

# Separation and Characterization of Flavonoids from Purple Onion Skin and Their Potential Application for Natural Dyeing of Cotton Loose Fiber

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## Abstract

The natural dye extracted from purple onion skin using absolute ethanol and the optimal extraction conditions for natural dye were investigated by the single-factor analysis as 80% aqueous ethanol mixed solvent system with a material-liquid ratio of 1:20, a temperature of 70°C, a time of 2 h, and a pH value of 5. This work aims to explore the pre-separation of purple onion skin dyes using thin-layer chromatography, and develop a developing agent ratio that can effectively separate quercetin and kaempferol dyes. Flavonoid was separated and purified by silica gel column chromatography to prepare quercetin after investigation of different amounts of silica gel filling height, purity level and different sample amounts. The isolated quercetin and kaempferol pigment were used as dyes to directly dye on modified cotton loose fiber. The optimal dyeing process was pH 6, temperature was 80°C, and dyeing time was 60 min. The fastness properties, both dyes showed good wash fastness and acceptable moderate light fastness, with Quercetin achieving a rating of 3 - 4 and Kaempferol being 3. This study explores a broader group of components separation and purification that can provide the palette with even more colors and new properties of the textile colored. It will be a key contributing factor to the transformation of the textile industry to more sustainable production practices aligning with larger global priorities to protect human health and ensure ecological integrity.

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## Keywords

Purple Onion Skin, Extraction, Thin Layer Chromatography, Column Chromatography, Separation, Dyeing, Fastness Properties

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## 1. Introduction

Recently, environmentally friendly natural dyes have started receiving much attention to avoid toxic, harmful, non-biodegradable, and bio-incompatible synthetic dyes in the textile industry. Natural dyes originate from a diverse array of sources, possess desirable color properties and express low risk to human health and the environment [1] [2] as well as their biodegradability [3]-[6], biocompatibility [7] [8], non-toxicity [9]-[11], and potential medical values [12]. They have been used in the dyeing of natural fibers, imparting vibrant hues to textiles. In addition, many natural colors have intrinsic antibacterial and antioxidant properties, which enhance the functionality and added value of natural fibers but most of the natural dyes lack color fastness, particularly when exposed to sunlight. This hampers the sustainable development of natural dyes in the modern market economy. Additionally, natural dyes often exhibit incomplete chromatography and high costs during the dyeing process. Therefore, there is a promising market for the development of abundant and affordable natural dyes. A larger amount of agricultural by-products from the crop processing and utilization industries are usually disposed of as wastes, resulting in the loss of natural pigment resources. Adopting certain technological approaches to recycle and reuse biomass wastes can turn these wastes into potential sources of natural dye for foods, cosmetics, and textile applications, thereby meeting the requirements of sustainable production and playing a vital role in protecting the environment. Scaling up the production of dyes from onion skins confronts several barriers. Regular and large-scale supply of onion skins is risky due to seasonal changes and market demand. The required collection, transportation and large-scale pre-processing are more challenging. Inconstancy in dye concentration due to onion variety and growing conditions can affect dye quality. Large-scale purification of specific compounds such as quercetin and kaempferol from complex extracts can be costly. Significant solid and liquid waste generation management is most important for environmental sustainability. Finally, economic durability must be competitive with existing dye sources and must consider all production costs. Onion (*Allium cepa* L.), belongs to the genus *Allium* of the Liliaceae family. It is a biennial herb and is widely grown in China and abroad. Onions are rich in nutrients and are known as the “Queen of Vegetables”. Onion skin mostly contains flavonoid polyphenols including quercetin, kaempferol, and myricetin [13] and the major colorant is quercetin, which imparts a yellowish-brown hue to cotton [14] with exhibiting functional characteristics such as antioxidant, UV-protective, anti-carcinogenic, anti-microbial, anti-mutagenic, and anti-diabetic properties [15].

The research focused on extraction of purple onion skin using absolute ethanol, explored pre-separation of purple onion skin dyes using thin-layer chromatography, and developed a developing agent ratio that can effectively separate quercetin and kaempferol dyes separated and purified by silica gel column chromatography to prepare quercetin dye. Silica gel filling height, sample amount and the purity were investigated. The isolated quercetin and kaempferol dye were used to directly dye modified cotton loose fiber by the optimal dyeing process without the use of inorganic salts and metal mordants for the cleaner production of environmentally friendly value-added products. The color characteristics, and fastness properties, both dyes showed good wash fastness and acceptable moderate light fastness.

## 2. Experimental Part

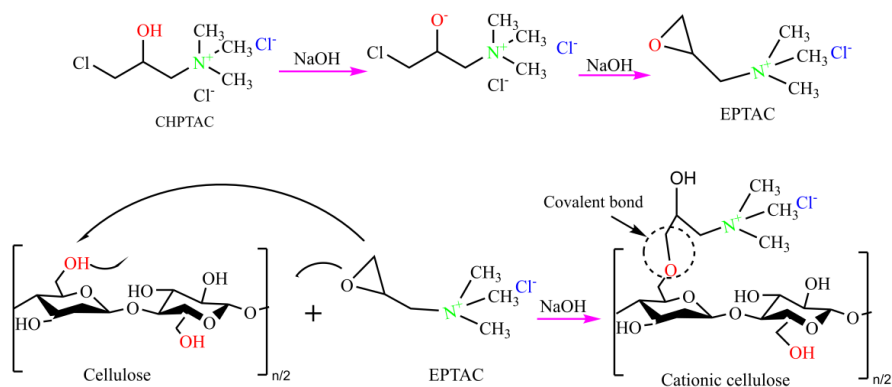
### 2.1. Experimental Materials and Instruments

Cotton loose fiber, purple onion skin, Absolute ethanol (Hangzhou Gaojing Fine Chemical Co., Ltd.), Anhydrous sodium carbonate (Shanghai Maclean Biochemical Technology Co., Ltd.), Modifier CHPTAC (Shanghai MacLean Biochemical Technology Co., Ltd.), Citric acid (Hangzhou Gaojing Fine Chemical Co., Ltd.), Standard Soap (Guangdong Zhongrun Chemical Co., Ltd.), Silica gels were purchased from Qingdao Ocean Chemical Co., Ltd.; ethyl acetate (Taicang Hushi Reagent Co., Ltd.), n-hexane (Sinopharm Chemical Reagent Co., Ltd.), methanol (Zhejiang Tengyu New Material Technology Co., Ltd.) all of the above drugs were used in this study.

