

Synthesis of blocking polyether silicone oil and silicone blocking waterborne polyurethane and application to cashmere knitted fabric finishing

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Abstract

Blocking polyether silicone oil is prepared through a reaction between amino-terminated polyether and epoxide-terminated polyether silicones. A fabric finishing agent made with cationic waterborne polyurethane modified by silicone oil is prepared by using polytetramethylene glycol as the soft segment, *N*-methyl diethanolamine as the hydrophilic unit, and tailor-made blocking polyether silicone oil as a chain extender for modification purposes. The finishing agent and the blocking polyether silicone oil are jointly used to treat cashmere knitted fabric. It is found that resistance to pilling of the treated cashmere knitted fabric is improved from a scale of 2–3 to 4 and its washing shrinkage rate is reduced from 11.2% to 3.3%. The treated cashmere knitted fabric has good hydrophilic ability, high water vapor and air permeabilities, and a soft handle. Moreover, the color of the fabric remains almost unchanged after finishing and the durability of the finishing properties is excellent.

Keywords

blocking polyether silicone oil, waterborne polyurethane, cashmere knitted fabric, finishing, resistance to pilling, shrinkage

Cashmere knitted fabric is generally considered as a textile that requires special care, as when the fabric is under tension and abrasion during wearing and washing, the fibers in the fabric tend to slip because of the directional friction effect, thus resulting in fabric felting shrinkage, fuzzing, and pilling.^{1–5} At present, the most commonly used means of finishing for resistance to felting shrinkage and pilling of cashmere knitted fabric, both locally and internationally, are to use a resin-finishing treatment or a combination of chlorination, oxidation, and biological enzyme treatment with resin-finishing.^{6–9} These methods can effectively protect the treated cashmere knitted fabric from shrinking and pilling during wearing, but consequently, there are negative effects on its original properties, such as softness, water vapor and air permeabilities, and so on.

The application of waterborne polyurethane (WPU) as a resin for textile finishing creates a tough film on the

fiber surface, which can reduce fiber slippage and strengthen the adhesion force between the fiber ends and yarn, thus effectively improving resistance to pilling and felting shrinkage of the treated fabric.^{10–12} In some of the literature, organosilicone modified WPU was investigated for use as a textile finishing agent, and fabric treated with this resin has a good handle, and thus poor handle caused by a finishing with the use of

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pure WPU can be avoided.^{13–15} This is mainly attributed to the gathering of the polysiloxane chains on the surface of the latex film that is formed by the organo-silicone modified WPU.^{16,17}

In this study, a blocking polyether silicone oil and a cationic WPU modified by using blocking polyether silicone oil are prepared and used as the finishing agent for cashmere knitted fabric. The influence of the structure of the WPU and the effect of the amount of finishing agent on the finishing properties are investigated.

Experimental details

Materials

Pure cashmere knitted fabric, provided by Ningxia Zhongyin Cashmere Co., Ltd (China), was used in the investigation. Polytetramethylene glycol (PTMG; Mn=2000 g/mol, industrial grade) was obtained from Jininghuakai Resin Co., Ltd (China). Isophorone diisocyanate (IPDI, industrial grade) was obtained from Bayer (Germany). *N*-methyl diethanolamine (MDEA, industrial grade) was obtained from Guangzhoufuchuang Chemical Co., Ltd (China). Amino-terminated polyether and epoxide-terminated polyether silicones (industrial grade) were both obtained from Dow Corning Silicones (China). Dibutyltin dilaurate (DBTDL, AR grade) and

1,4-butanediol (BDO, AR grade) were obtained from the Chengdukelon Chemical Reagent Factory.

Preparation of blocking polyether silicone oil

In the preparation of the blocking polyether silicone oil,^{18,19} epoxide-terminated polyether (A, Mn=900 g/mol) and amino-terminated polyether (B, Mn=10,500 g/mol) silicones, and isopropyl alcohol, were added into a three-neck flask (250 ml) equipped with a stirring apparatus, thermometer, and condenser at room temperature. The molar ratio between A and B was 1.1:1.0. The mixture was allowed to react for 8 h at 80°C and heated by using an infrared lamp. After cooling the system to a normal temperature, 70 wt% amino-terminated blocking polyether silicone oil (ammonia value 0.15, $\eta=700$ mpa·s) was obtained. Moreover, by-products, such as epoxide-terminated and α -amino- ω -epoxy blocking polyether silicone oils, may be found. The reaction structure of the blocking polyether silicone oil is shown in Figure 1.

