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# Evaluation of single-component chitosan fiber: from advanced materials to contemporary fashion manufacturing

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# Evaluation of single-component chitosan fiber: from advanced materials to contemporary fashion manufacturing

**Abstract**

Advancements in material science have driven the development of the textile industry. Chitosan has inherent advantages, such as biodegradability, nontoxicity, and antibacterial and wound acceleration properties. It has been applied in the textile industry as a coating on fabrics. Nevertheless, research on single-component chitosan fibers is scant. The advantages of single-component chitosan fibers include improved comfort, laundry durability, abrasion resistance, and structural flexibility. To fill the mentioned research gap, this study developed an industrial-manufacturing-based method for testing and analyzing chitosan fibers. This method was demonstrated by studying the physical and biological performance of commercial chitosan fibers. Studying the characteristics of the chitosan fibers revealed the influence of the fiber structure on their performance. Although chitosan fibers with a higher degree of deacetylation had a shorter molecular chain, they exhibited higher strength. Moreover, the molecular weight and deacetylation degree influenced biological performance. Chitosan fibers with higher deacetylation degree chitosan had better antimicroorganism performance on Staphylococcus aureus, Escherichia coli, and Candida albicans; however, no such relationship was observed for the solution form of chitosan. These results imply a difference in antimicroorganism mechanisms between the fiber (solid) form and solution form of chitosan.

**Keywords**

Chitosan, antibacterial, testing, evaluation system, management of manufacture, product meaning and consumer perception

## *Introduction*

In the past, textiles mainly provided basic coverage and packaging functions. Now these basic functions are no longer sufficient to meet the needs of people and the industry. Textiles with exceptional performance in strength, or with additional unconventional functions, have gradually become mainstream in the context of people's lifestyle changes. Growth in this area is furthered by the fact that cost is no longer the industry's primary concern, as the functional textiles industry is expected to hit 175 billion US dollars by 2020.

Functional textile raw materials include single-component functional fibers and functional components mixed functional fibers, while surface modification or mulch is performed after fabric formation. Chitosan is a good example to illustrate and demonstrate the testing and evaluation process of single-component functional fibers, as it been utilized in various applications due to its bio-functions.[1–3]

Compared with chitosan modified cotton/wool/manmade fibers, single-component chitosan fiber has advantages such as better comfort, laundry and abrasion resistant, and more structural possibilities (Figure 1). The production process of single-component bio-based fibers usually involves dissolving the raw material solvent into liquid or gel form, extruding the fiber through a spinneret, and washing the solvent.[4] Compared with common natural or manmade fibers, the mechanical properties of the bio-fiber are poor,[5] and the cost is more expensive.

As functional fibers enter the market, manufacturers in the middle and lower stream of the market need to carefully identify the raw materials. However, presently there is no system for testing and evaluating functional textile fibers. The detection of functional component mixed functional fibers usually involves the detection of an active component proportion, while the detection of single-component functional fibers contains the parameters of the material itself.

The molecular weight (Mw) and deacetylation degree (DD) are two important indexes of chitosan materials.[6–8] Due to specific requirements of the spinning process, the polymerization degree of chitosan used in fiber spinning is high. Unlike low molecular weight and ultra-low molecular weight chitosan (which are commonly in powder form) or thin film chitosan (which is soluble in water), high molecular weight chitosan can only be dissolved in an acidic environment. The deacetylation of chitosan in the fiber can be achieved up to 95%. Deacetylation has little effect on the spinning process. However, the processing environment of deacetylation affects the degree of polymerization in chitosan materials, which in turn affects the mechanical properties and biological properties of chitosan fibers.

Functional fibers, such as the chitosan fiber, are confronted with the challenge of translating fiber technology into a commercially salable product when promoted to the downstream. Therefore, building a systematic testing methodology is extremely important as it will allow the downstream and the terminal better understanding of the value of the fiber and educate downstream enterprises on the method of selecting high-quality raw material fibers amongst uneven qualities. With understanding, the advantages of the materials can be further promoted through the structure technology of the downstream. With this focus, this article systematically compares and analyzes six chitosan fibers, and proposes suitable methods for industrial testing to serve the industry, to enable the public to further enjoy the benefits of new materials.

## Experimental design

### *Materials*

Six fiber samples were purchased from manufacturers in Shandong (FH900, FH946, FH922, FJ922, FY902) and Tianjin (FZ915), China. All fibers were prepared by the wet spinning method, with dissolution, de-aeration, and filtration processes applied prior to spinning, and refinement, drying, and post-finishing after spinning.

### *Experiment methods*

The testing methods include the following aspects for manufactory needs: (1) content and key factor confirmation; (2) functional performance; and (3) non-functional (mechanical) performance (Figure 2). Such an approach has also been adapted in a previous study on the effect of demineralization on degradable chitosan fiber.[9] Other articles involve bio-performances, such as cell growth[10] and emission[11] promotion, acid resistance,[12,13] and chemical extraction.[14,15] Articles involving the anti-microbial performance of chitosan mostly focus on the solution state, and therefore in this study we conducted chitosan anti-microbial performance tests in both its fiber and solution states.

