
UPSCALING SUSTAINABLE COLOURATION: DESIGN, FABRICATION, AND PERFORMANCE EVALUATION OF AN INDUSTRIAL-SCALE NATURAL INDIGO EXTRACTION MACHINE FOR NIGERIA'S TEXTILE SECTOR

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ABSTRACT

The reliance of Nigeria's textile and cottage dyeing industries on imported synthetic colourants poses severe environmental hazards, while the importation of natural indigo dye from Europe and China continues to drain valuable foreign exchange reserves. To drive sustainable import substitution and foster local industrialization, this study presents the engineering design, fabrication, and physical performance evaluation of an indigenous, large-scale natural indigo extraction machine. Response surface methodology (RSM) based on a central composite design (CCD) was initially employed at bench scale to model process kinetics. Optimizing plant biomass fermentation (18 hours) and aeration duration (63 minutes at pH 12) predicted a maximum yield of 31.99 g/kg fresh biomass with a purity of 70.35%. Building upon these kinetic baselines, a mechanical extraction system was engineered to scale up the fermentation, liquid separation, and aeration phases into a controlled, high-efficiency mechanical process. Validation trials conducted across five operational runs processing 100 kg of fresh biomass per batch produced 3.21±0.7 kg of dry indigo flakes (equivalent to 32.13±0.7 g/kg biomass yield) with a purity of 71.12±1.3%, within 24-hour cycle. By replacing toxic synthetic dyes and bypassing expensive foreign colourant imports, this fabricated extraction system provides scalable, eco-friendly technological framework to revitalize Nigeria's historic textile clusters, directly supporting sustainable agricultural value addition and national economic self-reliance. (Note: In the first abstract submitted, the criteria set in the optimization software was "in range" for both fermentation time, pH and oxidation time and the predicted values were 26 hours fermentation time, pH 12, and 105-minutes oxidation time. After careful consideration, looking at Table 2, the highest crude yield was obtained at 18 hours fermentation time. Also, previous work reported that there is tendency for the fermented liquor to be contaminated after 24 hours fermentation thereby affecting pure yield. For this reason, the criteria for the fermentation time was changed from "in range" to "equals to 18". The software predicted a result where a combination of 18 hours fermentation time, pH 12 and oxidation for 63 minutes will result 31.99 g/kg and 70.35% crude and pure yield respectively as shown in Figure 3. It is a known fact that the ramp shown in the Figure 3 cannot be edited. It was copied directly from the software. With this development, large scale extraction using the fabricated machine had to be carried out using the new predicted parameters which resulted in different yields from the ones reported in the earlier abstract)

Keywords: indigo extraction; machinery fabrication; import substitution; sustainable development; natural dyes; textile engineering

INTRODUCTION

Accelerated depletion of fossil fuels and escalating environmental degradation necessitated intensified research into sustainable, renewable bio-resources (Cardon *et al.*, 2023). In the textile sector, global initiatives led by the European Union (EU) and international sustainability frameworks have actively sought to reintroduce efficient indigo-bearing crops into agricultural systems to develop scalable processes for natural dye production (Shin *et al.*, 2009; Mocquard *et al.*, 2022). Cultivating natural indigo (*Indigofera tinctoria*) supports sustainable agriculture by fixing atmospheric nitrogen restoring soil health in crop rotations, and yielding residual mass suitable for bio-fertilizers and biogas production, directly advancing Sustainable Development Goals (SDG) 12 (responsible consumption and production), SDG 13 (Climate Action) and SDG 15 (Life on Land) (Rao *et al.*, 2025). However, deploying natural dyes at an industrial scale requires overcoming key engineering bottlenecks,

particularly improving extraction kinetics and machinery efficiency to ensure economic viability (Pizzicato *et al.*, 2023).

Historically, natural indigo dyeing has been a prominent socio-economic enterprise across Northern Nigeria, centered in prominent urban hubs such as Kano, Sokoto, Zaria, and Bida (Akinbogun and Ogunduyile, 2009). Kano in particular, established regional prominence as early as the 14th century through its fine indigo-dyed textile products at traditional pit sites like Kofar Mata (Barau, 2007; Ezeanya-Esiobu, 2019P). Presently, most of the indigo dyers are out of business with only about 10 out of the 144 dye pits of “Kofar Mata” still functional (Abdullahi *et al.*, 2022). Despite this heritage, local dyeing operations have suffered a major decline, with remaining dyers relying on imported natural indigo powder from China, India, and Europe at exorbitant costs, reaching up to 350,000 per 25 kg container (personal communication). Local agricultural surveys in Kano state reveal an abundant, underutilized supply of *Indigofera tinctoria* biomass, with over 52 local farms producing an estimated 56,800 kg of fresh biomass per harvest cycle (Field work, 2022). Processing this local crop at an achievable 2% extraction yield could generate 1,136 kg of high-purity dye biannually (corresponding to 90.88 of 25 kg indigo bags), keeping nearly ₦32,000,000 (US\$26,666.8) within the local economy.

