THERMAL EXTRACTION OF PECTIN FROM THE STEM BARK OF ECO-WOOD HIBISCUS AND ITS APPLICATION TO CASHMERE FABRICS

by

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In this study, natural plant pectin was extracted from the stem bark of Hibiscus mutabilis and utilized to finish knitted cashmere fabrics. The performance differences between the fabrics before and after finishing were investigated. The results demonstrate that the extraction solution, comprising hibiscus stem bark tartaric acid at a pH of 2, extraction time of 90 minutes, and a material-liquid ratio of 1/30, yielded pectin at a yield of up to 13.24%. This yield is not as high as that of tartaric acid. Following the finishing of the knitted cashmere fabric with hibiscus pectin, a notable enhancement in the fabric's antibacterial and UV properties was observed. Following the application of a 7.5% hibiscus pectin solution, the transmission rate of UV light with a wavelength of less than 290 nm exhibited a tendency towards zero, while the ultraviolet protection factor value reached 43. Furthermore, the bacterial inhibition rate of Staphylococcus aureus was observed to reach 94.571%.

Key words: *hibiscus sabdariffa, stem bark, pectin, knitted cashmere fabric, finishing, UV protection, antibacterial property*

Introduction

Natural fibers always have some special properties, the most famous ones are the spider silk [1], silkworm cocoon [2], and animals hairs [3]. There is also a trend to fabricate nanofibers from natural silks, *e.g.*, sea-silk based nanofibers [4], silkworm-based nanofiber [5], snail-based nanofibers [6], latex-based nanofibers [7], mussel-based nanofibers [8], and spider-based nanofibers [9] by electrospinning technology [10-13]. This paper is to attract natural plant pectin from stem bark of Hibiscus mutabilis [14].

Hibiscus mutabilis, also known as the hibiscus flower, wood lily, is native to the Yangtze River basin in China and Southeast Asia. It is a deciduous shrub or small tree cultivated for its flowers and leaves, which can be used as a medicinal plant. The flowers and leaves are employed in the treatment of a variety of ailments, including those related to the

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blood, bleeding, heat, detoxification, pain, swelling, and other bodily functions. Additionally, they are non-toxic and devoid of any adverse side effects. Recent studies have demonstrated that Hibiscus sabdariffa contains flavonoids, which exhibit inhibitory effects on the growth of the typhoid bacillus and Staphylococcus aureus. The majority of studies on Hibiscus sabdariffa have focused on the flowers and leaves, with fewer investigations conducted on the stem bark and associated organisms. An optimal natural dyeing process with hibiscus was proposed [15]. The research group has demonstrated that pectin produced by degumming the stem bark of certain plants exhibits greater antibacterial and radiation protection. However, the UV resistance and antibacterial properties of the knitted cashmere fabric are of poor quality. In order to enhance the economic value of knitted cashmere products, the group employed the acid extraction process, acid dilution as the extraction solution, to study the effects of pH value, extraction time, material-liquid ratio, and other processing parameters on the extraction of pectin from the wood hibiscus stem bark. The extraction ratio and other processing parameters were optimized in order to enhance the extraction of pectin from wood hibiscus stem bark. The resulting pectin was found to be an optimal choice for knitting cashmere fabrics for functional finishing, which significantly enhanced the fabric's antibacterial and radiation resistance. The improvement in fabric quality was evident in the final product. The radiation performance of the product can be effectively enhanced, thereby conferring a strong practical application and promotional value.

Experimental

The following materials and reagents were utilized in this experiment:

- The stem bark of Hibiscus sabdariffa, sourced from the Dafeng Salt Soil in North Jiangsu Province, with a salt content of 6-10‰.
- Tartaric acid, ethanol, hydrochloric acid, and sulfuric acid, all of which were analytically pure and procured from the Shanghai Shenxiang Chemical Reagent Co.

The following instruments were utilized in the experimental process: A HHS-type constant temperature water bath was procured from Nantong Sansi Electromechanical Technology Co., Ltd. A 400Y pulveriser was sourced from Platinum Europe Hardware Factory. An FA2004 electronic balance was obtained from Shanghai Shunyu Hengping Scientific Instrument Co., Ltd. A pocket pH meter was purchased from Shanghai Bonte Instrument Co. Pipettes, flasks, glass rods, quantitative analysis filter paper, and other necessary equipment were sourced from Fushun Dongyang Industry and Trade Co.

