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# Study on the Modification of PP Nonwoven Fabric

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#### Abstract

This experiment aimed to graft PP nonwoven fabric with a silane coupling agent, then process it with chitosan or AgNO<sub>3</sub>, next to analyse and test it with related instruments, such as FT-IR, NMR, SEM, EDS, TGA etc., and finally study the influence of the modification of the silane coupling agent on the properties of the processed fabric. The results indicated that the silane-coupling agent could be effectively grafted onto the surface of the PP nonwoven fabric. On the other hand, the thermal stability of the PP non-woven fabric changed due to the processing of the silane-coupling agent and chitosan or AgNO<sub>3</sub>. In addition, the PP nonwoven fabric attained good antibacterial and water imbibitious properties through post-processing the nonwoven fabric with polyacrylic acid sodium (PAANa) and chitosan or AgNO<sub>3</sub>.

Key words: silane-coupling agent, chitosan, thermal stability, antibacterial, nonwoven.

#### Introduction

At present, among the artificial fibres used in nonwoven fabrics, polypropylene fibre accounts for 63%, polyester fibre 23%, viscose rayon 8%, acrylic fibre 2%, polyamide fibre 1.5%, etc. Therefore, we can see that the nonwoven fabrics of medical materials have great development potential.

PP nonwoven fabric is a thermoplastic material with excellent comprehensive performance; moreover, it is small in density, easy to produce and has low production costs. However, it also has some defects, such as a low melting point, low thermal deformation temperature, poor creep-resistant performance, poor dimensional stability, low temperature brittleness, etc.

On the other hand, PP nonwoven fabric itself is resistant to erosion by mildew, but it cannot directly kill it. Furthermore, its side-chain polymers have flexible molecule chains and are non-polar, with the latter attribute making it difficult to blend with other polymers, and the weak molecule combination making its heat resistance poor. Thus, in order to expand the functions of PP nonwoven fabrics, it is necessary to modify and process it.

Textiles have a variety of antibacterial processing methods [1 - 11]. For instance, the 'Moiskin' of Nisshinbo and the 'Ytopolei' of Fujibo were produced through spinning after adding chitosan powder to polymers. DC-5700, made by Dow Corning (USA), is an organic silicone resin quaternary ammonium salt with good antibacterial properties. Sandaulan SSN in Japan used biguanidine agents to produce antibacterial textiles,

while Toyobo processed the extracts of aloe into textiles with antibacterial, antiinflammation and anti-allergy properties, which was called 'Alobylgari' [12].

The processing of the above products are similar in that they are all filled with polymer, and so-called diversification cannot be realised from a small amount. Although some people have utilised the chitosan immersion method to carry out the process [13], the object used was natural fibres with function groups, which were not able to have subsequent reactions similar to PP fibres; therefore, modification was indispensable in order to allow PP fibres to take part in chemical reactions.

In the past, there were several techniques of the surface modification polypropylene fibre: 1) Chemical Oxidation: the surface of the fibre was oxidised with oxidizing reagents or gases to change the crudeness degree and content of the surface polar group. 2) Plasma Treatment: the advantage of this method was that the reaction was easy to control on the surface of the fibre, which caused little damage to the fibre and obviously changed the effect of the polymer with the inactive polar-end on the surface. The chemical reactions that emerged here included the fracture and ablation of molecule chains, surface cross-linking, the introduction of polar groups, etc.; although this method did not cause pollution, it incurred huge cost [14 - 15]. 3) Surface Grafting: because PP fibres have no reaction activity groups, its surface must be radiated or treated using chemical methods in grafting to produce grafting points. According to different induction modes, grafting treatment can also be divided into plasma grafting,

radiation grafting, chemical grafting, [16 - 17] etc. 4) Coupling Agent Treatment: the advantage of this method is that it will not damage the mechanical performance of the fibre, and it offers a good interface modification effect. Silane grafting also has unique advantages - it causes the fabrics to have functions of grafting and modification, as well as to possibly produce further hydrolysis, which can then form cross-linking structures, as shown in Figure 1, allowing the fabrics to have excellent physical properties. This technique can be applied for the modification of fibres without specific function groups or for the forming of common plastic. The cost of the equipment required is low, and the performance of the product is excellent.

However, the use of a silane-coupling agent to modify glass fibres and inorganic fillers, etc. was developed at that time, but more experimentation and studies on the modification process of PP fibres were still required.