Despite this agricultural potential, traditional bio-fermentation methods remain unviable for commercial scaling due to labour-intensive manual handling, slow liquid separation, uncalibrated aeration dynamics, and low overall dye recovery. Translating bench-scale chemical kinetics into pilot-scale production requires solving fluid mechanical mass transfer, and mechanical agitation challenges inherent in larger volumetric capacities. In alignment with the conference theme, “Innovating Tomorrow; Harnessing Engineering to Drive Nigeria’s Growth for Sustainable Development”, and specifically addressing the sub-theme “Sustainable Engineering Processes and Systems”, this study bridges the gap between laboratory optimization and industrial implementation. By transitioning traditional bio-fermentation into automated, low-energy mechanical process, this research demonstrates how cleaner production technology can be localized. The primary objective of this research is to present the mechanical design, structural fabrication, and operational performance evaluation of an indigenous, inductive-scale natural indigo extraction machine engineered as a sustainable system to revitalize Nigeria’s textile sector.

MATERIALS AND METHODS

Materials

Plant material (leaves and stems) of *Indigofera tinctoria* as identified by a taxonomist (Department of Biological Sciences, Ahmadu Bello University, Zaria), 3 months old (August to October), was harvested on 25th of October, 2023 from a farm in Yan Barau, Dawakin Kudu local government area of Kano State (see Figure A1 and A2, Appendix A), longitude 8.63° E and latitude 11.85° N with an altitude of 448 m above sea level. The plant biomass was cultivated under the following conditions: average daily air temperature, soil temperature, soil moisture, and rainfall were respectively 30.1 °C, 26.2 °C, 0.265 m³/m³ (28-100 cm), and 600 mm. Concentrated sulphuric acid, concentrated ammonia and standard synthetic indigo dye were purchased from Sigma Aldrich and used without purification. Coordinates of the farm were determined using hand-held Garmin78SC GPS receiver and pH was measured using Digital pH meter (Jenway 3310, UK).

Methods

Extraction of indigo dye

Fresh plant material with the leaves and stem (2 hours after harvest), 1 kg, was washed twice and placed in a transparent polyethene container and weigh down with 5 kg block to completely submerge the material in 10 l of water. The pH was adjusted to 3.5 using 20% concentrated sulphuric acid (this is to prevent indican from converting to indoxyl prematurely during fermentation). The container was covered to maintain anaerobic condition for fermentation to take place at different times. After a specified period of time, the pH of the fermented broth was measured, the solution was filtered and the pH was adjusted using concentrated ammonia (indoxyl forms indigo dye at an alkaline pH). The solution was then oxidized for a certain period of time using a blower at a flowrate of 3 l/min

(Figure A3, Appendix A). The solution was kept for 24 hours. to allow indigo slurry to settle at the bottom. The yellow green supernatant was gently drained (Figure A4, Appendix A) while the slurry was filtered using a 0.074 µm mesh (ASTM standard), placed on a tray (Figure A5, Appendix A) and allowed to dry at room temperature overnight in a cool dry place. After drying, the dye pellets were ground in to powder and washed with 2M HCl and then finally air dried.

Extraction experiments were designed with the aid of response surface methodology (RSM) using central composite design (CCD) which generated 15 runs in random order. The CCD is shown in Table 1. Crude dye (Figure A6, Appendix A) yield was calculated using Eq. 1. Crude indigo dye obtained from the 15 experiments is shown in Figure A7, Appendix A. To obtain the pure yield (dye content), standard synthetic indigo dye, 8 mg, was dissolved in 20 ml of concentrated sulphuric acid and then diluted to 500 ml with distilled water. The solution was then diluted to different concentrations viz., 0.1, 0.2, 0.4, 0.6, 0.8, 1.0 and 1.2 mg/l and the absorbance was measured at 610 nm to plot a standard curve. Crude indigo dye powder, 1 g, was dissolved in 20 ml concentrated sulphuric acid and diluted to 500 ml and the absorbance measured at 610 nm. From the standard curves, the pure yield (%) was calculated (Chanayath *et al.*, 2012). The data was analyzed using Design Expert® Software version 7 (DX7). After statistical analysis and optimization of extraction parameters using Desirability function, five sets of experiments were carried out at the optimized conditions to validate the results.

$$Crude\ yield\ \left(\frac{mg}{g}\right) = \frac{w1 - w2}{weight\ of\ the\ plant\ material} \dots \dots \dots Eq. 1$$

where w1=weight of tray containing dried indigo dye and w2= weight of empty tray.

