Optimization of Sonication Assisted Natural Dye Extraction

S. P. DEHANKAR[1,*], Dr. P. D. PATIL[2]

[1,*]Assistant Professor, Chemical Engineering, Department of Technology, Shivaji University, Kolhapur, (M.S.) India.

[2]Professor, Chemical Engineering, Department of Technology, Shivaji University, Kolhapur, (M.S.) India.

Abstract

The discharge of dyes into the aquatic ecosystem is a global environmental concern due to their negative toxical chemical effects. An important alternative to synthetic dyes is the dyes obtained from natural sources. Natural colorant from the petals of *Butea Monosperma* was extracted under different operating condition like extraction time, temperature and mass of petals by conventional extraction technique has been studied. For optimization of the extraction process and evaluation of interaction effects of different operating parameters, Response Surface Methodology (RSM) with help of design Expert version 7.1.6 was used. For optimum conditions; 3 g of flower mass and for extraction time 17.5 min, the yield of extraction is 1.57. UV-Spectrophotometer analysis is used to identify chemical groups in extracted dye.

Keywords: *Butea Monosperma*, Extraction, Response Surface Methodology, Spectrophotometer

Introduction

1.1. Background and Need of natural dyes

Today dyeing is a perplexing, particular science. About all dyestuffs are presently delivered from manufactured mixes. This implies expenses have been extraordinarily decreased and certain application and wear qualities have been significantly upgraded. Be that as it may, numerous experts of the specialty of regular passing on (for example utilizing normally happening wellsprings of color) keep up that common dyes have a far unrivaled stylish quality which is significantly more satisfying to the eye. [1].

Common dyes are gotten from normally happening sources, for example, plants (e.g., indigo and saffron); creepy crawlies (e.g., cochineal scarabs and lac scale bugs); creatures (e.g., a few types of mollusks or shellfish); and minerals (e.g., ferrous sulfate, ochre, and dirt) with no synthetic treatment1. A range of excellent normal hues going from yellow to dark exists in the above sources. These hues are shown by different natural and inorganic atoms (shades) and their blends are because of the retention of light in the unmistakable area of 400-800 nm. [2]

Since days of yore, common colors have been a fundamental piece of the human life and society. The deep rooted specialty of coloring with characteristic colors was regular in India, China, Egypt and Focal Asia. Nature gives us an abundance of plants which yield shading for the motivation behind coloring; many have been utilized since vestige. In old occasions, man utilized

characteristic assets like stem, bark, leaves, roots and blooms to extricate various hues yellow, orange, blue, red, green, dark colored, dim and so forth for coloring garments. [3].

1.2. History of *Butea monosperma*

Butea monosperma belongs to the plant family Fabaceae, ordinarily known as 'Palash' in 'Hindi'and 'Kesudo' in 'Gujarati'. It is likewise called "the fire of the woods" because of the splendid orange and red shading blooms. It pursues the exchange name "*Butea*" which has been taken from its logical name *Butea monosperma.* Truly, Dhak backwoods secured a significant part of the doab region between the Ganges and Yamuna, however these were cleared for horticulture in the mid nineteenth century as the English East India Organization expanded duty requests on the peasants. It is utilized for timber, pitch, grub, prescription, and color. The wood is filthy white and delicate and, being sturdy submerged, is utilized for well-controls and water scoops. Great charcoal can be gotten from it. [5]. The sort Butea incorporates *Butea monosperma, Butea parviflora, Butea minor and Butea superb* widely appropriated all through India. It holds a significant spot due to its restorative and different random employments of monetary worth. It is one of the most delightful tree has been put to some helpful reason. *Butea monosperma* is extensibly utilized in Ayurveda, Unani and Homeopathic drug and has turned into a cynosure of present day medication [6].

1.3. Relevance of the topic and the work objectives

The present research was performed with the express target of separating common color from the petals of Fire of woods blossom by fluid extraction technique and to examine the joined impact of various parameters, for example, extraction time, temperature and mass of petals utilized on the color extraction procedure utilizing Focal Composite Plan Accordingly Surface Approach (RSM).

