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〈研究論文(學術)〉

키토산을 이용한 부직포의 항미생물가공

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Antimicrobial Finish of Nonwoven Fabric by Treatment with Chitosan

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Abstract—Nonwoven fabric was treated with chitosan solution to impart antimicrobial activities by pad-dry method. Antimicrobial activity was measured by Shake Flask Method. Two chitosans of different molecular weight(Mw) with similar degree of deacetylation(DDA) were used : ca. 1,800(chitosan oligomer ; DDA 84%) and 180,000(DDA 86%). Chitosan oligomer displayed high antimicrobial activity against *P. vulgaris* at 0.01%, *S. aureus* and *E. coli* at 0.05% treatment concentration, showing above 90% of reduction rate. Chitosan of Mw 180,000 was effective against *S. aureus, E. coli and P. vulgaris* at 0.05% treatment concentration, showing almost 100% reduction rate. While chitosan of Mw 180,000 showed reduction rate above 75% against *K. pneumoniae* and *P. aeruginosa* at 0.5% treatment concentration, chitosan oligomer was not effective against them. Fabrics become stiffer and less air permeable as treatment concentration increases. Liquid strike-through time of the sample treated with 0.5% chitosan oligomer solution(3.0 sec) was comparable with a hydrophilic finished sample commercially available(2.6 sec).

1. Introduction

Nonwoven fabric is a rapidly growing segment of textile industry. The applications of nonwoven fabrics range from disposable hygienic products through sophiscated medical fabrics along with industrial uses. The fabrics selected for this study is polypropylene nonwoven for use as coverstock of hygienic products. Coverstock is topsheet of diaper and sanitary products and its side is contacted directly with skin. Skin troubles such as diaper rash become a frequent problem with baby wearing disposable diaper. Currently, there is increasing necessity for antimicrobial finishing due to consumer's great interest in health care.

It is well known that chitosan inhibits the growth of many bacteria including Gram-negative and Gram-positive ones[1~3]. Recently, chitosan oligomers

with low degree of polymerization have received much attention because of higher antimicrobial activity and water solubility comparing with chitosan of high molecular weight[2]. Chitosan itself is known to induce little skin reaction over a wide range of biomedical investigation[1]. This is an important advantage especially for the application onto next-to-skin fabrics.

In the present study, we investigated the applicability of chitosans including chitosan oligomer to impart antimicrobial properties to polypropylene nonwoven fabric for use as coverstock. The effect of molecular weight on the antimicrobial activity against various strains of bacteria(*Staphylococcus aureus, Escherichia coli, Proteus vulgaris, Klebsiella pneumoniae, and Pseudomonas aeruginosa*) was studied using a chitosan oligomer and a chitosan of high molecular weight. Additionally, performance properties of treated nonwoven fabrics including strength, stiffness, air permeability and liquid strike-through time were measured.

2. Experimental

2.1 Materials

100% polypropylene nonwoven fabric(thermal bonded, 26g/m^2, 0.13㎜ thickness) was used. Two chitosans of different molecular weight with similar degree of deacetylation(DDA) were used : ca. 1,800(Se-Hwa Co.) and 180,000(Protan Inc.).

2.2 Determination of molecular weight

The molecular weight of chitosan oligomer was determined by gel permeation chromatograph(JASCO, LCSS-905, Jasco. Co., Japan) with Shodex OHpak SB-801, SB-802, SB-803 columns using 0.1 M NaCl in 0.2% acetic acid as a mobile phase at a flow rate of 1㎖/min. Standard compounds used were pullulan(Mw ; 853,000, 95,400, 23,700, 5,800) and chitosan oligosaccharides(Mw ; 322, 483, 706, 876).

2.3 Estimation of DDA

Colloid titration technique[4] was used to estimate DDA. Colloid solution was made by dissolving 1g of chitosan in 100㎖ of deionized water and 100㎖ of 0.4M acetate buffer(18㎖ of 0.4M acetic acid/82㎖ of 0.4M sodium acetate). 1g of colloid solution mixed with 30㎖ of deionized water was titrated with 1/400N potassium polyvinyl sulfate solution after adding 2~3 drops of toluidine blue indicator, and then DDA was calculated.

