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Upcycled aquaculture waste as textile ingredient for promoting circular economy

Erh-Jen Hou [a,b], Chi-Shih Huang [a], Ying-Chou Lee [a,*], Hsueh-Ting Chu [c,d,**]

[a] Institute of Fisheries Science, College of Life Science, National Taiwan University, Taipei, Taiwan
[b] Inventor of UMORFIL® & Founder of Camangi Corp, Taiwan
[c] Department of Computer Science and Information Engineering, Asia University, Taichung, Taiwan
[d] Department of Medical Research, China Medical University Hospital, China Medical University, Taichung, Taiwan

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ABSTRACT

Fish farming (aquaculture) provides a stable source of nutritional protein, especially in Asia's coastline, distributed throughout the South China region of the mainland, southwestern Taiwan, and Southeast Asia, providing a continuous supply chain system. Novel textile material, from upcycling of fish scales, has been manufactured. The material constitutes of collagen peptide amino acid ingredients extracted from fish scales, which are used to create collagen modification polyester via a supramolecular method. The development of the collagen modification polyester, UMORFIL®T, can make a valuable contribution to the textile industry. After various experiments and characterizations, it was shown that the collagen modification polyester in this study contained a certain amount of collagen and retained the advantages of regular polyester. In addition, the characteristics of the original regular polyester were changed by the presence of collagen. The new properties included a natural champagne-gold color, better hand-feel, and odor control, which make the material a premium and sustainable choice for functional textiles. This study creates a new direction for a circular economy product, collagen modification polyester, that can protect the ecology, reduce environmental pollution, protect planet soil, provide premium value for the textile industry, and raise the value of aquaculture.

1. Introduction

The circular economy concept has been considered as a solution for sustainable development. The concept is not an innovation, but rather a shifting trend due to resource limitations. As a matter of fact, many industries have developed products by reuse, remanufacturing, or recycling in the past decades [1]. In the textile industry, the use of recycled plant fibers to improve textiles has a long history [2,3], and many textile materials that use regenerated plant fibers have been produced such as Viscose, Modal, Lyocell, Triacetate, Polynosic, Cupro. On the contrary, the use of animal amino acids to improve man-made textiles was rare in the past [4]. A novel collagen modification polyester, named UMORFIL®T, is introduced in this paper. It was inspired by the recycling of fish waste and successfully applied to the textile industry in this study. The recycling of food waste and the development of new textile materials based on the circular economy approach a good chance to become an important trend of the future.

Food waste is obtained from agricultural products, such as coffee or grape ground, and the necessary ingredients are extracted to make eco-friendly textile materials [5]. Owing to the success of the circular economy approach, future studies should focus on using other food-waste products since food is a fundamental basis of human existence [6]. Sustainable development in the agriculture, forestry, fishery, and stock farming industries is important for the future survival of economies and societies. Aquaculture and stock farming are the main industries that provide nutritional sources of protein; however, the fish industry has suffered unbalanced marine ecology and reduced marine biomass due to rapid climate change, dramatic population increase, and overfishing in the oceans. Therefore, the establishment of fish farms as an industry has become essential to provide nutritional protein, in many Asian countries [7,8].

Aquaculture provides a stable source of animal protein, especially in Asia's coastline, distributed throughout the South China region of the mainland, southwestern Taiwan, and Southeast Asia, providing a

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* Corresponding author.
** Corresponding author at: Department of Computer Science and Information Engineering, Asia University, Taichung, Taiwan.
E-mail addresses: i812@ntu.edu.tw (Y.-C. Lee), prof.htchu@gmail.com (H.-T. Chu).

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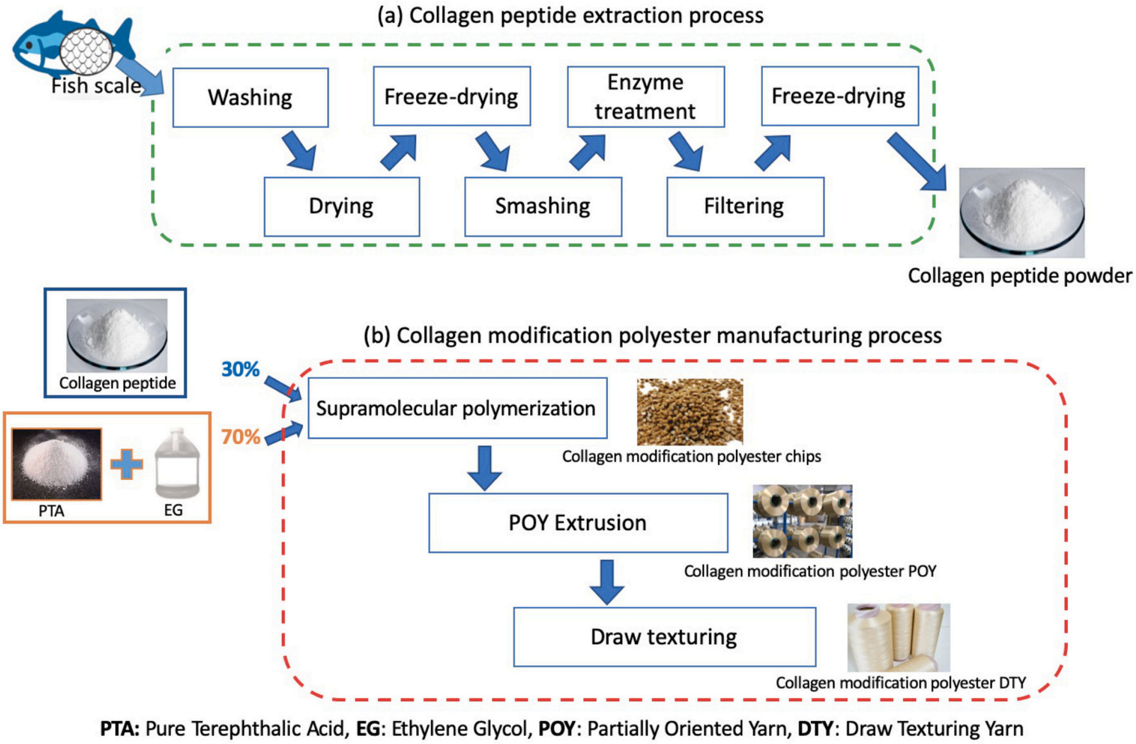


Fig. 1. Illustration of the fabrication of the collagen modification polyester with fish scales. (a) The first part is to obtain collagen from fish scales. (b) The second part is the polymerization process includes esterification reaction and polycondensation reaction to form a copolyester material as chips.

