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Sustainable Silk Yarn Dyeing using *Hibiscus rosa-sinensis*: Optimisation of Process Variables via Response Surface Methodology Approach

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Natural dyes offer an eco-friendly alternative to synthetic dyes, aligning with sustainable textile practices. This study aimed to evaluate the potential of *Hibiscus rosa-sinensis* flower extract as a natural dye for silk yarn and to optimise its dyeing parameters. *H. rosa-sinensis* natural dye was extracted using an acidified (4% citric acid) water extraction method, which was used to dye the silk yarn. The dyeing parameters, including pH, temperature, weight/volume ratio, and dyeing duration were evaluated using Response Surface Methodology (RSM). The optimum dyeing conditions were determined at pH 3, 30 °C, for 30 minutes, and a weight/volume ratio of 0.06 g/mL, resulting in a colour intensity of 30.35. The experimental outcomes were consistent with the predicted colour intensity of 32.05, confirming the reliability of the optimised parameters. The study further explored the effects of pH on the hue of the dyed yarn, showing a vibrant red at pH 3, which gradually shifted to greenish-brown at higher pH. The UV-visible spectrometry confirmed the stability of the anthocyanin pigments at lower pH levels. The findings demonstrate the potential of *H. rosa-sinensis* as a sustainable dye and provide insights into optimising parameters for natural dyeing processes. Future research could expand on using other mordants and explore co-pigmentation strategies to enhance dye stability and colour properties.

Keywords: *Hibiscus rosa-sinensis*; Response Surface Methodology (RSM); silk yarn; natural dye

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Dyeing process involves an interaction between a dye and a fibre, where the dye molecules penetrate the fibre structure, resulting in the production of coloured fabrics and textiles [1]. This process is critical in textile manufacturing as it provides colour with varying degrees of permanence and fastness. The ability to achieve a diverse palette of colours and patterns on various materials underscores the significance of dyeing in the textile industry. The quality and performance of dyeing agents significantly impact the durability of colour and, consequently, the overall quality of the dyed product. Dyeing agents can be generally classified into two categories: natural and synthetic dyes, each offering unique benefits and challenges in practical applications.

Natural dyes, derived from renewable sources, such as minerals, plants, animals, and microorganisms have been used in textile production across diverse cultures, for over 5,000 years. For example, ancient Chinese texts document the use of plant-based dyes from roots and leaves [2], while India's textile history features dyes from indigo and other botanicals, as evidenced by the Indus Valley Civilisation [3]. In ancient Egypt, plants like madder and woad were utilised to colour linen [4], and indigenous South America communities traditionally used dyes from animals and plants such as cochineal and annatto in textile art [5, 6]. Despite the richness of sources such as roots, leaves, stems, flowers, and fruits, the use of natural dyes declined with the advent of synthetic dyes due to their limitations in reproducibility and colour fastness [7].

H. rosa-sinensis, commonly known as the Chinese hibiscus, is an ornamental plant from the Malvaceae family, valued not only for its aesthetic appeal but also for its medicinal properties. Traditionally, the plant extracts have been used to treat various ailments, such as wounds, inflammation, fever, cough, diabetes, and infections due to their antioxidant, antimicrobial, and anti-inflammatory compounds [8-11]. Beyond medicinal uses, *H. rosa-sinensis* has demonstrated potential as a natural dye

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due to the presence of anthocyanins, which produce red to purple hues depending on pH [12, 13]. Several studies have demonstrated the effectiveness of its flower extract as a dyeing agent. Hayat and Jacob [12] extracted a red pigment using 5% acetic acid and dyed cotton and wool fabrics, which demonstrated colour fastness with the aid of metal salt mordants. The chemical analysis by Mejía et al. [13, 14] confirmed the presence of cyanidin-sophoroside and other anthocyanins in the petals, which are responsible for its distinctive pH-sensitive colour behaviour. Bronce et al. [15] further highlighted its viability as a food-grade colourant due to high pigment yields (~330 mg/L) and good consumer acceptance. In addition, anthocyanins from hibiscus have demonstrated good wash fastness on natural fibres such as silk, cotton, and wool, making them suitable for sustainable textile dyeing [16].

Despite the widespread acceptance of synthetic dyes in the textile industry due to their vibrant colours and consistency, concerns regarding their environmental impact persist. The production of synthetic dyes often involves hazardous chemicals that pose risks to both human health and ecological systems [17]. As awareness of the non-biodegradable and potentially carcinogenic nature of synthetic dyes grows, the demand for natural dyes is also increasing. Natural dyes, derived from renewable resources and yielding lower ecological footprints across their life cycles are perceived as more environmentally sustainable options. This study aims to investigate the potential of *H. rosa-sinensis* extract as a natural dye on silk yarn and to optimise the key dyeing parameters. Anthocyanins are highly sensitive to pH levels, exhibiting high stability below pH 3 and becomes progressively unstable above pH 4.5 [18]. Hence, an acidic extraction process was employed to ensure the preservation of anthocyanin pigments. Response Surface Methodology (RSM) was utilised to systematically optimise the dyeing parameters, with various conditions evaluated for their effects on colour intensity.

