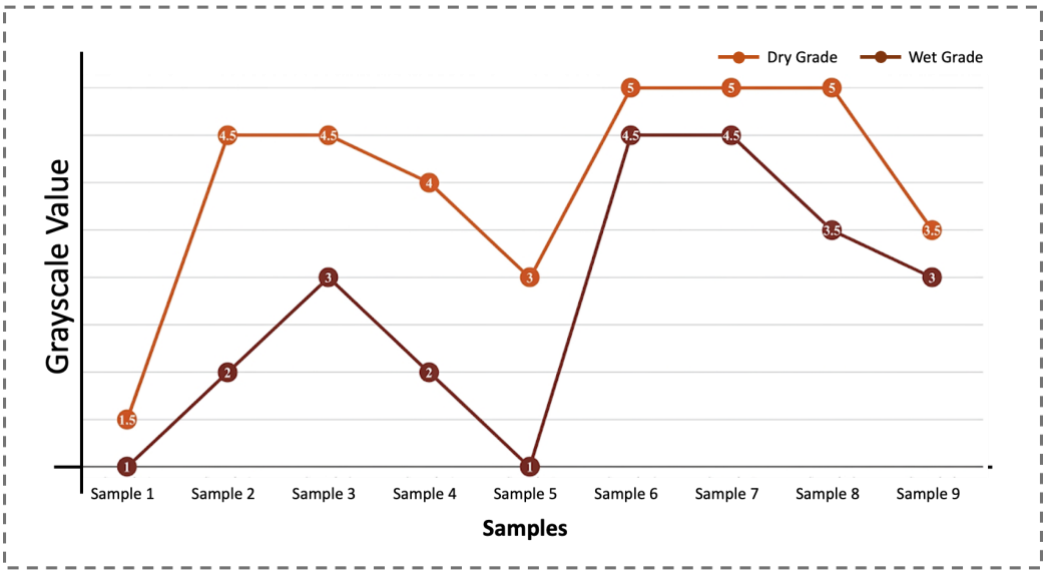


Figure 9. Rubbing fastness (dry and wet) of Red vat-dyed samples under varying reagent ratios (D:C:H)

3.1.3| Violet (H–C–D Sequence)

The Violet dye system demonstrated comparatively stable performance across tested ratios (Figure 10). Dry rubbing fastness values ranged between 4.0 and 5.0, with consistently high values observed across multiple samples (Samples 3–8). Wet rubbing grades ranged from 2.0 to 3.5, with the highest mean value (3.5 ± 0.5) recorded for Sample 9 (1:5:1, D:C:H). Overall variation in this series remained within a narrower band compared to the Red system.

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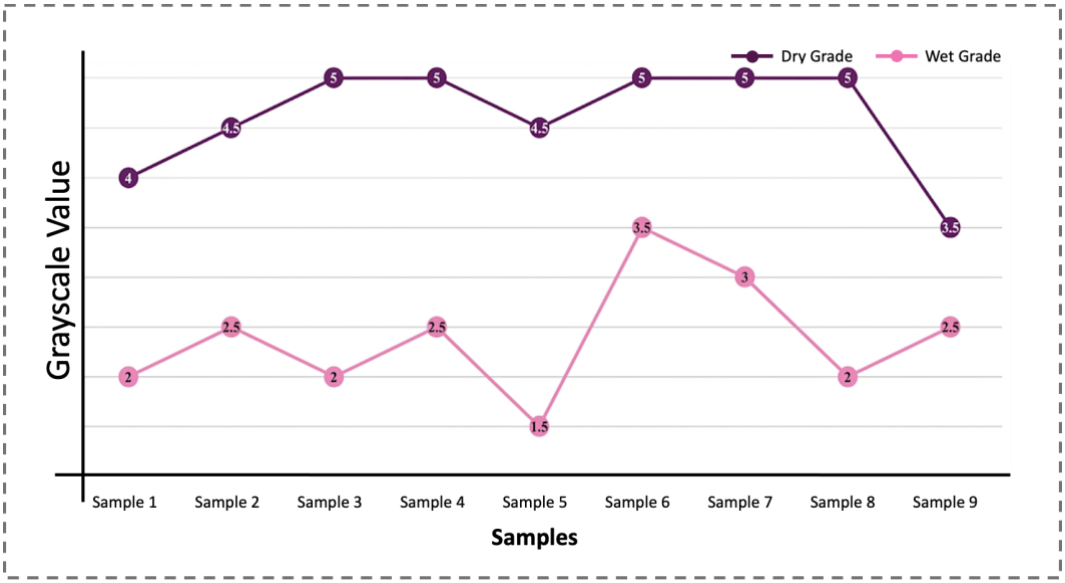