The extraction of pectin from the stem bark of Hibiscus mutabilis is a multi-step process. The first step is the pretreatment of the stem bark, which involves the removal of impurities and the reduction of moisture content. This is followed by heating and reflux extraction, filtration, concentration, alcohol precipitation, pectin precipitation, and drying. Finally, the pectin is isolated from the stem bark of Hibiscus mutabilis.

The pretreatment of the wood hibiscus stem bark involves the introduction of the dried material into a 400Y crusher for 30 seconds, after which the resulting powder is weighed according to the experimental regulations. The weight of the powder is recorded as 5 g.

The configuration of the tartaric acid extraction solution is: A volume of 500 mL of distilled water is to be measured, after which a few drops of the tartaric acid solution are to be pipetted into the injection. The pH is then to be adjusted to the set value of the test programme.

Heating reflux extraction is a process whereby a substance is extracted from a sample by heating it in a reflux apparatus. The pretreated hibiscus sabdariffa stem bark powder

was loaded into a 300 mL flask, with the volume of extract solution set in the test protocol. The powder was then gently stirred with a glass rod to ensure complete moistening. The mouth of the flask was sealed with cling film. The flask was placed in a constant temperature water bath heated to 100 °C and the recording time was set according to the test protocol. The flask was reflux extracted, with regular shaking to prevent internal charring [16].

Pectin precipitation. The extract was filtered through medical-grade skimmed knitted cashmere fabric a total of two or three times to remove impurities, and then filtered through filter paper to remove any remaining impurities. The filtrate was fixed to 30 mL, and a 7-fold volume of 95% ethanol solution was added. The mixture was then gently shaken and stirred well, left to stand for half an hour, filtered, and the extract was washed several times with 95% ethanol to remove any excess pigment. The sample was then dried in a constant temperature oven at 75 °C for one hour and weighed.

Based on the results of previous experiments, the material-liquid ratio was set at 1:30. A three-factor, three-level orthogonal experiment was designed to identify the optimal process for the extraction of wood hibiscus pectin, with the factors being the type of extracting acid, extraction time, and extraction pH. The selected factors and levels are presented in Table 1, which depicts the results of the orthogonal experiment [17].

Table 1. Orthogonal experiment factor levels

	Factors		
	A Acid	B Extraction pH	C Extraction time/min
1	hydrochloric acid	2.0	60
2	sulphuric acid	2.5	90
3	tartaric acid	3.0	120

The following materials were utilized in the experimental process: boiled 1+1 rib-knitted cashmere fabrics, wood hibiscus pectin, distilled water, Staphylococcus aureus (S. aureus) ATCC6638, SCDLP liquid medium, and counting medium (EA), all provided by Nanwei Yuda Fiber Technology Company.

The experimental apparatus. The following apparatus was employed in the course of this study: a HHS-type constant temperature water bath (Nantong Sansi Electromechanical Science and Technology Co., Ltd.), a UV transmission and sun protection tester (Shanghai Shen'an Medical Instrument Factory), a vertical pressure steam steriliser (Shanghai Shen'an Medical Instrument Factory), a biosafety cabinet (Shanghai Sujing Industry Co., Ltd.), and a watertight incubator (Shanghai Yihang Scientific Instrument Co.).

The extracted hibiscus pectin was subjected to a drying process at room temperature, resulting in the formation of a powdered pectin. A specific quantity of pectin powder was then weighed and placed into a measuring cup, followed by the addition of a specific volume of distilled water. This process resulted in the creation of a finishing solution with mass fractions of 0.0%, 2.5%, 5.0%, and 7%. The pectin solution was prepared at a concentration of 5% and the finishing solution bath ratio was 1:50. The configured finishing solution was placed in a well-set constant temperature water bath, with a glass rod constantly stirring. The solution was observed for complete dissolution, which was confirmed by immersing four pieces of knitted cashmere fabrics in the corresponding mass fraction of the finishing solution. The tim-

ing commenced with the commencement of the fabric's complete infiltration and was conducted with a glass rod in a constant, stirring motion. After 30 minutes, the fabric was removed, rinsed with distilled water, and placed in a clean, dry room temperature environment. Once the fabric has dried, it is advisable to conduct a test to ascertain its UV protection performance.