This study planned to perform grafting modifications of PP nonwoven fabrics with a silane-coupling agent, such as VTES (vinyl triethoxysilane), and then conduct further processing with different anti-bacterial materials, in which the 3 ethoxy of VTES reacted with the anti-bacterial materials, while at the same time a cross linking reticular structure would be formed in the fibre molecules. Thus, the heat resistance of nonwoven fabrics could be improved.

# Experiment

# Materials

The Benzyl Peroxide (99%, BPO), Tetrahydrofuran (THF), Acetic Acid and 2-Propanol used were all drug-test grade, purchased from Wako Pure Chemical Industries, Ltd.; the Triethoxyvinylsilane (97%, VTES) and Dibutyltin dilaurate were also drug-test grade, purchased from Acros Organics (USA); the Silver Nitrate was high-grade drug-test, purchased from Wako Pure Chemical Industries, Ltd. (Japan); the Tri(nonyl phenyl) phosphate was industrial grade. purchased from Go Yen Chemical Industries Co., Ltd. (Taiwan); Chitosan (D.C = 85%, Mv = 240,500, Taiwan Kaohsiung Applied Chemistry Co. Ltd.) and Poly (acrylic acid sodium salt) (PAANa, Mw = 8,000, 45% solution in water), pur-

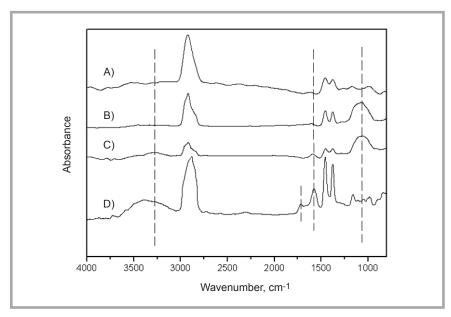


Figure 1. FT-IR of PP nonwoven fabrics  $(A - P_0, B - P_1, C - P_{11}, D - P_{21})$ .

chased from Aldrich Chemical Industries was also used.

#### Methods

# Modification of PP nonwoven fabrics

Firstly, 10 cm<sup>2</sup> of PP nonwoven fabric was cleaned with acetone until the weight remained constant, then 0.25 g of benzyl peroxide (BPO), VTES (1.5 g or 3.0 g), 0.1 g of the catalyst, and 0.1 g of antioxidant were prepared, then. THF was also added until the total weight amounted to 100 g. Next PP nonwoven fabrics were put into a reaction flask and subsequently infused with nitrogen gas. Afterwards they were placed in a temperature of 70 °C to react for 2 hours, then the PP grafted fabrics were immersed in THF for 30 minutes, and finally the fabrics were dried and re-immersed using the same method. This process was repeated three times.

# Antibacterial processing of the modified PP nonwoven fabrics

**Table 1.** Definitions of the various symbols of the PP nonwoven fabrics.

| Symbols                          | Definitions   |
|----------------------------------|---|
| P <sub>0</sub>                   | Greige PP nonwoven fabrics                                |
| P <sub>1</sub> or P <sub>2</sub> | P <sub>0</sub> modified with 1.5 g or 3 g VTES            |
| P <sub>11</sub>                  | P <sub>1</sub> processed with Chitosan                    |
| P <sub>12</sub>                  | P <sub>1</sub> processed with AgNO <sub>3</sub>           |
| P <sub>21</sub>                  | P <sub>1</sub> processed with Chitosan and PAANa          |
| P <sub>22</sub>                  | P <sub>1</sub> processed with AgNO <sub>3</sub> and PAANa |
| CHT                              | Chitosan  |

We used two types of the antibacterial processing of the modified PP nonwoven fabrics: one was to dissolve 2 g of chitosan in 500 ml of Acetic acid solution with a concentration of 2%, then the modified-PP fabrics were immersed in the above solution for 10 minutes at room temperature. The other was to add 100 ml of 0.01% AgNO<sub>3</sub> in 25 ml IPA, then the modified-PP fabrics were immersed in the above solution for 10 minutes at room temperature. All the fabrics above were treated by the 2 dip 2 nip process at a pick-up of about 90%, below 80 °C, then pre-dried for 5 minutes, and finally cured for 3 minutes below 100 °C. Then the properties of the treated fabric were tested. In the two processing methods above, proper amounts (0.5 g - 3 g) of PAANa polymeric material needed to be added.