MATERIALS AND METHODS

The 120/2 100% silk yarn was purchased from TCB Batik & Songket Sdn. Bhd, Malaysia. Sodium hydroxide (NaOH) and HCl were purchased from Merck. Citric acid, potassium alum sulphate (alum) and soap nuts were purchased from local suppliers. All chemicals were used without any purification.

Sample Collection

The *Hibiscus rosa-sinensis* flowers were collected from areas surrounding the International Islamic University Malaysia, Kuantan, Pahang, and were identified by a botanist. The voucher specimens (IIUM 520) were deposited in the Herbarium, Kulliyyah of Science, International Islamic University Malaysia. To preserve the samples, the hibiscus flowers were subjected to a drying process in an oven at a temperature of 40°C for 24 hours.

Silk Scouring

Soap nut (0.8 g) was soaked in 1.0 L of water for 24 hours. 1 gram of silk yarn was initially immersed in distilled water for 2 hours. Then, the silk yarn was washed in the soap nut for 30 minutes, employing a material-to-liquor ratio of 1:40, and maintaining the temperature at 50°C. The scoured yarn was thoroughly rinsed with distilled water until no more bubbles were observed. The yarn was then kept in a wet condition for further processing.

Mordanting

The scoured silk yarn was pre-mordanted using 0.15 g of alum (15 % on weight of fabric, OWF), with a material-to-liquor ratio of 1:40, at a temperature of 50°C for 1 hour. The yarn was then washed three times with distilled water and maintained in a wet state for subsequent procedures.

Response Surface Methodology (RSM)

The experimental design and analysis of parameters were conducted using the statistical software package Design-Expert 13.0.5. A Central Composite Design (CCD) was employed to explore the impacts of pH, temperature, time, and hibiscus weight/volume on the colour intensity of silk dyed with hibiscus. A four-factor and four-level central composite design, resulting in 28 experimental runs, was utilised to optimise the dyeing parameters, with the specific parameter variations (Table 1). The output variable for the study is the colour intensity, calculated as the average of four measurements. The outcome of the result focused on determining the main effects of individual factors, as well as examining the interactions between these factors. Analysis of regression and variance (ANOVA), and graphical optimisations were performed to identify the optimal conditions for the process variables.

Table 1. The variability in parameters for the dyeing process generated by Response Surface Methodology (RSM).

Run	Temperature (°C)	pH	Time (min)	Weight/Volume (g/mL)
1	30	3	30	0.02
2	30	3	30	0.06
3	30	3	120	0.02
4	30	3	120	0.06
5	30	9	30	0.02
6	30	9	30	0.06
7	30	9	120	0.02
8	30	9	120	0.06
9	70	3	30	0.02
10	70	3	30	0.06
11	70	3	120	0.02
12	70	3	120	0.06
13	70	9	30	0.02
14	70	9	30	0.06
15	70	9	120	0.02
16	70	9	120	0.06
17	50	6	75	0.02
18	50	6	75	0.06
19	50	6	30	0.04
20	50	6	120	0.04
21	50	3	75	0.04
22	50	9	75	0.04
23	30	6	75	0.04
24	70	6	75	0.04
25	50	6	75	0.04
26	50	6	75	0.04
27	50	6	75	0.04
28	50	6	75	0.04

Extraction

The preserved sample was weighed at different weight/volume ratios (0.02, 0.04, and 0.06) as determined by the parameters generated in the Response Surface Methodology (RSM). Each sample was immersed in 50.0 mL of 4% citric acid solution in distilled water for 6 hours at a temperature range of 50–60°C. Throughout the heating process, the beaker was covered with aluminium foil to prevent solvent evaporation. Following the extraction, the solution was left for 24 hours at room temperature. The extract was subsequently filtered to remove residual hibiscus solids. The extract was then filtered using Whatman No. 1 filter paper under gravity to remove the hibiscus residue. The obtained extract was subsequently used for the silk yarn dyeing process.

Dyeing

The silk yarns were dyed following the parameters (pH, temperature, weight/volume and time) generated from the RSM. The pH values were set at 3, 6, and 9, adjusted using a buffer solution of 1.0 M NaOH and 1.0M HCl. The temperature variations tested were 30, 50, and 70 °C, while the dyeing duration spanned 30, 75, and 120 minutes. Following the dyeing process, the dyed yarn was washed thoroughly with distilled water and subsequently air-dried at room temperature.