Preparation of WPU

Dehydrated PTMG, the catalyst DBTDL and IPDI were added into a dried three-neck flask (500 ml) equipped with a stirring apparatus, thermometer, and condenser to react for 1 h under a temperature of 78–80°C. After that, BDO was added as a chain

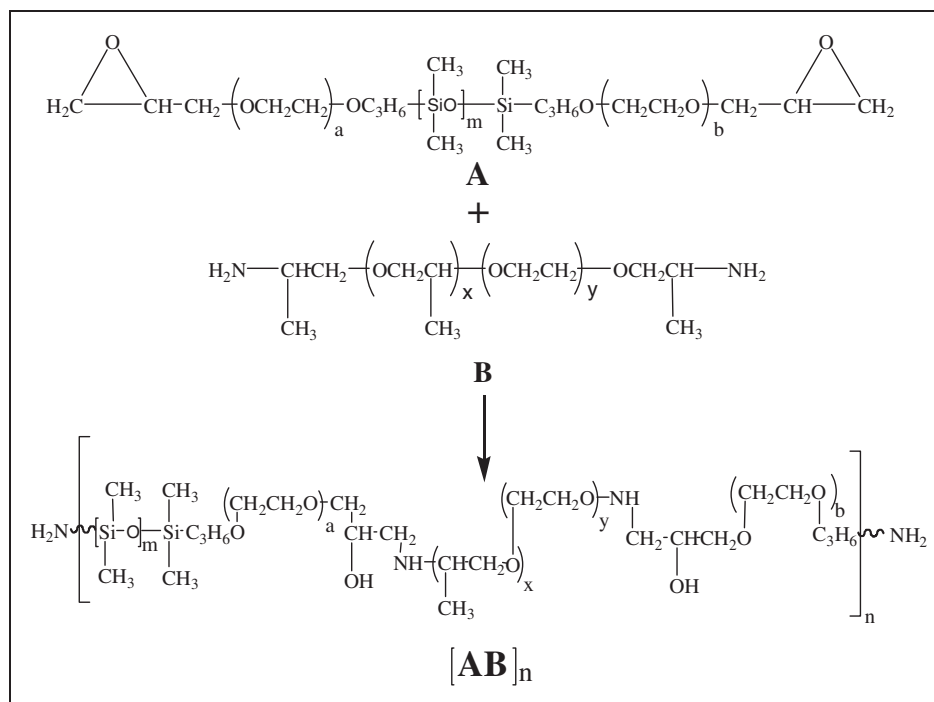


Figure 1. Synthesis scheme of blocking polyether silicone oil.

extender to react for 1 h at the same temperature, and then MDEA was added as a cationic hydrophilic chain extender to react for 1 h to obtain the WPU prepolymer after the whole system was cooled down to 50–55°C. Under high-speed stirring, a mixture of blocking polyether silicone oil and isopropyl alcohol (weight ratio of 1:3) was added for the grafting of the WPU prepolymer, and allowed to continue to react for 1 h under a temperature of 50–55°C. A WPU polymer emulsion (35 wt%), modified by the blocking polyether silicone oil, was obtained after neutralizing the system with a certain amount of acetic acid, which was emulsified for 30 min with deionized water under vigorous stirring.

In this research work, the appropriate combination of raw materials blended in the WPU was determined to be as follows: 39 wt% IPDI, 20.5 wt% PTMG, 0.08 wt% DBTDL, 8.0 wt% MDEA, 2.5 wt% BDO and 20 wt% blocking polyether silicone oil (AB) with an *R*-value ($-\text{NCO}/-\text{OH}$) of 1.6. The synthesis scheme is shown in Figure 2.

Finishing method on cashmere fabric

Cashmere fabric was treated at 40°C for 30 min in a finishing bath that consisted of water, acetic acid, and a finishing agent. The pH value of the bath solution was