2.Literature Review

Dyes got from characteristic sources have developed as a significant option in contrast to manufactured dyes. Along these lines, there is a requirement for growing better strong fluid extraction methods for draining common colorants from plant materials for applications in plant explore, nourishment just as dyeing businesses. The impact of ultrasound on characteristic colorant extraction from various potential dye yielding plant materials has been examined in correlation with attractive blending process as control. The outcomes show there is a critical 13–100% improvement in the extraction effectiveness of the colorant acquired from various plant materials because of the utilization of ultrasound. Accordingly, this technique could be utilized for removing shading materials from plant materials in a quicker and viable way. Dyes are utilized for shading the textures. Dyeing is a technique which confers excellence to the material by applying different hues and their shades on to a texture. Dyeing should be possible at any phase of the assembling of material fiber, yarn, texture or a completed material item including pieces of clothing and attire. The property of shading quickness relies on two components determination of legitimate color as indicated by the material to be colored and choice of the strategy for coloring the fiber, yarn or texture. [7].

The uncontrolled release of engineered dyes into the oceanic biological system is a worldwide ecological worry because of their negative ecotoxicological impacts. Dyes acquired from various normal sources have risen as a significant option in contrast to engineered colors. In this

examination, regular colorant from the petals of the Fire of woods (*Butea monosperma*) bloom was separated under various working conditions, for example, extraction time (45-120 min), temperature (60-90^{o}C) and mass of the petals (0.5-2 g) by traditional extraction method. Reaction surface philosophy (RSM) was utilized for enhancement of the extraction procedure and assessment of cooperation impacts of various working parameters. The effectiveness of extraction under these ideal conditions was seen as 8813.67 mg. Further, Fourier Change Infrared Spectroscopy was utilized to distinguish the real substance bunches in the separated color [8].

The overall interest for characteristic dyes is these days of incredible enthusiasm because of the expanded mindfulness on restorative properties of common dyes openly. Common colors are derived from normally happening sources, for example, plants, creepy crawlies, creatures and minerals. A few engineered colorants have been restricted in light of the fact that they cause hypersensitivity like indications or are cancer-causing agents. Among the every characteristic color, plant-based shades have wide scope of therapeutic qualities. Albeit known for quite a while for dyeing just as therapeutic properties, the structures and defensive properties of common colors have been perceived distinctly in the ongoing past. A significant number of the plants utilized for dye extraction are named restorative and a portion of these have as of late been appeared to have amazing antimicrobial movement [2].

The coloring of cotton texture utilizing Eclipta as regular dye has been examined in both customary and sonicator strategies. The impacts of coloring show higher shading quality qualities acquired by the last mentioned. Dyeing energy of cotton textures were looked at for both the strategies. The time/dye take-up uncovers the upgraded dye take-up indicating sonicator productivity. The consequences of quickness properties of the colored textures were reasonable for good. CIELAB qualities have additionally been assessed [9].

The dyeing of cationized cotton textures with lac normal dye has been examined utilizing both regular and ultrasonic methods. The impacts of dye shower pH, salt focus, ultrasonic power, dyeing time and temperature were contemplated and the subsequent shades acquired by dyeing with ultrasonic and regular strategies were looked at. Shading quality qualities got were seen as higher with ultrasonic than with customary warming. The results of speed properties of the dyed textures were reasonable for good. Coloring energy of cationized cotton fiber with lac color utilizing customary and ultrasonic conditions was looked at. The benefits of dyeing rate consistent, half-time of dyeing and standard liking and ultrasonic productivity have been determined and talked about [10]. The dyeing of cationized cotton texture with Solfix E utilizing shading matter removed from Cochineal dye has been contemplated utilizing both traditional and ultrasonic strategies. Components influencing dye extraction, for example, ultrasound control, molecule size, extraction temperature and time were examined. The outcomes showed that the extraction by ultrasound at 300W was more compelling at lower temperature and time than ordinary extraction. The impact of different variables of color shower, for example, pH, salt fixation, ultrasound control, dyeing time and temperature were examined [11].