Table 1: Central composite design of extraction parameters

Variable	Unit	Level	
		Low (-1)	High (+1)
pH	-	9	13
Oxidation time	Minutes	30	120
Batching time	Hours	18	72

Fabrication of indigo extraction equipment

The equipment was designed to suit the purpose of the extraction process for indigo and other natural dyes. The extraction equipment was constructed using stainless steel and consists of 9 components including 2 steeping/soaking tanks, 1 oxidation tank, 1 filtration tank, and 2 evaporators, 2 condensers and a steam boiler. The whole equipment (Figure B1, Appendix B) is controlled by an electric panel. Each of the steeping tanks is of 1,000 litres capacity and is equipped with mechanical presser (diameter= 1.16 m, height= 1.22 m) whose purpose is to weigh down (submerge) the plant material completely inside water (see Figure B2, Appendix B). Direct steam can be injected into the tank through by a steam blower at a rate of 100 kg/hr. to raise temperature where necessary. The steeping/soaking tank is connected to an oxidizing tank via a 25 mm pipe in which the fermented broth is transferred with the aid of 1 HP pump.

The oxidizing tank is fitted with a stirrer controlled by a 1 HP electric gear motor which turns at 360 rpm (see Figure B3, Appendix B). At the bottom of the tank is a root blower controlled by a 3 HP electric motor. Valve, 25 mm, is fixed at the outlet of the blower to control the flowrate. For a batch process, liquid is siphoned off through an upper outlet and the sedimented indigo at the bottom is collected via a tap attached to the bottom of the oxidizing tank and a mesh just beneath the tap. For continuous process, the oxidized solution is pumped into a filtration tank where 3 filter cartridges trap the dye particles under pressure. The trapped dye is then collected and passed to the evaporators (equipped with a stirrer) to separate indigotin and indirubin. Steam is also passed through the evaporator to dry the final product. The solvent used to dissolve the crude indigo is recovered by the condensers. The flowchart showing the steeping and oxidizing tanks is shown in Figure 1.