UV-Visible Spectrophotometry

The hibiscus dye extracted at various pH values (3, 6, and 9) was diluted to a 1:100 ratio with distilled water before being subjected to analysis using the UV-Visible Spectrophotometer (UV-1900i Shimadzu). The spectra were recorded at wavelengths from 400 to 800 nm.

Colorimetric Analysis

The dried dyed silk yarn was examined using a handheld colorimeter to measure the colour intensity in terms of L, a, b values. The CIE Lab* colour space is a colour model defined by the Commission Internationale de l'Eclairage (CIE). It describes colours based on three coordinates: L*, a*, and b*. These coordinates represent lightness (L*), redness-greenness (a*), and yellowness-blueness (b*), respectively. The colour intensity of the dyed silk yarn was measured through C_{ab}, representing the distance of the colour point from the L*-axis, and was calculated using the following equation:

$$C_{ab} = \sqrt{a^2 + b^2}$$

RESULTS AND DISCUSSION

Extraction of *Hibiscus rosa-sinensis*

In this study, the dye pigment from *H. rosa-sinensis* flowers was extracted using an acidified (4% citric acid) water extraction method. The incorporation of citric acid served a dual purpose: it maintained an acidic environment to stabilise the anthocyanins, preventing their degradation, and functioned as a co-pigment, thereby enhancing the colour intensity of the extracted dye [19]. The amount of hibiscus flower material for extraction was varied while keeping the solvent volume constant at 50 mL. Different weight-to-volume (w/v) ratios were tested, including 0.02 g/mL, 0.04 g/mL, and 0.06 g/mL. Among these, the highest ratio of 0.06 g/mL produced a visibly more intense and concentrated colour compared to the lower ratios, indicating a higher yield of extracted anthocyanins.

Maintaining a moderate temperature was critical to preserving the integrity of the pigment throughout the extraction process. Elevated temperatures are known to accelerate anthocyanin degradation due to the increased activity of peroxidase enzymes, which can lead to pigment breakdown [20]. Thus, the extraction temperature was controlled within the range of 50 °C to 60 °C, which should be sufficient to facilitate the release of anthocyanins without risking thermal degradation, and the extraction was carried out for 6 hours at 50–60 °C to obtain a vibrant red-coloured solution (Figure 1).

Alum was chosen as the mordant in this study because of its well-documented ability to form complexes with anthocyanins, facilitating a strong bond between the dye and the silk yarn. This strong chemical interaction enhances the overall colour retention and fastness of the dyed fabric [21, 22]. In addition, alum is known for its non-toxic properties, making it a safer alternative for sustainable textile applications. Although other mordants, such as stannous chloride, were considered, previous research suggests that while stannous chloride can produce high colour intensity, it often causes undesired colour shifts towards purple and results in uneven dye distribution on the fabric [21]. A pre-mordanting method was used in the dyeing process, where the silk yarn was pre-treated by immersing it in a heated alum solution at 60 °C. This technique allowed the mordant to effectively bind to the fibre prior to the dyeing process, thereby enhancing the subsequent uptake of the dye and improving the uniformity of the final colouration.

Figure 1. Hibiscus extract in acidified water.

a) b)

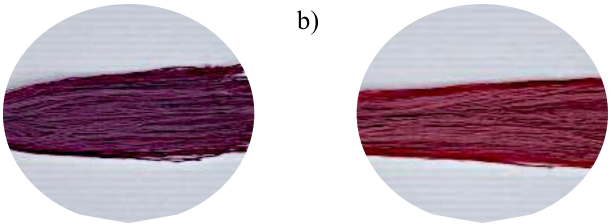


Figure 2. The silk yarn dyed with hibiscus flower extract using a) Stannous chloride ($SnCl_2$) and b) Alum.

Table 2. Effect of pH on colour intensity.

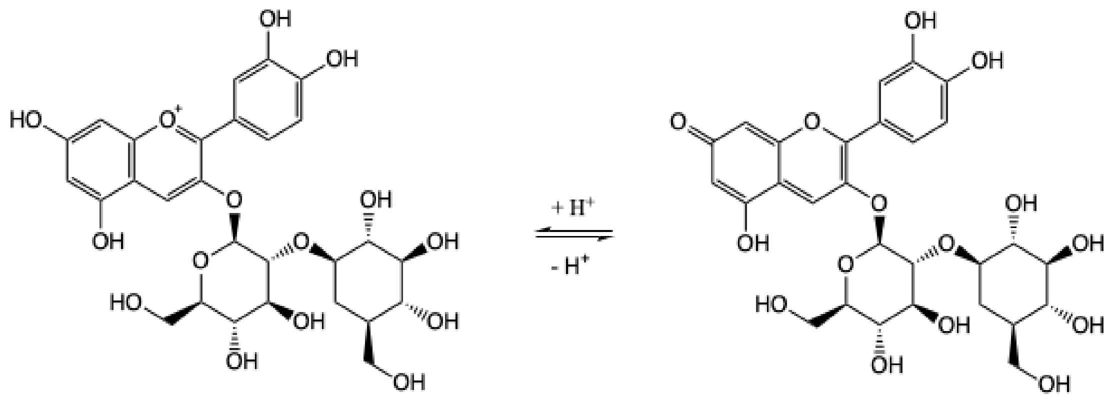
Time (min)	Weight/Volume (g/mL)	Temperature (°C)	pH	Colour intensity	Colour appearance
75	0.04	50	3	26.45	
75	0.04	50	6	14.57	
75	0.04	50	9	11.81	