2.4 Treatment of nonwoven fabrics

Chitosan oligomer(Mw 1,800) was dissolved in distilled water and the other chitosan(Mw 180,000) in 2% acetic acid solution. Fabric samples were padded with chitosan solution of 0.01~ 1.0% (w/v) to give 100% wet pick-up. The padded samples were dried at 100℃ for 3min. The samples treated with chitosan of Mw 180,000 were rinsed in distilled water.

2.5 Evaluation of antimicrobial activity

Antimicrobial activity of the treated fabrics was evaluated by Shake Flask Method[3] in terms of bacteria reduction rate. Five strains of bacteria were used ; *Staphylococcus aureus*(ATCC 6538), *Escherichia coli*(ATCC 8473), *Proteus vulgaris* (ATCC 881), *Klebsiella pneumoniae*(ATCC 4352), and *Pseudomonas aeruginosa*(ATCC 13388).

2.6 Evaluation of performance properties

Performance properties of the treated samples were evaluated using published standard procedures including breaking strength in machine direction, IST(INDA standard test) 110.4 ; stiffness, IST 90.1 ; air permeability, IST 70.1 ; liquid strike-through time using simulated urine, ERT 150.2-93

recommended by EDANA(European disposable and nonwoven association). Simulated urine was discharged at a rate of 25mℓ/3.5sec through the funnel fitted with magnetic valve.

3. Results and Discussion

3.1 Molecular weight and DDA

The molecular weight of chitosan oligomer is distributed in the range of 1,400~2,300 and the weight average molecular weight is 1,814. Its DDA was 84%. The polydispersity index of chitosan of Mw 180,000 was 2.43 and its DDA was 86%.

3.2 Antimicrobial activity of treated fabrics

Five strains of bacteria are selected in this study because they are distributed commonly in our environment and cause cross infections. All of them except *S. aureus*, which is Gram-positive, are Gram-negative. *K. pneumoniae* causes lung fever. *P. vulgaris* and *P. aeruginosa* decompose urea and cause skin problems such as diaper rash. *S. aureus* and *E. coli* cause infection and diarrhea, respectively.

Figs. 1~2 show antimicrobial activity of the samples treated with chitosan oligomer and chitosan of Mw 180,000, respectively. The samples treated with chitosan oligomer display antimicrobial activity above 90% of reduction rate against *P. vulgaris* at 0.01% treatment concentration, *S. aureus* and *E. coli* at 0.05% treatment concentration. The samples treated with chitosan of Mw 180,000 display high antimicrobial activity against *S. aureus, E. coli* and *P. vulgaris* at 0.05% treatment concentration, showing almost 100% reduction rate.

Comparing antimicrobial activity, chitosan oligomer is more effective against *E. coli* and *P. vulgaris* than chitosan of Mw 180,000. They show similar effectiveness against *S. aureus*. Surprisingly, chitosan oligomer is not significantly effective against *K. pneumoniae* and *P. aeruginosa* below 1.0% treatment concentration, showing 30~40% of reduction rate. Whereas, chitosan of Mw 180,000 gives above 75% of reduction rate against *K. pneumoniae* and

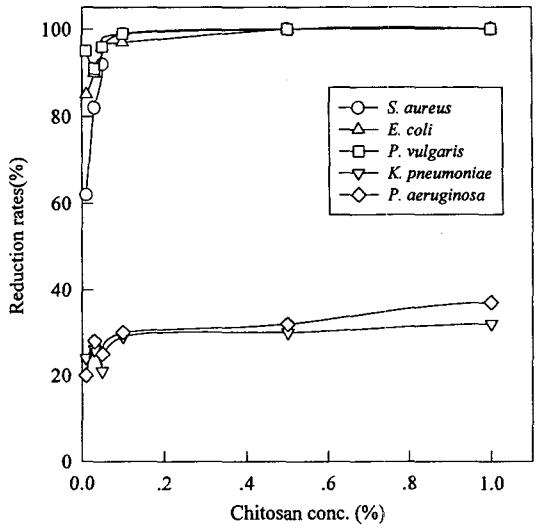

Fig. 1 Antimicroial activity of the samples treated with chitosan oligomer.