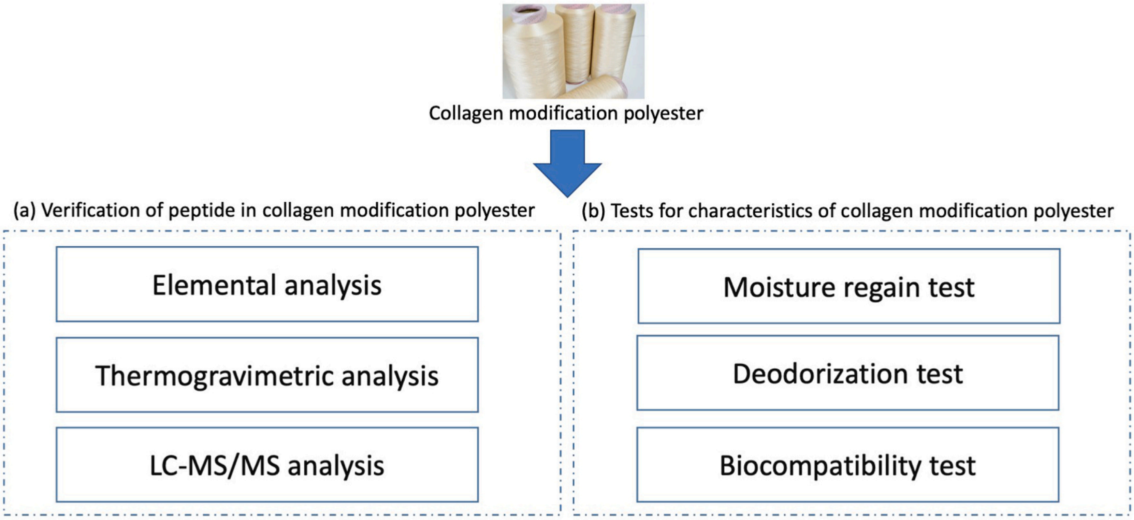


Fig. 2. Experiments for the collagen modification polyester: (a) verification of peptides in the collagen modification polyester, (b) tests for characteristics of the collagen modification polyester.

continuous supply chain system. According to the Food and Agriculture Organization of the United Nations (FAO), as of 2018, global aquaculture had produced 82 million tons of fish [9]. In addition, based on data from FAO, it has been revealed that following the careful treatment of a single fish, approximately 35% of its total weight, including bones, internal organs, and scales, is discarded as waste [10]. However, fish bones and internal organs can be processed into a source of feed [11]. Fish scales constitute between 1 and 5% of the total fish weight and are the only waste that cannot be reused directly. If a fish-filet processing factory directly buries fish-scale waste in the soil, it will result in acidification of the soil. Therefore, if fish scales can be recycled and reused to create new products of high added value, it will be an important milestone in the development of a circular economy [12–17].

Polyester is a widely used synthetic textile material with desirable properties such as light weight, durability, wrinkle resistance, and cost flexibility. Nevertheless, the downside of pure polyester fabrics limits their application as functional textiles. These shortcomings include hard hand feel, low odor resistance, low hygroscopicity, and limited dyeability [18,19]. Therefore, to overcome these shortcomings, increased research and development has focused on how to add value to polyester materials, considering both aspects of sustainability and functionality.

