



Dyeing Recipe Parameters and Fastness Properties of Fabrics Dyed with Artisanal Vat Dyes in Kano Metropolis, Nigeria

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ABSTRACT

Artisanal vat dyeing in historic urban hubs such as Kano metropolis, Nigeria, remains a vital socio-economic enterprise. However, local operations rely heavily on empirical, unstandardized chemical recipes that frequently compromise colourfastness performance and environmental sustainability. This study evaluated the operational dyeing parameters—specifically, temperature, sodium hydroxide (caustic soda) and sodium dithionite (hydros) concentrations, across 18 commercial dyehouses in Kano, assessing their direct impact on colourfastness. Statistical analysis using one-way Analysis of Variance (ANOVA) and Tukey’s HSD post hoc tests was conducted to quantify parameter variability within individual facilities (intra-dyehouse) and between different facilities (inter-dyehouse), benchmarking operational practices against a laboratory-optimized reference baseline. Colourfastness to washing and perspiration were determined according to ISO standard test protocols. Statistical results revealed significant variance ($p < 0.05$) in reagent concentrations both within individual dyehouses across different production batches and between distinct facilities. Field evaluations revealed severe operational chemical imbalances and uncalibrated thermal regimes, resulting in poor washing and perspiration fastness ratings (Grades 2-3) across multiple commercial workshops. Conversely, benchmarking local practices against a laboratory-optimized control recipe elevated washing, acidic perspiration, and alkaline perspiration fastness to maximum Grade 5 ratings. Standardizing local recipes to this optimized baseline cuts caustic soda expenditure by over 50%. It can also reduce thermal energy requirements, conserve process water, mitigate toxic effluent loading and provide a scalable operational framework to enhance the commercial value of Kano textiles for domestic and export markets.

Key words: vat dyeing; occupational dyers; Kano metropolis; caustic soda; hydros; colourfastness, recipe standardization



1. INTRODUCTION

Dyes are organic chromophoric compounds capable of selectively absorbing light radiation in the visible region of the spectrum while reflecting or scattering complementary wavelengths to impart durable colouration onto textile substrates (Berradi *et al.*, 2019). Dyes are either derived from natural bio-resources or synthesized from petroleum feedstocks, differing fundamentally in chemical structure, application methodology, and chemical/physical properties, and application methods (Croce *et al.*, 2017). Based on application mode, synthetic colourants are categorized into acid, basic, direct, disperse, reactive, and vat dyes (Alegbe and Uthman, 2024). Vat dyes are water-insoluble pigments that require chemical reduction into water-soluble leuco-salts to penetrate cellulosic fibres. Upon post-dyeing oxidation, the dye reverts to its insoluble parent form, trapped mechanically and via

intermolecular forces within the cellulose matrix to yield superior colourfastness (Hernandez-Sanchez *et al.*, 2026). Sodium dithionite (commercially termed hydros) is the universal reducing agent of choice due to its strong reducing potential across application temperatures starting from 30 °C (Vuorema, 2008), while sodium hydroxide (caustic soda) serves as the primary alkaline solubilizing agent. Synthetic indigo and indanthrone vat dyes represent the predominant class of industrial textile colourants which are widely used in denim manufacturing, luxury silk batik printing (Behary and Volle, 2025). They are also used for home furnishings and specialized technical textiles requiring high Near-Infrared (NIR) leaf-reflectance camouflage properties (Wardman, 2018; Goudarzi *et al.*, 2014; Wendel, 2015).

The ancient city of Kano has maintained global industrial prominence since the 14th Century through its historical indigo textile production



(Ezeanya-Esiobu, 2019). While traditional pit-dyeing sites (such as the historic Kofar Mata dye pits) have largely declined (Barau, 2007; Abdullahi *et al.*, 2021a), the incorporation of synthetic vat dyes and modern auxiliary chemicals since the 1930s marked a major technological transition within Kano's artisanal dyeing cluster (Byfield, 1994). Contemporary field survey (Abdullahi *et al.* 2021b) indicated that most occupational dyers in Kano metropolis utilize synthetic vat dyes, caustic soda and hydros formulated in an open vessel system using firewood combustion, volumetric dosing with uncalibrated spoons, and empirical temperature estimation by touch. This absence of process control introduces severe technical liabilities. Insufficient hydros concentration causes premature oxidation (causing dye precipitation out of solution) and poor rub/wash fastness. Conversely, excessive hydros dosing induces over-reduction, destroying the dye chromophore, reducing colour yield (Teli *et al.*, 2001), inflating production costs, and

excessively loading dyehouse effluent with hazardous sulphur species (Chakraborty, 1992).