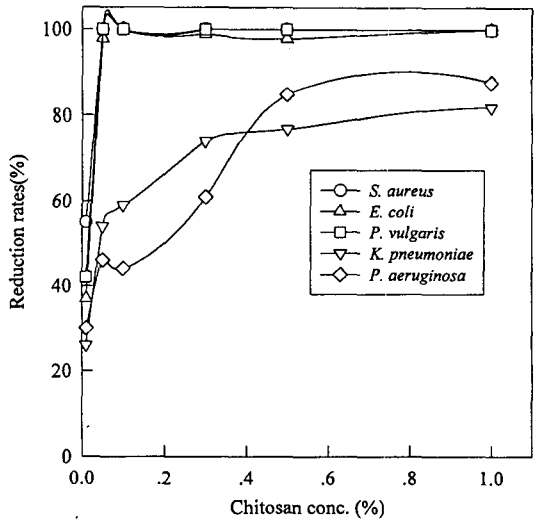

Fig. 2 Antimicroial activity of the samples treated with chitosan of Mw 180,0000

P. aeruginosa at 0.5% treatment concentration. The result indicates that the molecular weight of chitosan affects strains of bacteria being effected as well as effectiveness of inhibiting the growth of bacteria, because there is no significant difference in DDA between chitosan oligomer and chitosan of Mw 180,000. Seo et al. also reported that minimum inhibitory concentration of chitosan(Mw 3×10^5, DDA 80%) was different depending on the strains of bacteria and some bacteria were not effected significantly[1]. Uchida et al. studied the effect of molecular weight of chitosan oligomer on the antimicrobial activity and found that chitosan oligomer consisted of 4~7 unit inhibited the growth of *E. coli*, while oligomer mainly consisted of 3~4 unit did not retard the growth of *E. coli*[2]. On the other hand, Tokura et al. reported that although chitosan oligomer of Mw 9,300 was the growth inhibitor of *E. coli*, chitosan oligomer of Mw 2,200 was not growth inhibitor but growth accelerator[5]. Until now, the effect of molecular weight on the antimicrobial activity of chitosan is not clearly defined yet. Considering that DDA of chitosan is an important factor affecting antimicrobial activity, it is not reasonable to compare the results of these studies because DDA of chitosan oligomer being used by them was various.

In the case of *K. pneumoniae* and *P. aeruginosa*, their mechanism of growth inhibition by chitosan seems to be different from other three bacteria because chitosan of Mw 180,000 is effective against them but chitosan oligomer is not. In other study[6], we treated cotton fabrics with a series of chitosans with different molecular weight and similar DDA and obtained the similar results. There are two proposed mechanisms of antimicrobial activity by chitosan. In one mechanism, the polycationic nature of chitosan interferes bacterial metabolism by stacking at the cell surface[7]. The other mechanism is the binding of chitosan with DNA to inhibit mRNA synthesis[8]. In this mechanism, chitosan must be hydrolyzed to the molecular weight less than 5,000 which is easy to permeated into cell[5]. From the result, it is speculated that the growth of *K. pneumoniae* and *P. aeruginosa* is more effectively inhibited by the chitosan molecule of large size than that of small one by stacking on the cell wall. Further studies are required on the mechanism of antimicrobial action of chitosan including chitosan oligomer.