### Analysis and measurement

The ATR-FTIR spectra of the fabrics were recorded with a Bio-Rad Digilab FTS-200 spectrometer using an MCT detector. A diamond crystal was used as the internal reflective element. Single beam spectra were the result of 64 scans at a spectral resolution of 4 cm<sup>-1</sup>. The surface morphologies of the fabrics were observed with a JEOL Model JSM 6400 scanning electron microscopic. A gold coating was deposited on the samples to avoid charging the surface. The distribution of all atoms of the fabrics were analysed using JEOL JSM 6700F (Tokyo, Japan) energy-dispersive spectroscopy. The thermal resistance of the samples was measured by a DuPont 2200

weight-loss analyser. 5 - 10 mg samples were placed in alumina crucibles and tested using a thermal ramp within a temperature range of 30 - 600 °C, at a heating rate of 20 °C/min and nitrogen flow speed of 20 cc/min, after which their initial decomposition temperature (IDT) was obtained. The chemical shifts of the <sup>29</sup>Si of the fabrics were measured with a solid-state nuclear magnetic resonance (NMR) spectrometer. The samples were analysed using a Bruker Advance 400 <sup>13</sup>C-NMR spectrometer at 50 MHz, and the spectra were observed under cross-polarisation, magic angle sample spinning and power decoupling conditions with a 90° pulse and 4 s cycle time. The anti-bacterial properties of the same sample were tested according to method JIS1902-1998 of the Japanese Association for the Functional Evaluation of Textiles (JAFET). The water imbibitions of the processed fabrics were tested as follows [18]: the processed fabrics were put into a beaker with distilled water for 15 minutes, then the fabrics were taken out to be dehydrated with a centrifugal separator (4000 r.p.m.) for 10 minutes, and finally the fabrics were weighed. The water imbibitions were solved according to the following formula [19 - 20]:

Water imbibitions = 
$$= (W_1 - W_0)/W_0 \times 100\%$$

 $W_1$  - the weight of the processed fabric after dehydration,  $W_0$  - the weight of the processed fabric after conditioning.

Investigations on leaching behaviour were performed at 40 °C using a Rapid H-type dyeing machine. As a washing solution, a 1% aqueous solution of sodium lauryl sulfate SDS of pH 7 was used. After leaching for 20 minutes, the textile samples were rinsed intensively with water, dried at room temperature, and the physical properties were investigated again after 20 washes.

# Results and discussion

## ATR-FTIR

Figure 1 depicts an FT-IR analysis map showing the processing conditions of the PP nonwoven fabrics in different phases. In Figure 1.A, there are absorption peaks of -CH, -CH<sub>2</sub>, -CH<sub>3</sub> at 2925 cm<sup>-1</sup> and -CH<sub>2</sub>, -CH<sub>3</sub> at 1458 cm<sup>-1</sup> at 1377 cm<sup>-1</sup>, while in Figure 1.B, besides the absorption peaks above, there was the obvious absorption peak of Si-O-Si at 1072 cm<sup>-1</sup>.

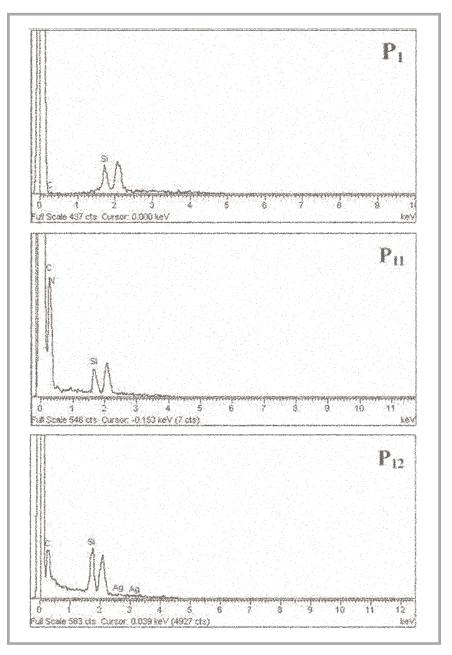


Figure 2. EDS of PP nonwoven fabrics; devinitions according to Table 1.

