

Original Research Article OPTIMIZATION OF TANNIN'S EXTRACTION FROM CASHEW NUT TESTA FOR USE IN LEATHER TANNING

ABSTRACT

Extensive researches have been conducted over the years, focusing on the use of vegetable tannins to reduce pollution caused by mineral tanning. Vegetable tannins have been utilized commercially, but they remain costly and are not easily accessible. The purpose of this study was to optimize the tannin's extraction from cashew nut testa using water in order to determine their suitability in leather tanning. Samples of cashewnut testa were collected randomly from cashewnut processors in Mtwara region, Southern Tanzania. They were shade-dried and ground into powder. Various ratio of powder/water (1:6, 1:9, 1:12 and 1:15) -on weight basis were made for extraction, maintained at a temperature of 70 °C for 90 minutes. Results showed that, depending on the extract ratios, different tannin concentrations were obtained which had an implication on tannin extracted from powder. Experiment was found solution with ratio 1:9 have shown to extract about 50.73% of total tannins presented on cashewnut testa powder followed by solutions with ratio 1:12, ratio 1:15, and ratio 1:6 extracted about 46.27%, 43.51% and 38.95% respectively. Suitability of various tannins solution extracted from cashewnut testa was tested using conventional tanning process on sheepskins. The physical properties of the resultant leathers were determined using the standard methods (TBS/ISO). Under given condition, it has shown that using the ratios of 1:6, 1:9, 1:12 and 1:15 resulted in leather with tensile strength ranging between 19.41±6.36 MPa and 43.00±8.18 MPa and elongation at maximum load in the range of 34.48±10.12 % to 77.56±10.96 %., average shrinkage temperatures were above 75°C, and flexing test gave no damage of leather at 100,000 flexes. The physical properties of leather recorded have shown to meet the minimum recommended values from the standard (ISO 14931-2021). The mixing ratio 1:9 of cashew nut testa powder/water has been recommended for extracting tannin for vegetable tanning.

Keywords: optimization, tannin extraction, cashew nut testa, leather tanning.

1. INTRODUCTION

Development of industrial activities has played a major role in increasing demand of raw materials including chemicals. Chemicals used in industrial processes include solvents, acids, and other toxic substances, which can have detrimental effects on the environment if are not well managed. Improper disposal of chemical wastes from industrial processes can result in long-term damage to ecosystems and natural habitats. When released to the environment, some of these chemicals and their wastes can also contribute to global issues such as climate change and ozone layer depletion (Bais *et al.*, 2018). Among the industrial activities that use chemicals include leather tanning, which has a significant impact on the environment as it generates a substantial amount of pollution and wastes. The use of chemicals such as chromium, formaldehyde, and dyes can lead to water and soil pollution if not properly managed (Hashmi *et al.*, 2017). Chrome tanning takes the lead among tanning methods, it alone contributes to 88 - 90% of the global leather production (Das *et al.*, 2022).

During leather processing, the pelt absorbs 54 - 57% of chromium, leaving the remaining percentage as liquid and solid waste (Adiguzel-Zengin *et al.*, 2017). This has led to growing pressures on industries to adopt more sustainable and environmentally friendly practices, such as reducing the use of chemicals, implementing better waste management strategies, and investing in cleaner production technologies (Ikram *et al.*, 2021). A shift towards cleaner and more responsible industrial processes is crucial for protecting the environment and ensuring a healthier and more sustainable future for all. Thus, for the sustainability and attaining green leather industry, chemical pollution should be minimized or avoided to enable tanneries to adhere to environmental standards (China *et al.*, 2020). The use of plant-based extracts in leather tanning aligns with environmental awareness by promoting biodegradable renewable sources, and reduced energy consumption, support of traditional practices, and minimized chemical usage.

Vegetable tannins are complex and heterogeneous as they constitute groups of polyphenolic secondary metabolites of higher plants with molecular weight between 500 and 20,000 Da. They share the ability to bind to and precipitate proteins, alkaloids and polysaccharides (Vuolo *et al.*, 2019). Some common sources of vegetable tannins include tree barks such as oak, chestnut, and hemlock, as well as plant materials like tara, mimosa, and quebracho. These tannins have been used in brewing and winemaking, ink production, and manufacturing of medicines, adhesives, textile dyeing materials, water treatment chemicals and many more (Chandrasekaran *et al.*, 2013). Vegetable tannins are used in the leather industry in the tanning of animal hides and skins to produce leather. Tannins' composition and concentration vary considerably with the species, age and part of the host plant (Rubert-Nason *et al.*, 2021). Depending on the source used, extracted tannins confer different organoleptic and chemical properties to the produced leather (Shirmohammadli *et al.*, 2018). Past observations and recent experimental studies have shown that chemical characteristics of tannins play an important role on leather stability (Fraga-Corral *et al.*, 2020).