Customary strategies utilized for the dissolvable extraction of regular items are related with longer extraction times and lower yields, utilization of enormous measure of natural solvents and poor extraction effectiveness. Ultrasound can be adequately used to improve the extraction rate by expanding the mass exchange rates and conceivable burst of cell divider because of arrangement

of microcavities prompting higher item yields with decreased preparing time and dissolvable utilization. The present work displays a comprehensive outline of various parts of ultrasound helped extraction (UAE) of different normal items. Component of UAE has been examined and proposals for ideal working conditions have been accounted for expanding the yield [12]. With consistently expanding interest for eco-accommodating, non-dangerous colorants, colors got from regular sources have risen as a potential option in contrast to moderately lethal manufactured colors. In the present work, microwaveassisted extraction of yellow-red characteristic color from seeds of Bixaorellana (Annatto) was contemplated. Reaction surface procedure (RSM) and fake neural system (ANN) were utilized to create prescient models for reproduction and advancement of the color extraction process [13].

Fluid concentrate and two flavanone glycosides named 5,7- dihydroxy- 4'- methoxy flavanone-5-Dglucopyranosyl-(1_2)- _-D-glucopyranoside(1)5,5'- dihydroxy-4',7-dimethoxyflavanone-5,5'-di-O-_-Dglucopyranoside (2) got from the blooms of *Butea monosperma* were read for assess their dyeing properties on cotton filaments. The stem bark of *Myricaesculenta* was utilized as characteristic severe while $SnCl_2$ and $FeCl_3$ as engineered mordants. The blend of dye with mordants indicated intriguing shades with great washing and light quickness properties [14].

3. Materials and Techniques

3.1. Bloom

The Fire of woodland is a medium estimated dry season-deciduous tree, developing to 15 m tall. The leaves are pinnate, with a 8-16 cm petiole and three handouts, every pamphlet 10-20 cm long. The blooms are 2.5 cm long, brilliant orangeered in shading, and roduced in racemes up to 15 cm long. The natural product is a case; around 15-20 cm long and 4-5 cm expansive. In the Indian province of West Bengal, its sprout denotes the beginning of spring (MarcheApril).

3.2. Bloom powder preparation

The blooms were taken from the trees and dried in sun for 1-2 days, at that point they were washed to expel any earth and different contaminants. After these the blossoms were kept in plate dryer for 2 hours at 65oC so they can be totally dried. Later these blooms were pounded in blender to make fine powder; screening was utilized to have a homogenous powder.

Weight of Petals before drying = 455 g

Weight of Dry Powder = 40 g

3.3. Experimental Techniques

3.3.1. Ultrasound Extraction

Ultrasound is characterized by recurrence go as power ultrasound (20–100 kHz) and analytic ultrasound (1–10 MHz). At the point when a fluid is lighted by ultrasound, microbubbles show up, develop and sway incredibly rapidly and even breakdown savagely if the acoustic weight is sufficiently high. The event of these crumples close to a strong surface will produce microjets and stun waves. [11]. The color was removed utilizing ultrasonication shower in which the diverse

synthesis arrangements were kept for various occasions according to the trial configuration sets given by Configuration direct

The yield of color was determined utilizing the accompanying articulation

1. Concentration = Absorbance / slope

2. Grams of colorant = mass of colorant/ molecular weight of colourant

3. Yield% = [weight of colorant/weight of raw material] x 10

3.4. Dye Analysis

3.4.1. Ultraviolet Spectrophotometer

Dye investigation were finished utilizing the bright spectrophotometer by estimating the absorbance of the various arrangements gotten by leading the trials from these the yield of the color was determined utilizing the above articulation for the conclusive outcome. To recognize the UV-VIS range profile of the concentrates from all the four sources, the concentrates were checked in the wavelength extending from 200-800 nm by utilizing UV-VIS spectrophotometer and the trademark pinnacles were distinguished.