Despite the economic footprint of Kano's cottage dyeing sector, previous academic literature has focused predominantly on powder toxicity (Abdullahi *et al.*, 2016; Sani *et al.*, 2018), or descriptive qualitative survey (Sani *et al.*, 2020; Abdullahi *et al.*, 2021a-c, 2022a-b). There remains a critical lack of quantitative data investigating real-time dyebath kinetics, intra- and inter-facility parameter variance, and their empirical correlation with ISO colourfastness standards. Therefore, the objectives of this study were to: measure real-time operational temperatures, and caustic soda and hydros concentrations across active commercial dyehouses in Kano metropolis, statistically evaluate batch-to-batch variance within single facilities (intra-dyehouse) and across distinct facilities (inter-dyehouse) using ANOVA and post-hoc testing, benchmark empirical dyehouse practices against a laboratory-optimized response surface control model to establish a



standardized, commercially viable dyeing guide for local dyers and industrial entrants, and correlate parameter fluctuations with ISO colourfastness ratings (washing and perspiration). For the purpose of this study, small-scale non-industrial hand-dyeing units are designated as dyehouses, the aqueous chemical mixture containing colourant, alkali, and reductant is termed the dyebath, and the resistance of dyed substrates to physical/chemical degradation is defined as colourfastness.

2. MATERIALS AND METHODS

2.1 Study Area, Sampling Design and Stratification

The field phase of this research was conducted within Kano Metropolis, Northern Nigeria (Latitude 10°-12°N and longitude 8°-9°E). The Metropolitan region comprises six urban Local Governments Areas (LGAs), Dala, Fagge, Kano Municipal, Nassarawa, Gwale, and Tarauni and two peri-urban LGAs, Kumbotso and Ungogo

(Balogun *et al.*, 2020). Serving as Sub-Saharan Africa's preeminent commercial textile hub, local hand-dyeing activities are fueled by heavy market demand from Kantin Kwari, a historical international textile market housing thousands of commercial merchants (Ibrahim, 2015). To obtain a representative cross-section of artisanal vat dyeing practices, a non-probability purposive sampling strategy embedded within a special cluster framework was deployed across 18 active-commercial dyehouses (designated dyehouses A through R) (see sampling sites in Figure 1). Selection was based on 4 strict inclusion criteria: continuous operational use of synthetic vat colourants requiring alkali-reduction systems, exclusive processing of 100% cotton woven fabrics, active commercial status with consistent daily batch production, and consent for real-time dyebath monitoring and chemical sampling. Within each selected facility, random batch sampling was conducted across different operational shifts to evaluate intra-facility process variation, while

comparisons across the 18 facilities yielded inter-facility variance data.

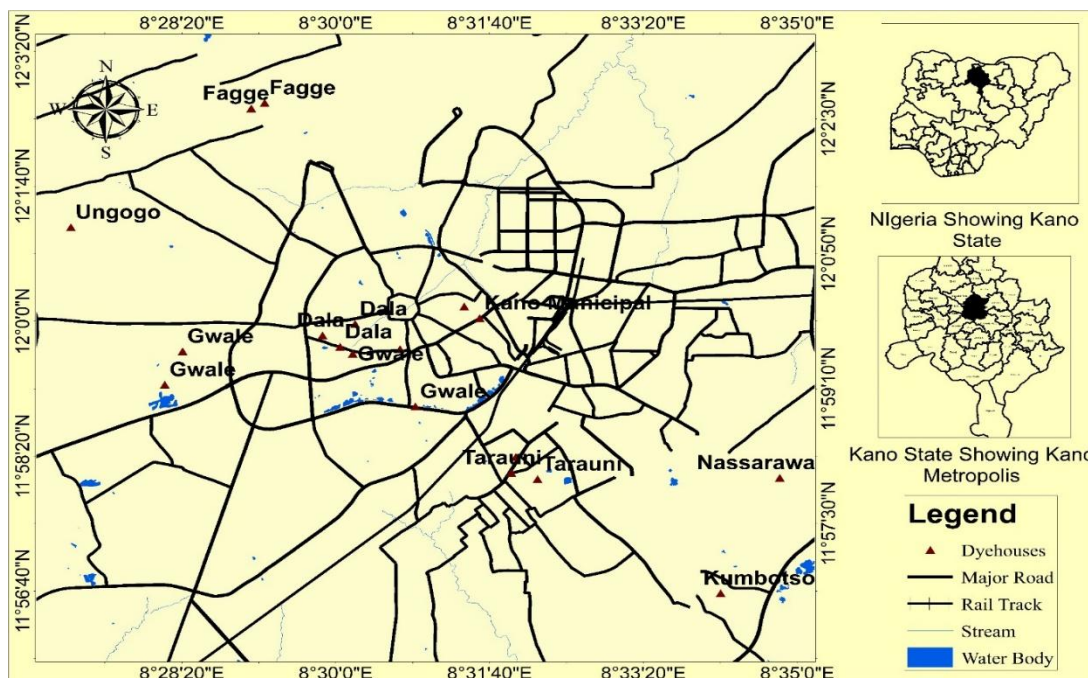


Figure 1: Map of Kano showing the 18 dyehouses sampled (Field work, 2020)