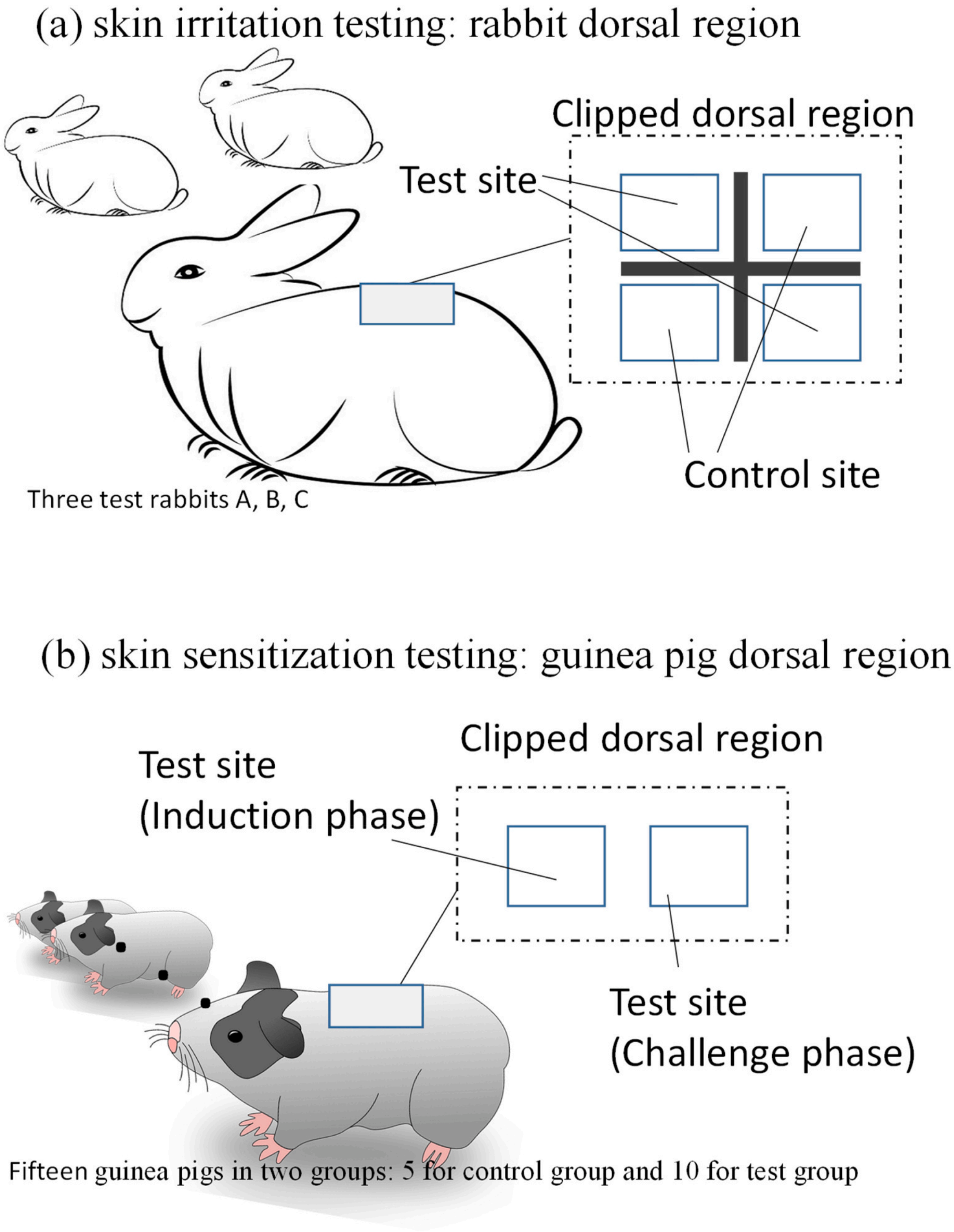


Fig. 3. Experimental design for biocompatibility tests of the collagen modification polyester products. (a) skin irritation test (b) skin sensitization test.

(a) Collagen modification polyester chips (b) Collagen modification polyester POY (c) Collagen modification polyester DTY

Fig. 4. Champagne appearance of collagen modification polyester products at different stages: (a) slices, (b) POY and (c) DTY.

To further enhance the characteristics of polyester fabrics, numerous nanofillers and dyes have been developed [20]. Polyester nanocomposites with different nanofillers such as nanodiamonds, fullerenes, carbon nanotubes, graphene, and graphene oxide can reinforce these important polymers to further enhance the final structural and physical characteristics of the fabric [21]. Moreover, dyeing with moisture

Table 1
Element content of different polyester DTY 75D/72F.

		Element content			
Sample	Weight	C (%)	H (%)	N (%)	S (%)
Regular polyester	9.422	62.057	4.063	0.125	0
	9.101	62.097	4.146	0.160	0
Collagen modification polyester	8.151	62.030	4.236	0.197	0
	9.273	62.128	4.170	0.204	0

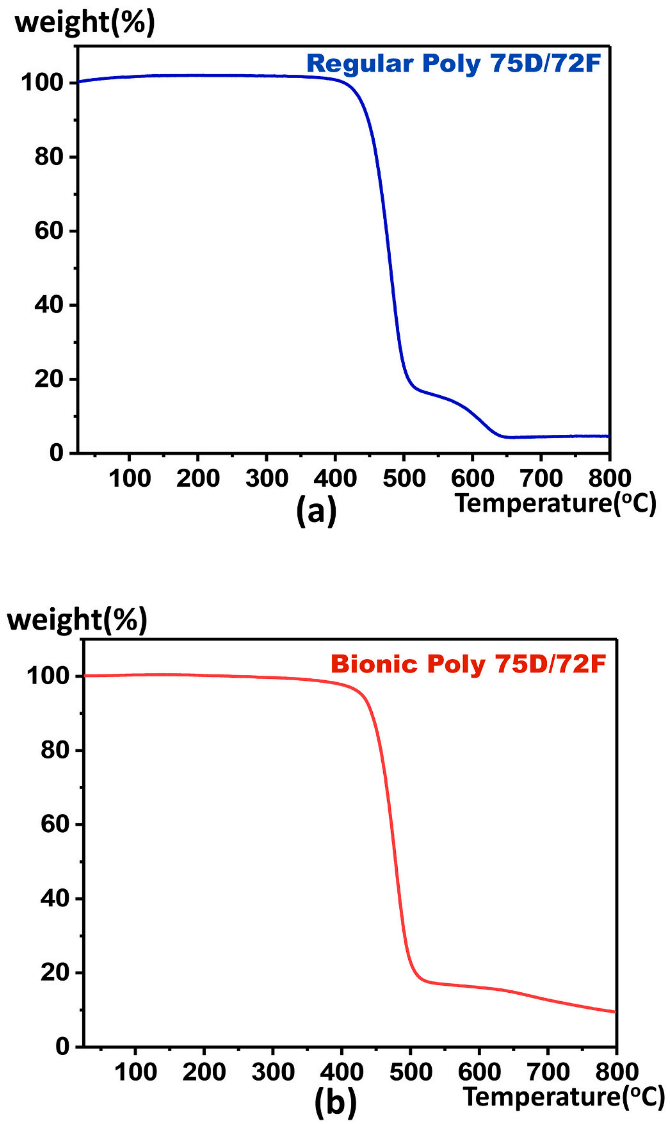


Fig. 5. The result of thermogravimetric analysis (a) Regular polyester DTY. (B) The collagen modification polyester DTY.