A QL2-200 high-speed universal pulverizer (Hangzhou Xingbiao Machinery Co., Ltd), Glass column (30mm\*300mm), G2, Pomex, ACO-6602 adjustable silent air pump, (Guangdong Hailea Group Co., Ltd.), an LA-652 DYER independent dyeing machine (Liuya Technology Co., Ltd.), a RE-2000B Rotary Evaporator (Hangzhou Xingbiao Machinery Co., Ltd.), a YTLG-10A Freeze dryer (Nanjing Xinyi Biotechnology Co., Ltd.), a Lambda 35 UV-Vis spectrophotometer (Elmer Perkin, USA), a Datacolor 600 Precision benchmark colorimeter (DATACOLOR, Inc., USA), a JA2003 Electronic Analytical Balance (Jiangxi Benoy Instrument Co., Ltd.), a WF611M Colorfastness to solar weather (Wenzhou Wanfeng Testing Equipment Co., Ltd.) were used in this study.

### 2.2. Pre-Treatment of Cotton Loose Fibers

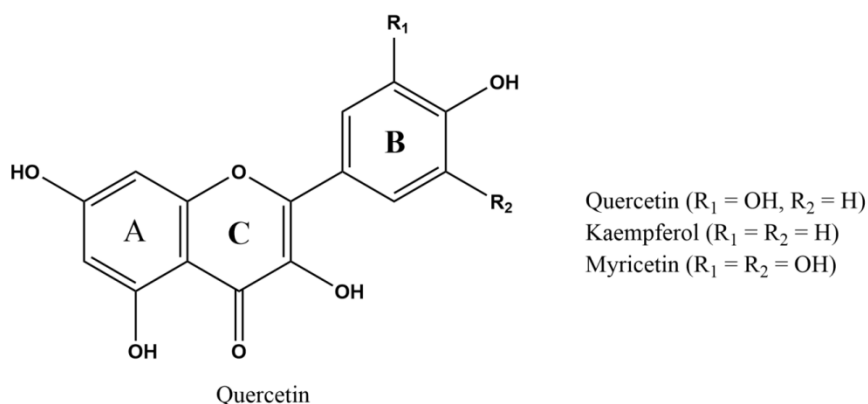
For cleaner, brighter, and suitable for further modification and dyeing processes as well as finishing applications, cotton loose fibers were treated with an optimal analysis as a mixture of 2.5 g/L amount of TF120EC and 6 g/L H<sub>2</sub>O<sub>2</sub> in water at a ratio of 1:15, with a temperature of 98°C and a reaction time at 40 minutes. Pre-treated cotton loose fibers were treated with an optimal analysis as 40 g/L CHPTAC and 10 g/L sodium hydroxide in water at a ratio of 1:50, with a temperature at 90°C for 30 minutes (**Figure 1**).



**Figure 1.** Modification of cotton loose fiber.

### 2.3. Extraction Method of Purple Onion Skin

Dry purple onion skin was ground into powder and immersed in a mixture of ethanol concentrations in deionized water with a dilution ratio of 1:20 for extraction, then heated to 70 °C at 2 °C /min, maintained at that time for 2 h, and then cooled at room temperature. Filter the mixture using a 300-mesh nylon mesh, spin steam to remove ethanol and fix the volume, centrifuge it at a speed of 6000 rpm for 15 min to eliminate the solids particles and obtain a dye solution, which is stored for further use. The chemical structures of the main components of the Purple onion skin extract and the major colorant is quercetin as shown in **Figure 2**.



**Figure 2.** The chemical structures of the main components of the purple onion skin extract.

### 2.4. Dyeing for Cotton Loose Fiber

Weighted 2 g modified cotton loose fiber, placed it into the purple onion skin extracted dye solution with a dye concentration of 40% at a liquor ratio of 1:50, the pH value was adjusted to 6, and raised the temperature to 80 °C at 2 °C /min for 60 min.

### 2.5. Column Chromatography

Column chromatography consists of a vertical glass column (diameter of 25 mm \* 400 mm) filled tightly with a solid stationary phase by weigh of 40 g of silica gel

(200 mesh - 300 mesh), add an appropriate amount of hexane and stir evenly. Extracted liquid natural dyes are poured at the top of the silica gel, dissolved in a mobile phase solvent such as, hexane, ethyl acetate and methanol, and as the solvent flow down the column, components move at different rates based on their interactions with the stationary phase. After separating the pure colored components from the dye sample and collecting the sample in tube, the sample is examined by detecting equipment.

## 2.6. Test Methods

### 2.6.1. UV-Visible Spectra

UV-visible spectra of the mixed dye solutions were acquired using a Lambda 35 UV-VIS spectrophotometer (Elmer Perkin, USA) in the wavelength range of 200 nm - 800 nm.

### 2.6.2. Thin Layer Chromatography

TLC plates 25\*75 mm (Qingdao Ocean Chemical Co., Ltd.) were activated in a preheated oven at 60°C for 30 minutes. A small sample was applied near the bottom of the TLC plate and then placed in the developing agent solvent, which was pulled by capillarity and the spotted silica gel plate was placed in a dryer to dry and finally identify the different components.