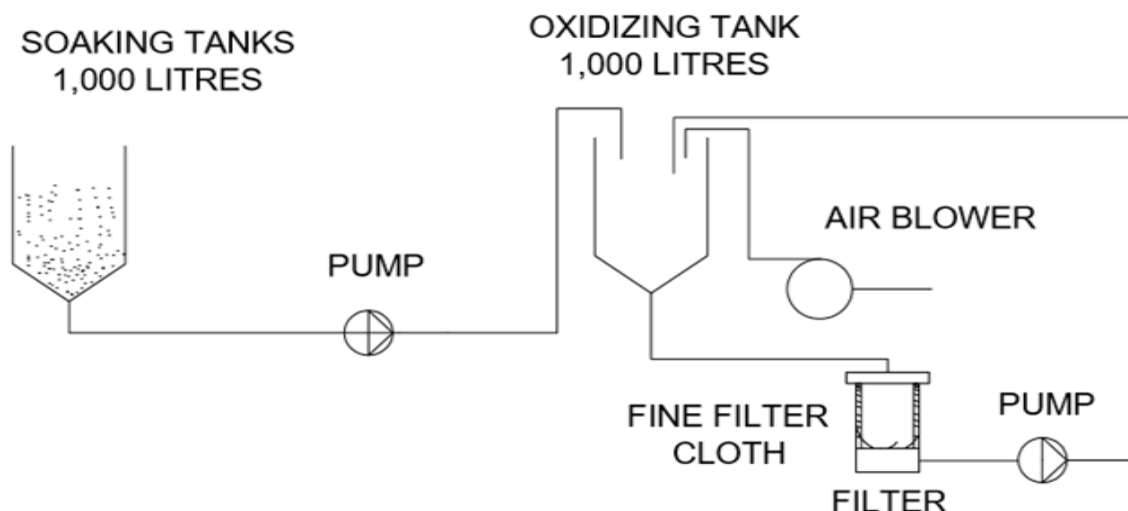


Figure 1: Flowchart showing soaking and oxidizing tanks

RESULTS AND DISCUSSION

Extraction and Yield of Indigo Dye

The extraction procedure was a modification of the method described by Stoker et al (1998) who used similar extraction technique to produce indigo on a large, commercial scale, from *Isatis tinctoria* leaves. Indigo precursor in *Indigofera tinctoria* is indican. The precursors are water soluble and on hydrolysis easily release free indoxyl which on aeration readily form indigo which is insoluble and therefore sedimentable. The identity of the extracted indigo was confirmed by UV-Visible spectroscopy, infrared spectroscopy, mass spectroscopy as well as proton and carbon nuclear magnetic resonance spectroscopy (not shown). Central composite design (containing 5 levels of each factor viz. low and high factorial, low and high axial, and centre) was used to fit a model, develop regression equations, and then optimize key factors influencing the quantity and quality (crude and pure yield) of the dye produced. The results obtained for crude and pure yield from different combinations of fermentation period as well as pH and oxidation time (Table 2).

Table 2: Crude and pure yield obtained from different combinations of fermentation time, pH and oxidation time

Run No.	Batching time (hr.)	pH	Oxidation time (min.)	Crude yield (g/kg)	Pure yield (%)
1	45	11	68	15	44.1
2	45	11	120	19	49.7
3	45	13	68	17	45.3
4	45	9	68	11	30.0
5	45	11	15	14	40.1
6	64	10	105	4	25.3
7	72	11	68	4	24.1
8	18	11	68	31	68.5
9	45	11	68	13	48.9
10	45	11	68	14	38.9
11	64	12	30	6	26.8
12	45	11	68	14	42.8
13	26	12	105	30	69.8
14	26	10	30	23	44.6
15	45	11	68	20	43.8

The highest crude yield, 31.0 g dye per kg of plant biomass (3.1%), was obtained when the batching/fermentation time was 18 hr. and where the pH was adjusted to 11 before oxidation for 68 min. The lowest yield, 4 g/kg, was obtained when the fermentation period was 64 hrs. (oxidation for 105 min. at pH 10) and 72 hrs. (oxidation for 68 min. at pH 11). The yield decreases with increase in fermentation time but increases with both increase in pH and oxidation time. Fermentation time for *Indigofera tinctoria* was suggested to be 15 to 16 hrs. as longer extraction time leads to contamination of the steep water with plant-derived materials (Pattanaik *et al.*, 2018; Pattanaik *et al.*, 2020a-b, Pattanaik *et al.*, 2021). The yield measured in this study, 3.1%, appears reasonable since John and Angelini (2023) reported that exceptional samples of *Indigofera tinctoria* might yield indigo to a value of 3.5%. Mussak and Bechtold (2009) also reported that traditional method of producing indigo has a maximum yield of 37 g/kg dry weight of leaf. Stoker et al (1998a) obtained a crude yield of 20 g/kg on a pilot scale from *Isatis tinctoria* leaves.

The purest sample among the 15 runs in the experiment has a purity of 69.8% which was obtained after 26 hrs. fermentation and oxidation for 105 min. at pH 12. The pure yield also shows the same trend as crude yield where lowest yield was obtained when the fermentation period was 64 hrs. (yield was 24.26.8%). The yield also increases with increase in pH and oxidation time but decreases with increase in fermentation period. Synthetic indigo is usually higher than 90% pure while natural indigo is 20-90% pure where a good sample of Bengal indigo was 61.4% (Perkin, 1900). Dutta et al (2017) obtained highest pure yield after 24 hrs. of fermentation. Natural indigo dye from *Indigofera tinctoria* plant biomass is believed to provide the highest purity of indigo dye (Pattanaik *et al.*, 2018). John (2004) obtained an average purity of 74±10% with an average yield of 0.5g/kg fresh leaves. Stoker et al (1998) achieved a pure yield of 20-40% from *Isatis tinctoria* leaves. Washing raw indigo in 2 M HCl after grinding the dried product could have improved the purity of the extracted dye. Dramatic increase in purity has been obtained from 3 large scale preparations of indigo by a wash with dilute HCl from 21-71%, 0.6-63%, and from 19-72% (John, 2004). Indigo can be extracted from woad with a purity of 90% if the leaves contain sufficiently high yield of indigo precursors and the leaves are rinsed free of soil (John, 2004; Garcia-Marias and John, 2004).

Model Fitting and Statistical Analysis

Analysis of variance (ANOVA) for the responses “crude yield” and “pure yield” was generated by Design Expert Software version 7 (DX7). Lack of fit shown in Tables 3 for both crude and pure

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yield compares residual error to the “pure error” from replicated design points. The selected model should have insignificant lack of fit. Lack of fit in this case is not significant and therefore the model can be used as a response predictor. The linear models selected exhibits low standard deviation, high coefficient of determination (94.72% and 93.69% respectively for crude and pure yield) and low PRESS. As shown in Tables 3, the model F-values imply that the selected models are significant. Values of “Prob>F” less than 0.05 will indicate that the model terms are significant.