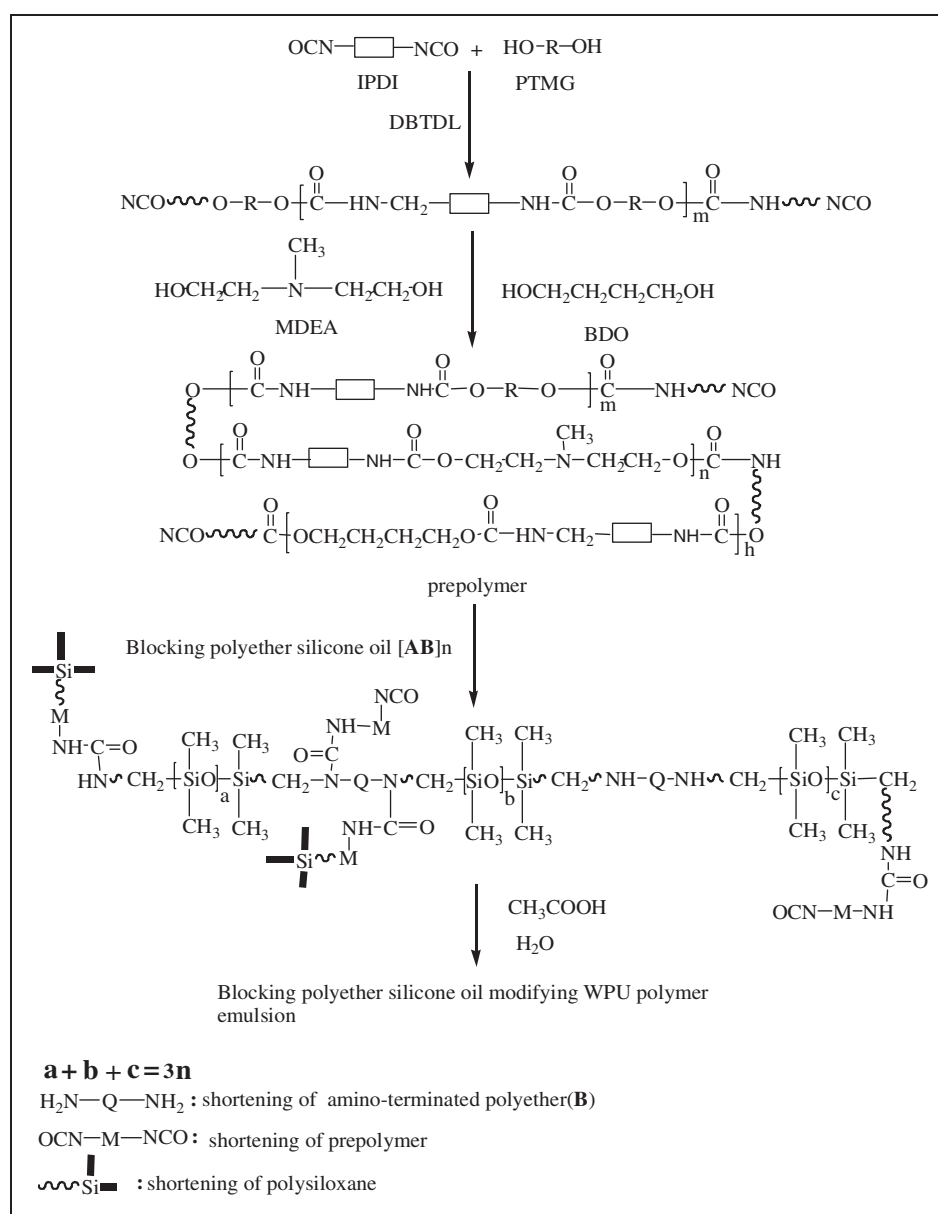


Figure 2. Synthesis scheme of waterborne polyurethane.

adjusted to 5–6 by using the acetic acid and the bath ratio was set at 20:1. After the finishing process was carried out, the cashmere fabric was dried at 80°C in an oven after it was dried by using a clothes dehydrator. The fabric was then ironed and pressed at a temperature of 150°C for 1 min.

Testing and characterization

Infrared spectroscopic analysis. Fourier transform infrared (FT-IR) spectrometry was used to produce the FT-IR spectrums of the films, which were produced by using a Nicolet 5700 FT-IR spectrometer (Nicolet Instrument Inc., USA) in the transmission mode. The films were evenly grinded with potassium bromide (KBr) for the testing.

Surface elementary analysis. The surface elementary analysis of the fabrics was carried out by using x-ray photoelectron spectroscopy (XPS). The fabric specimens that were treated with WPU before and after washing were analyzed with an XSAM800 multi-function surface analysis system (Kratos Analytical Ltd, UK) under the Al target.

Scanning electron microscopy. Scanning electronic microscopy (SEM) of the cashmere knitted fabric was carried out with an X-650 microscope (model JSM-5600LV), and operated at an acceleration voltage of 10 kV. The specimens were sputter coated with gold prior to observation. The cashmere knitted fabric specimens were inspected at a magnification of 5000× to detect the surface morphological changes.

Color difference analysis. By taking the original sample as the control, the color differences of the specimens were tested by using a color difference measuring instrument called Color i7, which was obtained from X-Rite (USA).

Washing treatment

The washing treatment of the cashmere fabric was carried out in accordance with American Association of Textile Chemists and Colorists (AATCC) Test Method 135-2012 – Dimensional Changes of Fabric after Home Laundering.