Figure 10. Rubbing fastness (dry and wet) of Violet vat-dyed samples under varying reagent ratios (D:C:H)

3.2| Washing Resistance Performance

Washing resistance results, obtained under simulated domestic laundering conditions, exhibited trends comparable to those for rubbing fastness. Fastness grades ranged from:

- Grade 2 to Grade 5

Higher washing resistance values (Grades 4–5) were observed in samples with relatively balanced reagent ratios, including:

- 5:1:1 (D:C:H)
- 2:1:1 (D:C:H)

Lower grades (2–3) were recorded in samples with:

- Excess alkali (e.g., 1:5:1, D:C:H)
- Excess reductant (e.g., 1:1:5, D:C:H)

Results are reported as mean values across three replicates, with variability remaining within ±0.5 grade.

3.3| Ratio Variation Outcomes

Comparative analysis across all dye systems indicates that the reagent ratio significantly influences fastness performance. Ratios approximating balanced proportions between NaOH and $Na_2S_2O_4$ consistently yielded higher fastness grades, whereas extreme deviations in reagent proportions resulted in reduced and more variable performance. Across all experimental conditions, the following trends were observed:

- ratios with balanced reductant–alkali proportions exhibited higher and more consistent fastness values.
- excess or deficiency of either naoh or $na_2s_2o_4$ corresponded to reduced fastness grades.
- variability in performance was more pronounced in wet rubbing and washing resistance than in dry rubbing

Although the mixing sequence (D–C–H, C–H–D, H–C–D) was included as a variable, its effect cannot be evaluated independently in this study because it is confounded with dye type. Observed performance differences therefore reflect the combined effects of dye chemistry and reagent ratio rather than sequence alone.

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4 | DISCUSSION

4.1| Effects of Over-Reduction, Under-Reduction and Incomplete Solubilisation

The results demonstrate that deviations from balanced reductant levels significantly affect dye performance. Ratios characterised by excess sodium hydrosulphite (e.g., 1:1:5, D:C:H) exhibited reduced rubbing and washing resistance. This behaviour is consistent with findings by Patra et al. (2018) and Chakraborty (2011), who report that over-reduction destabilises vat dye chromophores and interferes with effective re-oxidation. From a molecular perspective, excessive reduction promotes structural degradation of anthraquinone and indigoid dye systems, leading to fragmentation and reduced substantivity (Rippon et al., 2011; Broadbent, 2001). The resulting leuco species are less stable and more susceptible to removal during washing, which explains the lower wet fastness observed in this study. Figure 11 presents empirical evidence from the experiment.