Viscosity average molecular weight was calculated according to the Mark–Houwink equation, with viscosity detected by a Ubbelohde Viscometer (model K23798-OS Kinematic Viscosity Bath, Koehler). Then, the deacetylation degree of chitosan in the fiber samples were tested by colloid titration. This titration mechanism was based on the electricity neutralization reaction between the chitosan solution and polyvinylsulfuric acid potassium salt (PVSK). The chitosan fiber with a weight of ($W$) was dissolved by acetic acid (HOAc) to obtain a cationic polyelectrolyte solution. After the addition of a blutene indicator, the PVSK was added in drops to neutralize the cation. At the end of the electricity neutralization reaction, the color of the blutene indicator turned from blue to purple. In brief, 99 g of HOAc solution (3%) was added into 1.0 g of the fiber sample in a 250 mL conical flask. The solution was then stirred at room temperature until the fiber was well dissolved and then two to three drops of blutene indicator (0.1%) was added and mixed well. This was followed by the addition of 2.5 mM of the PVSK solution drop by drop. Once the color of the solution changed from blue to purple, the addition of PVSK was immediately stopped. The other three samples were dried at 105°C until they reached constant weights to calculate the moisture ratio.

The mechanical properties were tested with an Instron universal testing machine (type Instron 5566, Instron) (condition: 20°C, 50%RH; load: 10 N; speed 20 mm/min).

An antimicroorganism test based on the testing standard ASTM E2149-10 Standard Test Method for Determining the Antimicrobial Activity of Antimicrobial Agents Under Dynamic Contact Conditions was conducted. Antimicroorganism test methods in the textile industry can be grouped into the following categories: contact, transfer, absorption, and dynamic contact methods (Figure 3). Contact methods provide qualitative results, and transfer and absorption methods require strong antimicroorganism effects. Chitosan fibers inhibit microorganism growth but do not kill microorganisms exponentially. Therefore, a dynamic contact method was adopted. Moreover, to maximize the difference in microorganism inhibition performance, the contact time was set to 18 hours.

In brief, an incubated test bacterial inoculum in nutrient broth (Luria-Bertani, or LB broth obtained from Sigma-Aldrich, St. Louis, MO, USA) was diluted with nutrient broth. A total of 5 mL of the diluted inoculum was then added to a 250 mL conical flask containing a fiber sample of 1 g and 70 mL of diluted nutrient broth, which were previously sterilized by autoclaving. The test samples were incubated in an incubator shaker for 18 hours at a temperature of 24 ± 1°C and a speed of 250–300 r/min. After incubation, the solutions were diluted to a ratio of 1:5 with neutralizing solution, and then a serial dilution from $10^{-1}$ to $10^{-3}$ with distilled water was performed. The serial dilution was duplicated. Diluents from $10^{-1}$ to $10^{-3}$ of each sample were used and 100 μL of each diluent was added and spread evenly onto the agar plate (LB agar obtained from Sigma-Aldrich, St. Louis, MO, USA). The agar plates were incubated for 48 hours and the number of colonies was recorded. The antimicroorganism activity was expressed as a percentage reduction of the microorganism after incubation with testing samples compared to that after incubation with control cotton fibers. The protocol for the *Canidia albicans* (CaA) test is similar, but the contact incubation time was 60 hours. The chitosan fiber solution antimicroorganism activity test was based on a 0.02% chitosan/acetic acid solution, which was neutralized to pH 6.8–7.0 by NaOH after a contact time of 20 min.

## Results and discussion

Antimicroorganism and mechanical test results of the chitosan fiber are diverse and strictly related to the core parameter of chitosan, the degree of deacetylation, but the antimicroorganism performance of chitosan in solution form is less related to the degree of deacetylation. Based on the results of these tests, sample FH946 exhibited a relatively good biological and physical performance.

***Degree of deacetylation and molecular weight***

In the production process, higher temperatures and longer reaction times result in shorter molecular chains and weaker fibers. Differences can be seen under a scanning electron microscope (SEM) and by the naked eye. Samples FH946 (Mw: 345.1 Da, DD: 94.63%), FH922 (Mw: 760.5 Da, DD: 92.20%), and FJ922 (Mw: 524.5 Da, DD: 92.23%) have less shimmer and more curl than the other three samples (visible to the naked eye), and they have more folds and unevenness on the surface (visible under a SEM). The handfeel of these three samples is stiffer and rougher, and they have a tendency to bunch together. Notably, samples FH946, FH922, and FJ922 have a DD higher than 92% (and sample FH946 has the highest DD of 94.63%). As shown in Figure 4(a), fiber sample FH946 is covered with small particles that are the result of a high degree of deacetylation.