Fabric evaluation

Resistance to pilling. The resistance to pilling of the cashmere knitted fabric was tested and evaluated in accordance with American Society for Testing and Materials (ASTM) standard D 3512-2005.

Handle test. The handle was evaluated in accordance with AATCC Evaluation Procedure 5-2007 – Fabric Hand: Guidelines for the Subjective Evaluation of. The specimens were ranked through comparative assessments by using the following scale: 5 – limp; 4 – least rough; 3 – moderately rough; 2 – most rough; and 1 – stiff.

Wetting time. The wetting time was assessed in accordance with AATCC 79-2000.

Water vapor and air permeabilities. The water vapor and air permeabilities are the main indicators for evaluating the wear comfortable of the cashmere knitted fabric. Thus, these two types of permeabilities were investigated. Water vapor permeability was assessed in accordance with ASTM E96-2005. Air permeability was determined in accordance with ASTM 737-2004.

Results and discussion

Infrared spectroscopic analysis

In the infrared spectrum of the amino-terminated polyether silicone (Figure 3(a)), the absorption peaks around 3550–3350 cm^{-1} represent the $-\text{NH}_2$ stretching vibration peak, and those around 1645–1590 cm^{-1} represent the $-\text{NH}_2$ deformation vibration peak. The absorption peaks that occurred at 2880, 1463, and 1354 cm^{-1} represent the $-\text{CH}$ vibration and twisting and wagging peaks in the repeat segment of the

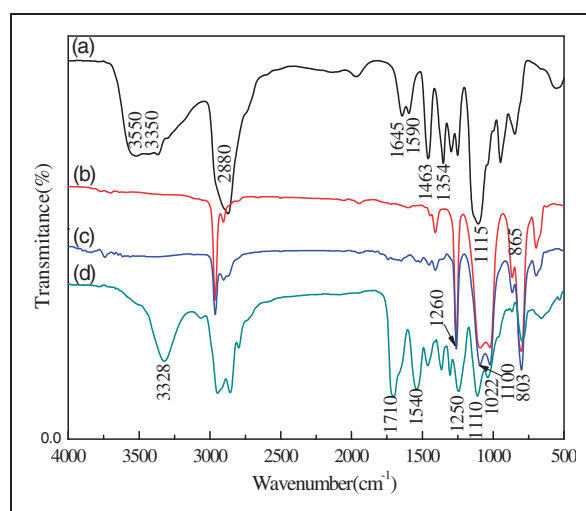


Figure 3. Infrared spectrums of the raw materials and water-borne polyurethane (WPU): (a) amino-terminated polyether silicone; (b) epoxide-terminated polyether silicone; (c) blocking polyether silicone oil; (d) WPU.

amino-terminated polyether, respectively. Furthermore, the absorption peaks at 1115 cm^{-1} belong to the -C-O-C- stretching and bending vibration peaks.

In the infrared spectrum of the epoxide-terminated polyether silicone (Figure 3(b)), the absorption peaks around $1100\text{--}1022\text{ cm}^{-1}$ represent the Si-O-Si stretching vibration peak. The absorption peaks at 1260, 865, and 803 cm^{-1} belong to the absorption peaks of the Si-C in Si-CH₃. In this study, the amount of the epoxy group in the epoxide-terminated polyether silicone is only 0.8%, thus the absorption peak of the epoxy group does not occur at 900 cm^{-1} in the spectrum.

In the infrared spectrum of the blocking polyether silicone oil (Figure 3(c)), the location of the group of characteristic absorption peaks of the blocking polyether silicone oil agrees with that of the epoxide-terminated polyether silicone except that in the latter, a stronger absorption peak at 2880 cm^{-1} was observed. The absorption peaks of -NH, -NH₂, and -OH did not occur in the infrared spectrum, as the total content of these groups in the blocking polyether silicone oil was only 0.6%.

In the infrared spectrum of the WPU (Figure 3(d)), the absorption peaks around 3328 cm^{-1} represent the N-H stretching vibration peaks of carbamido and those around 1540 cm^{-1} represent the N-H deformation vibration peaks of carbamido. The absorption peaks around 1710 cm^{-1} of the WPU are characteristic of the stretching vibration peaks of C=O. The peaks mentioned above indicate that carbamate formed in the polymers. The peaks that occurred at 1250 and 803 cm^{-1} belong to the Si-C absorption peaks in the WPU, which shows that the WPU has the feature structures of polyurethane and organic silicon.