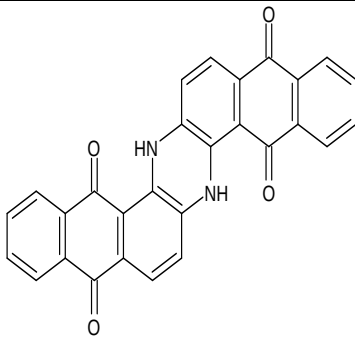
2.2 Materials and Reagents

Commercial grade of sodium hydroxide (99 %, UN No. 1823, Tianya chemicals, China) and hydros (85 %, UN No. 1384, Jinhe hydrosulphite Factory Co. Ltd., China), C. I. Vat Blue 4 (lot No. 101-196, Hangzhou Tiankun Chemical Co. Ltd., China), and hydrogen peroxide were

purchased from Al-Ameen Chemicals, Kano. C. I. Vat Blue 4 was chosen as the target dye because it represents the most frequently used vat colourant in the regional textile market. Undyed, starched 100% plain weave cotton fabric was sourced directly from Kantin Kwari market. The technical specifications of the fabric and dye are summarized in Table 1.

Table 1: Specification of the fabric and dye used in the study

Sample	Weave	Warp density (ends/cm)	Weft density (picks/cm)	Warp yarn count (Tex)	Weft yarn count (Tex)
100 % cotton	Derivative of plain weave	44 Cotton yarns	28 Cotton yarns	14.25	14.5

Blue dye	Commercial name	Colour Index name	Structure	Applicati on class	Chemical class
	Blue RSN	C. I. Vat Blue 4		IW	Indanthrone

2.3 Fabric Pre-treatment: Desizing and

Mercerization

Commercial fabrics in Kano are heavily starched during weaving and require pre-treatments to ensure uniform absorbency. Starch removal was executed by treating 30 cm by 30 cm fabric swatches in an aqueous bath containing 0.5 % hydrogen peroxide (50 % concentration) and 2 % sodium

hydroxide and then treating the fabric (30 by 30 cm) for 5 min., followed by thorough washing and rinsing with distilled water (Nkeonye, 1993). Desized swatches were subsequently mercerized by immersion in 250 g/l sodium hydroxide solution at 85 °C for 3 min. following optimized protocols established by Abdullahi (2023). Mercerized samples were washed thoroughly to neutralize residual alkalinity. Mercerization was integrated into sample



preparation because artisanal dyers in Kano routinely perform caustic pre-treatments; the process converts cellulose I to cellulose II, increasing the proportion of amorphous regions and accessible hydroxyl groups to maximize moisture regain and dye uptake (Wagaw and Chavan, 2012; Khalifa, 2017).

2.4 Determination of Empirical Operational Dyeing Parameters

To determine empirical chemical additions, dyers at each of the 18 sampled dyehouses were provided with 20 g swatches of pre-treated cotton fabric, 2 g of Blue RSN dye (10% shade), and bulk quantities of caustic soda and hydros. Dyers were asked to estimate the chemical quantities required to dye the fabric swatch in 400 ml of water, mimicking their routine operational practice (noting that some dyers calibrate additions relative to dye mass, whereas others calibrate relative to total bath volume). The estimated chemical quantities from each dyer were collected in triplicates (3 batches per dyehouse) in sealed polyethylene bags, weighed

on an analytical balance (with a precision of 0.01 g), and recorded. These triplicate measures provided the data for intra-dyehouse variance evaluation. The collected chemical batches were then returned to the respective dyers to prepare an open dyebath at a liquor-to-goods ratio of 20:1 (400 ml water per 20 g fabric). Immediately following bath preparation, dyebath temperatures were measured using a calibrated digital immersion thermometer. This protocol was executed across all 18 dyehouses to establish inter-dyehouse operational variance.