### 2.6.3. FT-IR Spectroscopy

FT-IR spectra of the purification dye were acquired in the 4000  $\text{cm}^{-1}$  - 500  $\text{cm}^{-1}$  wavenumber range using a Nicolet is 50 FT-IR spectrometers at a resolution of 4  $\text{cm}^{-1}$  using 32 scans.

### 2.6.4. K/S Value

Use SF600X Datacolor colorimetric spectrometer to test the K/S value of the loose fiber under D65 light source.

### 2.6.5. Fastness Test

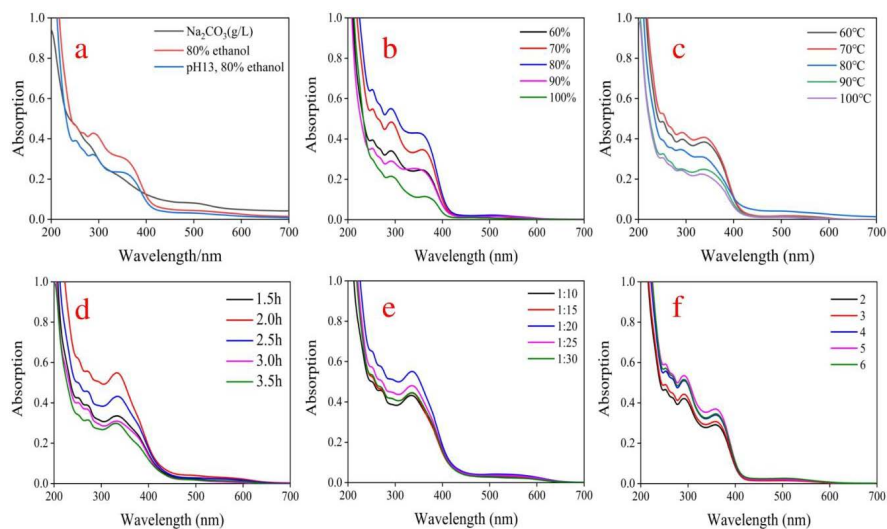
Referred to GB/T 8427-2008 "Textiles Color Fastness Test to Artificial Light Color Fastness: Xenon Arc" to measure the sunlight fastness of dyed samples; refer to GB/T 3921-2008 "Textiles Color Fastness" "Test color fastness to soaping" method, use a gray sample card to evaluate the discoloration of the sample and the stain fastness of the backing loose fiber.

## 3. Results and Discussion

### 3.1. Extraction Process Parameters Optimization

The ability of a solvent system to extract a natural dye is dependent on its properties. Because the molecule has a higher partition coefficient in the organic extraction solvent, making it more soluble, using polar organic solvents improves the extraction yield [16]. As seen in **Figure 3(a)**, The extraction efficiency of purple onion skin natural dye was significantly improved by using 80% aqueous ethanol solutions compared to 10 g/L sodium carbonate, and 80% absolute ethanol at pH

13 in deionized water under the same other process conditions (material-liquid ratio of 1:10, temperature of 70°C, time of 4h, and pH of 4). This is because of the fact that 80% aqueous ethanol strikes an effective balance between polar and non-polar properties, which leads to dissolve a difference of pigments. Its mildly acidic environment assists to protect sensitive pigments, while alkaline conditions can cause damage.



**Figure 3.** Optimization of purple onion skin extraction parameters, (a) Different extraction method, (b) Solvent system, (c) Temperature, (d) Time, (e) Material-liquid ratio, (f) pH.

**Figure 3(b)** shows a positive correlation with the solvent concentration, with the most effective concentration being around 80% by using different compositions of aqueous ethanol solutions under the same other process conditions (material-liquid ratio of 1:10, temperature of 70°C, time of 4 h, and pH of 4). This is because it can dissolve useful flavonoid pigments such as quercetin and kaempferol, and it has polar, non-polar, and non-toxic properties. Under the given extraction concentration parameters, 80% was considered the highest pigment concentration. The decline observed at higher concentrations could be attributed to the solvent's capacity to extract more contaminants and undesirable chemicals, potentially affecting the dye's color intensity. For the subsequent experiments, we selected the effective concentration of 80% aqueous ethanol solutions and changed only one of the remaining parameters each time, keeping the other variables constant. The adsorption curves of the dye at different temperatures (material-liquid ratio 1:10, time 4 hours and pH 4) are given in **Figure 3(c)**, where the maximum extraction was obtained at 70°C. A higher temperature will reduce the viscosity of the solvent and increase the molecular kinetic energy, so that more dye can enter the solvent [17]. Dye degradation at 70°C was probably minimized by optimizing the minimum extraction time required for effective dye release, maintaining a slightly acidic pH (5 - 6), and using 80% ethanol. Temperature affects the stability of the dye; while higher temperatures initially increase