**Table 1.** EDS of PP nonwoven fabrics; devinitions according to Table 1.

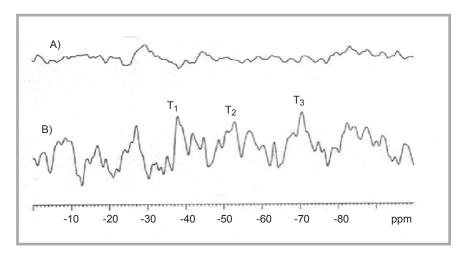
| Fabrics         | Element content, % |       |       |      |  |
|-----------------|--------------------|-------|-------|------|--|
|                 | С                  | N     | Si    | Ag   |  |
| P <sub>1</sub>  | 95.76              | 0     | 4.24  | 0    |  |
| P <sub>11</sub> | 78.51              | 17.42 | 4.07  | 0    |  |
| P <sub>12</sub> | 26.36              | 0     | 69.06 | 4.58 |  |

Thus, it was verified that the VTES silane-coupling agent was successfully grafted onto the PP nonwoven fabrics.

In *Figure 1.C*, we can see that there was an absorption peak of amide at 3279 cm<sup>-1</sup>, whereas those of the -CONH-group in Chitosan were at 1662 cm<sup>-1</sup> and 1585 cm<sup>-1</sup>. From these two absorption

peaks, we judged that chitosan existed in the PP nonwoven fabrics.

An absorption peak was found at 907 cm<sup>-1</sup>, the reason for which was that a portion of the chitin was not deacetylated [21 - 23]. Additionally, in *Figure 1.D* an absorption peak of >C=O was found at 1715 cm<sup>-1</sup>, which was the special absorption peak of PAANa [24]. Hence, it was proven



**Figure 3.** NMR of PP nonwoven fabrics; devinitions according to Table 1,  $(A - P_0, B - P_1)$ .

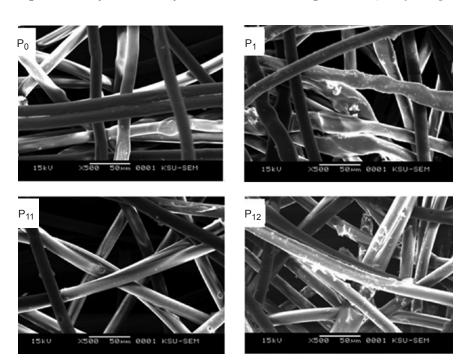


Figure 4. SEM of PP nonwoven fabrics; devinitions according to Table 1.

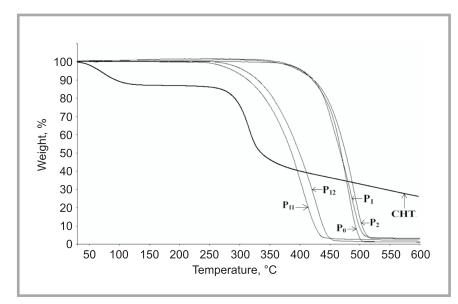


Figure 5. TGA of PP nonwoven fabrics; devinitions according to Table 1.

that PAANa was able to combine with the Chitosan through ionic bonding, and then be fixed to the fabrics. However, we could not completely verify the existence of Chitosan using FTIR analysis, hence we employed the EDS test for further confirmation.

#### **EDS**

Figure 2 and Table 2 display EDS analysis charts showing the processing conditions of PP nonwoven fabrics in different phases. From these charts, we can see that sample P<sub>1</sub> definitely exhibited Si, indicating that the VTES silane-coupling agent had been grafted onto the PP nonwoven fabric. Sample P<sub>11</sub> showed N, indicating that PP nonwoven fabrics were actually coated on the surface of fibres after being processed with chitosan. Sample P<sub>12</sub> showed Ag, indicating that there was Ag coating on the surface of fibres after processing with AgNO<sub>3</sub>.

#### **NMR**

Figure 3, the <sup>29</sup>Si-NMR chart shows the modification conditions of the PP nonwoven fabrics with VTES. Figure 3.B shows absorption peaks of -39 ppm ~ -41.9 ppm (T1), -53 ppm ~ -56.8 ppm (T2), and -70.6 ppm (T3) after the PP nonwoven fabric was modified with the VTES silane-coupling agent. These were the absorption peaks of Si-OH after hydrolysation and VTES modification [25 -26].

Figure 3.A shows the PP nonwoven fabrics without modification. From the map, we were unable to find the above absorption peaks. From the tests and analysis of FTIR and NMR, we know that the PP nonwoven fabrics and VTES silane-coupling agent had actually combined. VTES proved simple to hydrolyze and condense to form SiO<sub>2</sub> reticular structures of T<sub>3</sub> and T<sub>2</sub>, which did well for the heat resistance of the PP nonwoven fabrics.