Cashew (*Anacardium occidentale L.*) is a perennial tree crop, originated from South America, and now widely grown in the tropics, including Tanzania. It is a multipurpose tree crop, from which almost all parts (roots, stem, bark, leaves, apples and nuts) are used (Kapinga *et al.*, 2010). The nut has two shells: a hard exterior shell and a testa. The testa, which is thin and brown in color wrapped around a slightly curved fleshy kernel. It protects the kernel from penetration by atmospheric oxygen, which may cause oxidative rancidity as the testa contains antioxidant and it presents nearly 3% of the total cashew nut weight (Trox *et al.*, 2011). The testa is removed during processing and mostly considered as a waste product (Zafeer *et al.*, 2023). Quantitative analysis has shown that cashewnut testa contains about 19.9 - 22.1 percent tannins (Ukoha *et al.*, 2010).

Tanzania is among the major producers of cashewnuts in the world, with production volumes varying from year to year. The 2019/20 Census results have shown that, the total cashewnuts production in Tanzania was 391,119 tons, with 390,403 tons alone from smallholder farmers and 707 tons from large scale farms. Almost all the smallholder farms are in the mainland Tanzania, distributed as follows: Mtwara region (51.6%), Lindi region (27.3%), and Ruvuma region (10.0%) (URT: *National Sample Census of Agriculture 2019/20*). From the large mass of cashew nut produced annually, the amount of cashew nut testa that could be gathered during cashew nut processing as a byproduct could be high.

Bioactive compounds from cashewnut testa could be used to produce value-added products in the different fields (Zafeer *et al.*, 2023). Extraction of such bio-active compounds requires proper selection of a suitable solvent, the extraction method and sample preparation methods as prime issues for attaining a good recovery. Different solvents can also be used for the extraction of phenolic compounds from by-products such as water, ethanol, methanol, acetone and ethyl acetate (Patra *et al.*, 2022). Different solvents combinations can also be used to extract polyphenols (Sulaiman *et al.*, 2011). The selection of proper method for tannin extraction depends on the specific characteristics of the plant material, the desired

quality of the extract, and the available equipment and resources. The efficiency of extraction is influenced by factors such as the chemical nature of phytochemicals, the extraction method used, the solvent used, sample particle size, and the presence of interfering substances (Do *et al.*, 2014). Water extraction method is considered the best for extracting tannins due to its safety, environment considerations, cost-effectiveness, and the ability to preserve the desired properties of the extracted tannins (Gbashiet *al.*, 2017). Moreover, the extraction of tannins from plant material is greatly influenced by temperature, time and solvent to sample ratio (Kardel *et al.*, 2013).

Therefore, it is necessary to conduct research on how to use cashew nut testa as a source of tannins, especially as a tanning agent. The purpose of this study was to optimize the extraction of tannins from cashew nut testa using water to be used for the tanning of skins into leather.

2. MATERIAL AND METHODS

The study was carried out at Kiwango Leather Cluster Company, located in Mwanza District, Kilimanjaro region (Coordinates 3.70341° S, 37.66593° E).

2.1 Materials used

Cashew nut testa was collected randomly from different cashewnut processors in Mtwara region, Southern Tanzania. Sheepskins were purchased from Mwanza abattoir in Kilimanjaro region, where environmental factors for growth were taken into consideration. Water as a solvent, and all reagents with analytical grade were used.

The equipment used were rotary drum (Cap: Max 50pcs, 4rpm), thermometer, weigh balance, pH meter, heated agitator reactor (Cap: Max 80 Litres, 5000rpm), vortex mixer (M37610-26), UV-Visible spectrophotometer (X-ma3000, ROK), hammer mill, and muslin cloth.

2.2 Tannin extraction

Cashew nut testa was shade-dried for three days, followed by grinding using a hammer mill and sieving to obtain a powder with particle size less than 1 mm.