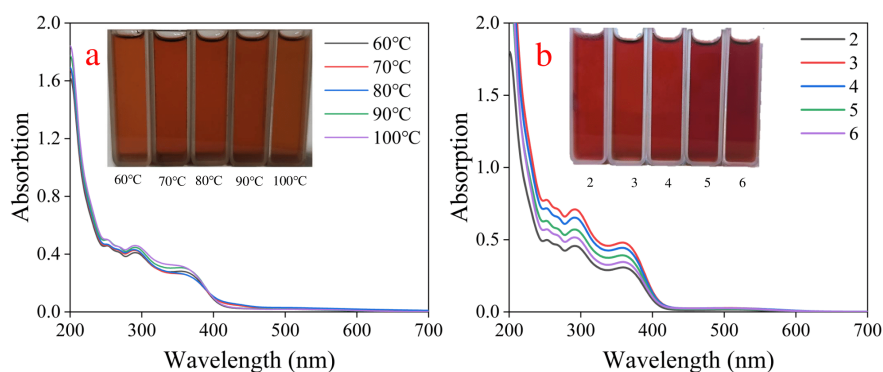
the extraction efficiency, above certain limit dye degradation is accelerated by oxidation, moisture analysis, and equilibration, leading to a decrease in dye concentration and color change. The temperature of 70°C was probably chosen to maintain a balance between effective extractions while minimizing thermal degradation of the target compounds. **Figure 3(d)** shows that the absorption value reaches the maximum, when the time was 2 hours, and after reaching a certain level, the extraction efficiency remains essentially constant or decreases slightly, with other parameters kept constant. In view of combined energy and time expenditure, the optimal extraction time was 2 hours. The effect of the solid-liquid ratios is shown in **Figure 3(e)**, the results showed that when the material-liquid ratio is 1:20, the absorbance value reaches the maximum, as the material-liquid ratio continues to increase, the absorbance value decreases. Therefore, the solid-liquid ratio was selected as 1:20 for follow-up experiments. **Figure 3(f)** indicates the maximum extraction capacity at pH 5. A slightly acidic environment (pH 5 - 6) is crucial for preserving the properties of the dye because flavonoids such as quercetin and kaempferol are more stable under these conditions, which prevents the degradation that occurs in alkaline environments. This pH range probably also inhibits the activity of enzymes present in onion skin that can break down the dye molecules. In addition, it probably optimizes the solubility of the target compounds in 80% ethanol. The optimum pH was probably determined by conducting the extraction at different pH levels (range between 2 to 6). The extracts obtained were analyzed for dye yield, purity, and stability using techniques such as spectrophotometry and chromatography to identify the pH range that gave the best results and minimized degradation. Therefore, choose to store the dye solution under weakly acidic conditions.

### 3.2. Stability Analysis of Natural Dye

The concentrated dye stock solution was diluted to a certain multiple and divided into five parts, which were placed at 60°C - 100°C for 2 h and shaken. It can be seen from **Figure 4(a)** that within a certain temperature range (60°C - 100°C), the maximum absorption wavelength position of purple onion skin natural dye remains unchanged, and the absorbance of the extract increases slightly. This is because the solubility of the dye increases as the temperature increases. It shows that the structure and properties of the pigment do not change under the conditions of 60°C - 100°C, and the purple onion skin dye has good thermal stability.

As shown in **Figure 4(b)**, the color of the dye solution extracted at different pH values changes. Under pH values of 2 - 6, the color of the natural dye from purple onion skin, the maximum absorption wavelength changes, and the absorbance value increases. The natural dye from purple onion skin has a minimum stability under weak acidic condition. This is because, softens the plant tissue making the dye compounds more accessible, and enhances the solubility of those compounds which simplifies their separation and elution into the solvent under weak acidic conditions. Therefore, the dye solution is stored under strong weak acidic condition.





**Figure 4.** Stability effect of purple onion skin dye liquor; (a) Thermal stability; (b) pH stability.

### 3.3. Separation and Purification of Purple Onion Skin Plant Dye Components

#### 3.3.1. Pre-Separation of Purple Onion Skin Natural Dye by Thin Layer Chromatography

In order to screen out the developing agent and eluent that can separate the different pigment components of purple onion skin. A mixture of hexane, ethyl acetate and methanol was selected as the developing agent, and the different extraction phases of purple onion skin were pre-separated by thin layer chromatography. The development results are shown in **Figure 5**.



**Figure 5.** TLC development of purple onion skin in developing agent.

As seen in **Figure 5**, when the purple onion skin extract is extracted with hexane as the extraction phase, two clear spots are obtained, and there is a small amount of residue in the starting point sample; this is because hexane has a greater polarity and more components in the purple onion skin extract are dissolved in the hexane phase development results, the  $R_f$  value of quercetin is 0.74, the  $R_f$  value of kaempferol is 0.38.



**Table 1.** Effect of different migration ratios on TLC Rf value.

Extraction phase	Developing agent	Rf value	
		Quercetin	Kaempferol
Hexane phase	5:2:0.1	0.35	0.15
	5:3:0.1	0.42	0.22
	5:4:0.2	0.58	0.30
	5:5:0.3	0.74	0.38
	5:5:0.4	0.85	0.41

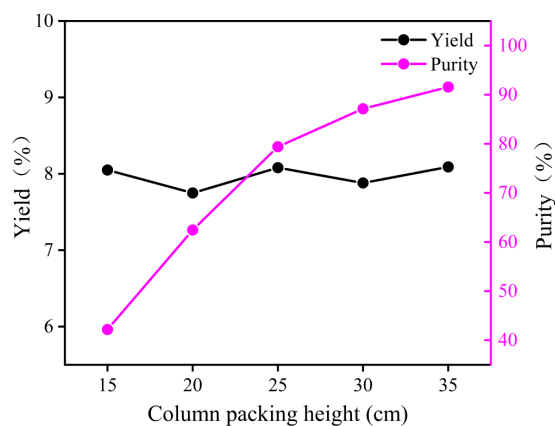
Note: Developing agent: hexane: ethyl acetate: methanol.

From **Table 1**, the Rf value is the ratio of the solute migration distance to the mobile phase migration distance. According to the general thin layer separation requirements, it is believed that a good separation effect can be achieved when the Rf value is between 0.3 and 0.5, and an acceptable separation effect can be achieved when the Rf is between 0.2 and 0.8. In the hexane phase development results, when the ratio of hexane: ethyl acetate: methanol is 5:2:0.1, the Rf value is small and the separation effect is poor. The reason is that the polarity of the developing agent is too small; the spot climbing speed is slow, resulting in a low spot Rf value. When the ratio of hexane: ethyl acetate: methanol is 5:5:0.3, the Rf value of quercetin is 0.74, the Rf value of kaempferol is 0.38, The Rf value is between 0.3 and 0.5, and the separation effect is good.