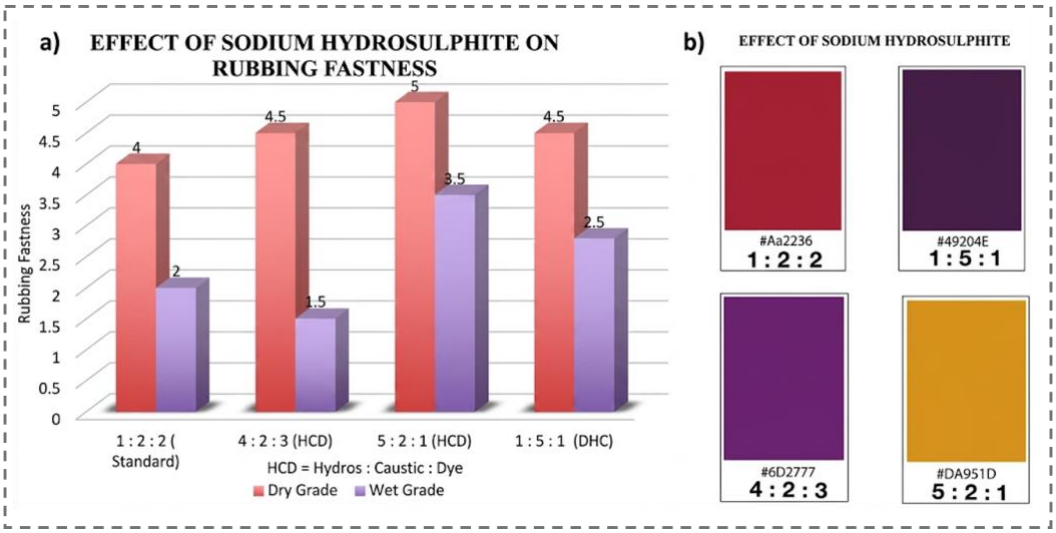


Figure 11. Effects of Over-reduction on Vat Dyes: (a) Effect of sodium hydrosulphite on rubbing fastness, (b) Sampled evidence of dyed fabrics

Conversely, under-reduction conditions (e.g., 5:1:1, D:C:H; 3:1:2, D:C:H) produced uneven colouration and reduced wet fastness. Incomplete reduction limits the formation of soluble leuco dye, thereby restricting fibre penetration and increasing the proportion of surface-bound dye. Similar observations have been reported by Rippon et al. (2011), where insufficient reductant availability resulted in poor dye diffusion and weak fixation. Moreover, these findings align with Abebe and Gelebo (2019), who demonstrated that incomplete solubilisation reduces chromophore regeneration efficiency during oxidation. Collectively, the results confirm that both excess and deficiency of sodium hydrosulphite disrupt dye–fibre interaction, emphasising the necessity of controlled reductant dosage.

4.2| Effects of Alkali Excess and Strongly Alkaline Dyebaths

The influence of sodium hydroxide concentration is similarly critical. Ratios with excess alkali (e.g., 1:5:1, D:C:H) corresponded to slight reductions in dry rubbing fastness, particularly under strongly alkaline dye bath conditions. This observation is consistent with Paul et al. (2017), who reported that high NaOH concentrations accelerate cellulose chain scission, weakening fibre integrity. Under such conditions, increased fibre swelling enhances dye diffusion but simultaneously promotes cellulose hydrolysis, disrupting intermolecular hydrogen bonding within the fibre structure. Santhi (2016) further noted that highly alkaline environments can destabilise the leuco form by promoting premature oxidation, thereby reducing effective dye uptake. The combined effect is reduced dye fixation efficiency and mechanical durability during rubbing. These outcomes are illustrated in Figure 12, corroborating the studies of Paul et al. (2017) and Santhi (2016).

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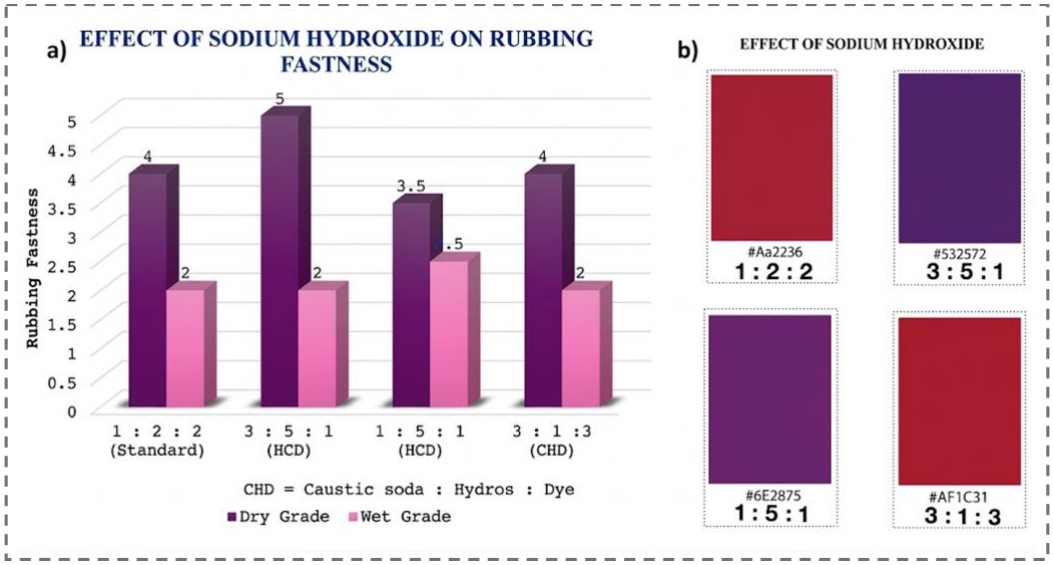


Figure 12. Effects of Over-alkaline on Vat Dyes: (a) Effect of sodium hydroxide on rubbing fastness, (b) Sample evidence of dyed fabrics

These results support Santos-Silva (2022), who emphasised that excess reagent concentrations disturb redox equilibrium, leading to inconsistent dyeing outcomes. The present findings reinforce that optimal performance depends on balanced alkalinity rather than maximum alkalinity.