Dyeing Process of Silk Yarn

The colour intensity and appearance of silk yarn dyed with *Hibiscus rosa-sinensis* extract were found to vary considerably depending on the pH levels of the dye bath. As the pH increased, a noticeable reduction in colour intensity was observed, with the resulting hues shifting from vibrant red at lower pH values to more muted brownish tones at higher pH levels (Table 2).

This phenomenon is primarily attributed to the instability of anthocyanins at elevated pH conditions. At lower pH levels, anthocyanins predominantly exist in their flavylium cation form, which imparts a rich red hue. However, as the pH rises, these pigments gradually convert into less stable structures such as quinoidal bases and other deprotonated forms (Figure 3), leading to a decrease in both stability and colour vibrancy [23, 24].

This behaviour is reflected in the UV-visible spectroscopic analysis of the *Hibiscus rosa-sinensis* pigment, which showed a maximum absorbance (λ_{max}) at 515 nm when the dye bath was maintained at pH 3. However, at higher pH values of 6 and 9, the λ_{max} shifted to a range of 494–497 nm (Figures 4a–4c). These shifts in the λ_{max} values further demonstrate the sensitivity of anthocyanins to pH-induced transformations, reinforcing the importance of maintaining an acidic environment to preserve the desired colour properties during the dyeing process.



Flavylium cation

Quinoidal base

Figure 3. pH-dependent structural transformation of anthocyanins.

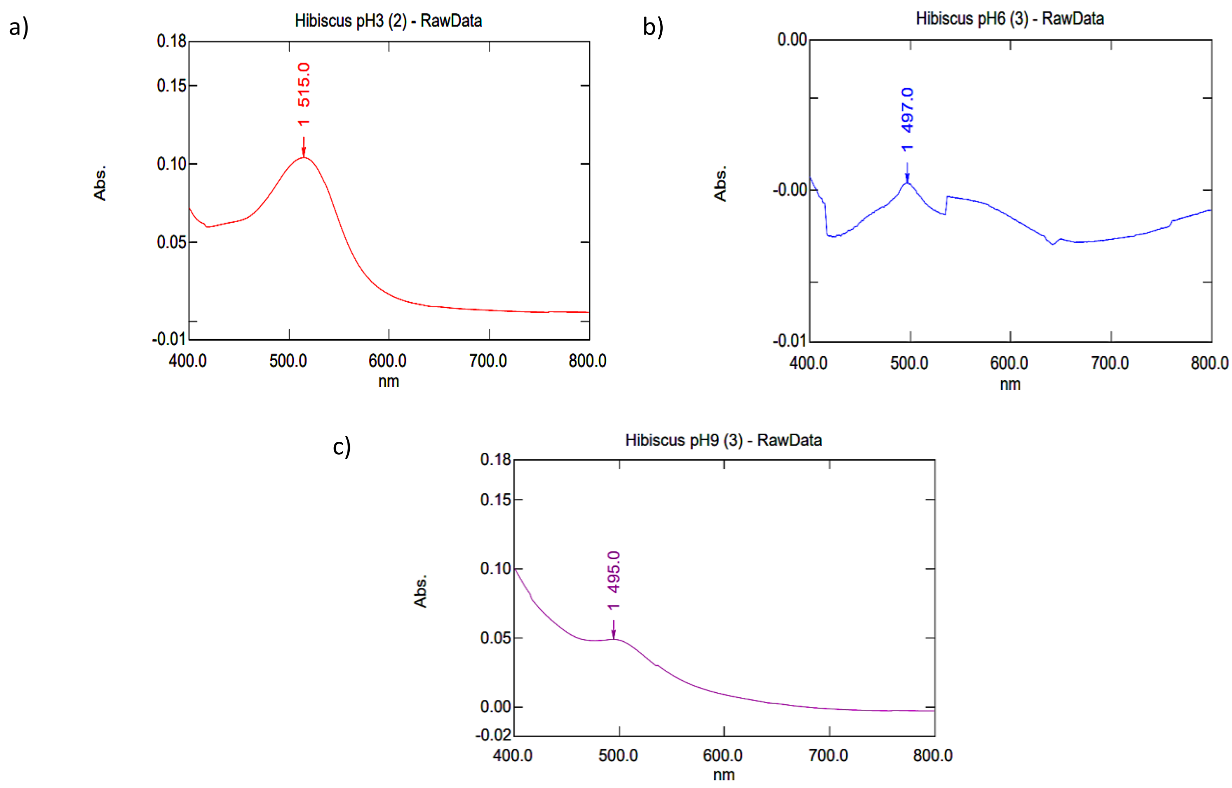


Figure 4. The UV-Visible absorption spectra of anthocyanin at a) pH 3 b) pH 6 c) pH 9.