Figure 1a; Cashewnuttesta

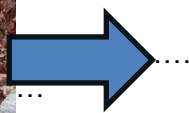


Figure 1b; Cashewnuttesta powder

Cashew nut testa powder was mixed with water in various ratio of powder/water based on weight in four batches (1:6, 1:9, 1:12 and 1:15) and continuously agitated in the reactor maintained at a temperature of 70 0C for 90 minutes. The solutions obtained were cooled for two hours and filtered using a muslin cloth. The pH of the extracted tannin solutions was measured. The extraction process was repeated twice in each ratio and five replicates of sample solution were taken randomly for tannin analysis.

2.3 Determination of tannin content

Stock standard solution of 1mg/ml of tannin acid was prepared by dissolving 100 mg of accurately weighed tannic acid in distilled water. The calibration curve was established using tannic acid (0 – 0.6 mg/ml) for the quantification of tannins in solution samples.

The total amount of tannin in extracted solution was measured using Folin-Ciocalteu phenol reagent method where 1 ml aliquot of the extracted tannin solution samples from ratio 1:6, 1:9, 1:12 and 1:15 were taken into 10 ml clear test tubes separately. 0.5 ml of Folin-Ciocalteu phenol reagent, 1 ml of saturated sodium carbonate solution (7.5%) were added and made up to 10 ml with distilled water to each test tube. All the reagents in each test tube were mixed properly using vortex mixer for 10 sec and kept undisturbed for 30 min for color development. The absorbance was read at wavelength of 760 nm against blank reagent using UV-Visible spectrophotometer (Soni *et al.*, 2018). Five replicates were taken per ratio to get reproducible results.

Moreover, the total amount of tannin in cashewnut testa powder was determined, where 1 g of sample was taken and the same procedures mentioned previous of Folin-Ciocalteu phenol reagent method applied. Five replicates were taken to get reproducible results.

2.4 Data analysis

The results obtained from tannin extraction were analyzed using StatPlus LE statistical packages version 7.7. One-way ANOVA, descriptive statistic was used to test the level of significance for the concentration of tannins of the cashewnut testa solutions. The p-Value < 0.05 was used to indicate the level of significance between the means of the tannin concentration obtained.

2.5 Tanning trial

The trials were conducted according to the procedures that involved skin soaking, liming, delimiting, bating, pickling, tanning, retanning and fatliquoring (*appendix 1*). All tanning trials received the same treatment except in tanning where various ratios of tannin solution were applied independently. Trials were conducted using the extracted tannin solutions (1:6, 1:9, 1:12 and 1:15) whereby the weight of solution applied was three times that of the pickled sheepskins. The retanning were done using equal weight of extracted tannin solution and tanned leather. Physical properties of the tanned leather including tensile strength and elongation at a break, shrinkage temperature and flex endurance were determined as among the leather quality attributes in comparison with standards.

2.5.1 Tensile strength and Elongation

Tensile strength and percentage of elongation of leather samples were measured by using Instron, Series IX AMTS 8.25.00 and Bluehill 3 version 3.41 software according to the official method (TZS 212/ISO 3376). Leather samples were cut parallel and perpendicular to the backbone using a dumbbell shaped press knife, set with jaws arranged 50 mm apart for standard testing and jaws separating at a rate of 100 mm/min. The greatest force recorded was taken as the breaking force (N) and the distance between the jaws at break relative to the original distance was taken as the percentage extension at break. The tensile strength (MPa) was calculated as the breaking force per area (width by thickness) of the strip. Each sample (crust) was measured ten times.

2.5.2 Shrinkage temperature

The shrinkage temperature of the tanned skins was measured using SATRA STD 114 test apparatus according to the official method (TZS 201/ISO 3380). Strips of leather 50 mm × 3 mm were cut from the tanned leather for assessment. The specimens were cut along and across the backbone. Holes were punched at the ends of the leather to allow the specimen to be held vertically in the test chamber filled with water and a small weight was attached to the lower end. The position of the lower end was indicated by an adjustable marker outside the tube to help judge when the shrinkage occurred. The apparatus was then closed and

water heated at approximately 4°C/min by applying the external heat source to the boiler components. The temperature at which the leather started to shrink was taken as the shrinkage temperature.