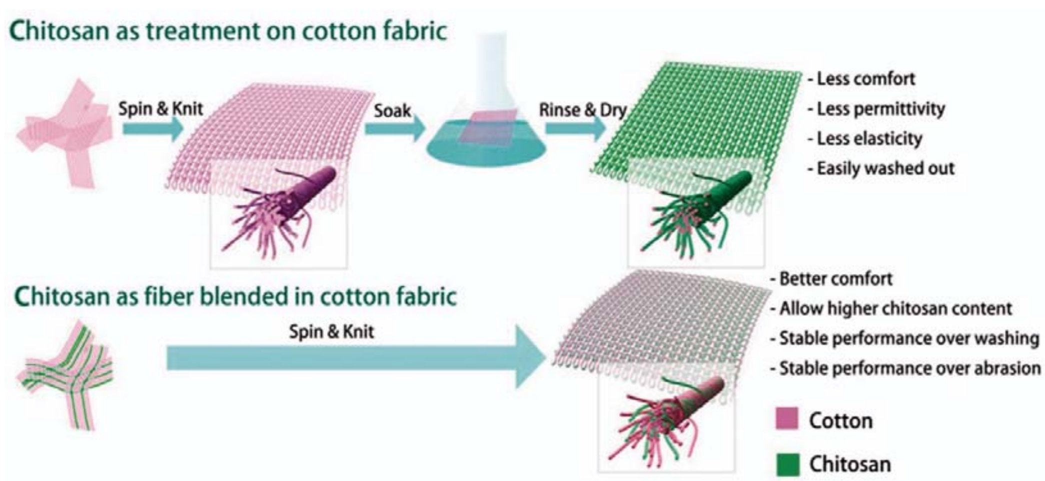



**Figure 1.**

Fabrication process and comparison of chitosan modified fabric and chitosan fiber-blended fabric.

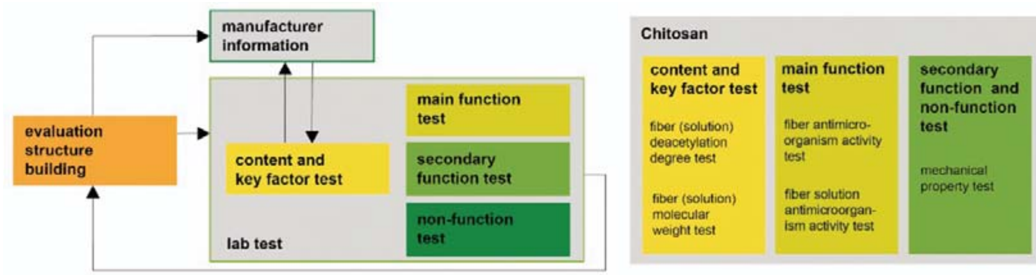



**Figure 2.**

Proposed evaluation process of functional fibers.