Table 3. Effect of weight/volume on colour intensity.

Time (min)	pH	Temperature (°C)	Weight/Volume (g/mL)	Colour intensity	Colour appearance
120	3	30	0.02	22.39	
120	3	30	0.06	30.35	

Table 4. Effect of time on colour intensity.

pH	Weight/Volume (g/mL)	Temperature (°C)	Time (min)	Colour intensity	Colour appearance
6	0.04	50	30	14.32	
6	0.04	50	75	13.80	
6	0.04	50	120	13.70	

Table 5. Effect of temperature on colour intensity.

pH	Weight/Volume (g/mL)	Time (min)	Temperature (°C)	Colour intensity	Colour appearance
9	0.06	120	30	15.81	
9	0.06	120	70	16.33	

The weight-to-volume (w/v) ratios of the *H. rosa-sinensis* samples significantly influenced the colour intensity of the dyed silk yarn. Increased weight/volume ratio corresponds to the rise in colour intensity observed. Specifically, the 0.06 g/mL sample achieved a colour intensity value of 30.35, compared to a significantly lower intensity of 22.39 for the 0.02 g/mL sample. A higher concentration of dye compounds in the extraction medium at elevated sample weights increases the overall availability of anthocyanins for binding with the fabric. Moreover, the greater amount of hibiscus sample provides a larger surface area, facilitating enhanced contact between the dye extract and the silk fibres, thereby improving the efficiency of dye uptake.

Silk yarn dyed for 30 minutes exhibited the highest colour intensity, reaching a peak value of 14.32 (Table 4). Although prolonged dyeing times are generally associated with improved dye absorption, the results indicate that the process of dye uptake is not linear over extended durations. The initial 30 minutes likely represent a period of rapid dye adsorption, during which the available binding sites on the silk fibres become saturated. Beyond this point, an equilibrium is reached, and the rate of desorption may begin to resist further adsorption, leading to a reduction in overall colour intensity. This observation suggests that extending the dyeing time beyond 30 minutes could result in dye molecule release from the fibre matrix, thereby diminishing the observed colour intensity.

The colour intensity of the dyed silk yarn exhibited only slight variations across different temperatures (Table 5), with a maximum intensity of 16.33 recorded at 70 °C, compared to a slightly lower intensity of 15.81 at 30 °C. These findings suggest that while increasing the dyeing temperature generally enhances colour intensity, it also introduces a risk of anthocyanin degradation due to thermal instability [25]. The improved solubility of anthocyanins at elevated temperatures may facilitate better dye dispersion within the dye bath, resulting in increased accessibility and penetration of the dye molecules into the silk fibres. However, prolonged exposure to high temperatures can destabilise the anthocyanin structures, leading to pigment breakdown and subsequent colour loss. Therefore, optimising the dyeing temperature is essential to balancing enhanced colour uptake with minimising pigment degradation.

Statistical Result of Response Surface Methodology (RSM)

Response Surface Methodology (RSM) was utilised to optimise the dyeing process, examining the interactions between multiple variables to determine their influence on the colour intensity of the dyed silk yarn. A Central Composite Design (CCD) was applied to evaluate the interactive effects of four critical operating variables: weight/volume ratio, pH, temperature, and dyeing time. The analysis of variance (ANOVA) results (Table 6) indicates that the model was statistically significant, with an F-value of 14.70 and a p-value < 0.05. Moreover, the model's coefficient of determination (R^2) was found to be 0.9406 (Table 7), suggesting that 94.06% of the variation in colour intensity could be explained by the selected factors. While this high R^2 value reflects a strong correlation and model fit, minor discrepancies between the predicted and actual colour intensity values (Figure 5) indicate that the model may still benefit from further refinement. These deviations could be due to unaccounted-for interactions or nonlinear effects, warranting additional experimental validation to improve the predictive accuracy.

Table 6. Variance Analysis (ANOVA) of the response surface model.