3.3 SEM analysis

Fig. 3 shows SEM pictures of the treated samples. More surface deposition is observed in the samples treated with chitosan of higher concentration and higher molecular weight, as expected. While small scattered surface deposition is observed in the samples treated with chitosan oligomer, extensive surface deposition is observed in 0.1%/Mw

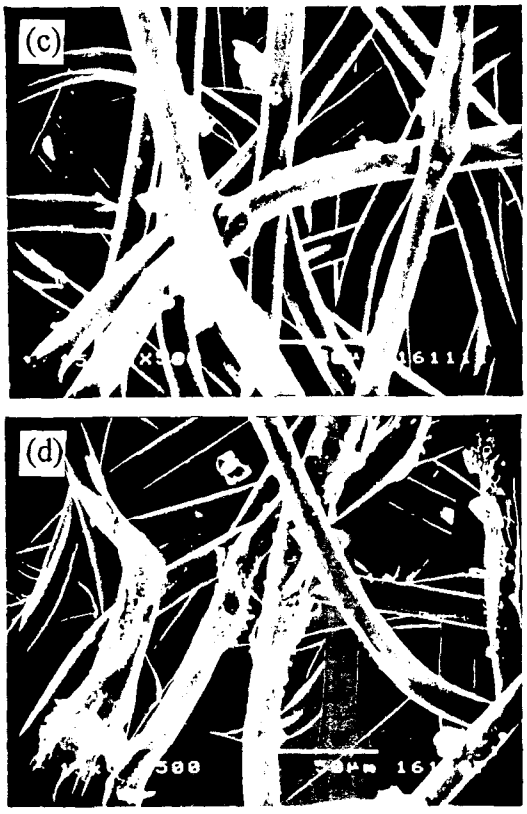

Fig. 3 SEM picture of the treated samples.
a ; 0.05%/Mw 1,800 b ; 0.1%/Mw 1,800
c ; 0.05%/Mw 180,000
d ; 0.1%/Mw 180,000

180,000 treated sample. This change in surface morphology of fabrics due to deposition affects air permeability and liquid strike-through time as well as stiffness and strength of the treated samples.

3.4 Performance properties of treated fabrics

Table 1 shows performance properties of treated fabrics. Stiffness of treated samples increases with the increase of molecular weight of chitosan and treatment concentration. Chitosan of Mw 180,000 gives more surface deposition than chitosan oligomer as shown in Fig. 3, and thus makes fabrics stiffer. Breaking strength in the machine direction, corresponding to the warp direction of woven fabrics, decreases up to 18%. Breaking strength in the machine direction is important in manufacturing hygienic products by continuous process. Usually, 3.5~4.0kg/5cm is necessary for continuous process. Although strength in the machine direction decreases slightly, strength of the treated samples is enough for use as a coverstock.

Fig. 4 shows the effect of chitosan concentration on air permeability of the treated samples. Air permeability of the treated samples decreases with the increase in treatment concentration. Especially, the samples treated with chitosan of Mw 180,000 show sharp decrease in air permeability, resulting from blocking pores by extensive surface deposition. Decrease in air permeability might be affected adversely to the comfort of hygienic products.

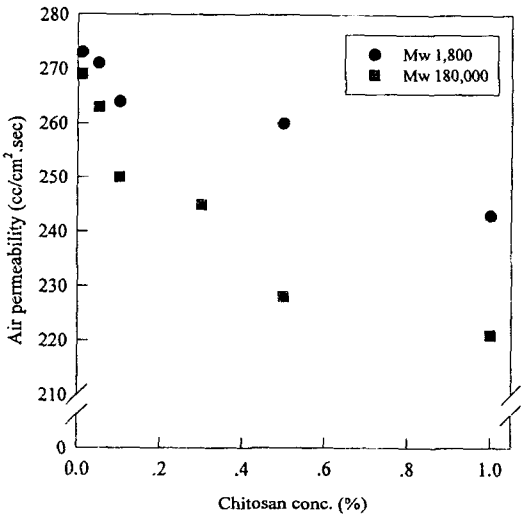

Fig. 4 Effect of chitosan concentration on air permeability of the treated samples.