Table 3: Analysis of variance (ANOVA) test for significance for crude yield and pure yield

Statistical Metric	Response 1: Crude yield	Response 2: Pure yield
Suggested model type	Linear	Linear
Model <i>p</i> -value	0.0001	0.0001
Ordinary R ²	0.9472	0.9369
Adjusted R ²	0.9328	0.9197
Predicted R ²	0.9190	0.8802
Lack of Fit (<i>p</i> -value)	0.8995	0.4373
Adequate Precision	26.321	24.373
Standard deviation	2.11	3.85
PRESS	75.24	309.67

$$\text{Crude yield} = +15.67 + 2.19 \cdot A + 1.51 \cdot B - 10.15 \cdot C \dots \text{Eq. 2a}$$

$$\text{Pure yield} = +42.85 + 6.04 \cdot A + 4.66 \cdot B - 15.64 \cdot C \dots \text{Eq. 2b}$$

For both responses, the predicted determination coefficient (Pred R²=0.9190 and R²=0.8802 for crude and pure yield respectively) are in reasonable agreement with the adjusted determination coefficient (Adj R²=0.9328 and R²=0.9197 for crude and pure yield respectively) which is satisfactory to confirm the fitness of the model. The “Adeq Precision” ratios (26.321 and 24.373 for crude and pure yield respectively) indicates an adequate signal which implies that model for the responses could be used to navigate the design space. The negative sign in front of the terms in a first order regression model indicates antagonistic effect whereas a positive sign shows synergistic effect. The responses, crude and pure yield, increase with an increase in oxidation pH and oxidation time but decrease with increase in batching/fermentation time. There is however, no interaction among the extraction parameters investigated. Interaction occurs when the effect of one factor on the response variable depends on the level of another factor (Anderson and Whitcomb, 2007). The relationship between crude and pure yields with oxidation pH and time as well as fermentation time established in this study (Eq. 2a and 2b respectively in coded form) can be used as a model for indigo dye extraction from *Indigofera tinctoria* biomass.

The consistency of the data for responses “crude yield” and “pure yield” was confirmed from normal probability plot of residuals shown in Figures 2a and 2b respectively. In both cases, the points are fairly close to a straight line indicating the validity of the experimental data since the residuals are normally distributed. As shown in Tables 4, the value of residuals (difference between experimental data and the predicted value) is low showing the precision of the experiments (Wagaw and Chavan, 2012; Myers *et al.*, 2009).