3.5. Experimental design

3.5.1. Response Surface Methodology

The present work includes streamlining of various parameters overseeing the extraction procedure. The general routine with regards to deciding these optima is by shifting one parameter while keeping the others at a vague consistent level. The real burden of this single variable enhancement is that it doesn't think about the intuitive impacts among the factors; along these lines it doesn't delineate the net impacts of different parameters on the response rate [3], [15]. Reaction Surface Technique (RSM) was utilized to structure the trials to test the reaction; there are various strategies which can be utilized for planning the examinations. We utilized Focal composite structure since it tests the parameters for a greater number of qualities and is more effective than different techniques. The anticipated qualities given by CCD are effectively good with the real once. We took two factors and read reactions for absorbance, and the yield as per the plan we have to direct 13 examinations.

3.6. Dyeing

The removed color test which has given the best outcome was utilized for coloring. Little portions of cotton texture were taken of same size. For staying of color shade/colorant the texture was pre-treated with Alum as a modarant. A severe or color fixative is a substance used to set (for example tie) colors on textures by framing a coordination complex with the color, which at that point appends to the texture (or tissue). It might be utilized for coloring textures or for escalating stains in cell or tissue arrangements.

Weight of Texture Strip = 0.29 g

Weight of Alum = 10 g in 100 mL Water

Soaking Span = 20-25 Minutes

Temperature = 45° C

The Strips were taken off and crushed to expel the overabundance water. After that strips were placed in the dye separate which was warmed at 60°C for around 1 hour with customary mixing to maintain a strategic distance from the inconsistent coloring and to get uniform shade on the material surface then the texture was washed and the example was prepared also was accomplished for the example which was removed without ultrasound and the outcomes were looked at.

3.6.1. Texture Speed

The speed of a shaded material is, subsequently characterized as its protection from these progressions when exposed to a specific arrangement of conditions. It pursues that shading quickness must be indicated as far as these progressions and communicated as far as their size. The colored cotton textures were tried by standard strategies: the particular tests were for shading quickness to washing IS-687-79, shading speed to scouring IS-766-88, shading speed to light IS-2454-85 and shading speed to sweat IS-971-83.

Following are various strategies for testing the speed of the colored materials.

Washing quickness

Shading quickness to washing of the colored texture tests was resolved according to Seems to be: 764 – 1984 strategy utilizing a Foremost washes O-meter following IS-3 wash speed technique.

Light speed

Shading speed to light introduction was resolved according to Seems to be: 2454-1984 technique. The example was presented to counterfeit light in a Vital blur O-meter (having 500 watt Philips mercury bulb tungsten fiber light reproducing sunlight) alongside the eight blue fleece norms. The blurring of each example was seen against the blurring of blue fleece principles

Rubbing speed

Shading speed to scouring (dry and wet), was surveyed according to Seems to be: 766-1984 technique utilizing a physically worked container meter and dark scale according to ISO-105-AO3 (extent of staining).

Perspiration quickness

Shading quickness to sweat was surveyed by IS 971-1983 composite example was set up by putting the test example between two adjoining bits of cotton texture and sewed all among four sides.

4. Data collection and Analysis

4.1. Utilization of Response Surface Methodology

Design of experiment

We utilized Central composite Design (CCD) for this investigation, the parameter fluctuated were *Butea onosperma* concentration (0.17 to 5.33 g), Time (0 to 35.17min). In CCD, we can either give esteems as far as high (+1) and low (- 1) or as far as alpha (+/ -) for the autonomous factors which are called as coded values. The qualities given are demonstrated as follows.