Fig. 5 shows liquid strike-through time of the treated samples. Liquid strike-through time is the time taken for a known volume of liquid(simulated urine) applied to the surface of a test piece of nonwoven coverstock, which is in contact with an underlying standard absorbent pad, to pass

Table 1. Physical properties of the treated fabrics

Sample (Mw)	Treatment conc.(%)	Stiffness (cm)	Breaking strength[a] (kg)[b]
Control	–	2.55	5.38
1,800	0.01	2.63	5.20
	0.05	2.75	4.39
	0.50	2.87	4.68
	1.00	2.98	4.53
180,000	0.01	2.65	5.10
	0.05	2.70	4.92
	0.50	3.82	4.73
	1.00	3.95	4.76

a : machine direction b : kg/5cm

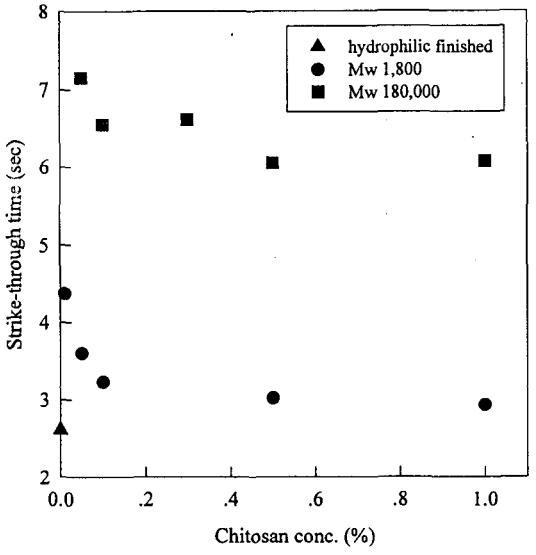

Fig. 5 Effect of chitosan concentration on liquid strike-through time of the treated samples.

through the nonwoven. Liquid strike-through time of the treated samples decreases with the increase of treatment concentration up to 0.5% and reaches to equilibrium thereafter. Hydrophilicity of the treated samples increases as treatment concentration increases because chitosan has hydroxyl and amine groups providing reactive sites with water. The samples treated with chitosan of Mw 180,000 show higher liquid strike-through time than the samples treated with chitosan oligomer. Chitosan of high molecular weight deposited extensively on the surface of fabric and blocked the porous structure of fabric, resulting in the decrease of wicking property and consequently liquid strike-through time increases. Liquid strike-through time of the sample treated with 0.5% of chitosan oligomer, showing 100% reduction rate against *S. aureus*, *E. coli* and *P. vulgaris* is 3.0sec. This is comparable with 2.6sec of a hydrophilic finished sample commercially available. Usually, less than 3.0 sec of liquid strike-through time is required for coverstock.

4. Conclusions

PP nonwoven fabric was treated with chitosan solution of different molecular weight with similar DDA to impart antimicrobial property for coverstock of diaper and hygienic products.

The samples treated with chitosan oligomer display high antimicrobial activity against *P. vulgaris* at 0.01% treatment concentration, *S. aureus* and *E. coli* at 0.05%, showing above 90% of reduction rate. The samples treated with chitosan of Mw 180,000 show high antimicrobial activity against *S. aureus*, *E. coli* and *P. vulgaris* at 0.05% treatment concentration, showing almost 100% reduction rate. While chitosan of Mw 180,000 is effective against *K. pneumoniae* and *P. aeruginosa* showing reduction rate above 75% at 0.5% treatment concentration, chitosan oligomer is not effective against them.

Nonwoven fabrics become stiffer as treatment concentration increases and chitosan of high molecular weight produces stiffer fabric. Breaking strength in the machine direction decreases slightly, but it is enough for use as coverstock. Air permea-

bility decreases as treatment concentration increases. Liquid strike-through time increases to 0.1% of treatment concentration and reaches to equilibrium thereafter. Liquid strike-through time of the samples treated with 0.5% of chitosan oligomer shown 100% reduction rate against *S. aureus, E. coli* and *P. vulgaris* is 3.0.sec, which is comparable with 2.6sec of a hydrophilic finished sample commercially available.

From the results, it is concluded that chitosan oligomer is applicable to impart antimicrobial properties for polypropylene nonwoven coverstock of diaper. And optimum molecular weight of chitosan should be selected according to the end use of textile products.

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