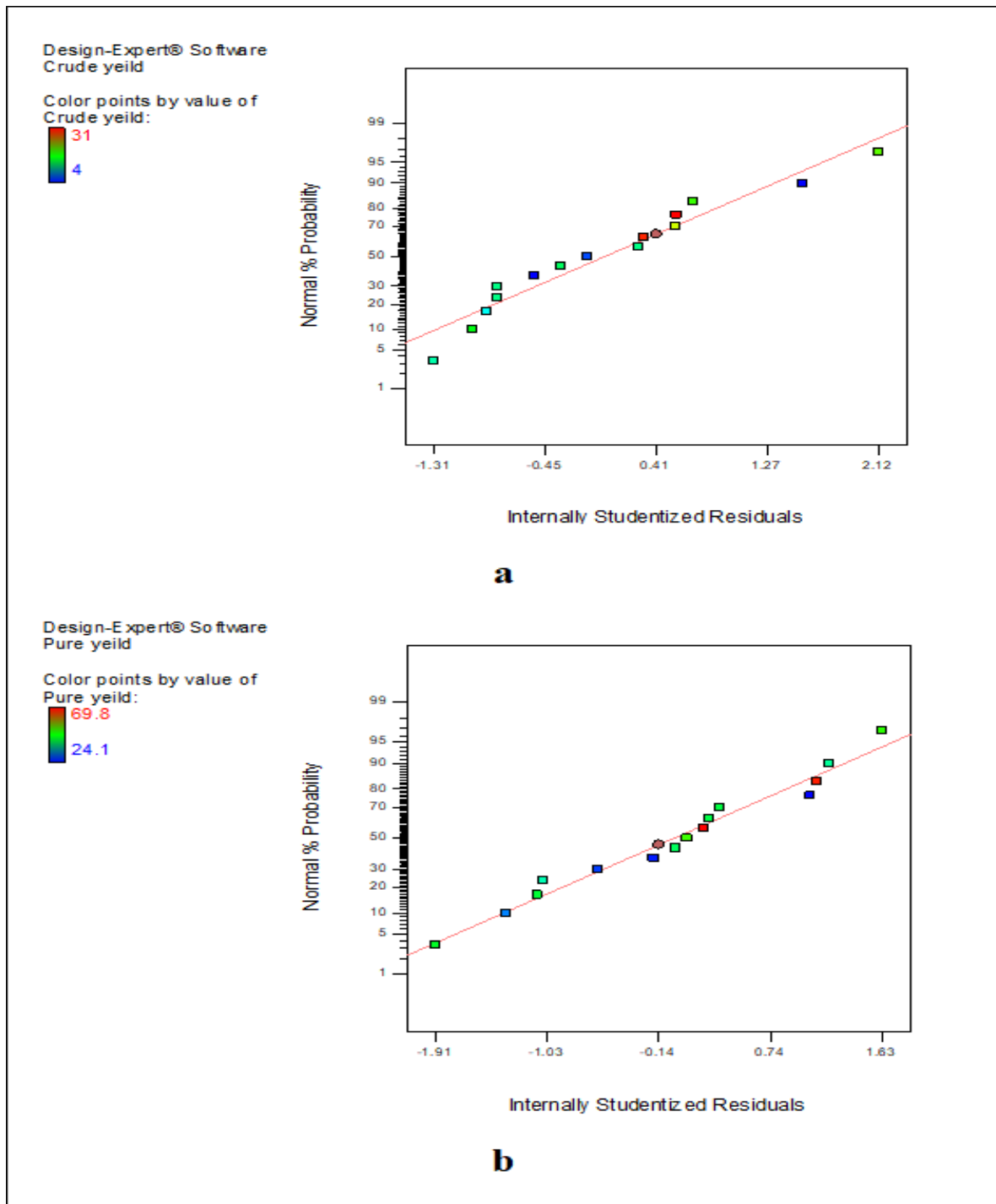


Figure 2: Normal probability plot of residuals (a) crude yield (b) pure yield

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Table 4: Diagnostic case statistics showing residuals for crude and pure yield

Crude yield				Pure yield			
Actual value	Predicted value	Residual	Run number	Actual value	Predicted value	Residual	Run number
30.00	29.51	0.49	13	69.80	69.18	0.62	13
6.00	6.20	-0.20	11	26.80	28.59	-1.79	11
4.00	4.84	-0.84	6	25.30	25.83	-0.53	6
23.00	22.12	0.88	14	44.60	47.78	-3.18	14
11.00	12.58	-1.58	4	30.00	34.30	-4.30	4
17.00	18.76	-1.76	3	45.30	51.39	-6.09	3
14.00	13.53	0.47	5	40.10	36.26	3.84	5
19.00	17.80	1.20	2	49.70	49.44	0.26	2
31.00	30.02	0.98	8	68.50	64.96	3.54	8
4.00	1.32	2.68	7	24.10	20.73	3.37	7
14.00	15.67	-1.67	10	38.90	42.85	-3.95	10
20.00	15.67	4.33	15	43.80	42.85	0.95	15
13.00	15.67	-2.67	9	48.90	42.85	6.05	9
14.00	15.67	-1.67	12	42.80	42.85	-0.047	12
15.00	15.67	-0.67	1	44.10	42.85	1.25	1

Optimization of Extraction Parameters for Large Scale Production

The objective of this optimization is to simultaneously make crude and pure yield as high as possible. In numerical optimization, desired goal is chosen for each factor and response. A multiple response method was applied for optimization of combinations of three factors namely oxidation pH, oxidation time and batching time. The 2 responses optimized were average crude and pure yield. The criterion was set as follows: “in range” for pH, “equals to 18” for batching time (because 18 hours batching time resulted in the highest crude yield and fermentation for more than 24 hours results in microorganisms contaminating the liquor affecting pure yield), “in range” for oxidation time, “maximize” crude yield, and “maximize” pure yield. Figure 3 shows the ramp of optimal parameters for crude and pure yield. The dot on each ramp reflects the factor setting or response prediction for that solution. Batching for 18 hrs. and oxidizing for 63 min. at pH 12 will result in 31.99 g/kg crude yield and 70.35% pure yield with a desirability of 1.000. The optimum conditions predicted were checked experimentally on a large-scale extraction using 100 kg of plant biomass 5 times. The experimental values obtained for crude and pure yield (for five replicates) using the predicted values of the extraction parameters was respectively 32.13 ± 0.7 g/kg and $71.12 \pm 1.3\%$. Small errors between these values indicated that the experimental values obtained were in good agreement with the values predicted from the models. Physicochemical parameters of indigo dye extraction including fermentation conditions such as temperature and time (Rahayuningsih *et al.*, 2022; Syafaatullah and Mahfud, 2020; Dutta *et al.*, 2017; Hidayati *et al.*, 2018) as well as oxidation conditions comprising of oxidation pH, oxidation time, and air flowrate (Pattanaik *et al.*, 2021) has been optimized previously using design of experiment to maximize indigo yield.