4.3| Stoichiometric Balance and Redox Efficiency

The highest fastness values observed in this study correspond to reagent ratios approaching stoichiometric equilibrium, particularly 5:1:1 and 2:1:1 (D:C:H) (Figure 13). These ratios consistently produced superior dry and wet rubbing performance as well as improved washing resistance. At these ratios, the balance between sodium hydrosulphite and sodium hydroxide facilitates:

- complete reduction of the vat dye to its soluble leuco form,
- stable diffusion into the amorphous regions of cotton fibres, and
- controlled re-oxidation to the insoluble pigment form

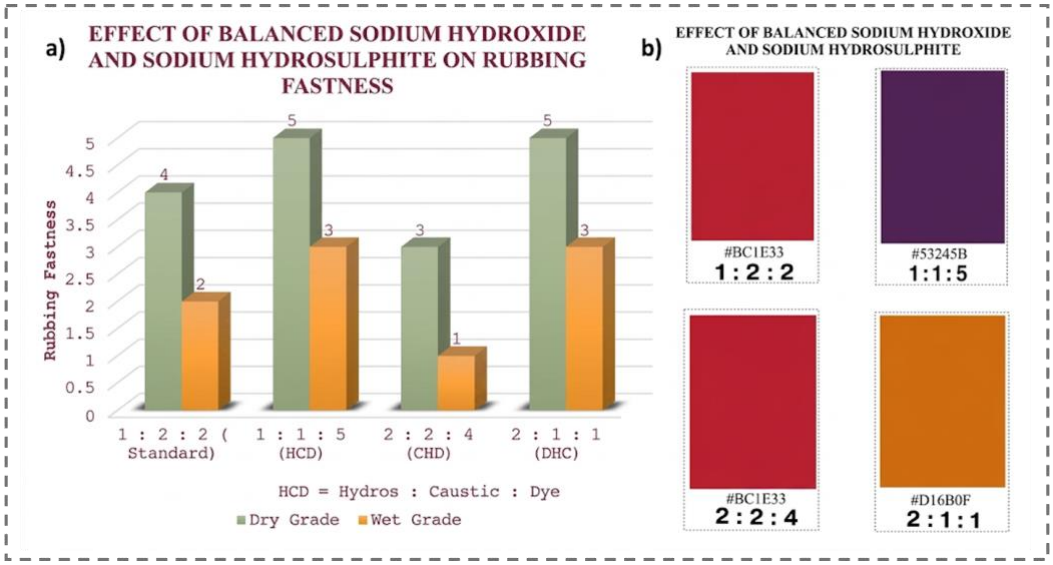


Figure 13. Stoichiometric Balance and Redox Efficiency: (a) Effect of balanced sodium hydroxide and sodium hydrosulphite on rubbing fastness, (b) Sampled evidence of dyed fabrics

This behaviour is consistent with stoichiometric principles governing redox systems (Maulik et al., 2022; Oriakhi, 2021), where electron transfer efficiency is maximised under balanced reactant conditions. Comparable findings have been reported in vat dyeing studies by Rippon et al. (2011) and Kim and Jang (2010), who showed that optimal redox potential and pH conditions enhance dye substantivity and fixation. Similarly, Verma et al. (2022) and Jaxel et al. (2020) observed that balanced reductant–alkali systems improve dye penetration while limiting molecular degradation. Recent studies (Wang et al., 2024; Zhou et al., 2022) further demonstrate that stoichiometric control reduces side reactions and

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improves dye utilisation efficiency. The present results extend these findings by providing empirical validation within a structured ratio-based framework, applicable to practical dyeing conditions.