Bacterial culture. The fabric sample was divided into pieces, weighed to an accuracy of 0.05 grams, and then divided again into smaller portions. Each portion was wrapped in sterile material and sterilized at 103 kPa, 125 °C for 15 minutes. The sample piece was then placed into a 10 mL test tube, to which 0. A 2 mL preparation medium bacterial suspension was added to the test tube, which was then fixed on the test tube rack at a temperature of 20-25 °C. The test tube was cultured for 24 hours after sampling, after which a pipette was used to suck up 1 mL of solution. This was done 10 times using the method of dilution. The sample is then shaken, 1 mL is sucked up and added to a sterilized petri dish, the counting medium (EA) is poured in at a volume of approximately 15 mL, and the dish is allowed to solidify at room temperature. The dish is then inverted and incubated at 37 °C for 24 hours. Samples are then taken for testing the antibacterial properties.

Experimental results and analysis

As illustrated in fig. 1, the quality of pectin extracted from different pH values varies considerably. When the pH value is 2.0, the pectin extraction effect is optimal, with an extracted pectin quantity of 0.735 g. Between pH 2.0 and 3.0, the weight of pectin decreases rapidly, while between pH 3.0 and 4.0, the amount of extraction decreases more gradually. The primary rationale for this phenomenon is that an increasingly acidic extracting solution is conducive to the hydrolysis of cross-linked polysaccharides in water. Pectin, the primary component of pectin, is a galacturonic acid-based polysaccharide, which is also an acidic substance. Consequently, as the pH value declines, the quantity of pectin extracted diminishes significantly.

The pH value of the selected extraction solution was 2.0, and other conditions remained unchanged, as can be seen from fig. 2. It can be observed that, under the condition of other conditions being the same, the weight of extracted pectin increased with the prolongation of time before the extraction time was 90 minutes. In this period, the weight of extracted

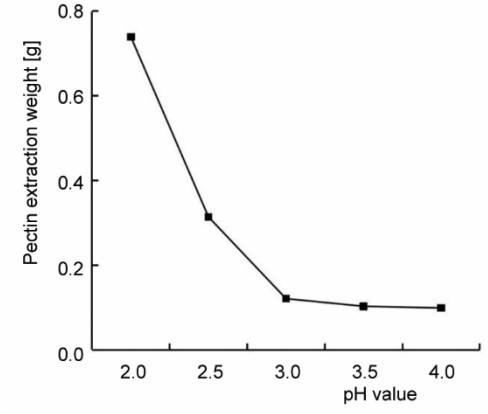

Figure 1. Pectin extraction weight at different pH values

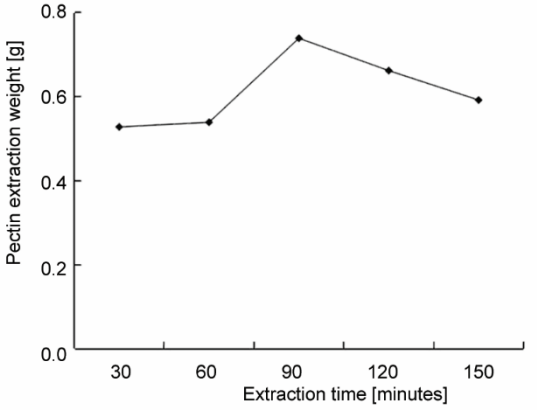

Figure 2. Pectin extraction weight at different extraction times

pectin was the fastest from 60-90 minutes. Conversely, the amount of extracted pectin decreased with the prolongation of time after 90 minutes. The highest amount of pectin extracted was 0.739 g in an extraction time of 90 minutes. The rationale behind this phenomenon is that as the acidity of the solution is increased, the extraction time is prolonged, allowing for greater penetration of tartaric acid into the stem bark of Hibiscus sabdariffa. This, in turn, enhances the hydrolysis of polysaccharides in the aforementioned tissue. However, when the extraction time exceeds a certain threshold, the hydrolysis of pectin polysaccharides is nearly complete, and the subsequent production of galacturonic acid is no longer observed.