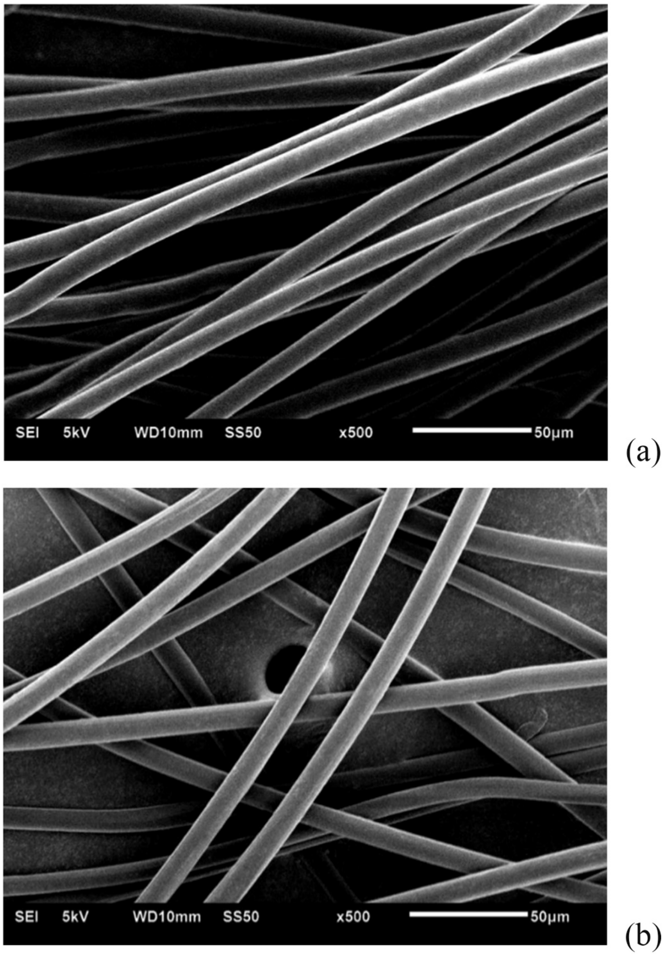


Fig. 6. The SEM image of the polyester fabrics, (a) Regular polyester, (b) the collagen modification polyester, × 1500.

Table 2
Outcome of moisture regain test.

Sample	Dry weight (g)	Wet weight (g)	Moisture regain (%)
Regular polyester	29.8260	30.0113	0.62
	29.8086	30.0070	0.67
	29.7783	30.0188	0.81
Average			0.70 ± 0.10
Collagen modification polyester	29.6651	30.0083	1.16
	29.7132	29.9591	0.83
	29.6713	29.9562	0.96
Average			0.98 ± 0.17

The significantly different level between two treatments as α = 0.1.

control chemicals can be used to improve the comfort and esthetic properties of polyester fibers [22–24].

Currently, a novel textile material exists. It originates from upcycled collagen peptide amino acids extracted from fish scales, which are then used to create a collagen modification polyester via a supramolecular method. The collagen modification polyester has functional characteristics from collagen peptide amino acids, and when compared with regular polyester, it provides properties such as increased comfort, odor control, and better moisture retention. These attributes result in a higher premium value for textile products [25]. Additionally, collagen is widely used in the food industry. However, in recent years, due to mad cow disease (BSE), foot and mouth disease (FMD), and avian influenza, collagen extracted from animals has gradually been replaced by other sources [26]. Aquaculture waste materials are an alternative source of collagen [27–29]. In addition, the nutritional value of collagen peptides from fish scales has been studied extensively [30,31]. Collagen is composed of three α-chains that assemble into complex hierarchical fibers or other structures [32]. Extraction methods of collagen peptides from fish skin and scales have been investigated [33]. Compared with collagen from terrestrial animals, collagen from fish scales has the advantages of being free of fat, antibiotic(s), and prion(s) [34].

After upcycling the fish scales and extracting the high-value collagen peptide amino acids, the residual material can be processed as raw material for the production of fertilizers used in the food and agriculture

Table 3
Outcome of deodorization test.

After 10 washes	Time	Blank Test (ppm)	Testing sample (ppm)	Reduction (%)
Ammonia (ISO 17299-2)	0 h	100	100	
	After 2 h	84	76.5	8.9
Acetic acid (ISO 17299-2)	0 h	30	30	
	After 2 h	17	4.5	73.5
Isovaleric acid (ISO 17299-3)	0 h		38	
	After 2 h		0.4	99
2-Nonenal (ISO 17299-3)	0 h		14	
	After 2 h		1.7	88

industries, truly placing the discarded ingredients in the highest value chain and adopting a circular economy [35].

This study investigated the application of aquaculture waste in the textile industry. Protein-type textile fibers, such as wool and silk, usually provide better thermoregulation, softness, and more comfortable features. Therefore, the purpose of this study was to explore the characteristics of a collagen modification polyester produced using collagen peptides from fish scales, from a circular economy point of view, by comparing it to regular polyester fibers, with the aim of providing a premium and sustainable raw material choice for the textile industry.

2. Material and methods

Regular polyester fiber was obtained by the polymerization of aromatic polyethylene terephthalate according to industrial practice [36]. The procedure of the new modified polyester involved two steps: the extraction of collagen peptides from fish scales and modification to form the collagen polyester.

Recycled tilapia fish scales from fish farms were washed, dried, freeze-dried, smashed, and then broken down into short amino acids by using an enzyme (protease 7307–1) that was obtained from a bacterial strain [37]. The method used for collagen extraction from fish scales was adapted from a previously developed method [38–40]. Finally, collagen peptides were obtained after filtering (Fig. 1a).