2.5 Dyeing Execution, Sample Collection and Fastness Testing

Dyeing trials were conducted across all facilities using the pre-measured empirical chemical averages for each respective dyehouse. Pre-treated cotton samples (20 g) were dyed to a 10% depth of shade (2 g dye) for 10 min. in an open stainless-steel vessel under real-time temperature tracking. The dyebaths were prepared by dissolving dye, caustic soda, and



hydros in water and stirring with a stainless-steel spatula for 30 sec. prior to inserting the fabric. Upon completion of the 10 min. immersion, dyed samples (labelled SDA through SDR for dyehouses A through R) were removed, thoroughly rinsed in cold running water, air oxidized to restore the insoluble pigment structure, and line-dried in the shade. Dyed swatches were evaluated for colourfastness to washing and acidic and alkaline artificial perspiration, wet treatments being the most common agencies that result in the fading of dyed articles in the study area (Abdullahi, 2023), according to standard protocols as follows:

Wash fastness: The specimen (10 cm× 4 cm) was covered on both sides with two pieces of undyed outer-lining white cotton and wool fabrics (5 cm× 4 cm) and the three pieces stitched round the edges leaving 5 cm×4 cm exposed. Wash fastness test was carried out using the ISO-3 test under the following conditions: washing with 5 g/l soap and 2 g/l sodium carbonate in a LR of 50:1 at 60±2 °C for

30 mins. The composites specimen was removed, rinsed and the components separated and dried. The change in colour of the dyed specimen and the staining of the adjacent cloths were assessed using the appropriate grey scale. wash fastness test was carried out according to ISO 105-C10:2006 (E).

Fastness to perspiration: Samples were prepared as described in wash fastness test and then treated in alkaline and acidic artificial perspiration solutions using the following conditions: thoroughly wetting the composite samples with 0.5 g/l l-histidine monohydrochloride monohydrate, 5 g/l sodium chloride, 2.5 g/l disodium hydrogen orthophosphate dihydrate and adjusting the pH to 8 or 5.5 using 0.1 Normal sodium hydroxide or acetic acid, respectively, in a LR of 50:1 for 30 mins. at room temp. The treated samples were placed between 2 glass plates under a weight of 4.5 kg for 4 hours at 37±2 °C (body temperature), removed, dried at 60 °C and then the change in colour and degree of staining of



under-lining fabrics assessed as in wash fastness. Fastness to perspiration was carried out according to ISO 105-E04:2013.

2.6 Optimized Control Recipe and Literature Discrepancy

To evaluate local empirical practices against scientific baselines, a control reference was established using Response Surface Methodology (RSM) with the aid of central composite design optimized by Abdullahi (2023). The optimized control parameters used to dye the control sample in this study were: dyeing using 20 g/l hydros, 10 g/l caustic soda, at 60 °C for 10 min. in a liquor-to-goods ratio of 20:1 in an open vessel. Standard textile literature (Aspland, 1992a-b, 1997; Chakraborty, 2010; Lacasse and Baumann, 2004; Broadbent, 2001) classifies C. I. Vat Blue 4 under the IW (indanthrene warm/hot) application class, which typically recommends dyeing temperatures between 40 °C and 50 °C, vatting for 10 min. and dyeing for 45 min. to 1 hour, with specific salt additions in closed or semi-enclosed

industrial machines. However, classical industrial parameters cannot be directly applied as a benchmark for Kano artisanal dyehouses because local dyers operate entirely in an open pot. Therefore, the RSM-optimized open-pot dyed-control was selected as the sole valid benchmark to evaluate local occupational practices.

2.7 Statistical Analysis

All experimental data were analyzed using Minitab Statistical software (Minitab® version 19.11). Data distributions were first evaluated for normality using the Shapiro-Wilk test (and visual assessment of normal probability plots), while equality of variances across dyehouses was confirmed using Levene's test. One-way analysis of variance (ANOVA) was subsequently conducted to evaluate significant variance across sample means for dyebath temperature, sodium hydroxide concentration, and hydros concentration. Tukey's Honest Significant Difference (HSD) post-hoc test ($\alpha=$



0.05) was applied for pairwise mean comparisons to isolate significant intra-, and inter-dyehouse variations. The following hypothesis were tested:

Hypothesis 1

H₀₁: There is no significant difference in the mean operating conditions (hydros concentration, caustic soda concentration, and dyeing temperature) among the sampled dyehouses.

3. RESULTS AND DISCUSSION

3.1 Field Survey and Statistical Variance of Operational Parameters

Across the 18 sampled dyehouses, hydros concentration ranged from 15.33 g/l to 21.67 g/l with an overall mean of 18.99 g/l. The overall mean temperature was 76.39°C with mean

H₁₁: There is a significant difference in at least one of the mean operating conditions among the sampled dyehouses.

Hypothesis 2

H₀₂: The mean local operating conditions do not differ significantly from the optimized control conditions of 20 g/l hydros, 10 g/l caustic soda, and 60 °C dyeing temperature.

H₁₂: The mean local operating conditions differ significantly from the optimized control conditions of 20 g/l hydros, 10 g/l caustic soda, and 60 °C dyeing temperature

temperature ranging from 68.66 °C to 82.59 °C.