### 3.3.2. Separation of Purple Onion Skin Plant Dye Components

#### (1) Effect of column packing height on column chromatography separation performance

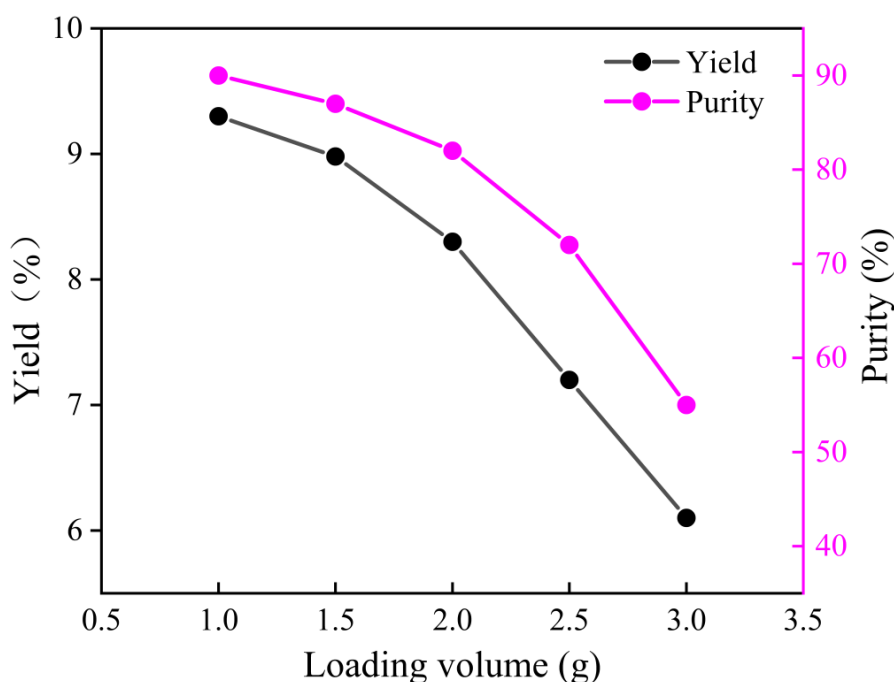
The eluent was petroleum ether-ethyl acetate system, the silica gel mesh size was 200 mesh - 300 mesh and the sample amount was 2.0 g. The effect of column height on the yield and purity of quercetin was investigated during one loading. The experimental results are shown in **Figure 6**.

**Figure 6.** Effect of different column packing heights on column chromatography separation performance.

Shown in **Figure 6**, when the height of the silica gel column increases, the yield of quercetin remains basically unchanged, but the purity of quercetin gradually increases. The reason for this phenomenon is that when the column height is too low, the substances to be separated cannot be completely separated in the stationary phase; as the column height increases, the number of theoretical plates in the silica gel column increases, and the separation purity gradually increases, but when the column height reaches a certain range, the growth rate of shikonin purity tends to be stable and the separation efficiency decreases. Comprehensive analysis shows that when the silica gel column height is 35 cm for one sample loading, the yield of quercetin can reach 8.09% and the purity is 91.56%.

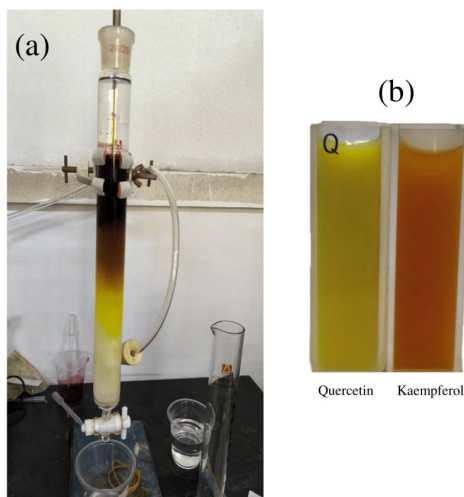
## (2) Effect of sample loading on column chromatography separation

The eluent was hexane-ethyl acetate, the silica gel mesh size was 200-300 mesh and the column height was 30 cm. The effect of the sample load was investigated. The experimental results are shown in **Figure 7**.



**Figure 7.** Separation of purple onion skin by column chromatography with different sample loading amounts.

As shown in **Figure 7**, when the sample load increases, the yield of quercetin decreases, and the yield is 6.1% when the sample load is 3 g. The reason for this phenomenon is that when the column height is constant, when the sample load is greater than 2 g, the silica gel column is overloaded, the substances to be separated cannot be completely separated in the silica gel stationary phase, and the purity of quercetin decreases significantly. After comprehensive consideration, the sample load is selected as 1.0 g, at which time the yield of quercetin is 9.3% and the purity is 90%.

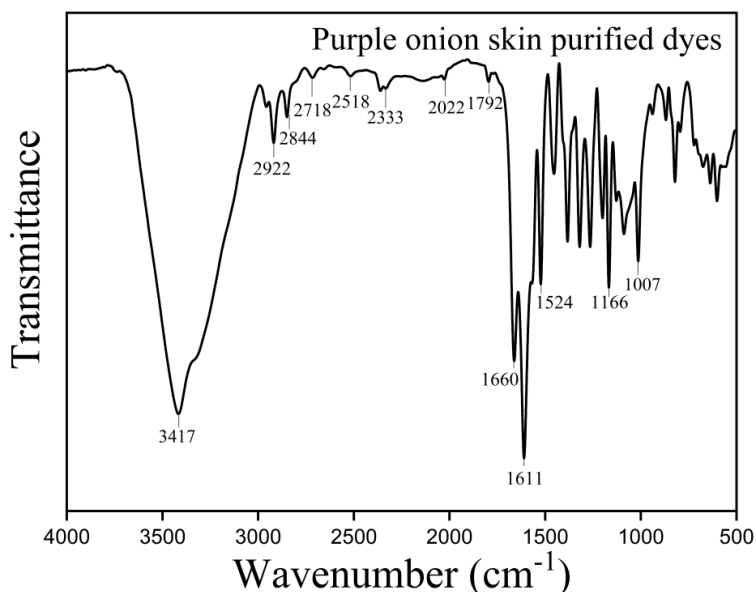


**Figure 8.** Separation of purple onion skin extract by Column chromatography; (a) Column chromatography process; (b) Separation components.