2.5.3 Flex Endurance

The flex endurance test was conducted according to the official method (TZS 206/ISO 5402) using a bally flexometer. Leather specimen of dimension 70 mm x 45 mm was folded and fixed to the jaws of the instrument in such a manner that the grain side remained outside with a fold on the specimen. The motor was switched on with one clamp fixed and the other moved backward and forward causing folds in the specimen to run along it. All ten samples were subjected to 300,000 flex cycles to assess the cracks and damage. The sample test specimens were observed after every 10,000 flex cycles.

3. RESULTS AND DISCUSSION

3.1 Tannin content obtained from extracted solution before and after leather tanning trial

Table 1 presents the quantities of tannins extracted from cashewnut testa powder in the four ratios of the tannin solution. Tannin concentration was obtained from the calibration curve ($y = 4.954x + 0.304$ with $R^2 = 0.9913$, where y is absorbance and x is concentration of standard tannic acid solution in mg/ml). The results show total tannins content found in the cashewnut testa powder was measured to be 268.32 ± 4.72 g/kg.

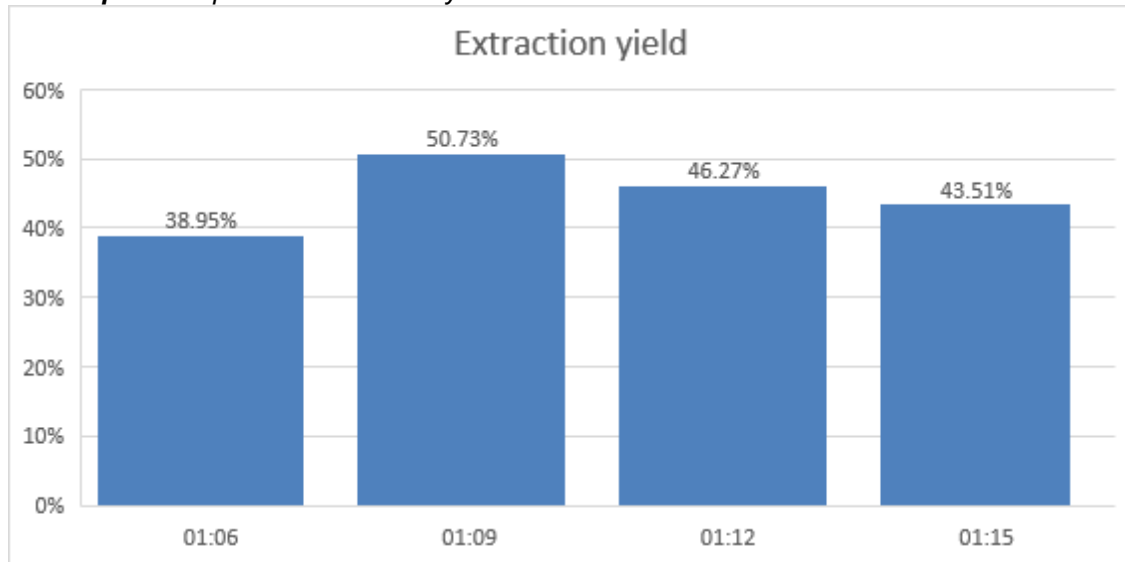
A comparison of the four ratios solution indicated significant difference in tannin concentration ($P > 0.05$). Different concentrations of tannin obtained from various ratios of powder/water have shown the amount of solvent used have an effect on tannins' extraction. The concentration of the solution is an outcome of the ratio of solute quantity within the specified volume of solvent.

Table 1: Tannin content obtained from extracted solution before and after leather tanning trial

		Tanning solution				p-Values from ANOVA
		1:6	1:9	1:12	1:15	
Concentration of tannin obtained on solution, g/litre	Before tanning	17.42±0.06	15.12±0.12	10.34±0.25	7.78±0.08	0.0000
	After tanning	4.16±0.08	3.35±0.10	2.58±0.10	1.77±0.11	0.0000
Implication of extraction yield of tannins from cashewnuttesta powder, g/kg	Before tanning	104.52±0.39	136.14±1.10	124.17±3.09	116.75±1.25	0.0000
	After tanning	24.99±0.49	30.15±0.94	31.05±1.24	26.64±1.70	9.7625E-7