Caustic soda concentration spanned a wide operational range (15.47-33.39 g/l) with overall mean of 21.43 g/l. All the 3 operational parameters investigated highlight severe empirical inconsistency across Kano metropolis (See Table 2).

Table 2: Operating dyeing parameters among the occupational dyers

S/No.	Dyehouse	Parameter (Mean±SD)		
		Hydros (g/l)	Caustic Soda (g/l)	Temperature (°C)
1	DA	16.58 ± 0.34 ^{acd}	15.47 ± 0.37 ^b	79.60 ± 0.40 ^a
2	DB	21.40 ± 1.11 ^b	21.58 ± 0.32 ^a	75.62 ± 0.38 ^{bc}
3	DC	19.52 ± 1.51 ^{abcd}	21.53 ± 0.37 ^a	79.60 ± 0.40 ^a
4	DD	20.74 ± 0.47 ^{ab}	21.59 ± 0.41 ^a	74.63 ± 0.38 ^b
5	DE	20.64 ± 1.06 ^{ab}	22.45 ± 0.32 ^a	68.66 ± 0.35 ^d
6	DF	21.67 ± 0.26 ^b	22.67 ± 0.38 ^a	72.64 ± 0.37 ^e
7	DG	19.61 ± 1.25 ^{abd}	21.42 ± 1.13 ^a	75.62 ± 0.38 ^{bc}
8	DH	19.15 ± 0.55 ^{abcd}	21.65 ± 0.38 ^a	77.61 ± 0.39 ^{fg}
9	DI	19.55 ± 0.80 ^{abd}	21.76 ± 0.77 ^a	72.64 ± 0.37 ^e
10	DJ	19.58 ± 0.32 ^{abd}	21.33 ± 0.23 ^a	76.62 ± 0.39 ^{cf}
11	DK	20.53 ± 0.48 ^{ab}	20.90 ± 0.38 ^a	68.66 ± 0.35 ^d
12	DL	21.57 ± 0.40 ^b	21.59 ± 0.35 ^a	74.63 ± 0.38 ^b
13	DM	17.20 ± 1.21 ^{acd}	21.28 ± 0.50 ^a	79.60 ± 0.40 ^a
14	DN	16.56 ± 0.44 ^{acd}	18.19 ± 0.51 ^{ab}	79.60 ± 0.40 ^a
15	DO	19.48 ± 3.83 ^{abcd}	33.39 ± 6.80 ^c	78.61 ± 0.40 ^{ag}
16	DP	15.33 ± 1.54 ^c	20.05 ± 0.50 ^{ab}	82.59 ± 0.41 ^h
17	DQ	16.65 ± 2.15 ^{acd}	20.38 ± 1.28 ^{ab}	82.59 ± 0.41 ^h
18	DR	16.11 ± 1.59 ^{cd}	18.57 ± 1.40 ^{ab}	75.62 ± 0.38 ^{bc}
Mean± SD		18.99±2.05	21.43±3.46	76.39±4

Means within a column sharing at least one superscript letter are not significantly different. This is much easier to interpret than listing all 153 pairwise comparisons. The mean values obtained are from 3 batches per dyehouse.

according to Tukey's HSD test at $p < 0.05$. The superscript letters are the Tukey HSD grouping based on the uploaded pairwise results.

One-way Analysis of Variance (ANOVA) revealed statistically significant differences ($p < 0.001$) across the 18 sampled commercial dyehouses (DA-DR) for all three evaluated parameters: dyebath temperature, caustic soda

concentration and hydros concentration (see Table 3). Field measurements confirmed that 100% of the monitored artisanal facilities rely on empirical estimation using uncalibrated spoons rather than gravimetric measurement. This absence of volumetric standardization leads



to severe batch-to-batch operational instability. Therefore, the null hypothesis that there was no significant difference in the mean operating conditions among the dyehouses was rejected. The alternative hypothesis was accepted, indicating that the mean operating conditions differed significantly among the sampled dyehouses. The Tukey's HSD results shown in Table 4 provide further insight into the significant ANOVA results. Overall, the Tukey's HSD analysis is the primary inferential tool as it controls the family-wise error rate across all pairwise comparisons (including the control). The test confirms that the dyehouses do not follow a uniform set of operating conditions. Temperature exhibited the greatest degree of between-dyehouse variation, followed by caustic soda and then hydros concentration. Differences in these operating conditions may contribute to differences in dyeing performance and process efficiency.