Source	**Sum of square**	**df**	**Mean square**	**F-value**	**P-value**	
Model	993.13	14	70.94	**14.70**	<0.0001	Significant
A-Weight/Volume	61.35	1	61.35	12.72	**0.0034**	
B-time	601	1	601	0.0125	0.9128	
C-pH	590.53	1	590.53	122.41	**<0.0001**	
D-temperature	265	1	265	0.0055	0.9421	
AB	2.81	1	2.81	0.5816	0.4593	
AC	15.33	1	15.33	3.18	0.0980	
AD	1.39	1	1.39	0.2886	0.6002	
BC	1.33	1	1.33	0.2765	0.6078	
BD	2.13	1	2.13	0.4419	0.5178	
CD	22.85	1	22.85	4.74	**486**	
A^2	9.03	1	9.03	1.87	0.1944	
B^2	0.1831	1	0.1831	0.0380	0.8485	
C^2	74.84	1	74.84	15.51	**0.0017**	
D^2	0.2617	1	0.2617	0.0543	0.8194	
Residual	62.71	13	4.82			
Lack Of Fit	60.31	10	6.03	7.53	617	not significant
Pure Error	2.40	3	0.8009			
Cor Total	1055.84	27				

Table 7. Table of Correlation Coefficient.

Source	**Standard deviation**	**R^2**	**Adjusted R^2**	**Predicted R^2**	**PRESS**	
Linear	0.6175	0.6175	0.5510	0.4407	590.53	
2FI	0.6609	0.6609	0.4614	-0.2396	1308.79	
Quadratic	**0.9406**	**0.9406**	**0.8766**	**0.6039**	**418.27**	**Suggested**
Cubic	0.9825	0.9825	0.9052	-2.7813	3992.42	**Aliased**

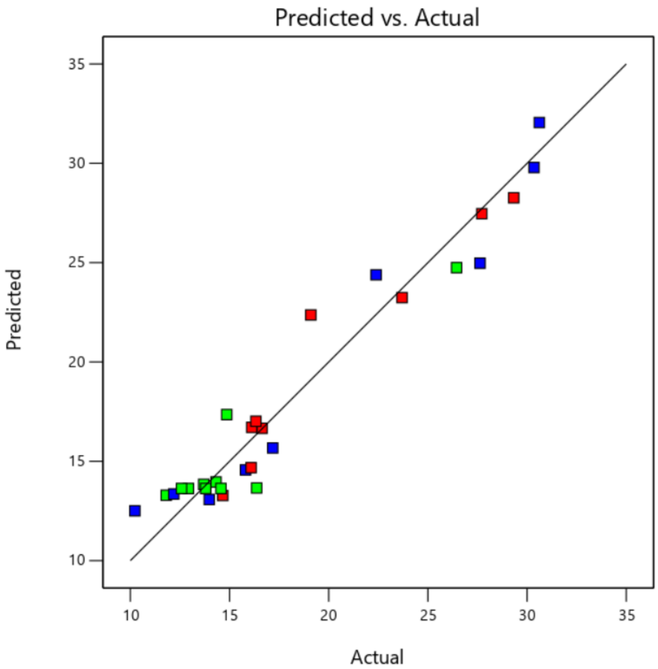


Figure 5. The predicted versus actual data for colour intensity.

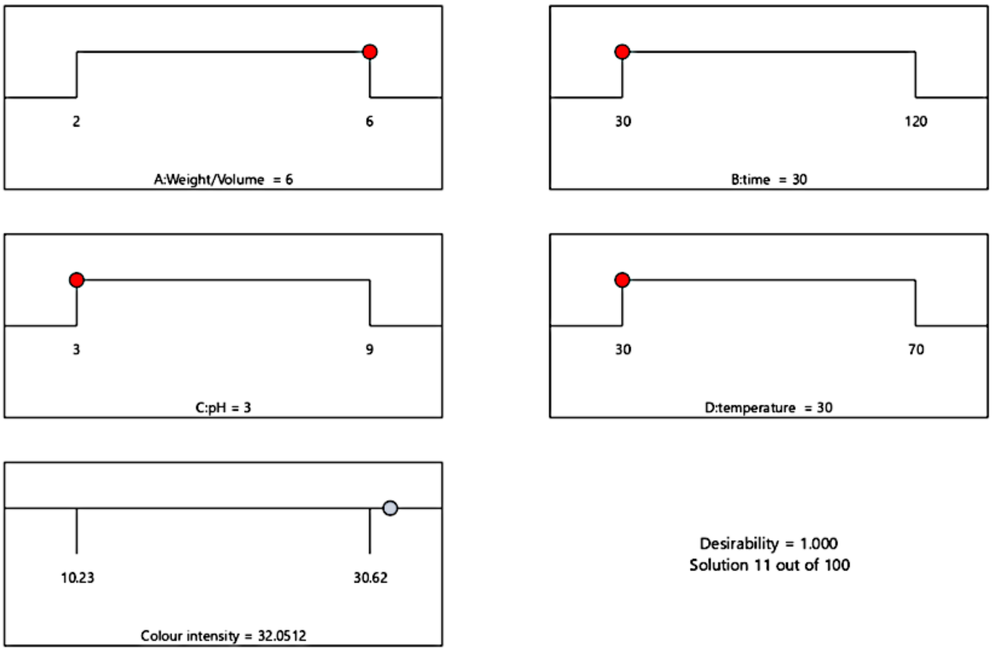


Figure 6. The optimised parameter of dyeing process.