Table 4.1.a: Experimental coded levels of variables using CCD

Name	Symbol	Unit	-alpha	-1 level	0 level	+1 level	+ alpha
Powder Mass	X	Gram	0.17	1	3	5	5.83
Time	Y	Min	-0.18	5	17.50	30	35.18

Table 4.1.b: CCD design matrix for three independent variables and their experimental response

Run	Time (min)	Concentration (gram)	Absorbance
1	0	0	3.84
2	0	- α	2.9
3	0	+ α	3.1
4	- α	0	2.67
5	0	0	3.81
6	0	0	3.79
7	+ α	0	3.6
8	+1	+1	3.8
9	0	0	3.76
10	+1	-1	3.5
11	-1	-1	3
12	0	0	3.87
13	-1	+1	2.5

Using the experimental design, for each response quadratic model were developed which can be represented in terms of following equations:

Equation of coded parameters

$$P = \beta_0 + \beta_1 w_1 + \beta_2 w_2 + \beta_1 2 w_1 w_2 + \beta_1 w_1^2 + \beta_2 w_2 2$$

Where, β_i and β_{ii} are the regression coefficients and wi, wii are variables

Equation of actual parameters

$$R = \mu_0 + \mu_1 X + \mu_2 Y + \mu_{12} XY + \mu_{11} X^{2+} \mu_{22} Y2$$

Where, μ_i and μ_{ii} are the actual regression coefficients and X and Y are powder mass and time respectively.

4.2. ANOVA Analysis

ANOVA examination was accomplished for contemplating of the relapse models. ANOVA investigation is a device which uses factual examination for the advancement of the model. The reaction of absorbance demonstrated noteworthy outcomes. The Model F-estimation of 39.05 infers the model is huge. There is just a 0.01% shot that a "Model F-Worth" this enormous could because of clamor Estimations of "Prob> F" under 0.0500 show model terms are critical. For this situation X, XY, X_2, Y_2 are critical model terms. Qualities more prominent than 0.1000 demonstrate the model terms are not significant. If there are numerous unimportant model terms (not including those required to help hierarchy), model decrease may improve your model. The "Absence of Fit F-esteem" of 17.05 infers the Absence of Fit is significant. There is just a 0.96%chance that an "Absence of Fit F-esteem" this enormous could happen due to noise. Significant absence of fit is terrible - we need the model to fit. The "Pred R-Squared" of 0.7678 is in sensible concurrence with the "Adj R-Squared" of 0.9407."Adeq Accuracy" measures the sign to commotion proportion. A proportion more noteworthy than 4 is desirable. Your proportion of 15.435 shows a sufficient sign. This model can be utilized to explore the design space.

Table 4.2.a: ANOVA analysis for the response

Response	Source	Sum of Squares	DOF	F value	P value
Absorbance	Model	2.82	5	39.05	<0.0001
	Lack of Fit	0.094	3	17.05	0.096
	Pure error	7.320E-003	4	-	-

Table 4.2.b: Values of regression coefficients

Response	Source	R-squared	Adjusted R-Squared	Predicted R-Squared
Absorbance	Quadratic	0.9654	0.9407	0.778

4.3. Response surface models

The reaction surface models were created from the ANOVA investigation. For examination shapes and charts are utilized. The reaction surface models demonstrate the ideal conditions for the parameters with correlation with different parameters.

The Anticipated versus genuine chart is appeared underneath which holds for the given parameters and conditions and supposedly is huge which is a proof that our model is huge and can give streamlined outcomes.