Influence of WPU structure on fabric finishing properties

MDEA content. The amount of MDEA in the WPU has a crucial impact on the finishing effect of the cashmere knitted fabric, such as the affinity between the WPU and cashmere fibers, absorption ability of the WPU, and properties of the WPU films wrapped on the fiber surface. In this test, the amount of MDEA in the WPU was set as a variable parameter, the sum of the number of moles of the MDEA and PTMG was not changed, and other parameters were maintained to agree with the raw material composition mentioned above. The cashmere knitted fabric was treated with 2% (owf) WPU and the results are shown in Figure 4.

In comparison with the untreated fabric, the water vapor and air permeabilities (Figure 4(a)) of the treated fabric were slightly reduced and the washing shrinkage, wettability (Figure 4(b)), and resistance to pilling (Figure 4(c)) were obviously improved, while the handle (Figure 4(c)) of the treated fabric was almost unchanged. These are mainly a result of the film formed by the WPU on the fiber surface. As for the increase in the amount of MDEA in the WPU, the hydrophilicity and density of the WPU cationic, affinity, and gravity charge between the WPU and cashmere fibers, absorption quantity of the WPU on the cashmere fibers, and the toughness and integrity of the WPU film on the fiber surface all increased. When the WPU film wrapped onto the surface of the fibers, the effect mentioned above is a result of the increase in the amount of MDEA in the WPU, which led to an improvement in the water vapor and air permeabilities, and resistance to pilling. However, the amount of MDEA has little effect on the softness of

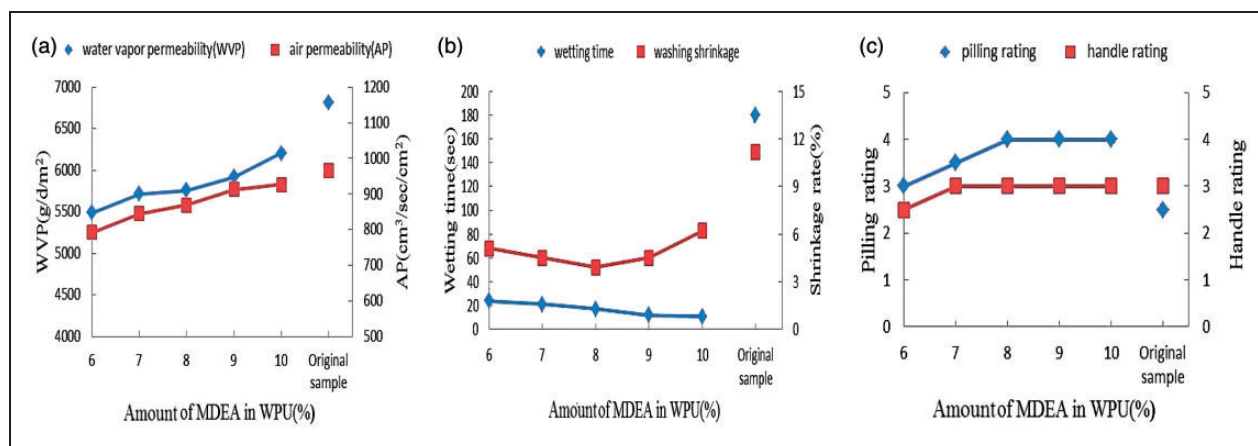


Figure 4. Influence of amount of *N*-methyl diethanolamine (MDEA) in waterborne polyurethane (WPU) on finishing properties. WVP: water vapor permeability; AP: air permeability; Original sample: untreated cashmere knitted fabric.