(Note: The software predicted the runs with combination of factors based on the information we provided in Table 1 as shown in Table 2. The columns crude and pure yield were originally empty with no values. After conducting 15 batch experiments and obtaining the samples shown in Figure A7, the crude and pure yields of each of the samples was determined and placed in the columns. It was after feeding the software with the results that statistical analysis and optimization were possible to obtain the predicted and optimized results).

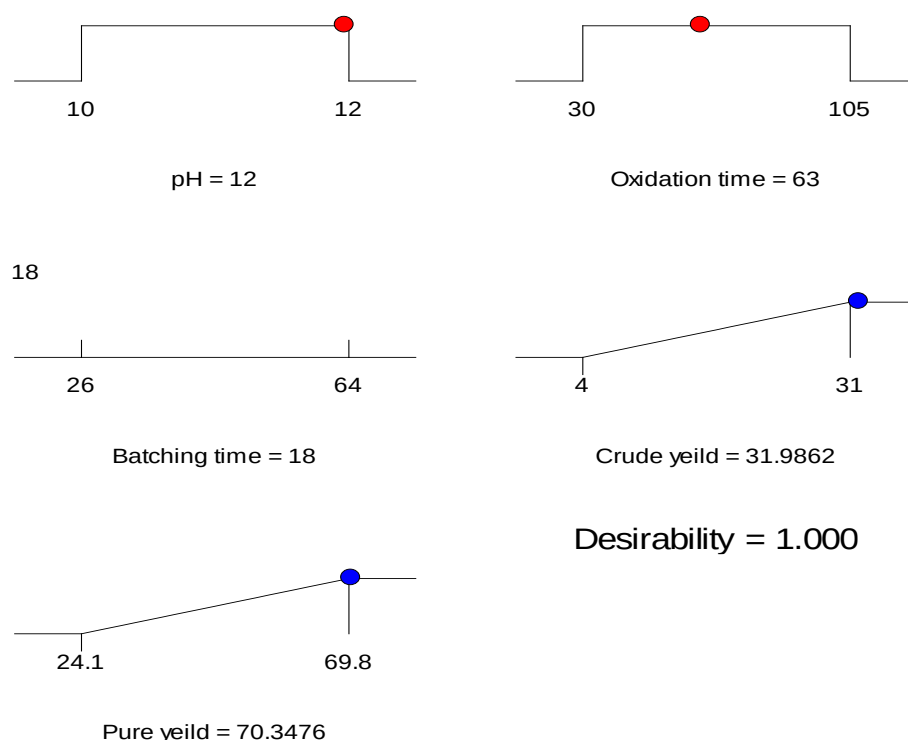


Figure 3: Ramp of optimal conditions for crude and pure yield

CONCLUSION

This study successfully designed, fabricated, and evaluated an inductive-scale natural indigo extraction machine, bridging the critical scale-up gap between laboratory-optimized bio-chemical kinetics and commercial pilot production. Tested across five consecutive validation runs under optimized operating conditions (18 hours fermentation, 63 minutes oxidation, and pH 12), the machine demonstrated exceptional operational fidelity and mass transfer efficiency. The inductive-scale unit achieved a crude pigment yield of 32.13 g/kg biomass and a pure yield of 71.12% (this progresses towards 90% purity obtainable with synthetic indigo and also the 90% purity sought by the European Union under sustainable production of plant-derived indigo), confirming that scaling up from bench parameters (31.99 g/kg crude yield and 70.35% pure yield) was accomplished without yield loss or kinetic degradation. The mechanical agitation, aeration dynamics, and volumetric mass transfer efficiency of the fabricated unit proved highly effective for pilot-scale processing. Furthermore, utilizing locally available engineering materials for construction demonstrates that sustainable industrial processing equipment can be manufactured cost-effectively within Nigeria, significantly reducing capital expenditure compared to foreign machinery imports. Industrially, this scalable extraction framework offers a viable technical pathway to commercialize natural indigo cultivation, substitute synthetic colourant imports, mitigate toxic effluent loading, and stimulate agrarian-industrial growth. Future iterations will focus on integrating automated pH monitoring and continuous-flow aeration controls.