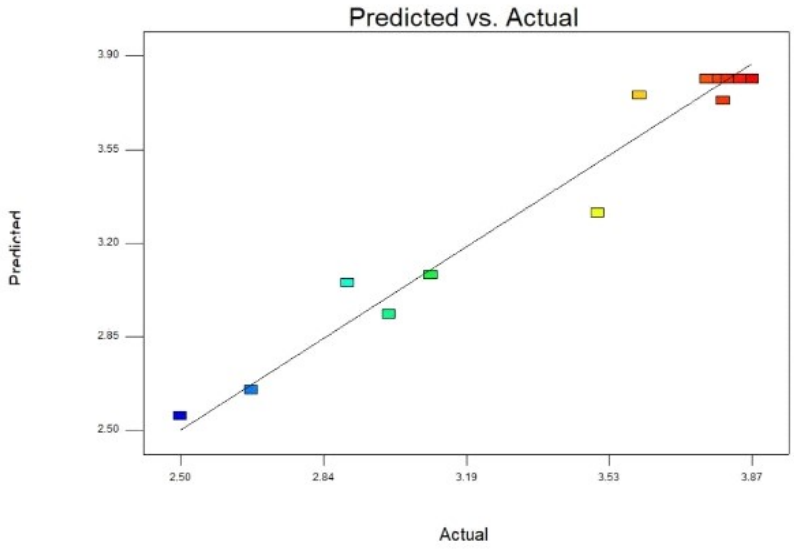


Figure 4.3: Actual versus Predicted plot

4.4. Dye Characterization and Ultraviolet Spectrophotometer Analysis

The UV spectrophotometer was utilized to examine the examples of concentrate. Water was utilized as a standard reference and the absorbance of each example was estimated. We got tops at 220,435 and 360nm which demonstrated for the nearness of Butein (C15H12O5) compound. The color samplegave red shading when treated with watery sodium hydroxide which is the test for the presenc eof chalcone compound.

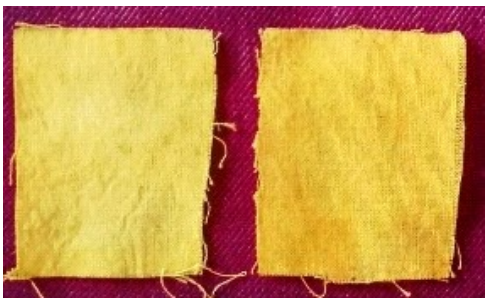
Figur 4.4: UV spectrophotometer absorbance plot

4.5. Dyeing Analysis

The advanced outcome parameter concentrate was utilized for coloring one was removed utilizing ultrasonication and other was separated without ultrasonication every single other condition were kept consistent, the outcomes gave significant variety. Without sonication gave a yellow shading though the one with ultrasonication gave a solid and profound yellow shade which demonstrates the ultrasonication helps in extraction of the color and furthermore expands the efficiency of the system.

(a)

(b)



(c)

Figure 4.5: Cotton Fabrics after dyeing – (a) Fabric dyeing without Ultrasonication (b) Fabric dyeing with Ultrasonication (c) Comparison of both samples

The following analysis was done after dyeing two fabrics one standard and other sample:

Table 4.5.a: Strength and K/S value

Parameter	Standard value	Sample value
% Strength	100	148.6
K/S	178	214

Table 4.5.b: Fastness value

Parameter	Standard value	Sample value
Wash Fastness	3-4	4
Perspiration fastness (1-5)	4	4-5
Light Fastness (1-7)	6	7
Rubbing Fastness (1-5)	-	-
Dry Fastness	3-4	4-5
Wet Fastness	3	4

4.6. Yield Calculations

The yield was calculated as: Concentration = Absorbance / slope

Concentration = 3.87 / 0.302

Concentration = 12.81 moles per liter

The molecular weight of the colorant compound present in the dye sample was found out to be 272. Therefore the mass of colorant was found out to be :

Grams of colorant = 12.81 / 272

Grams of colorant = 0.047 g per liter

We took initial of 3 g of raw material, so yield of the colorant is

Yield% = [Colorant weight / raw material weight] x 100

Yield% = [0.047/3] x 100

Yield% = 1.57

Therefore, the optimum sample use for the extraction of natural dye using ultra sonication is producing a yield of 1.57.

5. Results and Discussion

Natural dye from Fire of Woods blossoms was extricated by following fluid extraction method and the extraction procedure was done at fixed temperature, diverse extraction times and with various mass of bloom petals with an unequivocal goal of deciding the ideal extraction conditions. Color extraction from bloom relies upon both the measure of blossom utilized and the extraction time. With increment in the mass of bloom utilized for extraction (1 g to 5 g) just as the extraction time (5min to 30min), the aggregate sum of color separated additionally increment.