The one-sample t-test results presented in Table 5 show that concentration of hydros had a local

mean of 18.99 g/l, compared with the optimized value of 20.0 g/l, representing a difference of 1.01 g/l (5.04%) below the optimized level. This difference was statistically significant ($t_{53} = -3.210$, $p = 0.002$), indicating that the observed local hydros concentration was significantly lower than the optimized control value. The largest deviation was observed for caustic soda concentration (Table 5). The mean local concentration was 21.43 g/l, compared with the optimized concentration of 10.0 g/l, giving a mean difference of 11.43 g/l, equivalent to a 114.33% higher concentration than the optimized level. This difference was highly statistically significant ($t_{53} = 22.839$, $p < 0.001$). The result indicates that the sampled dyehouses were using substantially more caustic soda than specified under the optimized condition. Dyeing temperature also differed substantially from the optimized condition. The local mean temperature was 76.39 °C, compared with the optimized temperature of 60°C, representing an average increase of 16.39 °C or 27.32%. The



difference was highly significant ($t_{53} = 30.150$, $p < 0.001$). Thus, the prevailing local practice involves operating at considerably higher temperatures than the optimized condition. Consequently, the null hypothesis that local operating conditions did not differ significantly from the optimized control conditions was rejected. The findings demonstrate that the current local practices deviate significantly from the optimized recipe, with the largest deviation observed in caustic soda concentration, followed by dyeing temperature, while hydros concentration was comparatively close to the optimized level.

Table 3: One-way analysis of variance (ANOVA) of dyehouse operating conditions

Operating parameter	Grand mean	df Between	df Within	F-value	p-value	Decision
Hydros conc. (g/l)	18.99	17	36	6.711	<0.001	Significant
Caustic soda conc. (g/l)	21.43	17	36	12.038	<0.001	Significant
Temperature (°C)	76.39	17	36	334.740	<0.001	Significant

One-way ANOVA was conducted at 5% significance level. The mean values obtained are from 3 batches per dyehouse

Table 4: Tukey's HSD post-hoc comparison of dyehouse operating conditions

Operating parameter	Number of significant pairwise comparisons	Most notable significant differences	Interpretation
Hydros conc. (g/l)	27	Dyehouse 16 vs 6: -6.34 g/l ($p < 0.001$); dyehouse 16 vs 12: -6.24 g/l ($p < 0.001$); dyehouse 16 vs 2: -6.07 g/l ($p < 0.001$); dyehouse 18 vs 6: -5.56 g/l ($p = 0.002$)	Significant differences occurred among several dyehouses, particularly those with relatively low hydros concentrations compared with dyehouses 2, 6 and 12.
Caustic soda conc. (g/l)	29	Dyehouse 15 vs 1: 17.91 g/l ($p < 0.001$); dyehouse 15 vs 14: 15.20 g/l ($p < 0.001$)	Dyehouse 15 was particularly distinct, recording substantially higher caustic soda concentration than many other

		0.001); dyehouse 15 vs 18: 14.82 g/L ($p < 0.001$); dyehouse 15 vs 16: 13.34 g/l ($p < 0.001$)	dyehouses.
Temperature (°C)	125	Dyehouse 5 vs 1: −10.95°C ($p < 0.001$); dyehouse 11 vs 1: −10.95°C ($p < 0.001$); dyehouse 5 vs 16: −13.93°C ($p < 0.001$); dyehouse 11 vs 17: −13.93°C ($p < 0.001$)	Temperature showed extensive pairwise differences, indicating very strong variation in operating temperature among the dyehouses.

Significant differences are based on Tukey's HSD adjusted p-values at $\alpha = 0.05$. A total of 153 pairwise comparisons were possible for each parameter (18 dyehouses).

Table 5: Comparison of local operating conditions with the optimized control recipe

Parameter	Local mean	Optimized control	Mean difference	Deviation (%)	95% CI	t (53)	p-value
Hydros (g/l)	18.992	20.0	−1.008	−5.04	18.363– 19.622	−3.210	0.002
Caustic soda (g/l)	21.433	10.0	+11.433	+114.33	20.429– 22.437	22.839	<0.001
Temperature (°C)	76.394	60.0	+16.394	+27.32	75.303– 77.485	30.150	<0.001

The one-sample t-test compares the mean local operating conditions with the optimized control value. Statistical significance was assessed at 5% level.

3.2 Dyebath Kinetics and Thermochemical Over-Saturation Mechanics

Field observations revealed a widespread practice of maintaining excessive alkalinity (above 20 g/l) under uncontrolled thermal conditions (above 70 °C) over open wood fires.

Thermodynamically, while an alkaline environment ($\text{pH} > 12$) is mandatory to generate the water-soluble leuco-enolate anion (Chakraborty, 1992) of C. I. Vat Blue 4, exceeding 10 g/l caustic soda introduces an excess of sodium counter-ions into the dyebath.