Based on the extraction condition applied with known amount of *cashewnuttesta* powder and water, the yields of extraction by various ratios increased in the following order 1:6 < 1:15 < 1:12 < 1:9. Solution with ratio 1:9 extracted about 136.14 ± 1.10 g of tannin, followed by ratio 1:12 with 124.17 ± 3.09 g, 1:15 with 116.75 ± 1.25 g and 1:6 with 104.52 ± 0.39 g based on one kilogram of the *cashewnuttesta* powder per each. Extraction yields ranged from 38.95% for ratio 1:6 to 50.73% for ratio 1:9 (Plot 1). High extraction yield from ratio 1:9 is favorable by extraction condition applied which influence high solubility of molecule in that ratio. At the molecular level, solubility is controlled by the energy balance of intermolecular forces between solute-solute, solvent-solvent and solute-solvent molecules (Gong et al., 2007).

Graph 1: Response of extraction yield obtained from different ratios of tannin solution



The pH value is important as leather is produced with a pH of about 4.5 to 5.5, this pH ensures that the fat and tannins bound in the leather remain. The pH value of the extracted tannin solutions ranged from 4.5 to 5.0. Binding of tannins extracted from cashewnut testa in the four ratios of the tannin solution to the skin in tanning were above 75% of tannins concentration obtained in the solution.

3.2 Leather physical testing

Suitability of extracted tannin in leather tanning was assessed using physical test, considering that tannins consist fraction of materials so that can affect the quality of the leather (Covington, A. D, 2009). The leather is characterized as an anisotropic material, the strength and stretch properties change directionally and locational over the area of the leather (Mutlu *et al.*, 2014). Physical properties of leathers tanned with the different ratios of tannin solution are presented in Table 2.

Table 2 Characteristics of the sheepskin tanned using Cashewnut testa tannin solution

Property	Item	Tanned with tannin solution of				Recommended value(ISO 14931-2021)
		1:6	1:9	1:12	1:15	
Tensile strength, MPa	Sample 1	25.48 \pm 6.31	25.57 \pm 5.35	38.92 \pm 8.49	43.00 \pm 8.18	> 12
	Sample 2	21.99 \pm 5.08	19.41 \pm 6.36	30.19 \pm 5.03	33.35 \pm 10.86	
Elongation at Maximum Load, %	Sample 1	48.02 \pm 14.47	34.48 \pm 10.12	68.93 \pm 27.37	64.25 \pm 14.77	40 - 90
	Sample 2	35.10 \pm 13.32	40.60 \pm 13.73	77.56 \pm 10.96	42.85 \pm 12.87	
Shrinkage temperature, °C	Sample 1	80.80	76.90	78.33	76.50	> 75
	Sample 2	79.57	76.57	78.07	80.43	
Flex endurance	For both samples	No damage at 100,000	No damage at 100,000	No damage at 100,000	No damage at 100,000	No damage at 100,000 flexes

The tensile strength of leathers obtained from this experiment ranged from of 19.41 ± 6.36 MPa to 43.00 ± 8.18 MPa. The highest tensile strength was given by leather tanned using ratio 1:15 are 43.00 ± 8.18 MPa, while the lowest was given by leather tanned using ratio 1:9 are 19.41 ± 6.36 MPa. However, the tensile strength for all experimental tanned leather met the recommended minimum value of 12 MPa. The assessment of tensile strength is vital in determining leather's capability to withstand stretching forces, ultimately impacting its structural integrity and suitability for various leather applications.

The results show that elongation at maximum load of the experimental leathers ranged from 34.48 ± 10.12 % to 77.56 ± 10.96 %. Except for sample 1 of ratio 1:9 and sample 2 of ratio 1:6, the elongation at maximum load obtained from the tanned leather are within the recommended range of 40 % to 90 %. Apart from the effect of tannin solution to the tanned leather, elongation influenced with original properties of the skin, as the variability in the physical properties of leather is determined by the distinct characteristics of individual animal within the same species, this led to consistent applied of tannin solution on the skin of the same species yielding different results of leather properties. The highest elongation at maximum load was obtained from the leather tanned using ratio 1:12 while ratio 1:9 resulted in the lowest value. The elongation value of leather is a critical quality parameter, as a low value increases its susceptibility to tearing, whereas a high value can result in excessive deformation, compromising the usability of leather goods.