5.1. Impact of time and Concentration on dye extraction

The extraction of dye is legitimately relative to the time as the time expands the measure of color extricated continues expanding however upto a specific level after that it stays steady. The mass or grouping of powder utilized incredibly impacts the color extraction yet is time reliant, increasingly mass of powder with enormous time of extraction gives better return of color removed. The form plot demonstrates the consolidated impact of mass of petals utilized and extraction time on the aggregate sum of color extricated. Clearly, both the free factors strongly affected the extraction procedure. The reaction surface model for absorbance is demonstrated as follows. In fig we can consider that to be the time and centralization of Butea monosperma increases the absorbance esteem additionally increments and here we get the best outcome for 17.50 minutes and 3 gram powder, which is only the ideal point for the model.

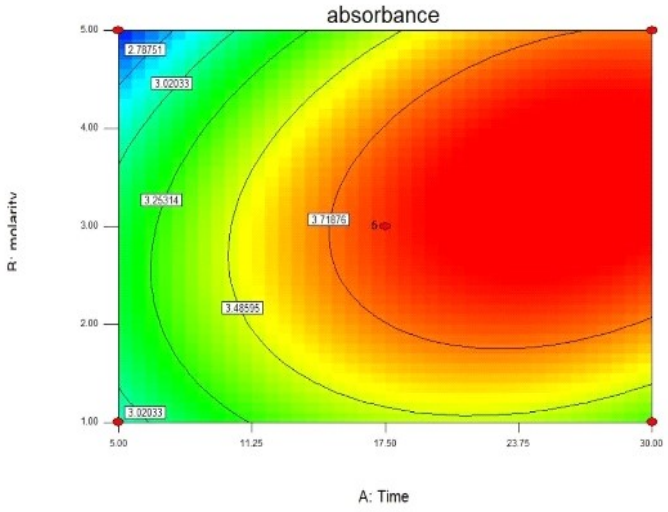


Figure 5.1: Contour Plot of Concentration vs Time

5.2. Results of UV spectrophotometer

The UV profile of plant concentrate taken at the 200 to 800 nm wavelength demonstrates the sharp top at 220 nm with absorbance esteems 3.87, 3.84 and 3.85 for control, ideal and novel extraction. The plant concentrate has two more retention groups at 430 and 365. Normally

flavonoids and their subordinates have the sort of retention groups. In this way, the absorbance at this wavelength affirms the flavonoid idea of colorant. The UV profile of plant concentrate was taken at the 200 to 800 nm wavelength. The sharp crest at 220 nm is watched. A profile with such assimilation groups is trademark for flavonoid and its subsidiaries. In this way it affirms the flavonoid nearness in the example. The atomic recipe, and sub-atomic load of the colorant are $C_{15}H_{12}O_5$ and 220 separately.

5.3. Texture dyeing Results

The texture was dyed one with ultrasonication and other without it, the one without ultrasonication was considered as standard for correlation. The quality of test is seen as 148.6 regarding 100 of the standard, the k/s worth is 214 while standard has 178, these worth demonstrates the predominant nature of color removed utilizing ultrasonicator. The speed estimations of the example were additionally higher than the standard and the primary explanation for this is the utilization of ultrasonication and the ideal conditions.

6. Conclusion

Butea monosperma flower can be adequately utilized for the extraction of common color, and with the guide of ultrasonication procedure the outcomes demonstrated that the pace of extraction and the colorant separated expanded with the utilization of ulrasonication strategy additionally the profundity of colorant extricated was way better prevalent in quality then the one removed without ultrasonication. The RSM gave the best ideal outcomes for 17.5 minutes extraction time and 3 grams of blossom powder and the model are seen as noteworthy with the relapse coefficient around 0.9654. The absorbance of the colorant shade shows pinnacle estimation of 3.87. The yield of the ideal example utilized for the extraction is 1.57. The color test was applied to texture and indicated enormous outcomes so the trial demonstrates that normal color separated utilizing ultrasonication with no utilization of synthetic substances and engineered materials and strategies can be utilized for material application successfully.

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