The materials used to produce the collagen modification polyester included collagen peptides from fish scales, benzenedicarboxylic acid (PTA), ethylene glycol (EG), and catalysts. The molar ratio of collagen peptides to PTA was 3:7. The benzenedicarboxylic acid was terephthalic acid (TPA) or isophthalic acid (IPA), or a combination of TPA and IPA. The catalysts were Sb_2O_3 and TiO_2, and the ratio range of parts-per-million concentration between Sb_2O_3 and TiO_2 was approximately 25:4. Titanium dioxide (TiO_2, 97% purity) particles were obtained from Chemours (Chemours Ti-Pure R-104). Antimony trioxide (Sb_2O_3, 99.9% purity) particles were obtained from Gredmann (Gredmann GM-CA). Benzenedicarboxylic acid (PTA, 99.9% purity) was supplied by Formosa Chemicals & Fiber Corp. Ethylene glycol (EG, 98% purity) was supplied by Nan-Ya Plastics.

Following the above-mentioned super molecular polymerization, collagen modification polyester chips were produced, and then extruded into collagen modification polyester partially oriented yarn (POY), and finally textured to bionic silk draw textured yarn (DTY) (Fig. 1b).

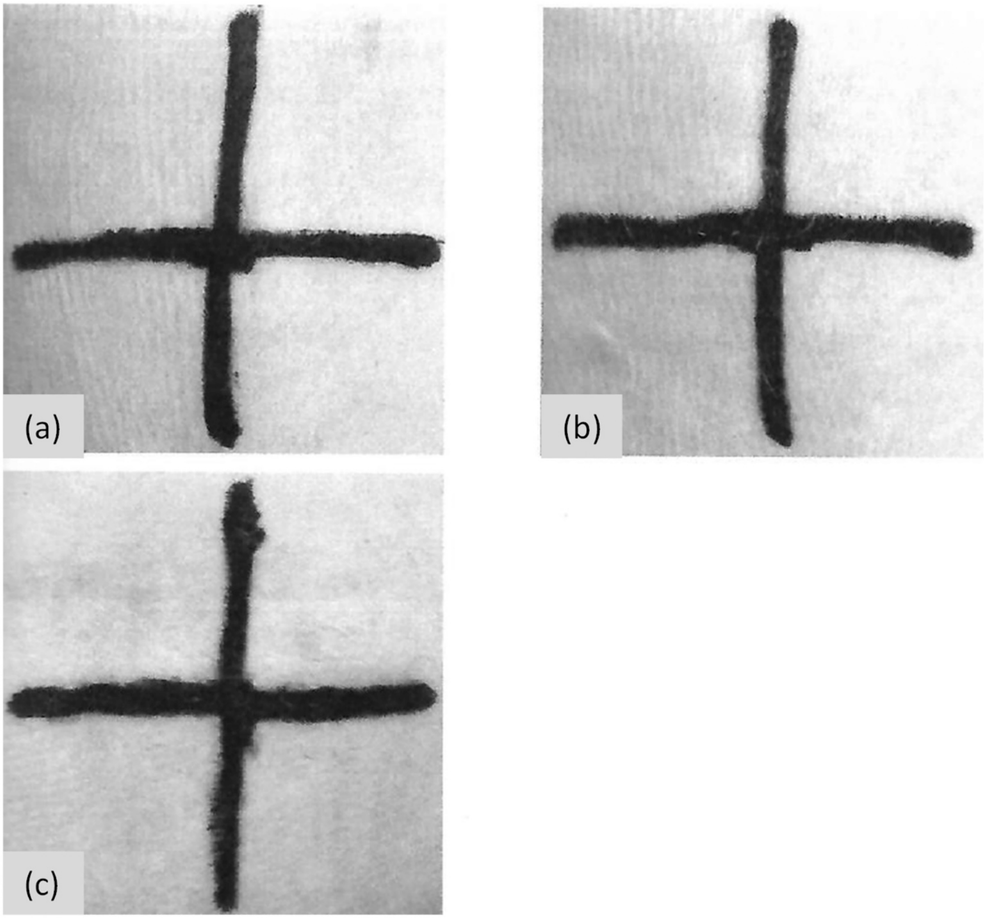


Fig. 7. The result of rabbit skin irritation test at 72 ± 1 h of the observation period for the three rabbits (a), (b) and (c).

Table 4
Individual skin reaction of guinea pigs.

Group	Sex	Animal ID	Grade of skin reaction 24 ± 1 h	48 ± 1 h	Sensitization Rate	Sensitization Capacity
Control	Male	351	0	0	0%	Weak
		352	0	0		
		353	0	0		
		354	0	0		
		355	0	0		
Test	Male	356	0	0	0%	Weak
		357	0	0		
		358	0	0		
		359	0	0		
		360	0	0		
		361	0	0		
		362	0	0		
		363	0	0		
		364	0	0		
		365	0	0		

2.1. *Scanning electron microscopy (SEM) analysis*

The appearance of the collagen modification polyester was observed using SEM [41]. SEM images of the samples were obtained at an accelerating voltage of 30 kV, a magnification of 10× up to 400.000×, and a 3.5 nm resolution for wavelength.

2.2. *Elemental analysis and thermogravimetric analysis*

In order to verify the ingredients in the collagen modification polyester material, we conducted elemental analysis and thermogravimetric analysis (Fig. 2a). A Vario EL cube elemental analyzer (Elementar, Langenselbold, Germany) was used to identify and analyze the following elements: carbon (C), hydrogen (H), nitrogen (N), and sulfur (S). During elemental analysis, the DTY collagen modification polyester was combusted, and the resulting oxides of carbon, hydrogen, and sulfur along with nitrogen were sequentially analyzed.

Thermogravimetric analysis was carried out using a Perkin Elmer TGA thermal analyzer, with a platinum measuring cell to handle the polyester samples [42]. The measurements were performed in air at a heating rate of 10 °C/min. Samples were heated to 800 °C starting from 100 °C. The thermogravimetric analyzer controlled sample weights before and during the measurements.