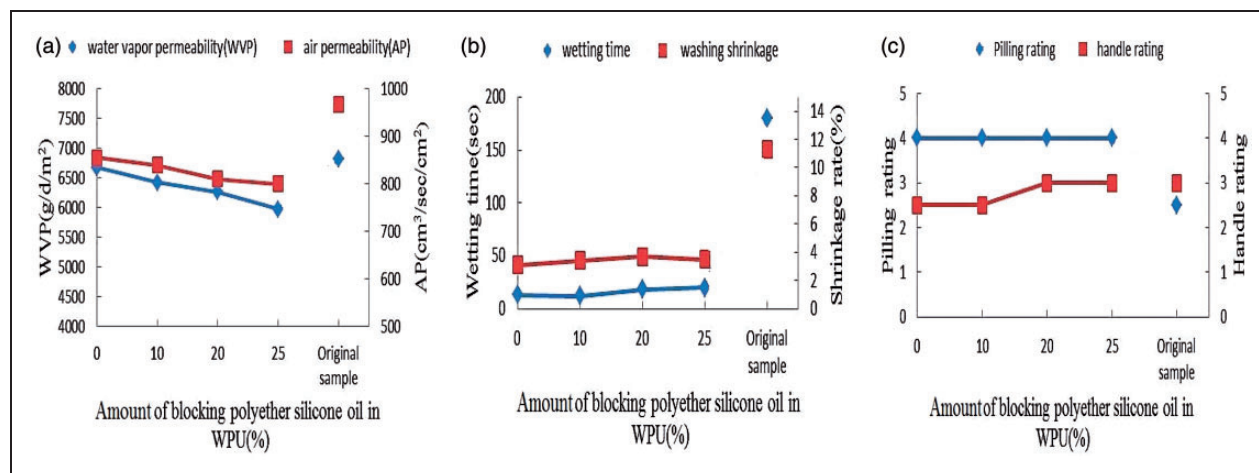


Figure 5. Influence of the amount of blocking polyether silicone oil in the waterborne polyurethane (WPU) on finishing properties of cashmere knitted fabric.

the WPU latex film, and thus the handle of the treated fabric was almost the same. Resistance to washing shrinkage was first increased and then reduced with an increase in the amount of MDEA, because when there is too much MDEA, the WPU film formed onto the fiber surface has strong hydrophilicity and easily dissolves during washing. In conclusion, it is more appropriate that there is 8.0 wt% of MDEA in the WPU.

Blocking polyether silicone oil content. The main purpose for introducing the blocking polyether silicone oil into the WPU was to improve the softness of the treated cashmere knitted fabric. In the testing, the amount of blocking polyether silicone oil in the WPU was taken as a variable parameter, and the other parameters were those of the raw material composition mentioned above. The cashmere knitted fabric was treated with 2% (owf) WPU and the influence of the amount of blocking polyether silicone oil in the WPU on the finishing properties of the cashmere knitted fabric are shown in Figure 5.

With an increase in the amount of the blocking polyether silicone oil in the WPU, the water vapor and air permeabilities (Figure 5(a)) and wettability (Figure 5(b)) of the treated fabric were slightly reduced and resistance to washing shrinkage (Figure 5(b)) and resistance to pilling (Figure 5(c)) remain almost the same, while the handle of the treated fabric was slightly improved (Figure 5(c)). Since the blocking polyether silicone oil was grafted into the WPU, polysiloxane chains gather together on the WPU film surface when forming the film because of the phase separation caused by the thermodynamic incompatibility between polysiloxane and polyurethane.^{20,21} Therefore, the handle of the treated fabric can be improved with the gathering of the polysiloxane chains in the blocking polyether

silicone oil on the surface of the WPU film. With an increased amount of blocking polyether silicone oil in the WPU, the effect tends to be even more obvious.^{15–17} The polyether segment, a basic feature structure of the blocking polyether silicone oil, has a certain hydrophilicity; thus, the introduction of the blocking polyether silicone oil into the WPU makes little difference to the wettability, as well as the water vapor and air permeabilities of the treated fabric. Moreover, the properties of the resistance to washing of the treated fabric can be improved with the introduction of the silicone oil.^{13,22} In terms of the amount of blocking polyether silicone oil in the WPU, 20 wt% is the optimal amount for obtaining the best effect for the finishing of the cashmere knitted fabric.

Influence of amount of WPU on finishing effect

The cashmere knitted fabric was treated with different amounts of WPU and the results are shown in Figure 6.

The water vapor and air permeabilities (Figure 6(a)) of the treated fabric decreased, while the handle (Figure 6(c)) was negatively affected. The washing shrinkage, wettability (Figure 6(b)), and resistance to pilling (Figure 6(c)) of the treated fabric first improved and then remained the same with an increase in the amount of WPU. Also, with an increase in the amount of WPU, the toughness and integrity of the film formed by the WPU on the surface of the treated fiber, yarn, and fabric improved, thus resulting in a reduction of the directional friction effect and less fiber movement. Therefore, resistance to pilling and washing shrinkage of the treated fabric are improved while water vapor and air permeabilities are slightly decreased. When the WPU was modified by including blocking polyether silicone oil and then applied to the