## **SEM**

Figure 4 depicts an SEM chart showing the processing conditions of the PP nonwoven fabrics in different phases. In Figure 4.A the surface of the fibre was smooth, whereas in Figure 4.B, the surface of the fibre was obviously rough and had the phenomenon of coating, which verified again that the VTES silane-coupling agent was successfully grafted onto the PP nonwoven fabric, as mentioned in the FTIR analysis. By respectively comparing Figures 4.C and

**4.D** with *Figure 3.B*, we know that there were relatively few coats on the surface shown in *Figure 4.C*; however, we were able to judge whether chitosan existed through EDS.

Besides roughness, the PP nonwoven fabrics had the phenomenon of cracking on the surface after being processed with AgNO<sub>3</sub>, which may have been caused by the oxidation of AgNO<sub>3</sub>

#### **TGA**

Figure 5 and Table 3 show the TGA analysis charts of various PP nonwoven fabrics. From Table 3, we can see that the initial decomposition temperature of the PP nonwoven fabrics (P<sub>1</sub>, P<sub>2</sub>), which were modified with VTES, were slightly higher than that of the PP nonwoven fabrics  $(P_0)$ , and the decomposition temperature of the fabrics modified with high-concentrations of VTES were obviously high; the main reason being that the PP nonwoven fabrics had a great deal of VTES silane-coupling agent on the surface, which made the decomposition temperature rise slightly. In addition, if the modified PP nonwoven fabrics were then processed with chitosan (as in  $P_{11}$ ), the decomposition temperature would apparently decline, which was due to the decomposition temperature of chitosan being low. Meanwhile, the maximum decomposition rates followed this order:  $P_2 > P_1 > P_0 > P_{12} > P_{11} > CHT$ , the reason for which was the same as above. Contrarily, the residual amounts followed the order of CHT  $> P_2 > P_1 > P_{11} > P_0 >$ P<sub>12</sub>. From the above, we learned the following: 1) a great deal of chitosan was left when it was heated to 500 °C, but its maximum decomposition temperature and decomposition rates were the worst. This may be attributed to the inorganic substances remaining from the manufacturing process of chitosan, which left high residual amounts; but chitosan containing 40% moisture could not be dried, hence the phenomenon of weight loss would appear before 100 °C [27]. 2) Modification with VTES led to an increase in the stability of the heat resistance of the PP nonwoven fabrics.

# Water imbibitions and antibacterial properties

From *Table 4*, we know that the water imbibitions of the PP original fabric was approximately 19%, despite the fact that, logically speaking, PP nonwoven fabrics are unable to absorb water. However, the

**Table 3.** Thermal properties of PP nonwoven fabrics; 1) at 500 °C.

|                 | Thermal properties            |                          |                              |  |
|-----------------|-------------------------------|--------------------------|------------------------------|--|
| Fabrics         | Decompositiom temperature, °C | Decomposition rate, %/°C | Char yield <sup>1)</sup> , % |  |
| P <sub>0</sub>  | 420.19                        | -0.757                   | 3.212                        |  |
| P <sub>1</sub>  | 422.65                        | -0.708                   | 8.822                        |  |
| P <sub>2</sub>  | 424.56                        | -0.637                   | 14.248                       |  |
| P <sub>11</sub> | 313.70                        | -0.921                   | 5.601                        |  |
| P <sub>12</sub> | 329.51                        | -0.916                   | 1.465                        |  |
| CHT             | 294.84                        | -1.038                   | 32.949                       |  |

Table 4. Water imbibitions of PP nonwoven fabrics; devinitions according to Table 1.

| Fabrics         | PAANa, g | Moisture imbibitions, % |  |
|-----------------|----------|-------------------------|--|
| P <sub>0</sub>  | 0        | 10.4                    |  |
| P <sub>11</sub> | 0        | 73.1                    |  |
| P <sub>21</sub> | 0.5      | 116.7                   |  |
|                 | 1        | 164.9                   |  |
|                 | 2        | 198.5                   |  |
| P <sub>12</sub> | 0        | 64.2                    |  |
|                 | 0.5      | 97.1                    |  |
| P <sub>22</sub> | 1        | 121.7                   |  |
|                 | 2        | 144.3                   |  |

**Table 5.** Wash fastness of the water imbibition and antibacterial properties of PP nonwoven fabrics; <sup>1</sup> Water imbibitions, <sup>2</sup> bacteriostatic value, <sup>3</sup> bactericidal value.

| Fabrics         | Before washing |                |                | After washing 20 times |     |     |
|-----------------|----------------|----------------|----------------|------------------------|-----|-----|
|                 | M.I.,1 %       | A <sup>2</sup> | B <sup>3</sup> | M.I., %                | Α   | В   |
| P <sub>0</sub>  | 10.4           | 0              | <0             | 18.3                   | 0   | <0  |
| P <sub>21</sub> | 164.9          | 4.5            | 3.0            | 107.5                  | 3.1 | 1.7 |
| P <sub>22</sub> | 121.7          | 4.5            | 2.4            | 81.3                   | 3.0 | 1.5 |

structure of the nonwoven fabric was, to some extent, loose, which permitted the infiltration of water molecules.