The pH value of the extraction solution was selected as 2.0, the extraction time was 90 minutes, and all other conditions remained unchanged. The results are presented in fig. 3. As illustrated in the figure, the pectin extraction weight exhibited a rapid increase with the bath ratio increasing from 1:20 to 1:30. However, the extraction amount decreased rapidly from 1:30 to 1:40, and then exhibited a gradual increase from 1:40 to 1:60. Consequently, the bath ratio of 1:30 reached the highest extraction weight of 0.739 g, indicating that the material-liquid ratio of 1:30 is optimal for extracting the greatest pectin weight. The reason for this is that the bath ratio is low, and the tartaric acid content in the extracting solution is also low. Consequently, the contact rate between the stem bark of Hibiscus sabdariffa and the tartaric acid is also low. Conversely, when the bath ratio increases, the tartaric acid content in the extracting solution gradually increases. As the pectin content of the stem bark of Hibiscus sabdariffa increases and the contact rate of tartaric acid rises, the efficiency of the reaction improves and the extraction amount increases. However, if the bath ratio is further increased, a portion of the pectin may become degreased. When the bath ratio exceeds a certain range, the pectin polysaccharides are extracted. When the bath ratio exceeds a certain range, pectin polysaccharides are almost entirely hydrolyzed, resulting in the partial degreasing of pectin and a significant decline in pectin extraction. Thus, the optimal ratio is 1:30.

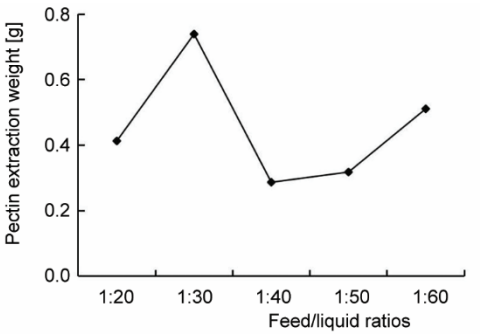

Figure 3. Weight of pectin extracted at different feed/liquid ratios

Previous experiments have demonstrated that hydrochloric acid, sulfuric acid, and tartaric acid are effective in extracting pectin from Hibiscus sabdariffa. The selected extract bath ratio was 1:30, and the pH value of the extract solution and the extraction time were varied in a three-factor, three-level orthogonal experiment. The results are presented in tab. 2.

As illustrated in tab. 2, the pectin yield extracted with hydrochloric acid ranged from 4.28% to 6.64%, that extracted with sulfuric acid from 7.3% to 11.7%, and that extracted with tartaric acid from 2.42% to 13.24%. The pH value of the extraction solution had the greatest effect on the pectin yield. As the pH value decreased, the acidity of the extraction solution increased, which effectively and rapidly promoted the hydrolysis of pectin. However, if the pH value was too low, the hydrolysis of pectin in the stem bark of Hibiscus sabdariffa was more severe, which could easily result in the defatting of pectin, and thus the pectin yield decreased. The extraction time has the least effect on the pectin yield. The optimal extraction time for different acidic reagents varies. The pectin yield is higher for 6# and 9# extractions. The experiment demonstrated that extraction stability was highest for 7#. The optimal extraction effect for the stem bark pectin of Hibiscus sabdariffa was achieved when a tartaric acid

solution was used as the extraction solution, the pH was 2, and the extraction time was 90 minutes.

Table 2. Extraction results of stem bark pectin of Hibiscus mutabilis

No.	A	B	C	Pectin yield [%]
1#	1	1	1	6.64
2#	1	2	2	5.64
3#	1	3	3	4.28
4#	2	1	2	7.30
5#	2	2	3	9.74
6#	2	3	1	11.70
7#	3	1	3	13.24
8#	3	2	1	8.74
9#	3	3	2	2.42
I_1	16.56	27.18	27.08	
I_2	28.74	24.12	15.36	
I_3	24.40	18.40	27.26	
K_1	5.52	9.06	9.03	
K_2	9.58	8.04	5.12	
K_3	8.13	6.13	9.09	
R	4.06	2.93	3.97	

Knitted cashmere fabric is renowned for its breathability, skin-friendliness, and comfort. It is the most popular traditional fabric. However, the ultraviolet radiation permeability of knitted cashmere fabrics is considerable. Ultraviolet irradiation of the fiber results in macromolecular chain fracture, which damages the internal structure of the fiber. This, in turn, leads to a high transmittance of the fabric and poor light resistance characteristics.