These surplus cations compress the electrical double layer surrounding the cellulose fibres, suppressing electrostatic repulsion and triggering premature ionic screening and leuco-dye aggregation (Chakraborty, 2010). Additionally, over-reduction occurs when excessive concentration of hydros is used at elevated temperatures. Over-reduction increases as the concentration of caustic soda used is increased up to the point where it is such as to promote the crystallization of the leuco compound (Brassard, 1943). The pH level of the dyebath solution in vat dyeing process is high by the presence of caustic soda that there should be an electrostatic repulsion force between anionic leuco vat dye and cellulose fibre which would show negative charge in the surface of the fibre in strong alkali solution (Kim *et al.* 2013) resulting in surface aggregation. In woven cotton yarns, these large molecular aggregates cannot diffuse into the sub-microscopic pores of the fibre matrix, accumulating instead on the

outer yarn sheath, a phenomenon known as “ring dyeing”.

Furthermore, large amounts of highly concentrated caustic soda embedded on mercerized cellulose which would invariably go into the dyebath and saturate it with sodium ions, could have reduced the solubility of the leuco vat dye. Yoe and Edgar (1922) have similarly shown that the higher the caustic soda concentration in the dyebath, the lower the concentration of the reduced dye as hydroxyl ion favours the rate of solution while Na^+ retard it. Thermal spikes (70 to 80 °C) accelerate the oxidative decomposition of hydros in open vessels (Broadbent, 2001), dropping the redox potential below the critical -750 mV threshold required for complete reduction (Chakraborty, 1992). This causes the leuco-dye to re-oxidize prematurely into insoluble pigment within the liquid bath prior to fibre absorption. This kinetic behaviour underscores the vital role of substrate morphology. While literature regarding electrospun regenerated cellulose nanofibers



(Khatri *et al.*, 2017) reports high colour yields and fastness at 20 g/l caustic soda due to the fully accessible, non-woven nanofibrillar surface area, conventional woven cotton requires a strictly calibrated 10 g/l caustic soda baseline. The dense, twisted yarn structure of woven fabrics makes them highly susceptible to ionic salting-out, surface precipitation, and fastness failure.

3.3 Comparative Fastness Evaluation and Benchmarking

The physical consequences of operational imbalances are evident in the ISO colourfastness evaluations. Samples produced across commercial dyehouses (DA-DR) yielded poor-to-moderate wash fastness (ISO 105-C10) and perspiration fastness (ISO 105-E04) ratings of Grades 2-3 (see Table 6). Because the dye in these samples resides predominantly as surface-bound clusters, mechanical agitation and detergent action during testing strip the pigment easily, causing severe staining on adjacent test fabrics. Over-reduction chemically alters the dye

chromophore, often permanently damaging the conjugated double-bond system or modifying the amine/carbonyl groups (Chakraborty, 2010; Broadbent, 2001; Teli *et al.*, 2001; Vuorema, 2008). During washing, these modified molecules easily bleed out of the fibre, leading to massive colour loss and severe staining. Additionally, the over-reduced chemical species are chemically unstable. Exposed to acidic (pH 5.5) or alkaline (pH 8.0) synthetic sweat under warm conditions (37 °C), the altered dye easily desorbs and bleeds onto neighboring fibres.

Benchmarking local practices against the RSM-optimized laboratory control recipe (20 g/l hydros, 10 g/l caustic soda and dyeing at 60 °C) elevated wash, acid perspiration and alkali perspiration fastness to maximum Grade 5 ratings. In open-air, non-insulated vessels without salt additions, the dyebath experiences rapid atmospheric oxygen ingress and thermal dissipation. In an open vessel, rapid surface aeration causes atmospheric oxygen to consume sodium dithionite much faster than in enclosed



machinery, hence more hydros will be needed to maintain a stable negative redox potential.

Also, under open-pot atmospheric exposure, a temperature of 60 °C without salt addition provides the necessary kinetic energy and reduction stability to maintain leuco-solubility while compensating for continuous surface re-oxidation (Fox, 1948; Abdullahi *et al.*, 2021c). Standardizing alkalinity to 10 g/l maintains a sufficiently

high pH (>12.5) for complete solubilization while preventing sodium ion screening. Paired with 20 g/l hydros to scavenge surface atmospheric oxygen in open pots, single

monomolecular leuco-anions diffuse completely into the inner amorphous core and lumens of the cellulose fibres. Subsequent air oxidation permanently locks the re-crystallized indanthrone pigment within the fibre matrix, delivering maximum durability. Table 7 shows cluster comparison of local operational parameters and fastness ratings benchmarked against control. While washing and perspiration fastness directly measure dye stripping under aqueous/ionic stress, future work will quantify mechanical surface abrasion via ISO 105-X12.