2.3. *LC-MS/MS analysis*

The collagen content in the product of the fabrication process was further analyzed using liquid chromatography coupled to tandem mass spectrometry (LC-MS/MS) (Fig. 2a) [43]. The samples were simply diluted and injected onto a hydrophilic interaction LC column (HILIC) coupled to an LC/MS/MS system operated in positive and negative

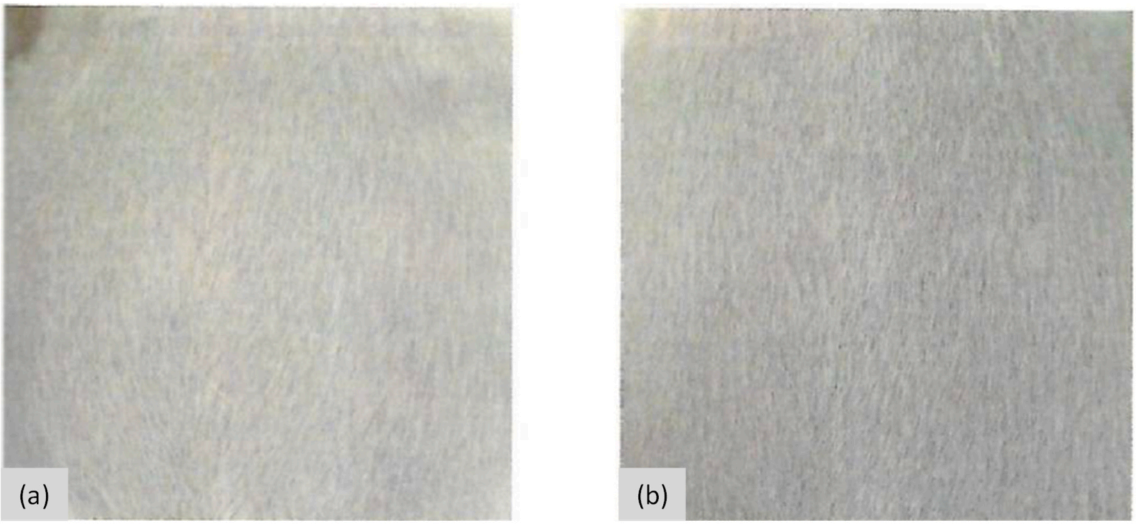


Fig. 8. The result of guinea pig sensitization test at 48 ± 1 h of the observation period. (a) Control group animal. (b) Test group animal.

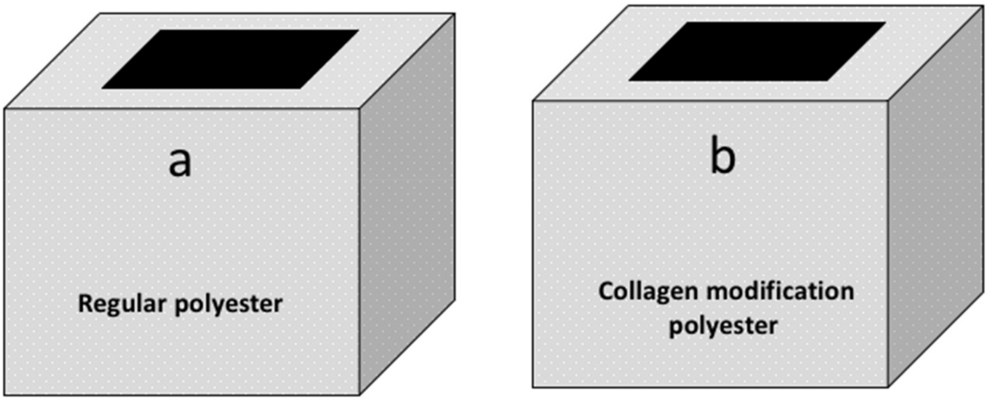


Fig. 9. Compare the hand feeling of fabric test boxes: (a) Box with regular polyester fabric, (b) Box with collagen modification polyester fabric.

Table 5
Score from compare the hand feeling of fabric testers.

No.	A (Regular polyester)	B (Collagen modification polyester)
No.1	4	5
No.2	3	4
No.3	3	5
No.4	3	4
No.5	4	5
No.6	3	4
No.7	3	5
No.8	3	4
No.9	4	4
No.10	4	4
No.11	4	5
No.12	3	4
No.13	4	4
No.14	3	4
No.15	3	5
No.16	4	4
No.17	4	5
No.18	3	5
No.19	3	4
No.20	3	4
No.21	3	4
No.22	4	5
No.23	3	5
No.24	4	5
No.25	3	4
Average	3.40 ± 0.50	4.44 ± 0.51*

[*] $p < 0.01$.

Table 6
Characteristics list of regular polyester and collagen modification polyester.

Characteristics	Regular polyester DTY	Bionic silk polyester DTY
Color	Raw white	Champagne-like
Tenacity (g/d)	3.8–4.8	3.94–4.21
Elongation (%)	22–25	30–35
Moisture regain (%)	0.2–0.4	0.8–1.2
Deodorization	N/A	Acid odor

polarity. Multiple reaction monitoring (MRM) by mass spectrometry was used for detection because of its high selectivity and sensitivity [43].

2.4. Moisture regain and deodorization tests

Compared with regular polyester products, the collagen modification polyester products were tested for moisture regain, deodorization, and biocompatibility (Fig. 2b). All the tests followed the latest industrial standards.

The moisture regain test was performed according to the JIS L1030–2 standard [44] and ASTM D2495–07 [45]. The collagen modification polyester, DTY 75D/72F, was dried in an oven at 105 °C until there was no further change in weight. The initial weight was recorded, and the sample was immediately placed in the environmental system. The change in mass was measured at 20 °C and 65% RH. The ratio of moisture regain was defined as:

$$\text{Moisture regain}\ (\%) = \frac{\mathrm{W} - \mathrm{D}}{D} \times 100, \tag{1}$$

where D is the weight of dry DTY (g), and W is the weight of wet DTY (g).