As seen in **Figure 8**, separation of purple onion skin extract by Column chromatography (a) Column chromatography process (b) separation components. Hexane and Kaempferol in purple onion skin are separated by silica gel column chromatography; the silica gel column is gradient eluted by an eluent, and quercetin can be effectively eluted when the volume ratio of hexane to ethyl acetate is 5:5:0.3, kaempferol can be effectively eluted when the volume ratio of hexane to ethyl acetate is 5:5:0.4.

### 3.4. FT-IR Spectroscopy

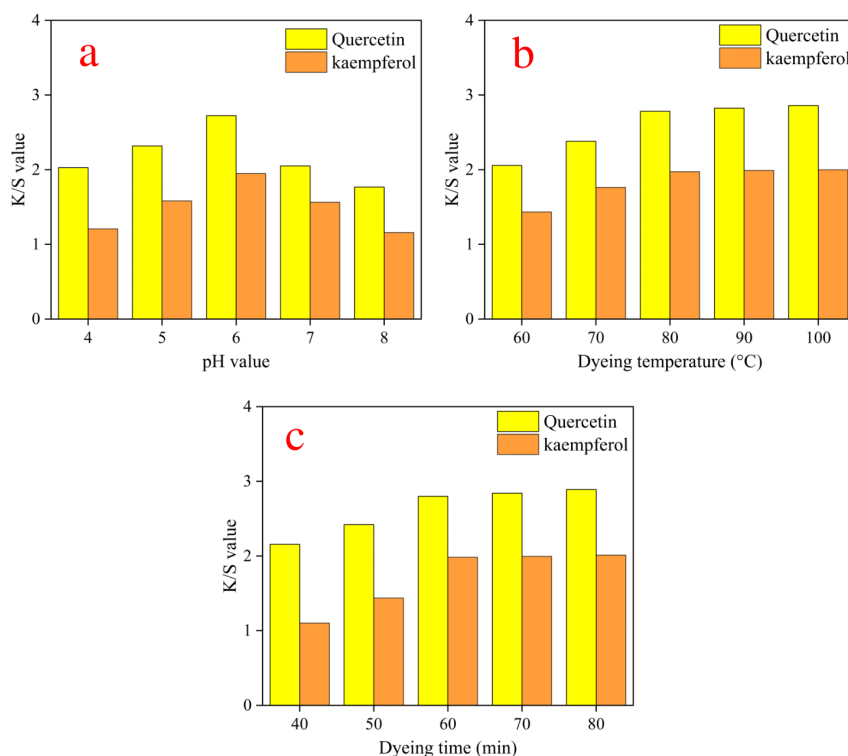
The isolated product of purple onion skin was subjected to FTIR analysis, and the results were shown in **Figure 9**.



**Figure 9.** Infrared spectra of purple onion skin separated natural dye extract.

From **Figure 9**, the FTIR spectrum of purified quercetin from purple onion skin shows important functional groups. A broad peak at  $3417\text{ cm}^{-1}$  indicates O-H stretching, likely from phenolic hydroxyl groups and water. The peaks at  $2922$  and  $2844\text{ cm}^{-1}$  relate to C-H stretching in alkyl groups. A strong peak at  $1660\text{ cm}^{-1}$  suggests C=O stretching, which may come from a carbonyl or ketone group in the quercetin structure. The peak at  $1611\text{ cm}^{-1}$  is likely due to C=C stretching, indicating the presence of aromatic rings in quercetin. Peaks between  $1500\text{ cm}^{-1}$  -  $1000\text{ cm}^{-1}$ , such as those at  $1524$ ,  $1166$ , and  $1007\text{ cm}^{-1}$ , show C-O stretching and aromatic ring vibrations, further supporting the identification of quercetin. These results confirm that a quercetin-rich fraction has been successfully separated from purple onion skin.

### 3.5. Effect of Separated Dye Solution on K/S Value of Cotton Loose Fiber Dyeing



**Figure 10.** Effect of separated dye solution parameters; (a) pH value, (b) Dyeing temperature and (c) Dyeing time on K/S value of cotton loose fiber dyeing.

As shown in **Figure 10(a)**, at pH 6, the K/S value of quercetin is 3.66, and the K/S value of kaempferol is 1.65. As the pH value increases, quercetin has good stability under weak acidic. The K/S values of kaempferol decrease significantly; the reason is that quercetin has a better affinity for cationic cotton fiber than kaempferol. Under alkaline conditions, the K/S values of the two pigments are the worst, because the pigments are poorly stable under alkaline conditions, and the pigments are destroyed, resulting in a decrease in dyeing performance.



From **Figure 10(b)**, at a temperature of 80°C, the K/S value of quercetin is 3.66, and the K/S value of kaempferol is 1.45. As the temperature rises to 100°C, the K/S value does not change much; this is because the dye component is dispersed in the dye solution. When entering the dyeing stage, the entire dye solution system gradually heats up. On the one hand, the dye molecules move actively and obtain more energy for diffusion under the action of heat. Therefore, 80°C is selected for subsequence process.

