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Optimization of alkaline extraction method for natural dye extraction from arecanut husk

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Abstract
Growing concerns over the environmental impact of synthetic dyes have accelerated research into renewable, plant-based colorants. This study aimed to optimize alkaline extraction of natural dye from arecanut (*Areca catechu* L.) husk using temperature and extraction time as key process variables. A factorial completely randomized design (FCRD) was employed with four temperature levels (70, 80, 90, and 100 °C) as the main treatment and five extraction durations (60, 90, 120, 150, and 180 min) as the sub-treatment. The efficiency of each extraction combination was assessed based on dye yield, color value (K/S), and tannin concentration. Statistical analysis revealed significant effects of both temperature and time, as well as their interaction, on all response parameters. Among the tested combinations, extraction at 80 °C for 120 min produced the highest pigment yield, maximum color strength, and elevated tannin content compared with other treatments. The optimized conditions demonstrated improved extraction efficiency while minimizing energy and chemical inputs, thereby enhancing the environmental sustainability of the process. The findings confirm arecanut husk as a promising bioresource for eco-friendly textile dyeing and provide a reproducible framework for scaling alkaline extraction methods to other plant-based residues. This study highlights the potential of applying statistically optimized processes to improve natural dye production and support circular bioeconomy initiatives in the textile sector.

Keywords: Arecanut, *Areca catechu* L., alkaline, pigment, optimization, arecanut husk

1. Introduction
India's tropical crop is the arecanut (*Areca catechu* L.). Common names for *Areca catechu L.* include betel nut tree and betel palm (Raghavan, 1957) [8]. Although its exact origin is unknown, arecanut is thought to be native to the Philippines or Malaysia. The genus *Areca* has no fossilized remnants. However, evidence from closely related genera in fossil records suggests that it existed during the Tertiary Period. Its native habitat in the Philippines, Malaysia, and Indonesia is suggested by the largest species diversity (24 species) and other markers (Bavappa, 1963) [3].
The arecanut fruit's mesocarp is fibrous, with endosperm ruminated in depth with a basal embryo. About 60-80% of the fresh fruit's weight and volume are made up of the husk. The fiber from the husk was made of cellulose with varying ratios of pectin, protopectin, lignin, and hemicellulose (Ramachandra *et al*. 2004) [10]. The main constituents of areca nut husk are cellulose (35-64.8%), hemicellulose, lignin (13-26%), and pectin (7%) (Rajan *et al*. 2005) [9].
The pursuit of new natural dyes derived from diverse plant materials is a component of the trend toward the use of natural dyes. This is because synthetic dye use has recently been prohibited due to the fact that their industries effluent is the main source of environmental pollution, in addition to the intermediates used in their production being (Al Amoudi and Osman, 2009) [1]. To maximize the colour yield on textile fabrics and assess the dyeing properties of any textile substrate, it is important to extract the colour component from natural sources (Mansour, 2009; Shaukat *et al*., 2009) [6, 11]. Furthermore, colour yield, extraction process cost, and dyeing cost are all impacted technically and commercially by standardizing the extraction process and optimizing the extraction variables.
Arecanut husk is an agro-waste product that contains a lot of polyphenols and may have good dyeing properties. It can be helpful to look into the feasibility of making natural dye from arecanut husk, an agricultural byproduct, and assess its potential as a sustainable

alternative to synthetic dyes. Given the increasing demand for eco-friendly textile processes and growing concerns about the environmental impact of synthetic dyes, present investigation is undertaken with aim to optimize the alkaline extraction of natural dye from arecanut husk.

2. Materials and Methods

2.1 Material

The arecanut fruits of Shriwardhanee variety were collected from local farmers for extraction of dye. The fresh husk was peeled and chopped into small pieces. Distilled water, Hydrochloric acid, Ethanol and Sodium hydroxide used were all of analytical grade.

2.2 Preparation of the Alkaline dye Extracts from Arecanut husk

Using the alkaline method, 100 ml 2% of NaOH solution was mixed with 5g of Arecanut husk, and the mixture was boiled for 120 minutes at 90 °C. The fine cotton fabric was used to filter the hot dye extract, which was then left to cool. At last, it was filtered through Whatman 40 filter paper once more. Then dye solution was collected and stored.

2.3 Characterization of Colour strength rating of dye (%)

The visible portion of the spectrum's light absorption is used to measure the colour strength. The ratio, expressed as a percentage, of K/S values for samples relative to a standard at the same wavelength is known as relative colour strength. The dyed sample's "K" and "S" are its absorption and scattering coefficients, respectively. The Kubelka-Munk equation is used to determine relative colour strength (%) from reflectance, R, as follows: $K/S = (1-R)^{2/}2R$ (Shimo and Smriti, 2015) [12].