Table 6: Fastness ratings of samples from occupational dyers to washing and perspiration

Sample	Wash fastness		Perspiration			
	Colour change	Staining	Acid		Alkaline	
			Colour change	Staining	Colour change	Staining
DA	3	3	4	3	3	3
DB	2	2	2	2	2	2
DC	2	2	2	2	2	2
DD	2	2	3	2	2	2
DE	2	2	2	2	2	2
DF	2	2	2	2	2	2
DG	2	2	3	2	2	2
DH	2	2	2	3	2	2
DI	2	2	2	2	2	2
DJ	2	2	2	2	2	2
DK	3	2	3	2	3	3
DL	2	2	3	2	2	2
DM	2	2	2	3	2	2
DN	3	3	3	4	3	3
DO	2	2	3	2	2	2
DP	3	2	2	3	3	2
DQ	3	3	3	3	3	3
DR	3	3	4	3	3	3

Grey scale ratings for wash fastness: 5-Excellent, 4-Good, 3-Fair, 2-Poor, 1-Very poor. Grey scale ratings for staining: 1-Much change, 2- Considerable change, 3-Noteceable change, 4-Slight change, 5-Negligible change. Grey scale for light fastness test: 1-Very poor, 2-Poor, 3-Moderate, 4-Faily good, 5-Good, 6-Very good, 7-Excellent, 8-Outstanding

Table 7: Cluster comparison of local operational parameters and fastness ratings benchmarked against control

Statistical cluster	Included dyehouses	Mean caustic soda range (g/l)	Mean temperature range (°C)	Wash Fastness (ISO 105-C10)	Alkali perspiration (ISO 105-E04)	Dominant kinetic mechanism
Group A (severe excess)	DB, DC, DD, DE, DF, DG, DH, DI, DJ, DL, DM, DO	21.28-33.39	68.66-79.60	Grade 2	Grade 2	Severe Na ⁺ screening→ heavy ring dyeing
Group B (moderate excess)	DK, DP, DQ	20.05-20.90	68.66-82.59	Grades 2-3	Grades 2-3	Moderate aggregation+ thermal hydros loss
Group C (low-end empirical)	DA, DN, DR	15.47-18.19	75.62-79.60	Grade 3	Grade 3	Sub-optimal reduction→ surface pigment buildup
Optimized control baseline	Laboratory reference	10.0	60.0	Grade 5 (max.)	Grade 5 (max.)	Monomolecular diffusion→ core trapping

3.4 Economic, Environmental, and Policy

Implications for Kano Metropolis

Adopting the 10 g/l caustic soda optimized baseline provides immediate economic, environmental, and institutional benefits for Kano's artisanal dyeing sector. Standardized local recipe cuts caustic soda expenditure by over 50%, significantly lowering chemical

overhead and improving profit margins for small-scale dyers. Reducing alkali loading, however, lowers the total dissolved solids (TDS), chemical oxygen demand (COD), and alkalinity of untreated wastewater discharged into municipal drainages, protecting local soil and groundwater ecosystems. Reducing the liquor-to-goods

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ratio by half (using 20:1) also reduces the effluent load. Elevating the fastness properties from Grade 2-3 to Grade 5 could transform Kano-dyed textiles from domestic craft items into high-value, export-grade materials suitable for international markets across West Africa.

CONCLUSION

This study establishes a quantitative baseline on occupational parameters and fastness properties of artisanal vat dyeing in Kano metropolis, Nigeria. The findings confirm that local dyers rely on uncalibrated, empirical protocols, resulting in significant chemical and thermal imbalances, specifically excessive caustic soda usage (21.43 g/l mean) and thermal overheating (76 °C mean). These uncontrolled conditions induce premature sodium dithionite decomposition and superficial dye aggregation, directly degrading fabric wash and perspiration fastness down to unsatisfactory ratings

(grades 2-3). Standardizing local recipes to the optimized laboratory baseline (10 g/l caustic soda, 20 g/l hydros, 60 °C) and 20:1 (liquor-to- goods ratio) removed unfixed surface pigment, elevating all fastness properties to maximum commercial standards (grades 5). Furthermore, maintaining a strict 20:1 liquor- to-goods ratio might have suppressed the oxidative breakdown of hydros by minimizing dissolved oxygen, while simultaneously conserving process water and reducing effluent loading. Adopting this standardized regime cuts caustic soda expenditure by over 50% (from >20 g/l to 10 g/l), offering immediate economic benefits for local dyehouses. To sustain these technical gains, relevant educational authorities should incorporate Artisanal Textile Chemistry and Dyeing Technology into the vocational and technical education (VTE) curricula of higher institutions to train dyers on precise volumetric and stoichiometric process



control. Additionally, promoting a strategic transition from synthetic dyers to locally cultivated natural indigo will reduce reliance on imported colourants, lower toxic wastewater impact, and position Kano

textiles for high-value, eco-certified international markets.

CONFLICT OF INTEREST

The author declared that there is no conflict of interest with regards to the paper.

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