The optimised dyeing conditions determined through Response Surface Methodology (RSM) recommend using 0.06 g/mL of the *Hibiscus rosa-sinensis* extract at pH 3 and a temperature of 30°C for a dyeing duration of 30 minutes. Under these parameters, the predicted colour intensity was calculated to be 32.05 (Figure 6), closely matching the actual experimental result, where the highest recorded colour intensity was 30.62 using the same conditions. This close alignment between predicted and observed values indicates the effectiveness of the RSM-derived parameters in achieving optimal colour intensity for the dyed silk yarn. The consistency between the two sets of results suggests that the optimised parameters are robust and reliable for maximising the colour intensity in the experimental setting.

The 3D response surface plots (Figure 7) provide visual insights into the interactions among the dyeing parameters. These plots indicate that colour intensity markedly decreases as the pH increases (Figures 7a-7c). The results show that the highest colour intensity was achieved at pH 3 within 120 minutes and at 30°C (Figures 7b and 7c). Furthermore, the dye uptake by silk yarn appears to be relatively independent of temperature and time, as increasing these variables did not significantly influence the overall colour intensity (Figures 7d and 7e). However, the colour intensity of the dyed silk yarn showed a noticeable increase when the weight/volume ratio of the dye extract was higher, indicating that the amount of dye in the extraction solution is a critical factor, whereas extended dyeing times had a minimal impact on the colour intensity (Figure 7f). These findings demonstrate that the weight/volume ratio and pH are the most influential factors in determining the final colour intensity, confirming their importance in the optimisation of dyeing process.