The deodorization test was used to measure how effectively the textile material (the collagen modification polyester fabric composed of 91% UMORFIL® T DTY and 9% Spandex) reduced the unpleasant smell of chemicals, produced by sweat and foot odors. The detector tube test was performed according to the standard test method, ISO 17299-2 [44]. The test method measures odor component chemicals, including ammonia, acetic acid, methyl mercaptan, and hydrogen sulfide. The test conditions included (a) amount of specimen: 10 × 10 cm (b) test vessel: Tedlar bag (5 L), (c) gas volume: 3 L in vessel (d) temperature and humidity: 20 °C, 65% RH (e) machine wash at 80 ± 5 °F under ATCC 135–2012 [46].

The gas chromatography (GC) test was performed according to the standard ISO 17299-3 test method [47]. This method applies to odor component chemicals, such as indole, isovaleric acid, nonenal, and acetic acid with added sodium chloride (NaCl). The test conditions included (a) amount of specimen: 50 cm^2 (b) test time: 2 h (c) machine wash at 80 ± 5 °F under ATCC 135–2012.

2.5. Biocompatibility tests

The biocompatibility tests of the collagen modification polyester were performed according to the ISO 10993-10: 2010 standard test method [48] which assesses the potential of the proposed material to induce irritation and skin sensitization (Fig. 2b). Regarding extraction methods for preparing test samples, this study followed the ISO 10993-12:2012 guidelines, and the test material was extracted using physiological saline at 37 ± 1 °C for 72 ± 1 h.

Three albino rabbits were used for the skin irritation test (Fig. 3a). Before the test, the fur on the shoulder of the test animal was shaved, and their skin was confirmed to be in good condition. For the experiment, 0.5 mL of the test material extracts and 0.5 mL control material were applied on the test and the control areas of the dorsal skin, respectively, using a gauze patch. After 4 h, the gauze was removed, and the irritation on the rabbit skin was evaluated at 1, 24 ± 1, 48 ± 1, and 72 ± 1 h.

For the skin sensitization test, 15 guinea pigs were used (Fig. 3b). Before the test, the fur on the shoulder of the test animals were shaved, and their skin was confirmed to be in good condition. The test animals were exposed to the test article extracts by intradermal injection and epidermal application as the induction phase. Following a rest period of 14 days, test article extracts loaded on a gauze patch were applied on the upper flank of all test animals for 24 ± 1 h as the challenge phase. The skin sensitization reaction of the upper flank of the test animals was evaluated at 24 ± 1 and 48 ± 1 h after the challenge phase.

3. Results and discussion

In the past ten years, many methods have been proposed to improve man-made fibers [49–51]. In this study, we have developed a process to improve polyester fiber with collagen additive and the modified polyester is better than the original polyester in terms of color and touch, as well as moisture regain and deodorization.

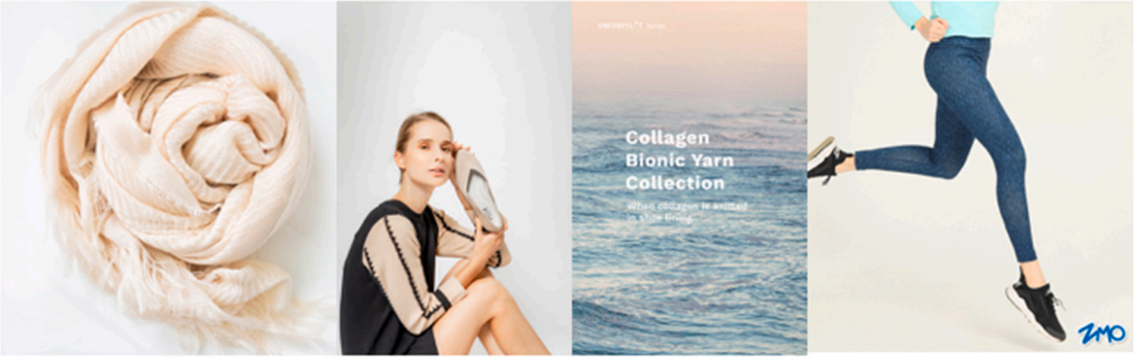
3.1. Characteristics of collagen modification polyester

Fig. 4 shows the collagen modification polyester's color has totally been changed from chips, the chip's color is dark champagne, after the spinning process chips will become POY of super gold color, it is the first form of yarn made directly from chips. POY is mainly used in texturizing to make textured yarn, also known as DTY. After the texturize process, the color of DTY became the champagne gold color. From the change in color for different stage in the high temperature spinning process, we can also confirm it's successfully modification by collagen.

Table 1 shows the results of CHNS (carbon, hydrogen, nitrogen, and sulfur) elemental analysis for both regular DTY polyester and the collagen modification DTY polyester. The nitrogen content in the collagen modification polyester was higher than that of the regular polyester. The increased nitrogen content shows that collagen peptides were incorporated into the chemical structure of the resultant polyester.

Peptide tests in collagen modification polyester yarn were conducted using LC-MS/MS [43]. An LC-MS/MS test performed by the SGS company (Report No. UG/2020/10248) revealed 40.0 mg/kg collagen content which was calculated based on hydroxyproline content. The ratio of hydroxyproline to collagen was 13.4%. These results confirm the effectiveness of supramolecular polymerization in the production of

Chips DTY yarn Fabric



Scarf Shoes lining Sport wear

Fig. 10. The collagen modification polyester product from chips to final product in the textile supply chain.

collagen modification polyester.

Thermogravimetric analysis (TGA) showed that the thermal weight of regular DTY polyester at 800 °C was less than 5%, while the thermal weight of the collagen modification DTY polyester was more than 10% at the same temperature (Fig. 5). The results showed an improvement in the heat resistant properties of polyester from collagen modification as shown by the increase in decomposition temperature.