4.4| Comparison with Conventional Dyeing Ratios

The conventional 1:2:2 (D:C:H) formulation produced moderate fastness performance relative to optimised ratios. While widely used in practice (Ericksson et al., 2021), this formulation appears to function as a general-purpose operational ratio rather than a chemically optimised equilibrium condition. The superior performance of 5:1:1 and 2:1:1 (D:C:H) indicates that stoichiometric requirements vary depending on:

- dye molecular structure,
- reduction potential, and
- electron-accepting capacity

This observation aligns with Santos-Silva (2022) and Sokolenko et al. (2020), who reported that vat dye systems exhibit dye-specific stoichiometric behaviour, necessitating adaptive optimisation rather than fixed formulations. By quantitatively evaluating multiple ratio conditions, this study contributes to ongoing research in precision dye chemistry, demonstrating that small adjustments in reagent proportions can significantly improve fastness performance.

4.5| Mechanistic Interpretation of Dye-Fibre Interaction

The observed performance trends can be explained through the underlying redox mechanism of vat dyeing. Sodium hydrosulphite ($Na_2S_2O_4$) functions as an electron donor, reducing the insoluble dye into its soluble leuco form, while sodium hydroxide (NaOH) maintains the alkaline conditions required for leuco stabilisation (Wang et al., 2024).

- Under balanced stoichiometric conditions:
- The leuco dye remains chemically stable,
- Diffusion into the fibre matrix is maximised, and
- Re-oxidation restores the insoluble chromophore within the fibre

This process results in strong dye–fibre interactions, supported by π–π stacking and van der Waals forces, as described by Kim and Jang (2010). These interactions contribute to the high dry fastness (Grade 5) and moderate wet fastness (≈3.5) observed in optimised samples. In contrast, reagent imbalance alters this mechanism:

- excess reductant promotes dye degradation,
- excess alkali damages the fibre structure,
- insufficient reductant limits dye solubilisation

These effects collectively reduce fixation efficiency and increase colour loss during mechanical or aqueous stress.

4.6 | Contextual Implications and Methodological Considerations

The findings have direct relevance for artisanal dyeing systems, where reagent proportions are often determined empirically. Studies by Appiah-Brempong et al. (2022) and Karim et al. (2021) highlight variability in dyeing outcomes due to non-standardised practices. The ratio-based framework developed in this study provides a structured and reproducible approach to dyebath formulation, enabling improved consistency without requiring advanced instrumentation. From a broader perspective, the results contribute to the development of sustainable textile processes by demonstrating that optimising existing chemical systems can enhance performance without introducing additional chemical complexity. The interpretation of mixing sequence effects is constrained by the experimental design, in which each dye system was associated with a single mixing sequence. Consequently, observed differences cannot be attributed solely to sequence variation. Future studies should isolate sequence as an independent variable using a single dye across all sequences.

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5 | IMPLICATIONS OF FINDINGS

5.1| Technological and Process Implications

The results confirm that maintaining stoichiometric balance between sodium hydroxide (NaOH) and sodium hydrosulphite ($Na_2S_2O_4$) enhances the reduction–oxidation efficiency of vat dyeing. Balanced reagent conditions promote complete dye reduction, effective fibre penetration, and stable re-oxidation, resulting in improved fastness performance. The identified ratios, particularly 5:1:1 and 2:1:1 (D:C:H), provide a reproducible basis for dyebath formulation. Their application reduces reliance on trial-and-error practices and supports greater consistency in dyeing outcomes. From a process perspective, these findings support a transition from empirically driven methods to quantitatively guided formulation strategies, improving operational control, dye utilisation efficiency, and repeatability in cotton vat dyeing systems.

5.2 | Environmental and Sustainability Implications

The results indicate that reagent imbalance contributes to inefficient chemical utilisation, with implications for waste generation and effluent composition. By maintaining stoichiometric proportions, the use of excess reagent can be minimised, thereby reducing the likelihood of residual chemicals entering wastewater streams. While the present study did not include direct effluent analysis (e.g., BOD or COD), the observed reduction in reagent excess under optimised conditions suggests a potential decrease in chemical load in dye effluents. This should be interpreted as an inferred outcome rather than a quantified reduction. This approach aligns with resource-efficiency principles under Sustainable Development Goal 12 (Responsible Consumption and Production) (Omondi, 2024; Ahiable & Triki, 2021), particularly in contexts where wastewater treatment infrastructure is limited. Importantly, optimisation is achieved using existing, low-cost reagents, making it accessible for small-scale producers without requiring technological substitution or additional capital investment.