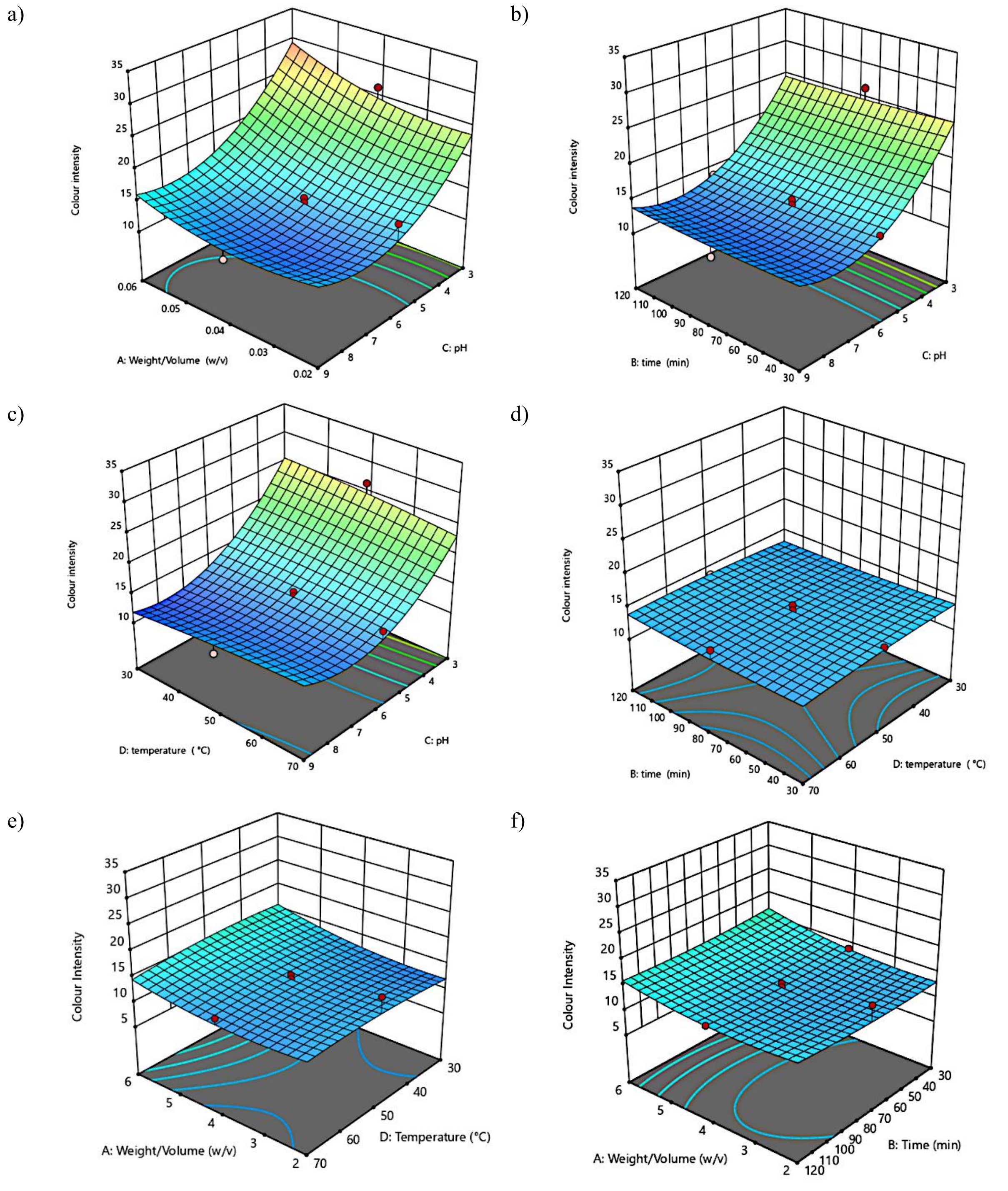


Figure 7. 3D Response Surface of a)Weight/Volume and pH , b) Time and pH , c) Temperature and pH , d) Time and Temperature, e) Weight/Volume and Temperature and f) Weight/Volume and Time.

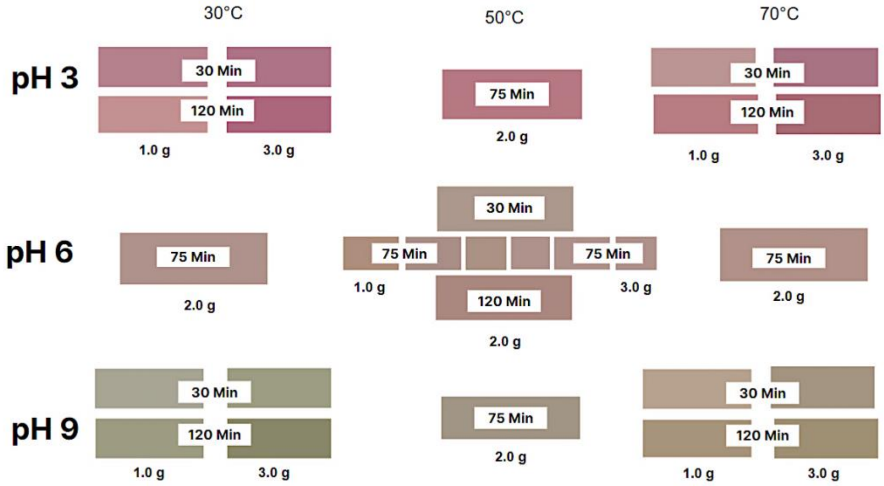


Figure 8. Summary of the colour appearances based on different parameters of dyeing process.

The effect of pH on the colour appearance of dyed silk yarn, as analysed through Response Surface Methodology (RSM), further supports the structural sensitivity of anthocyanins described earlier. At pH 3, the silk yarn displayed a vibrant red hue, indicating the dominance of the flavylium cation form under acidic environment. As the pH increased from 6 to 9, the colour shifted from reddish-brown to greenish, reflecting the gradual transformation of anthocyanins into less stable forms such as quinoidal bases. These pH-induced changes directly impact the chromophoric system, as visually evident in Figure 8. The ability to manipulate pH to achieve a spectrum of colours highlights the versatility of *Hibiscus rosa-sinensis* extract as a natural dye source. This characteristic is particularly advantageous for textile applications, where a diverse range of hues can enhance the elegant appeal of fabrics. These observations demonstrate the complex relationship between dyeing parameters, particularly pH, and their significant impact on the resulting hue and colour intensity of the dyed fabric.

CONCLUSION

The deep-red anthocyanin pigment extracted from *Hibiscus rosa-sinensis* using distilled water acidified with 4% citric acid demonstrated strong potential natural dye for silk yarn. Maintaining the extraction temperature between 50 and 60 °C was essential to prevent pigment degradation and preserve colour properties. The optimal dyeing conditions were found at pH 3 for 30 minutes and at 30 °C with a weight/volume ratio of 0.06 g/mL, yielding a maximum colour intensity of 30.35, which is closely aligned with the predicted highest intensity determined through Response Surface Methodology (RSM). Lower pH levels in the dye bath resulted in a vibrant red hue with robust colour intensity, while elevated temperatures were found to contribute to colour loss, resulting in a paler appearance, which further corroborates the careful balance required during the dyeing process. An increase amount of hibiscus used during extraction correlates with enhanced colour intensity in the dye bath. However, extended dyeing durations were observed to reduce the colour intensity. This study highlights the critical importance of optimised conditions determined via RSM in maximising the colour intensity of the silk yarn dyed with anthocyanin pigment extracted. Future studies could explore the use of alternative metal mordants to investigate their specific impacts on the silk yarn dyeing process. Additionally, incorporating co-pigmentation strategies may further enhance the extraction of anthocyanin pigment from the flower.

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The authors declare that they have no conflict of interest.

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