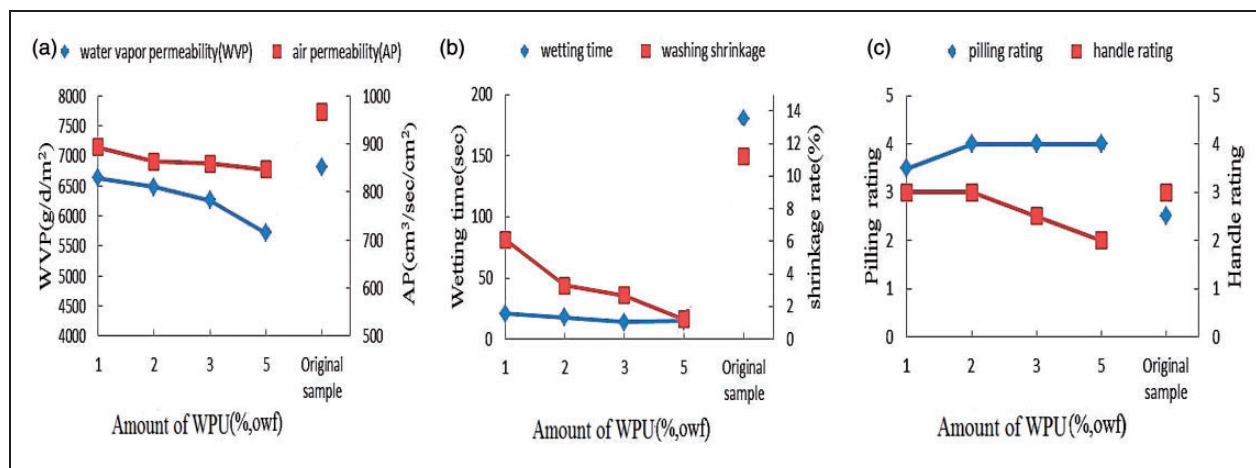


Figure 6. Influence of amount of waterborne polyurethane (WPU) on finishing properties.

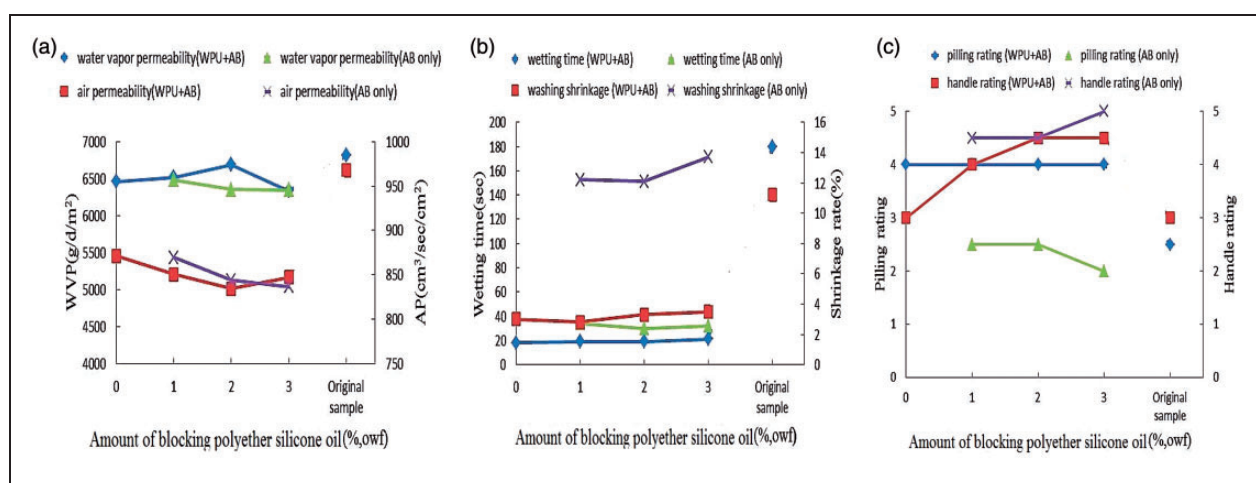


Figure 7. Influence of amount of blocking polyether silicone oil on finishing properties.

fabric finishing, the handle of the treated fabric was improved to a certain extent, but if the amount of WPU was too much, the rigidity of the film formed by the WPU on the fiber surface increased and the treated fabric was stiff, and thus the handle of the treated fabric was negatively affected. Finally, a concentration of 2% (owf) WPU was found to be optimal for all three properties: resistance to washing shrinkage and pilling, and the softness handle of the treated cashmere knitted fabric.

Influence of the amount of blocking polyether silicone oil on the finishing effect

The use of a softness finishing agent to treat cashmere fabric and improve its handle is one of the important steps in the finishing of cashmere knitted fabric. In this test, two different treatment methods are applied. One

method is where the cashmere knitted fabric was treated with blocking polyether silicone oil only (denoted as AB only) and the other method is where the cashmere knitted fabric is treated with both blocking polyether silicone oil and WPU (WPU 2% (owf) + AB). The influence of the treatment method on the finishing effect of the cashmere knitted fabric is shown in Figure 7.