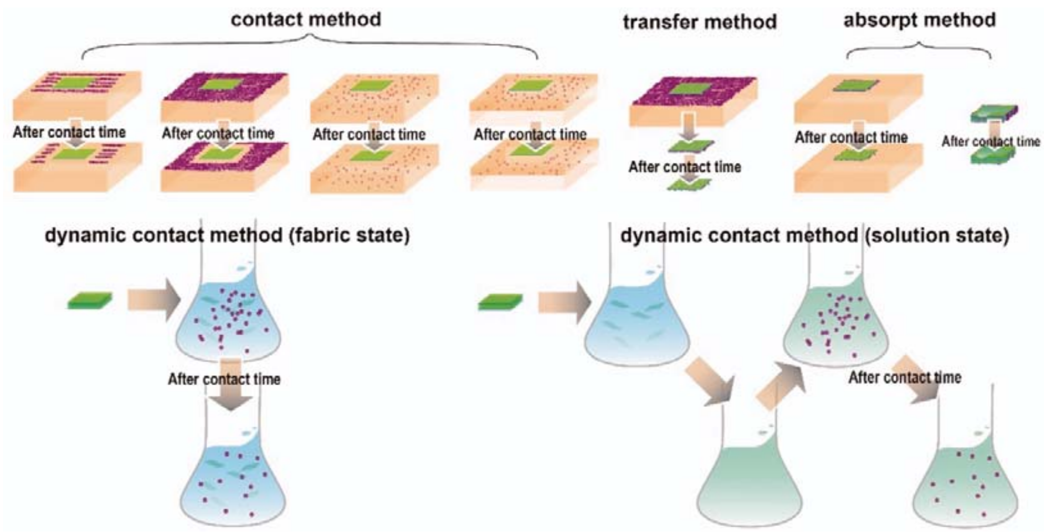
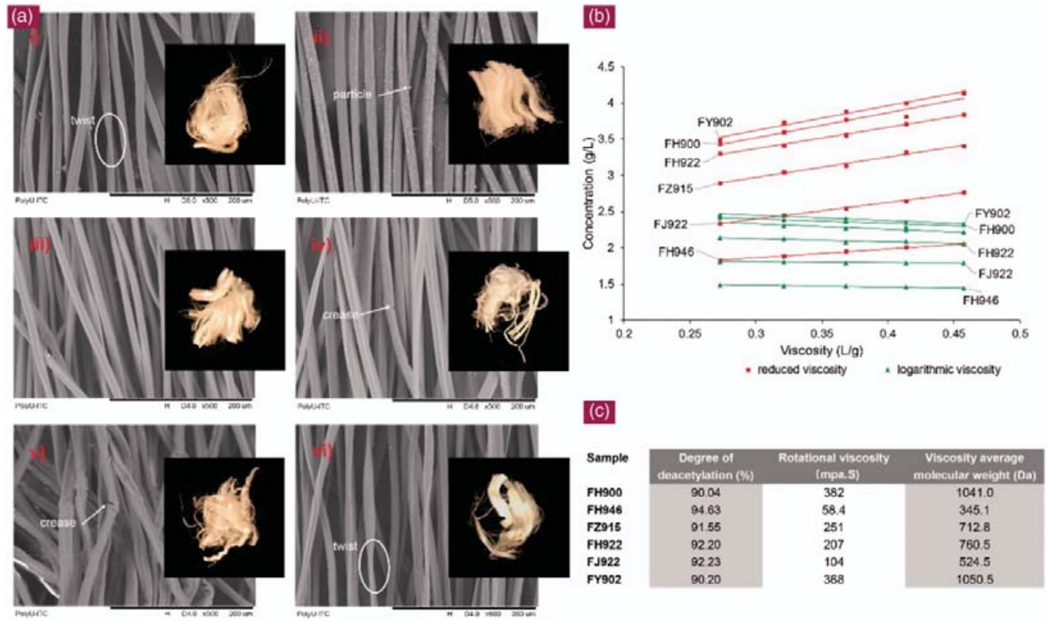



**Figure 3.**

Popular methods of textile antimicroorganism testing.

### *Mechanical properties*

The breaking strength of chitosan fibers ranges from 0.5 to 2 cN/tex, which is weaker than that of cotton fibers (1.8–3.1 cN/tex). Sample FJ922 has the highest breaking strength (1.63 cN/tex), and sample FH900 has the best uniformity of breaking strength (Figure 5). Sample FH946 has a special curve with a clear yielding point, and a significantly higher tensile modulus, which is due to the high degree of deacetylation and the resulting high crystallinity. Although the molecular weight of sample FH946 is lower, the deacetylation degree factor seems to take on more weight. Evidence of high crystallinity in sample FH946 is shown by the particles that appeared on the SEM image (Figure 4(a)), and the flaw on the fibers explains the low uniformity of the same sample, as the fibers tend to break at the crease point. The Young's modulus was not calculated since the fineness cannot be confirmed in this article. Yet, the fineness of the fibers is similar, and therefore it is safe to say that FH946 has the highest modulus.




| Sample | Degree of deacetylation (%) | Rotational viscosity (mpa.S) | Viscosity average molecular weight (Da) |
|---|---|---|---|
| FH900 | 90.04 | 382 | 1041.0 |
| FH946 | 94.63 | 58.4 | 345.1 |
| FZ915 | 91.55 | 251 | 712.8 |
| FH922 | 92.20 | 207 | 760.5 |
| FJ922 | 92.23 | 104 | 524.5 |
| FY902 | 90.20 | 368 | 1050.5 |

**Figure 4.**

(a) Photograph and scanning electron microscope image of fiber samples: (i) FH900; (ii) FH946; (iii) FZ915; (iv) FH922; (v) FJ922; (vi) FY902. (b) Reduced viscosity and logarithmic viscosity of six samples. (c) Degree of deacetylation, viscosity, and molecular weight of six samples.