Shrinkage temperature is among the most important parameters in characterizing thermal stability of leather. It provides information about the degree of tanning as the increase in the shrinkage temperature is due to good crosslinking reaction between collagen fibres and tannins (Wu *et al.*, 2019). Good quality leather should have a minimum shrinkage temperature of 75°C (Nalyanya *et al.*, 2018). Values of shrinkage temperature of leather tanned with all ratios of tannins solution have shown to be above the recommended minimum value of 75°C.

Flexing test is normally done on leather intended for making leather products that flex several times (Nalyanya *et al.*, 2018). Flexing test applied to the respective tanned leather gave no damage at flexes of 100,000 flexes as a standard up to 300,000 flexes. There was no any crack or damage observed on sample specimens after 300,000 flex cycles, implying that all leather specimens passed the flexing test. On the other hand, all the test values of the sheep leather fall within the required values. Therefore, it is evident that the sheep skins tanned with cashew nut testa's tannin has good quality and resistance when subjected to continuous flexing.

4. CONCLUSION

The objective of this work was to optimize tannin's extraction from four different ratios of cashewnut testa powder and water, to be used for leather tanning. It has shown that the solvent to sample ratio affect the extraction yield of tannin from cashewnut testa powder. The mixing ratio 1:9 of powder/water can be used to extract high amount of tannin from cashewnut testa under given conditions compared to other ratios tested. From this study, solution with ratio 1:9 have shown to extract about 50.73% of total tannins presented on cashewnut testa powder followed by solutions with ratio 1:12, ratio 1:15, and ratio 1:6 extracted about 46.27%, 43.51% and 38.95% respectively. Furthermore, leather tanned from four different ratio were subjected to leather physical testing and the result showed have met the standard required. Despite variations in feeding ratios during tanning, efforts can be made to enhance them in order to achieve the desired leather properties.

Cashewnut testa can be valuable and sustainable resources in leather industries, as an organic tanning agent source for production of leathers from skins. The findings of this study have underscored the potential use of cashewnut testa as a vegetable tanning material. Advantageous optimization of extraction of the tannin from cashewnut testa and the promising tanning results pave way for further investigation. To ensure successful and cost-

effective adoption of cashew nut testa tannin extracts for tanning, it is recommended to explore their benefits, which not only enhance the leather industry but also contribute to creating an eco-friendly environment.

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COMPETING INTERESTS

Authors have declared that no competing interests exist

AUTHORS' CONTRIBUTIONS

Mr Sharif Zuberi Mwangi designed the study, performed the statistical analysis, wrote the protocol, and wrote the first draft of the manuscript. Prof. Valerian Cosmas Silayo and Dr. Davis Naboth Chaula managed the analyses of the study and the literature searches. All authors read and approved the final manuscript.

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APPENDIX

Formulation of tanning process

Process	Product used	Percent	Duration (mins)	Remarks
Washing	Water	300	15	Drain
Soaking	Water	300	60	Drain
	Wetting agent	1		
Washing	Water	300	15	Drain
Liming	Water	300	285	Run 30' stop 30' (3X)
	Sodium sulphide	2		
	Lime	6	150	
Fleshing				Done mechanically
Washing	Water	300	15	Drain
Deliming, Bating and Degreasing	Water	200	90	Drain
	Ammonium chloride	2		
	Bating agent	0.5		
	Degreasing agent	2		
Washing	Water	300	15	Drain
Pickling	Water	100		Overnight
	Salt (sodium chloride)	10	15	
	Formic acid	1	(3 x 15)	

Tanning	1:6 tannin solution	300	(3 x 120) + 360	Solution divided in three equal portion and fed for every 120mins
	1:9 tannin solution			
	1:12 tannin solution			
	1:15 tannin solution			
Fixation	Formic acid	2	30	pH 3.5
Neutralization	Sodium bicarbonate	1	(3 x 15) + 60	pH 5
Washing	Water	300	15	Drain
Retanning	With respectively tannin solution	100	120	Drain
Fatliquoring	Water 45 °C	100	60	
	Lipoderm liquor	2		
Fixing	Formic acid	0.5	30	pH 3.5
	Anti-mold	0.02	15	
Washing	Water	300	15	Drain
Drying	Shade-dry	-	Two days	Drain
Staking	Hands-on (pulling)	-	-	Soften