2.4 Characterization of Percent yield of dye (%)

The Weight of extracted dye and Weight of sample used is calculated using measuring cylinder and weighing balance. Percent yield is calculated using formula:

$$\%\ yield = \frac{\text{Weight of extracted dye}}{\text{Weight of sample used}} \times 100$$

(Bekele *et al*., 2024) [4].

2.5 Determination of Tannin content of dye (mg TE/g)

Using a standard procedure, secondary metabolite qualitative phytochemical screening was carried out. Quantitative assessment of the total tannin content of *Areca catechu* husk extracts in aqueous form: The Folin-Denis method, with a few minor adjustments, was used to quantitatively analyze the total tannin content in the aqueous extract of *Areca catechu* husk. A stock solution containing 1 mg/ml of *Areca catechu* husk extract was made. For quantification, 0.5 ml of Folin-Denis reagent and 1 ml of distilled water were added to 0.1 ml of *Areca catechu* extract and thoroughly mixed. After adding one milliliter of 15% (w/v) sodium carbonate to this mixture, it was alkalinized and allowed to sit at room temperature for half an hour in the dark. At 700 nanometers, the absorbance was measured. As a standard, 1 mg/ml of pure tannic acid was used. All of the reagents except the extract make up the blank. The outcome was stated as mg TE/g (mg tannic acid equivalent) per gram of dry weight of extract (Vanimakhal and Balasubramanian, 2016) [15]. The total tannin was calculated by using the formula: Total tannin content = TE x V/m

Where,
TE = Tannic acid equivalence (mg/ml), V = Volume of the extract (ml), m = Weight of the pure plant extract (g)

2.6 Statistical analysis

A completely randomized design (CRD) was used to choose the treatments and replications as part of the experiment in order to increase experimental and statistical accuracy. The observations were conducted in five replications, and the mean value and ANOVA were used to compute important differences between the results of the analysis of different treatments. The data were analyzed and interpreted using a completely randomized methodology, and valid conclusions could only be drawn if there were significant differences between treatment means at the 5% level of significance (Panse and Sukhatme, 1985; Amdekar, 2014) [7, 2].

3. Results and Discussion

3.1 Colour strength value (%)

The color strength rating of a natural dye is crucial because it directly indicates how much color the dye can impart to a fabric or material. A higher color strength (k/s value) means a more intense and vibrant color can be achieved with the same amount of dye, while a lower value indicates a weaker, potentially more faded colour. The data for colour strength rating due to extraction temperature, time variation are presented in Table 1.

The significantly highest mean colour strength value was observed in Treatment T_2 (7.184%) followed by Treatment T_3 (7.074%) while lowest mean colour strength value was observed in Treatment T_4 (6.272%) which was at par with Treatment T_2 (6.301%). The significantly maximum colour strength rating of dye was observed at extraction time 120 minutes (7.703%) while lowest at 180 minutes (6.437%) which was at par with 90 minutes (6.473%). Colour strength rating of the dye increased significantly with increase in time upto 120 minutes. However, increase in extraction time from 120 minutes does not further increase the colour strength value. The interaction between different extraction temperature and time on colour strength value of dye was found to be statistically significant. The maximum colour strength value of dye was recorded in T_2 (8.689%) for 120 minutes extraction and minimum value of colour was recorded in T_1 (5.505%) for time 60 minutes.

The results shows that colour strength value was increased up to extraction temperature 80 °C for extraction time 120 minutes and wanes after it. Similar results were found by Tu Le, (2021) [14]. The results showed that the optical density increased as the extraction time increased and peaked at 120 minutes; if the extraction time exceeds 120 minutes, the optical density decreases. This may be because longer extraction times increase the optical density and improve the ability to separate the colorants. If the extraction period exceeds 120 minutes, the dye's structure may alter or other chemicals that influence the solution's color may separate, lowering the optical density. Since optical density (OD) directly affects how dark or intense a color looks, color strength rating and OD a measure of how much light a substance absorbs or attenuates are intimately related. A higher optical density, which usually indicates a higher concentration of light-absorbing substance, produces a brighter or deeper colour.

3.2 Percent yield (%)
The percentage yield of natural dyes is a critical factor in their economic viability and sustainability. Higher yield rates mean less raw material is needed, reducing costs and environmental impact. This is especially important because natural dye extraction often requires significant amounts of plant material and may involve environmentally impactful solvents. The data for percent yield due to extraction temperature, time variation are presented in Table 2.