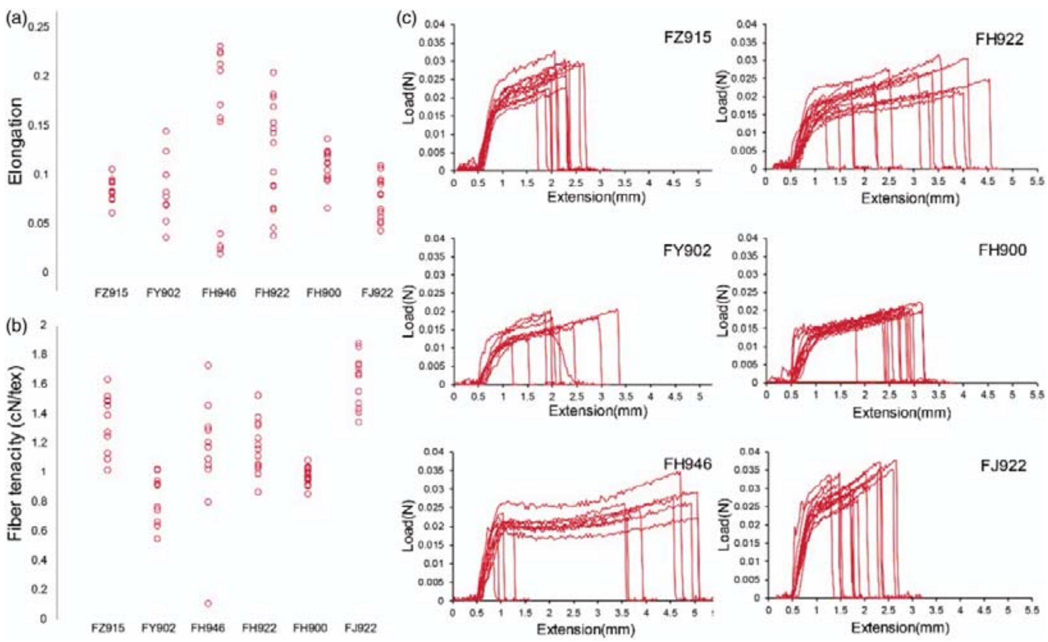



**Figure 5.**

(a) Breaking strength of fiber samples. (b) Elongation of fiber samples. (c) Stress–strain curve of fiber samples.

In the yarn spinning process, frequent breakage of the fiber was an issue, while the results of mechanical property tests revealed the reason for the difficulty in chitosan yarn spinning, poor yarn strength, yarn uniformity, and material loss. Meanwhile, the elasticity of the fiber greatly affects to the handfeel of the fiber, as well as the resulting fabric product. Sample FZ915 has soft handfeel, while FH946 has stiffer handfeel.

### *Antimicroorganism*

#### *Fiber state.*

Three representative microorganism species of *Staphylococcus aureus* (StA), *Escherichia coli* (EsC), and CaA were tested against six fiber samples with a dynamic contact method and contact time of 18 hours. All samples showed excellent inhibition effects over three microorganism strains. Over contact time, the viable count of the microorganisms declined, except for sample FH900 on StA. For all three strains, the performance of FH946 and FH922 ranked first and second, respectively, which can be explained by their high DD compared to the other samples. Sample FJ922, which has a similar DD to FH922, had a slightly worse reduction performance, as FJ922 (Mw: 524.5 Da) has a lower Mw than FH922 (Mw: 760.5 Da). Samples FZ915 (Mw: 712.8 Da) and FH922 (Mw: 760.5 Da) had similar Mw, but had significantly different antimicroorganism performance, as sample FH922 with higher DD performed better. Samples FH900 (Mw: 1041.0 Da, DD: 90.04%) and FY902 (Mw: 1050.5 Da, DD: 90.20%) both showed similar Mw and DD, but FY902 had a significantly higher microorganism killing rate on StA and CaA, while FY902 had a slightly higher DD. Based on these results, the antimicrobial properties of the chitosan fiber are more apparent on EsC if the differences needs to be demonstrated quickly and concisely.

The molecular weight and deacetylation degree are both reported to have influence on the antimicroorganism performance of chitosan in its solution form. Previous studies produced contradictory results.[16–20] Preparation of the fiber includes deacetylation progress, which reduces the chitosan molecule to shorter chains. Chitosan in six fiber samples showed a strong linear relationship between DD and Mw (Figure 6). By calculating the coefficient of determination, we are able to compare the impact of DD and Mw of chitosan in fiber form for antimicroorganism functions.

As shown in Figures 7(b) and (c), the relationships between the microorganism reduction rate and DD and between the microorganism reduction rate and Mw show a strong linear relationship between DD and Mw: DD is positively correlated to the microorganism reduction rate while Mw shows a negative correlation. However, comparing the two factors, DD $(R_S^2 = 0.7524\,;\ R_E^2 = 0.8506\,;\ R_C^2 = 0.3820)$ has a higher coefficient of determination than Mw $(R_S^2 = 0.5578 ; R_E^2 = 0.6494 ; R_C^2 = 0.2548)$, which indicates that the differences in the antibacterial function amongst the six samples are more likely to be caused by their DD.