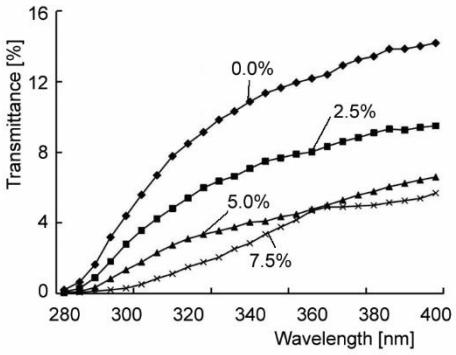

Figure 4. The UV transmittance of knitted cashmere fabrics after finishing

The experimental homemade wood hibiscus pectin was used to create a finishing solution with a concentration of 0.0%, 2.5%, 5.0%, and 7.5% of wood hibiscus pectin. This solution was applied to knitted cashmere fabric, and the results are shown in fig. 4. The UV resistance of the fabric was significantly enhanced with the increase in the concentration of the finishing solution. At a concentration of 7.5%, the wavelength of UV light transmission rate of less than 290 nm tends to zero, and the ultraviolet protection factor (UPF) value reached 43. As the quality points of the finishing solution increase, the fabric UV transmission rate of less than 290 nm tends to zero, with the UPF value reaching 43.

The finishing liquid mass fraction also increases, with the UVA transmittance of knitted cashmere fabrics being 11.76%, 7.80%, 4.71% and 4.61%, respectively.

Bacterial culture experiments demonstrate that the number of colonies on knitted cashmere fabrics decreases gradually with the increase in pectin solution concentration. Table 3 presents the bacterial inhibition of knitted cashmere fabrics before and after finishing by pectin solution. Table 3 presents the antibacterial efficacy of knitted cashmere fabrics before and after pectin solution finishing. As illustrated in tab. 3, the antibacterial efficacy of the fabric increases with the concentration of the pectin finishing solution. The greater the concentration of pectin, the more pronounced the antibacterial performance. The antibacterial efficacy of knitted cashmere fabric against Staphylococcus aureus following finishing can reach 94.571%, exhibiting a superior antibacterial effect.

Table 3. Bacteriostatic inhibition rate of pectin-finished knitted cashmere fabrics

Concentration of finishing solution [%]	Number of Staphylococcus aureus colonies [pcs]	Staphylococcus aureus bacterial inhibition rate [%]	Staphylococcus aureus bacterial inhibition value [%]
0.0	$1.05 \cdot 10^6$	Control sample	Control sample
2.5	$1.7175 \cdot 10^5$	0.7863	83.643
5.0	$6.225 \cdot 10^4$	1.2271	94.071
7.5	$5.7 \cdot 10^4$	1.2653	94.571

Conclusion

Hibiscus mutabilis can be utilized as a source of natural plant pectin, and the prepared pectin can be employed as a finishing agent to enhance the ultraviolet protection and antibacterial properties of knitted plush fabrics. Additionally, it has the value of promotion in other textile fabrics. The extraction of pectin from the stem bark of Hibiscus sabdariffa, when tartaric acid is used as the extraction solution, is optimized by adjusting the pH value to 2, extending the extraction time to 90 minutes, and employing a casting ratio of 1:30. This extraction method yields the highest pectin yield from Hibiscus sabdariffa. The pectin yield was found to be up to 13.24%. Additionally, sulfuric acid, hydrochloric acid, and tartaric acid were employed as reagents in the preparation and extraction of pectin from the stem bark of Hibiscus sabdariffa. However, orthogonal experiments demonstrated that tartaric acid was more effective than the other two acids in extracting hibiscus pectin, exhibiting greater extraction stability. Sulphuric acid and hydrochloric acid were found to be less effective. The antibacterial properties and anti-ultraviolet performance of knitted cashmere fabrics treated with hibiscus pectin finishing have been significantly enhanced. This is due to the strengthening of the finishing effect as the quality fraction of the finishing solution increases. In order to take into account the fabric stiffness, smoothness, crease resistance, and other performance characteristics, it is recommended that the finishing solution quality fraction be 7. At a concentration of 5%, the finishing of the knitted cashmere fabrics results in a zero UV transmittance for wavelengths below 290 nm, an UPF value of 43, and a zero UV light transmittance for wavelengths below 290 nm. The UPF value reaches 43, and the bacterial inhibition rate of Staphylococcus aureus can reach 94.571%.

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