Fig. 6 shows an SEM image of the polyester fibers. The surface of the collagen modification polyester was as smooth as that of the regular polyester fiber, and there were almost no small particles on the fiber surfaces. The surface of polyester fibers with nanofillers usually has nanoscale roughness such that this modification may cause damage to machinery during textile processing [21]. Therefore, compared to nanofillers, the collagen modification polyester has the potential to provide superior textile properties.

3.2. Properties of the collagen modification polyester

The synthesized polyester was successfully verified to contain collagen, and was composed of animal amino acids similar to wool and silk. Consequently, we termed it the bionic polyester, i.e., UMORFIL®T in this study. The advantages of the collagen modification polyester included better- moisture regain and deodorization capabilities.

A significant difference exists between the production of collagen modification polyester and existing technology that adds nanofillers and nanoparticles to regular polyester fibers for improved functionality [52–54].

Table 2 presents the results of the moisture regain test (%) for both samples (regular and collagen modification polyesters). It can be observed that the average moisture regain of the collagen modification DTY polyester was 0.98%, whereas that of the regular DTY polyester was 0.70% ($\alpha = 0.1$). The superior moisture regain of the collagen modification polyester increases its opportunities for application in various functional textiles, such as active wear, sportswear, shoes, and bedding.

The deodorization results of the collagen modification polyester fabric are illustrated in Table 3. The experimental samples demonstrated much better deodorization potential than the blank test after 2 h. The blank test underwent the same test procedure without being exposed to the sample in the vessel.

3.3. Biocompatibility of collagen modification polyester fibers

The skin irritation test of the collagen modification DTY polyester indicated that the primary irritation index (PII) was zero, and it was thus classified as non-irritant, as shown in Fig. 7. Therefore, the test material did not induce irritation on the skin of the test rabbits.

The skin sensitization test of the collagen modification polyester DTY indicated that the sensitization rate of both test and control groups were zero (Table 4), and thus, the collagen modification polyester DTY was classified as weakly allergenic (Fig. 8). The skin reaction was graded according to the Magnusson and Kligman scale [55]. Therefore, the test material did not induce a sensitization response on the skin of guinea pigs.

3.4. Better hand feel of collagen modification polyester

To compare the hand feel of collagen modification polyester and regular polyester, we synthesized two fabrics, one made of collagen modification polyester and the other made of regular polyester (Fig. 9). All production conditions, except the materials, were the same. The fabrics were placed in different boxes, box (a) contained regular polyester fabric, and box (b) contained collagen modification polyester fabric. Human testers placed their hand into each box to touch and feel the fabric and graded it from 1 to 5 (Table 5), with higher scores indicating better hand feel ($p < 0.01$).

3.5. Advantages of collagen modification polyester

The properties of regular polyester and the collagen modification polyester, such as color, tenacity (g/d), elongation (%), moisture regain (%), and deodorization are shown in Table 6: (1) the color of the collagen modification polyester chips was different from that of regular polyester chips, but the yarn surface, for both, was smooth when drawn into yarn, which will not cause damage to the weaving and knitting machines. (2) There are similar concepts already in the market with coating technology that coats soybean protein onto the surface of polyester fabric to enhance the hydrophilicity and softness, but the real effect depends on the coating rate [56]. Collagen modification is a new technology that imparts permanent features and functions to polyester. In our study, the collagen modification polyester showed higher moisture regain, and the fabric was softer and had better hand-feel than regular polyester. The textile finishing industries are chemical industries in which dyes, pigments, and auxiliaries are used in very large quantities with very large volumes of water [57]. The collagen modification polyester has a champagne-gold color and has naturally occurring protein fiber features, which can reduce environmental pollution caused by detergents and chemical use, in addition to reducing the environmental pollution concerns caused by the use of functional textile auxiliaries in the supply chain.

3.6. *Existence of collagen modification polyester in the textile market*

An investigation into textile market trends showed that an increasing number of brand manufacturers retain the original fabric structure and replace the original material with collagen modification polyester (Fig. 10), which can upgrade the fabric and product, enhance the product value, and create a new sustainable story for brand customer experience [58–61].

4. Conclusions

In this study, collagen modification polyester showed better hand feel and the ability to deodorize and reduce acid odor storage when compared to regular polyester. Collagen modification does not only extend the lifespan of textiles and reduce environmental pollution caused by detergent use, but also reduces environmental pollution problems caused using functional textile auxiliaries in the supply chain. In addition, this study concluded that the new collagen modification polyester fabrics are more skin friendly, have better moisture management, and are better suited for use in various functional textiles. On the other hand, with respect to the viewpoint of a circular economy strategy, the new collagen modification polyester textile can reduce environmental pollution by upcycling fish scale waste, and thus raise the value of aquaculture. This study creates a new direction for adopting a circular economy concept for modification of polyester, which modifies the synthetic fiber into a sustainable material for the future.

Abbreviations

DTY	Draw textured yarns
EG	Ethylene glycol
IPA	Iso-phthalic Acid
MR	Moisture regain
PET	Polyethylene terephthalate
PII	Primary irritation index
PTA	Purified Terephthalic Acid
POY	Partially oriented yarns
SEM	Scanning electron microscope
TPA	Terephthalic Acid

Author statement

Erh-Jen Hou conceived of the presented idea. Ying-Chou Lee leads the project. Hsueh-Ting Chu, Erh-Jen Hou, Chi-Shih Huang and Ying-Chou Lee discussed the project and jointly wrote the manuscript. All authors read and approved the final manuscript.

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Not applicable.

Declaration of Competing Interest

This research was sponsored by Camangi Corporation (Taiwan) and may lead to the development of products that may be licensed to Camangi Corporation (Taiwan), in which we have a business and/or financial interest. We have disclosed those interests fully to SAGE Publishing, and have in place an approved plan for managing any potential conflicts arising from this arrangement. The trade name of the proposed bionic silk polyester fiber is “UMORFIL T".

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