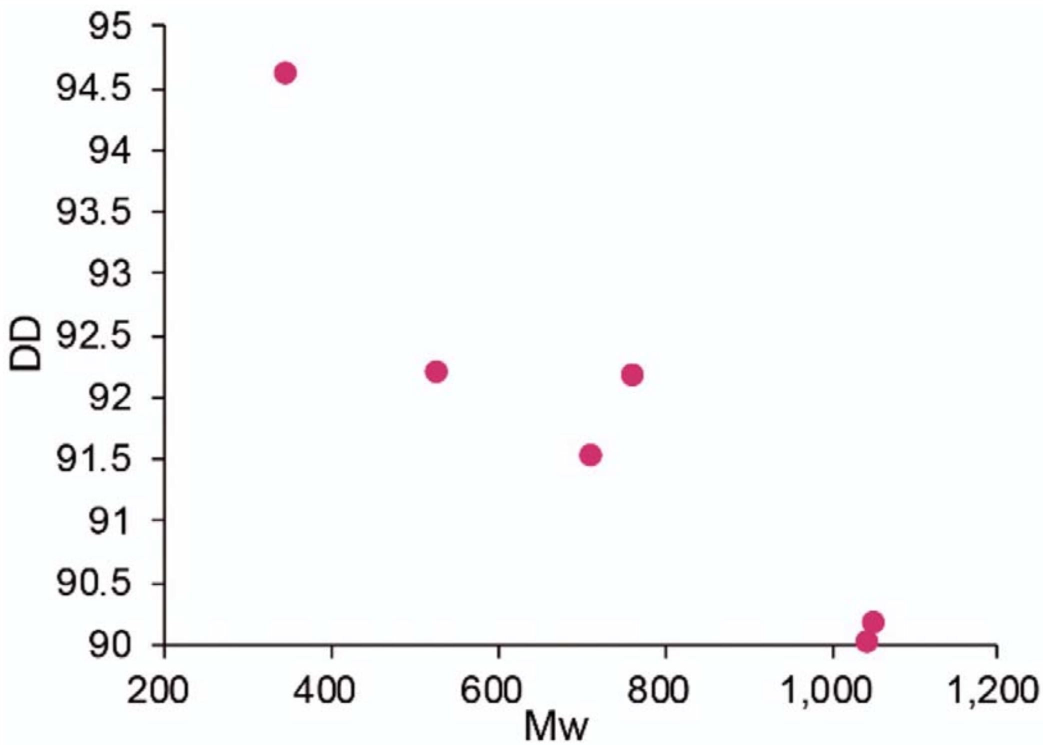



**Figure 6.**

Relationship of the deacetylation degree (DD) and molecular weight (Mw) of chitosan fiber material in six samples.