5.3| Educational and Capacity-Building Implications

The study demonstrates that stoichiometric principles can be operationalised within practical dyeing contexts, providing a foundation for improved technical training. Integrating stoichiometric reasoning into textile education can enhance understanding of dye–fibre interactions and process control. This approach enables practitioners to:

- diagnose dyeing inconsistencies systematically,
- adjust reagent proportions based on observed outcomes, and
- develop more consistent and predictable dyeing practices

Such integration strengthens the linkage between scientific knowledge and craft-based practice, contributing to competency-based training models in textile education.

5.4| Industrial and Economic Implications

The optimisation framework developed in this study offers a cost-effective pathway for improving dyeing efficiency. By reducing unnecessary reagent use and enhancing fastness performance, the approach supports both cost reduction and product quality improvement. Improved durability of dyed fabrics contributes to extended product lifespan and increased consumer satisfaction. For small- and medium-scale enterprises, this may enhance competitiveness in both local and export markets. Additionally, reduced chemical waste has implications for lowering downstream treatment costs and improving compliance with emerging environmental standards in textile production.

5.5| Theoretical and Research Implications

This study reinforces the applicability of stoichiometric equilibrium as a governing principle in textile dyeing systems. The findings demonstrate that stoichiometry provides not only a descriptive chemical framework but also a predictive and operational tool for controlling dye–fibre interactions. By empirically linking reagent ratios to measurable fastness outcomes, the study contributes to ongoing research in process optimisation and sustainable textile chemistry. The work also identifies avenues for further investigation, including:

- extension to other fibre types and dye classes,
- integration with instrumental colour measurement (e.g., δe analysis),
- coupling stoichiometric optimisation with environmental impact assessment

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These directions support the development of more comprehensive, data-driven approaches to textile process design.

6 | LIMITATIONS OF THE STUDY

While the study provides controlled experimental evidence on stoichiometric optimisation in vat dyeing, several limitations define the scope and generalisability of the findings.

6.1| Scope of Experimental Conditions

The experiments were conducted under controlled laboratory conditions using a fixed dyebath volume (300 mL) and A5-sized mercerised cotton samples. This enabled isolation of stoichiometric variables; however, such conditions may not fully represent the complexity of industrial or artisanal batch dyeing systems. Scale-up may introduce additional variables, including:

- thermal gradients and heat transfer variability,
- mechanical agitation differences, and
- variations in oxygen exposure during oxidation

These factors may influence redox dynamics and dye fixation efficiency, requiring validation under larger-scale or field conditions.

6.2| Range of Dye Types

The study utilised three vat dye systems (golden yellow, red, and violet) to represent a limited chromatic range. Although these dyes provided comparative insight, vat dyes vary in molecular structure, particularly between anthraquinone and indigoid classes. Such structural differences influence:

- reduction potential,
- leuco stability, and
- fibre affinity

Accordingly, the optimised ratios identified in this study should be interpreted as system-specific rather than universally applicable. Broader validation across additional dye chemistries is required.

6.3| Measurement Constraints and Environmental Assessment

Fastness evaluation was limited to:

- rubbing fastness (ISO 105-X12), and
- washing resistance using a simulated domestic laundering method.

While these provide reliable indicators of dye performance, the study did not include:

- light fastness (e.g., ISO 105-B02),
- perspiration resistance (e.g., ISO 105-E04), or
- long-cycle washing durability (e.g., ISO 105-C06).

Furthermore, fastness grading was based on visual grey scale assessment (ISO 105-A03/A02). Although widely accepted, this method is semi-quantitative. Instrumental colour measurement (e.g., spectrophotometric reflectance and ΔE values) would improve precision and reproducibility. Environmental impact was not directly quantified. Specifically, no effluent analysis (e.g., Biochemical Oxygen Demand (BOD) or Chemical Oxygen Demand (COD)) was conducted. Therefore, any inference regarding reduced chemical load is indicative rather than empirically verified.

6.4| Artisanal Implementation Context

Although the study is positioned within the context of artisanal and semi-industrial dyeing in Ghana, experimental validation was not conducted under field conditions.
Practical implementation may be influenced by:

- variability in reagent grade and purity,
- water hardness and local water chemistry, and
- manual inconsistencies in mixing and handling

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Field-based trials are required to evaluate the robustness and adaptability of the optimised ratios under real production conditions.