As shown in **Figure 10(c)**, when the dyeing time is 60 min, the K/S value of quercetin is 3.66, and the K/S value of kaempferol is 1.55. When the dyeing time is 60 min, the K/S value of quercetin and kaempferol is higher than at 70 min. The K/S value of the fiber increases first and then decreases with the extension of dyeing time. As the time rises to 60 min, the K/S value does not change much; this is because the dye component is stable in the dye solution. Therefore, 60min is selected for subsequence process.

### 3.6. Dyeing Properties and Dye Fastness of Cotton Loose Fiber with Separated Purple Onion Skin Dye

The CIELAB color space is a three-dimensional model representing the way people perceive colors in terms of lightness- $L^*$ , redness-greenness- $a^*$ , and yellowness-blueness- $b^*$  and fastness test results are shown in **Table 2**.

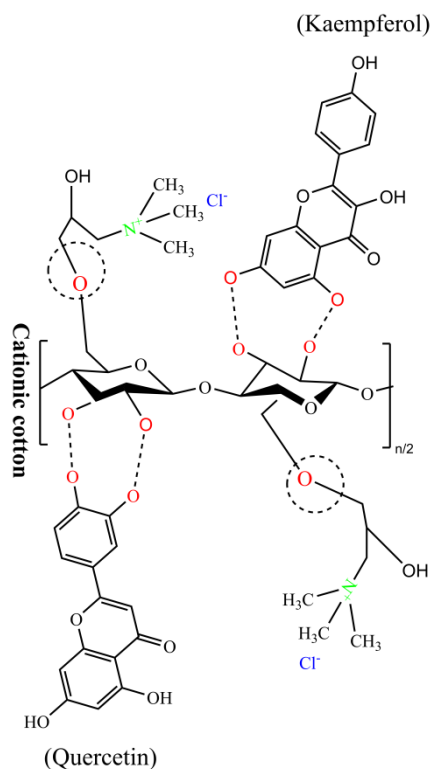
**Table 2.** Dyeing properties and color fastness of purple onion skin pigments dyed on modified cotton loose fiber.

Dye types	$L^*$	$a^*$	$b^*$	K/S	Wash fastness	Light fastness	Sample
Quercetin	71.17	15.46	19.35	2.72	4	3 - 4	
Kaempferol	64.54	16.69	13.31	1.97	4	3	

Note: dye concentration of 5% at a liquor ratio of 1:50, the pH value was adjusted to 6, and raised the temperature to 80°C at 2°C/min for 60 min.

**Table 2** presents that, the dyeing properties of two key pigments, such as Quercetin and Kaempferol extracted from purple onion skin, when applied to modified purple cotton loose fiber  $L^*$ ,  $a^*$ ,  $b^*$  value express that Quercetin resulted  $L^* = 71.17$ ,  $a^* = 15.46$  and  $b^* = 19.35$  and Kaempferol resulted  $L^* = 64.54$ ,  $a^* = 16.69$  and  $b^* = 13.31$ . Quercetin is a lighter and more potentially more yellow-toned purple compared to kaempferol. The K/S values indicate that quercetin expresses higher color strength (2.72) than Kaempferol color strength (1.97) under the test conditions. Regarding fastness properties, both dyes showed good wash fastness and acceptable moderate

light fastness, with Quercetin achieving a rating of 3 - 4 and Kaempferol is 3. This is because of better hydrogen bonding of dye with on the cotton fiber during dyeing.



**Figure 11.** Proposed chemical interaction between quercetin, kaempferol and cationic cotton loose fiber.

From **Figure 11**, the -OH on the quercetin, kaempferol can form a hydrogen bond with the -OH on the cotton fiber during dyeing. These findings suggest that purple onion skin can effectively dye on modified cotton loose fiber with acceptable fastness rating.

#### 4. Conclusion

In this study, we successfully extracted natural dye from purple onion skin and optimized the extraction process of the dye by single-factor analysis. Thermal and pH stability have been analyzed. For separation and purification of purple onion skin, thin layer chromatography was used to pre-separate purple onion skin by using hexane phase and the developing solvent ratio for effective separation and purification by silica gel column chromatography. FTIR data confirms the presence of quercetin-rich fraction. Meanwhile, direct dyeing with separated and purified quercetin and kaempferol was successfully carried out without using any inorganic salt and metal mordant for the cleaner production of environmentally friendly value-added products, good wash fastness and acceptable moderate light fastness, with Quercetin achieving a rating of 3 - 4 and Kaempferol is 3. The con-

tent of this research fully validates the feasibility of a new horizon through future studies that should focus on optimizing the extraction processes and exploring a broader group of component separation and purification which can provide the palette with even more colors and new properties of the textile colored. It will be a key contributing factor to the transformation of the textile industry to more sustainable production practices aligning with larger global priorities to protect human health and ensure ecological integrity.

### Data Availability

Data will be made available on request.

### Ethics Statement

Ethics statement for the use of human and animal subjects (may require consent to participate and consent to publish for human subjects): not applicable.

### Consent for Publication

Each author has consented to the publication of this study.