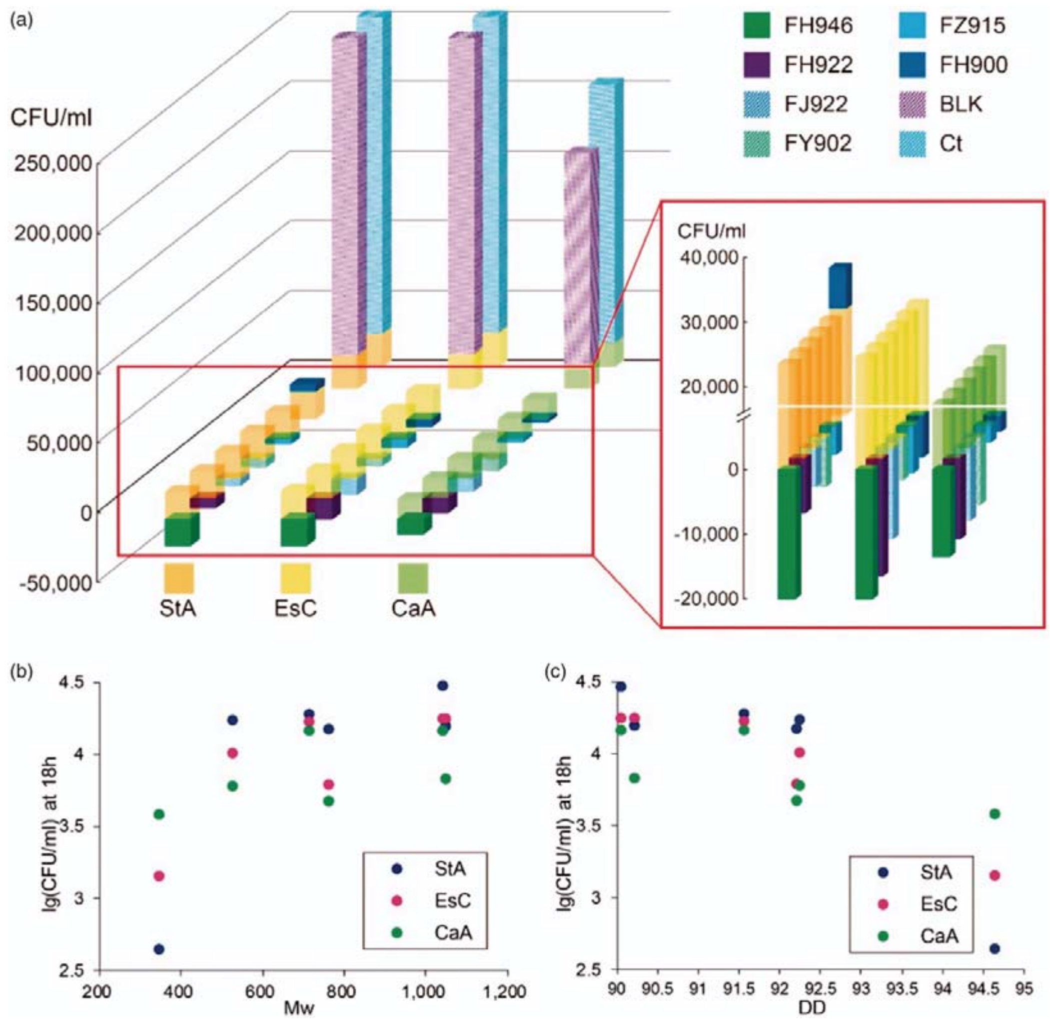



**Figure 7.**

(a) Antimicroorganism test results of fiber samples by inhibition rate; relationship of microorganism count and (b) molecular weight (Mw) and (c) deacetylation degree (DD) of chitosan fiber.

StA: *Staphylococcus aureus*; EsC: *Escherichia coli*; CaA: *Canidia albicans*.

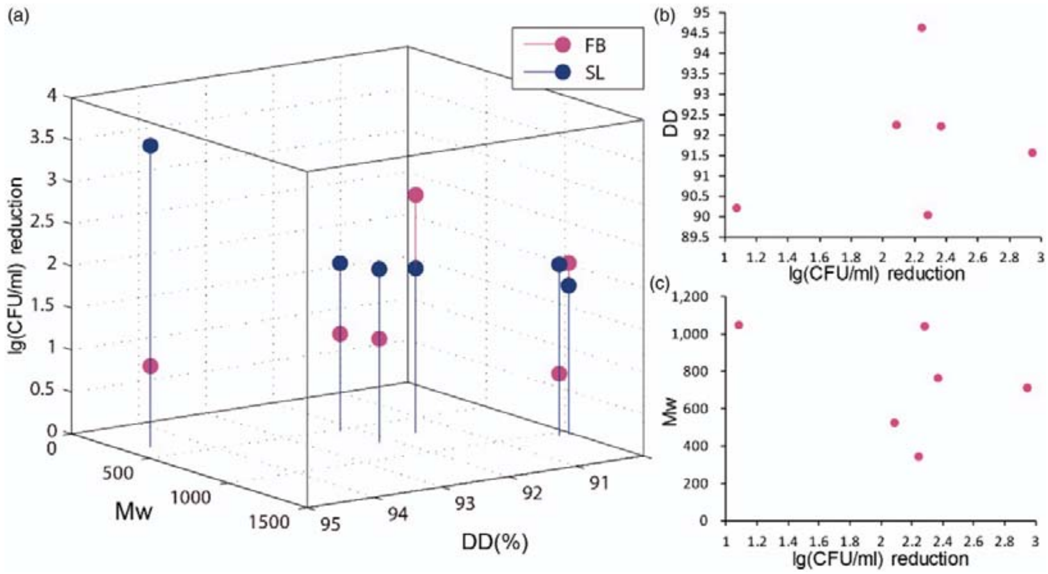



**Figure 8.**

(a) Inhibition on *Staphylococcus aureus* by fiber (FB) sample and fiber solution (SL) sample; relationship of microorganism reduction after 20 min and (b) deacetylation degree (DD) and (c) molecular weight (Mw) of chitosan fiber solution.


**Figure 9.**

Evaluation of chitosan fiber samples (a) and application on garment making (b).

The reduction rate of CaA shows less correlation to the DD and Mw of the chitosan fiber samples. Two pairs of samples (FH900 and FY902; FZ915 and FH922) that had similar DD and Mw had a diverse reduction effect. Although FH900 and FY902 also had diverse reduction performance on StA, the test results for the other samples are relatively closer to the fit line.

***Solution state.***

An antimicroorganism activity test was also administered on the chitosan/acetic acid solution of the chitosan fibers. The results show that the clear trend found in the fiber samples were not found in its solution state, and the results were inconsistent with a previous study.[19] The solution of sample FZ915 had significantly higher inhibition performance (inhibition factor 2.95), while the solution of sample FY902 had significantly lower inhibition performance (inhibition factor 1.08). The other fiber solutions produced similar inhibition factors (FH900: 2.28; FH946: 2.24; FH922: 2.37; FJ922: 2.09). Compared with the fiber state, the antimicroorganism activity of the fiber sample in the solution state had little relationship with the DD or Mw of the chitosan material (Figure 8).