6.5| Confounding of Mixing Sequence and Dye Type

The experimental design assigned specific mixing sequences to individual dye colours (Golden Yellow = D–C–H, Red = C–H–D, Violet = H–C–D). As a result, the effect of mixing sequence is confounded with dye-specific chemical behaviour, making it difficult to isolate sequence effects independently. As a result, the independent influence of mixing sequence on colour fastness cannot be conclusively determined. Any observed differences reflect combined effects rather than isolated sequence behaviour. Future studies should apply a single dye system across all mixing sequences to enable valid comparative analysis.

7 | CONCLUSION AND FUTURE STUDIES

This study examined the role of stoichiometric optimisation in enhancing the colour fastness of vat-dyed cotton fabrics through controlled variation of sodium hydroxide (NaOH) and sodium hydrosulphite ($Na_2S_2O_4$) ratios. The results demonstrate that stoichiometric balance between the reductant and alkali is a primary determinant of redox efficiency, governing dye reduction, fibre penetration, and subsequent oxidation stability. Across the experimental conditions, the ratios 5:1:1 and 2:1:1 (D:C:H) consistently produced superior rubbing and washing fastness. These findings empirically validate the theoretical premise that balanced reactant proportions optimise electron transfer processes in vat dye systems (Maulik et al., 2022; Oriakhi, 2021). In contrast, reagent imbalance, whether through reductant excess or excessive alkalinity, disrupted the redox cycle and reduced dye–fibre bonding efficiency, consistent with established studies (Paul et al., 2017; Rippon et al., 2011). The study demonstrates that stoichiometric control can be operationalised as a practical formulation strategy, offering a reproducible basis for improving dyeing performance. Within the scope of the present design, the mixing sequence did not exhibit a distinguishable effect; however, this observation is constrained by experimental confounding and should be interpreted with caution.

7.1| Practical and Theoretical Significance

Practically, the study provides an evidence-based framework for dyebath formulation using quantitatively defined reagent ratios. Adoption of optimised ratios can enhance process consistency, reduce unnecessary reagent use, and improve fastness performance without requiring complex analytical tools. Theoretically, the research extends the application of stoichiometric equilibrium from abstract chemical systems to applied textile processes, demonstrating its utility as a predictive and diagnostic model for dye–fibre interactions. This reinforces the role of stoichiometry as a governing principle in process optimisation and sustainable textile chemistry.

7.2| Policy and Educational Outlook

The findings support the integration of stoichiometric principles into textile training and practice. Embedding ratio-based formulation methods within vocational and technical education can improve process understanding and reduce reliance on purely experiential approaches. At the policy level, the results provide a basis for developing guidelines that promote efficient chemical use and improved process control in small- and medium-scale dyeing operations. Such integration may contribute to broader efforts toward sustainable textile production and environmental compliance.

7.3| Future Studies

Further research is required to extend and validate the findings of this study:

i. *Extended Fastness Evaluation*: Incorporate additional durability tests, including light fastness (ISO 105-B02), perspiration resistance (ISO 105-E04), and standardised washing protocols (ISO 105-C06), to assess long-term performance.
ii. *Broader Dye and Fibre Systems*: Evaluate the applicability of the optimised ratios across diverse vat dye chemistries and alternative textile substrates.
iii. *Environmental Quantification*: Conduct effluent analysis (e.g., BOD, COD, residual reagent profiling) to quantify environmental impacts associated with stoichiometric optimisation.
iv. *Alternative Reducing Systems*: Investigate bio-based or low-impact reductants as substitutes for sodium hydrosulphite within a stoichiometric framework.

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v. *Field Validation*: Implement controlled trials in artisanal and semi-industrial settings to assess the robustness of the proposed ratios under real production conditions.

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DATA AVAILABILITY STATEMENT

Data sharing upon reasonable request from the corresponding author.

ETHICAL APPROVAL STATEMENT

There is no ethical approval for this study.

CONFLICT OF INTEREST STATEMENT

The authors declare that there are no conflicts of interest related to the research, authorship, or publication of this article.

DECLARATION OF GENERATIVE AI AND AI-ASSISTED TECHNOLOGIES IN THE WRITING PROCESS

During the preparation of this manuscript, the author(s) utilised language and editing software, specifically ChatGPT, Grammarly, and Gemini to enhance the clarity, academic coherence, and rigour of the text, and to reduce possible redundancies. Following the use of these tools, the author(s) reviewed and edited the content as necessary and take(s) full responsibility for the final content of the publication.

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