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### Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

### References

- [1] Abate, M.T., Zhou, Y., Guan, J., Chen, G., Ferri, A. and Nierstras, V. (2020) Colouration and Bio-Activation of Polyester Fabric with Curcumin in Supercritical CO<sub>2</sub>: Part II—Effect of Dye Concentration on the Colour and Functional Properties. *The Journal of Supercritical Fluids*, **157**, Article 104703. <https://doi.org/10.1016/j.supflu.2019.104703>
- [2] Haddar, W., Baaka, N., Meksi, N., Elksibi, I. and Farouk Mhenni, M. (2014) Optimization of an Ecofriendly Dyeing Process Using the Wastewater of the Olive Oil Industry as Natural Dyes for Acrylic Fibres. *Journal of Cleaner Production*, **66**, 546-554. <https://doi.org/10.1016/j.jclepro.2013.11.017>
- [3] Jamee, R. and Siddique, R. (2019) Biodegradation of Synthetic Dyes of Textile Effluent by Microorganisms: An Environmentally and Economically Sustainable Approach. *European Journal of Microbiology and Immunology*, **9**, 114-118. <https://doi.org/10.1556/1886.2019.00018>
- [4] Girdthep, S., Sirirak, J., Daranarong, D., Daengngern, R. and Chayabutra, S. (2018)



- Physico-Chemical Characterization of Natural Lake Pigments Obtained from *Caesalpinia sappan* Linn. and Their Composite Films for Poly(Lactic Acid)-Based Packaging Materials. *Dyes and Pigments*, **157**, 27-39. <https://doi.org/10.1016/j.dyepig.2018.04.043>
- [5] Choudhury, A.K.R. (2018) Eco-Friendly Dyes and Dyeing. *Advanced Materials and Technologies for Environmental*, **2**, 145-176.
- [6] Patel, B. and Kanade, P. (2019) Sustainable Dyeing and Printing with Natural Colours vis-à-vis Preparation of Hygienic Viscose Rayon Fabric. *Sustainable Materials and Technologies*, **22**, e00116. <https://doi.org/10.1016/j.susmat.2019.e00116>
- [7] Baseri, S. (2022) Ecological Dyeing of Cotton Fabric with *Matricaria recutita* L. in the Presence of Human Hair Keratins as an Alternative Copartner to Metallic Mordants. *Sustainable Materials and Technologies*, **32**, e00405. <https://doi.org/10.1016/j.susmat.2022.e00405>
- [8] Rather, L.J., Zhou, Q., Ali, A., Haque, Q.M.R. and Li, Q. (2020) Valorization of Natural Dyes Extracted from Mugwort Leaves (*Folium artemisiae Argyi*) for Wool Fabric Dyeing: Optimization of Extraction and Dyeing Processes with Simultaneous Coloration and Biofunctionalization. *ACS Sustainable Chemistry & Engineering*, **8**, 2822-2834. <https://doi.org/10.1021/acssuschemeng.9b06928>
- [9] Al-Tohamy, R., Ali, S.S., Li, F., Okasha, K.M., Mahmoud, Y.A.-G., Elsamahy, T., *et al.* (2022) A Critical Review on the Treatment of Dye-Containing Wastewater: Ecotoxicological and Health Concerns of Textile Dyes and Possible Remediation Approaches for Environmental Safety. *Ecotoxicology and Environmental Safety*, **231**, Article 113160. <https://doi.org/10.1016/j.ecoenv.2021.113160>
- [10] Xu, X., Gong, J., Li, Z., Li, Q., Zhang, J., Wang, L., *et al.* (2020) Mordant Free Dyeing and Functionalization of Wool Fabrics with Biocolorants Derived from *Apocynum Venetum* L. Bast. *ACS Sustainable Chemistry & Engineering*, **8**, 12686-12695. <https://doi.org/10.1021/acssuschemeng.0c04757>
- [11] Imani, H., Gharanjig, K. and Ahmadi, Z. (2022) A Novel Efficient Method for Eco-Friendly Deep Dyeing of Wool Yarns by Extracted Madder Dyes in the Presence of Additives. *Industrial Crops and Products*, **183**, Article 114970. <https://doi.org/10.1016/j.indcrop.2022.114970>
- [12] Reddy, N., Han, S., Zhao, Y. and Yang, Y. (2012) Antimicrobial Activity of Cotton Fabrics Treated with Curcumin. *Journal of Applied Polymer Science*, **127**, 2698-2702. <https://doi.org/10.1002/app.37613>
- [13] Lee, K.A., Kim, K., Kim, H.J., Chung, M., Chang, P., Park, H., *et al.* (2014) Antioxidant Activities of Onion (*Allium cepa* L.) Peel Extracts Produced by Ethanol, Hot Water, and Subcritical Water Extraction. *Food Science and Biotechnology*, **23**, 615-621. <https://doi.org/10.1007/s10068-014-0084-6>
- [14] Karabulut, K. and Atav, R. (2020) Dyeing of Cotton Fabric with Natural Dyes without Mordant Usage Part I: Determining the Most Suitable Dye Plants for Dyeing and UV Protective Functionalization. *Fibers and Polymers*, **21**, 1773-1782. <https://doi.org/10.1007/s12221-020-9365-2>
- [15] Pal, C.B.T. and Jadeja, G.C. (2018) Deep Eutectic Solvent-Based Extraction of Polyphenolic Antioxidants from Onion (*Allium cepa* L.) Peel. *Journal of the Science of Food and Agriculture*, **99**, 1969-1979. <https://doi.org/10.1002/jsfa.9395>
- [16] Rather, L.J., Ali, A., Zhou, Q., Ganie, S.A., Gong, K., Rizwanul Haque, Q.M., *et al.* (2020) Instrumental Characterization of Merino Wool Fibers Dyed with *Cinnamomum camphora* Waste/Fallen Leaves Extract: An Efficient Waste Management Alternative. *Journal of Cleaner Production*, **273**, Article 123021.

<https://doi.org/10.1016/j.jclepro.2020.123021>

- [17] Fang, J., Meng, C. and Zhang, G. (2022) Agricultural Waste of *Ipomoea batatas* Leaves as a Source of Natural Dye for Green Coloration and Bio-Functional Finishing for Textile Fabrics. *Industrial Crops and Products*, **177**, Article 114440.

<https://doi.org/10.1016/j.indcrop.2021.114440>