The evaluation of functional fibers includes the evaluation of both functional and non-functional properties. Studies with a similar aim to evaluate performance of chitosan fiber also state the importance of both directions.[21,22] As some functional fibers have more than one function, the evaluation system should include the main functional performance evaluation and the non-functional evaluation. For example, chitosan fibers often have major antimicrobial properties and additional functions that promote cell growth and wound healing. The main and secondary functions change depending on the application, so the fiber evaluation system should be adjusted according to the application.

Similarly, for non-functional fibers, all kinds of performance evaluation should be included in the evaluation system of functional fiber. For example, in the case of yarn spinning, the yarn quality, difficulty of yarn spinning, and fiber mechanical properties are to be tested. Other factors to evaluate may include dyeability, color-fastness, handfeel, hygroscopicity, and comfort performance. Cost is also a factor that cannot be ignored in actual production.

Whether it is a single-component functional fiber or a functional component mixed functional fiber, a surface-modified or laminated functional fiber, there are parameters to adjust its functional performance. The functional components of multi-component functional fibers usually have poor mechanical performance or have a high cost. In production, functional and non-functional properties and costs should be equally considered. For example, the deacetylation of single-component chitosan fiber is the most important index affecting its biological function. Chitosan fiber with a higher deacetylation degree has better antimicroorganism properties and higher strength, but the cost is higher, the handfeel harder, and the fiber strength uniformity is worse with more tendency for breakage (Figure 9). In actual production, the production demand and the terminal demand should be considered comprehensively in order to choose the appropriate fiber raw materials. For example, fiber with chitosan film or chitosan particles needs to be tested not only for the content and functional properties of the active ingredients, but also for the durability after wash and wear.

For the chitosan fibers tested in this article, both mechanical properties or bio-performance are related to the core parameter of chitosan, the degree of deacetylation, which is also reflected in the appearance and handfeel. Therefore, it is possible to quickly identify single-component functional fibers through systematic testing and classification. It is important to note that even if the raw material is the same, the nature of the material and fiber may be different depending on the form that the fiber takes. Through the testing of the fiber in solution form, it can be found that the antimicrobial properties tested from the raw material under the commonly used system do not reflect the antimicrobial properties of the fiber products. Therefore, it is necessary to distinguish functional materials from functional fibers.

## *Conclusion*

A functional textile can be fabricated from a functional fiber or achieved by functional finishing on a non-functional textile. The function of fibers includes chemical functions (which includes promoting chemical reactions), physical functions (which includes electrical and electronic functions), thermal functions, optical functions, physical form functions, and material separation. Biological functions include antibacterial, compatible, and pro-growth functions. Some functional fibers have multiple functions, and besides the main target function, textile fabrication has basic requirements for fiber performance, especially physical performance. Therefore, functional fiber evaluation should consider both functional and non-functional performance. This article takes the chitosan fiber as an example to show how these performances interact with each other, and how the evaluation results can guide the selection for different needs.

The results of this study show that chitosan fiber samples with the highest degree of deacetylation exhibited relatively high mechanical strength and antimicroorganism performance levels, but their elongation weakness affected their softness and handfeel. Moreover, the difference in antimicroorganism performance between the fiber samples was more apparent for EsC than for the other microorganisms. All aspects of functions examined in this study were determined to be associated with the key index of chitosan, namely deacetylation degree. Therefore, evaluating fiber products by using relatively few tests and representative bacterial strains is feasible. Through the demonstration of the proposed industrial-manufacturing-based methods by using chitosan fibers, this study revealed how different dimensions can be reviewed and tested as well as how the test results can facilitate the generation of fast testing methods by focusing on several key aspects. Chitosan fibers were determined to differ in appearance due to their different proportions of functional components or different key factors, such as color at the macro level as well as gloss and surface morphology at the micro level. Determining or predicting the performance of samples in a rapid and simple manner is feasible. The results also reveal that the characteristics of the antimicroorganism performance of the fiber samples differed from those of chitosan in the solution state.

The sample evaluation process and method in this article can help participants in each link of the industry chain select products or adjust production factors. From the point of view of a middleman or a customer, a simple, quick, intuitive identification method without the need for equipment is ideal. In addition, they can select products according to the actual needs of functional and non-functional performance by referring to the results of sampling tests. For example, chitosan fiber has proven to be effective in anti-bacterial and wound healing functions when applied to wound dressings, while in daily clothing use, cost and other factors need to be considered more.

The promotion of functional textiles requires testing standards. However, due to diverse materials, complex functions, and various uses, it is difficult to have a universal system. Meanwhile, textiles involve many stages of products, such as the fiber. Current functional textile testing standards tend to be specific to fabrics, and the attributes of fabrics can be changed through structural changes. Furthermore, the test methods for fabrics may not be suitable for downstream producers. The method presented by this article can help the various links of the industrial chain to adjust their evaluation method, so that the development of material technology can better serve society.

## *Declaration of conflicting interests*

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