The PP nonwoven fabrics modified with VTES saw an apparent increase in their water absorption abilities, due to the -Si(OH)<sub>3</sub> values of the VTES retaining some -OH groups in its periphery after condensation, which combined with the hydrogen bonds of water molecules and helped improve the water absorption of the nonwoven fabrics. The PP nonwoven fabrics modified with VTES and added chitosan (P11), as well as the PP nonwoven fabrics modified with VTES and added AgNO<sub>3</sub> (P<sub>12</sub>) showed better water absorption effects than the PP nonwoven fabrics without modification  $(P_0)$ . Their ratios of water content were 73.1% and 65.4%, respectively, which was due to the Chitosan and AgNO3 having good moisture retention abilities, leading to an improvement in water absorption. However, AgNO<sub>3</sub> was poor in infiltration [28], therefore, P<sub>12</sub> was slightly lower in water imbibitions than P<sub>11</sub>. If (Chitosan+PAANa) or (AgNO<sub>3</sub>+PAANa) were used in the post-processing, the introduction of PAANa would increase the water absorption of the nonwoven fabrics by 3 to 10 times, which was due to the strong water absorption properties of PAANa. When the hydrophilic groups reacted with water, the polymer chain groups —COONa decomposed into COO- and Na<sup>+</sup>.

The carboxyl on the polymer chain was not able to diffuse in water, causing negative charges on the polymer chain. The electrostatic repulsion forces made the polymer chain expand, further improving the water imbibitions [29]. Therefore, we confirmed that the PP nonwoven fabrics, which were poor in water absorption, were greatly improved with the addition of PAANa. The nonwoven fabrics processed with chitosan and PAANa are better at water absorption because the chitosan contains –OH and -NH<sub>2</sub> groups, which can form hydrogen bonds with water.

From *Table 5*, we can see that the antimicrobial properties of the untreated fabrics were much worse than those of the fabrics treated with VTES and chitossan or AgNO<sub>3</sub>. The difference is presum-

ably because the amine group in the chitosan molecule is apt to form positively charged quaternary ammonium ions in an acidic medium that would, in turn, reduce the bacterial metabolism through the adsorption and stacking of chitosan polymer chains on the bacterial cell wall and through blocking DNA transcription by chitosan. This process would thereby enhance the antimicrobial property of the treated fabric. The other fabrics have the same antimicrobial properties, which were treated with AgNO<sub>3</sub>.

#### Washing fastness

Table 5 shows the changes in water imbibition, bacteriostatic value and bactericidal value of the fabrics after 20 washes. It shows that greige fabric does not change greatly in the aspect of retaining water imbibitions as well as antibacterial properties after washing, whereas treated fabrics P21 and P22 see an obvious reduction in water imbibitions and antibacterial properties; the former by about 34.8% and the latter - 33.2%, both showing a similar range of reduction. The reason may be that the PAANa was washed. Although the anti-bacterial value significantly decreased, it was still effective.

# Conclusion

This experiment utilised VTES to modify PP nonwoven fabrics, then the fabrics were post-processed using PAANa and chitosan, or AgNO3, and finally followed by an exploration of the influences of VTES and PAANa concentrations on related properties of the fabrics processed. From this experiment, we drew the following conclusions: 1) the VTES silane-coupling agent could be effectively grafted onto the surface of the PP nonwoven fabrics. 2) The PP nonwoven fabrics without modification were very smooth in appearance, whereas the modified and processed PP nonwoven fabrics had the disadvantages of the coating and cracking of surface fibres. 3) PP nonwoven fabrics can be improved with respect to the stability of their heat resistance by being modified with VTES. 4) If the modified PP nonwoven fabrics were post-processed with PAANa and the addition of Chitosan or AgNO3, the processed fabrics would apparently be improved in their antiseptic properties and water absorption.

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