It is recommended to incorporate a finer mesh than the one used in the fabricated equipment as this may likely enhance crude indigo yield. Indigo yield depends on climatic conditions which vary from season to season. It is therefore imperative to investigate seasonal variations and harvest time which could lead to changes in indigo yield. Indigo seed should be genetically modified and grown in the farms or in close proximity according to the principles of Good Agricultural and Collection Practices (GACP) which recommends on-farm production and post-production processes to address environmental, economic, and social sustainability. Considering the irregular power supply in the

country especially in remote areas like farms, solar panels are recommended to be installed on the equipment to have uninterrupted power supply which is mainly needed for the transfer of the fermentation broth from steeping to the oxidation tank and for blowing air into the oxidation tank.

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APPENDIX A

Extraction of indigo dye from plant biomass



Figure A1: Indigo farm at the point of harvest



Figure A2: Indigo farm 2 months after harvest



Figure A3: Oxidation of fermented solution during batch experiments





Figure A4: Residual fermented broth after oxidation in batch experiments



Figure A5: Indigo slurry drying on a tray



Figure A6: Dried indigo pellets



Figure A7: Samples obtained from batch extraction experiments

APPENDIX B

Large scale extraction of indigo dye using the fabricated equipment

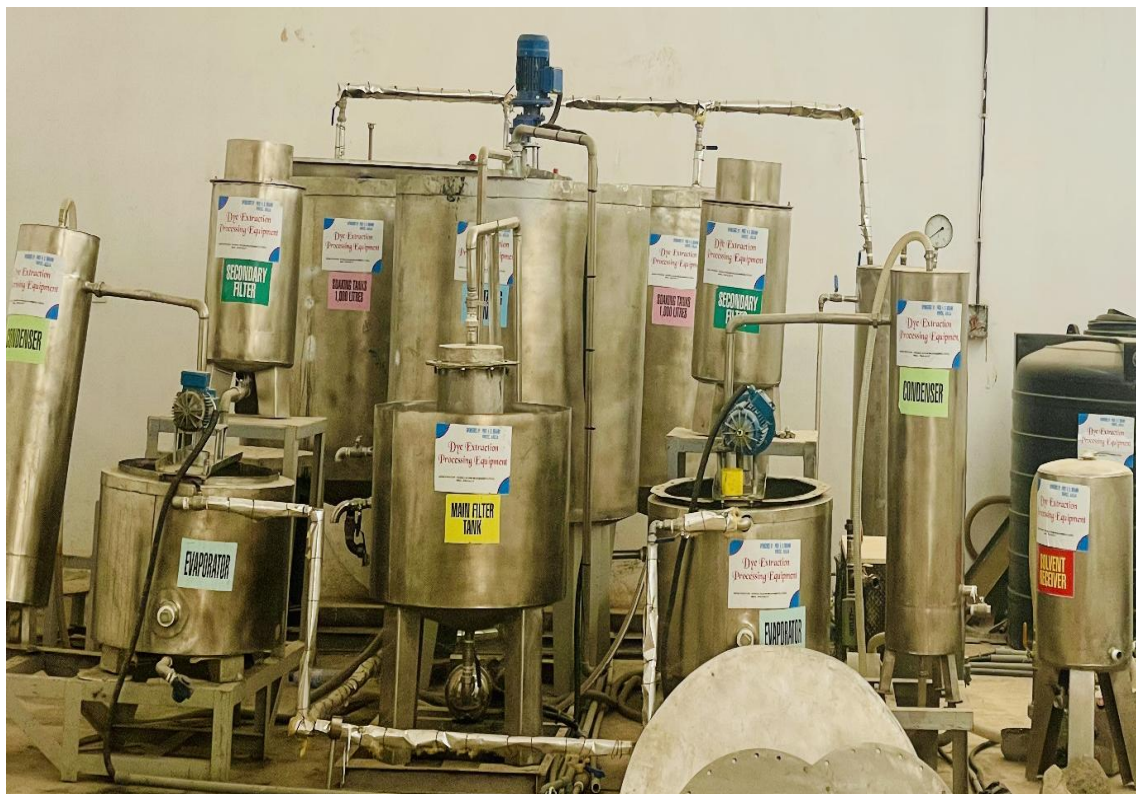


Figure B1: Fabricated indigo dye extraction equipment



Figure B2: Steeping/fermentation of plant biomass in the steeping tank



Figure B3: Oxidation of fermented broth in the oxidation tank