The highest mean percent yield was observed in Treatment T_2 (89.882%) which followed by Treatment T_1 (84.010%) while lowest mean percent yield was observed in Treatment T_4 (76.771%) which was followed by Treatment T_3 (78.804%). Percent yield of the dye increased significantly with increase in extraction time upto 120 minutes. However, increase in extraction time from 120 minutes does not further increase the percent yield. The significantly maximum percent yield was recorded at extraction time 120 minutes (85.594%) and minimum percent yield was noticed at extraction time 60 minutes (79.168%). The interaction between different extraction temperature and time on yield of dye was found to be statistically significant. The maximum percent yield of dye was recorded in T_2 (93.207%) at 120 minutes extraction and minimum value of percent yield was recorded in T_4 (74.297%) for time 90 minutes.

Increase in temperature and time have been demonstrated due to increase solute solubility and diffusion rate, which in turn increases extraction efficiency. However, sensitive compounds like phenolics may degrade if these parameters are increased past a certain point. At low temperatures (about 25 °C), tannins can withstand temperatures of up to 24 hours, but only 2 to 6 hours at high temperatures (80-120 °C). It often seems that the degradation or destruction of the target compounds as a result of extended exposure to high temperatures or other reaction circumstances is the cause of the yield decline after optimum conditions. Haggag *et al.* (2022) [5] extracted dye from mulberry leaves using conventional heating at 100 °C.

3.3 Tannin (mg TE/g)
The data for tannin due to extraction temperature, time variation are presented in Table 3.

The significantly highest mean tannin was observed in Treatment T_2 (5.239 mg TE/g) followed by Treatment T_1 (4.630 mg TE/g) while lowest mean tannin was observed in Treatment T_4 (3.652 mg TE/g) followed by Treatment T_3 (3.804 mg TE/g). Tannin of the dye increased significantly with increase in time upto 120 minutes. However, increase in extraction time from 120 minutes does not further increased the tannin content. The significantly highest tannin content was recorded at 120 minutes extraction time (4.506 mg TE/g) while lowest at 60 minutes extraction time (4.132 mg TE/g).The interaction between different extraction temperature and time on tannin content of dye was found to be statistically significant. The maximum tannin content was recorded in T_2 (5.447 mg TE/g) and minimum tannin content was recorded in T_3 (3.368 mg TE/g) for time 60 minutes.

Supardan *et al.* (2024) [13] noticed that without causing noticeable oxidation or degradation, 80 °C is enough to increase the solubility and diffusion of phenolic compounds from the plant matrix.

Table 1: Effect of different extraction conditions on the Colour strength value of the arecanut husk dye

Treatment (Extraction temperature °C)	Colour strength value (%)					Mean
	Extraction time (min.)					
	60	90	120	150	180	
T_1 (70 °C)	5.505	6.133	6.960	6.538	6.370	6.301
T_2 (80 °C)	6.502	6.812	8.689	7.072	6.846	7.184
T_3 (90 °C)	6.473	6.517	8.664	7.164	6.553	7.074
T_4 (100 °C)	6.304	6.431	6.498	6.146	5.980	6.272
Mean	6.196	6.473	7.703	6.730	6.437	
			S. Em±		CD at 5%	
Treatment (T)			0.037		0.105	
Extraction time (M)			0.041		0.117	
Interaction (T×M)			0.082		0.234	

Table 2: Effect of different extraction conditions on the Percent yield of the Arecanut husk dye

Treatment (Extraction temperature °C)	Percent yield (%)					Mean
	Extraction time (min.)					
	60	90	120	150	180	
T_1 (70 °C)	77.233	81.067	88.867	87.211	85.670	84.010
T_2 (80 °C)	88.470	90.456	93.207	92.408	84.867	89.882
T_3 (90 °C)	76.367	77.867	80.983	80.500	78.303	78.804
T_4 (100 °C)	74.603	74.297	79.317	78.113	77.523	76.771
Mean	79.168	80.922	85.594	84.558	81.591	
			S. Em±		CD at 5%	
Treatment (T)			0.132		0.380	
Extraction time (M)			0.148		0.425	
Interaction (T×M)			0.296		0.849	

Table 3: Effect of different extraction conditions on the tannin content of the Arecanut husk dye

Treatment (Extraction temperature °C)	Tannin (mg TE/g)					Mean
	Extraction time (min.)					
	60	90	120	150	180	
T_1 (70 °C)	4.363	4.666	4.817	4.757	4.548	4.630
T_2 (80 °C)	5.121	5.394	5.447	5.153	5.080	5.239
T_3 (90 °C)	3.368	3.992	4.032	3.975	3.653	3.804
T_4 (100 °C)	3.674	3.689	3.730	3.657	3.511	3.652
Mean	4.132	4.435	4.506	4.385	4.198	
			S. Em±		CD at 5%	
Treatment (T)			0.021		0.060	
Extraction time (M)			0.023		0.067	
Interaction (T×M)			0.047		0.135	

4. Conclusions
Based on the results of present study, it can be concluded that the extraction temperature 80 °C for 120 min is found to be best for alkaline dye extraction method as it demonstrated good amount of Colour strength, Percent yield and Tannin.

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