Blocking polyether silicone oil could provide the treated cashmere fabric with good wettability (Figure 7(b)) and softness (Figure 7(c)), but cannot improve its resistance to washing shrinkage (Figure 7(b)) and pilling (Figure 7(c)). When the cashmere fabric was treated with only blocking polyether silicone oil, and the amount of silicone oil was increased, the fibers in the treated fabric tended to slip easily under external force, thus resulting in a slight decrease in resistance to washing shrinkage

and pilling. In conclusion, the optimal concentration of blocking polyether silicone oil is 2% (owf). The application of blocking polyether silicone oil in WPU obviously improves the softness of the treated fabric, and does not affect resistance to washing shrinkage and pilling, wettability, and the water vapor and air permeabilities (Figure 7(a)) of the treated cashmere knitted fabric. It has been reported and proven in practice that the handle of cashmere fabric will not be satisfactory even though the fabric is treated with a large quantity of softness finishing agents after treatment when there are significant amount of shrinkage resistance and pilling resistance agents.^{23–25} When the concentration of the WPU and that of the blocking polyether silicone oil were both 2% (owf), the treated cashmere fabric obtained good softness properties with a similar handle to fabric that was treated with only 2% (owf) blocking polyether silicone oil for the softness finishing. In terms of the cashmere knitted fabric that was treated with both WPU and the blocking polyether silicone oil, the resistance to pilling improved from 2–3 to 4, the washing shrinkage rate was reduced from 11.2% to 3.3%, and the hydrophilicity and water vapor and air permeabilities were excellent.

Scanning electron microscopy

The cashmere knitted fabric was treated with both blocking polyether silicone oil and WPU (AB 2% (owf) + WPU 2% (owf)) and the SEM (magnification up to 5000 \times) results are shown in Figure 8.

The scales and the clearance of the untreated cashmere fibers can be clearly seen (Figure 8(a)). After treating the fabric samples with both blocking polyether silicone oil and WPU that had been modified with the addition of blocking polyether silicone oil, a smooth

film was wrapped around the fiber surface and fiber clearance was blocked (Figure 8(b)). The surface structure changed as the blocking polyether silicone oil and WPU films formed on the fiber surface, which led to increases in dimensional stability and smoothness and a better handle. The smooth fiber surface could reduce the tendency of the fabric to pill and shrink, but the water vapor and air permeabilities were somewhat decreased due to the existence of the films.

Influence of treatment agent on fabric color

The cashmere knitted fabric was treated with the same concentration of both WPU and blocking polyether silicone oil – 2% (owf). The original sample was taken as the control, and the color difference of the treated fabrics was then tested and is shown in Table 1.

It can be observed in Table 1 that for the cashmere knitted fabric treated with WPU and blocking polyether silicone oil, there is only a slight change in the color shade.

Durability of the finishing effect

The primary factor that negatively affected the finishing properties of the WPU and blocking polyether silicone oil treated fabric was washing. The cashmere knitted fabric that was treated with the same concentration of both WPU and blocking polyether silicone oil (2% (owf)) was washed and its finishing durability was investigated through a surface elementary analysis. The results are shown in Figures 9 and 10.

With an increase in the number of washings, the water vapor and air permeabilities (Figure 9(a)) and the handle (Figure 9(c)) of the treated fabric slightly improved. The resistance to washing shrinkage

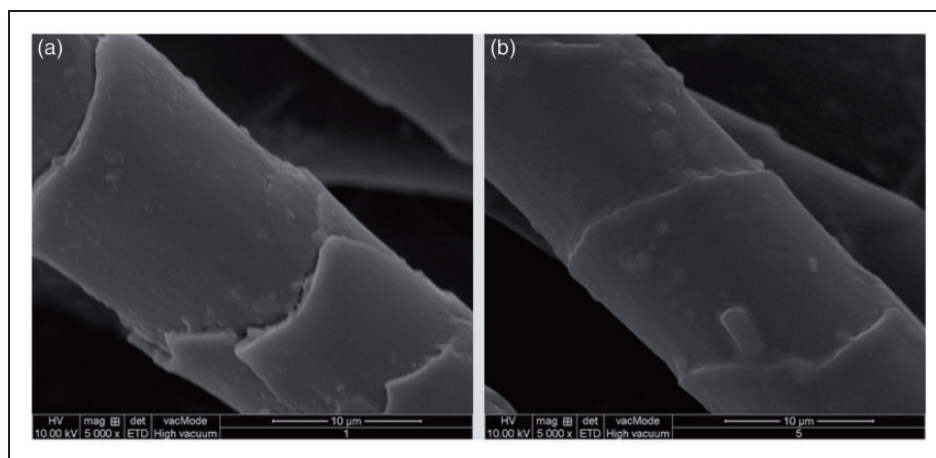
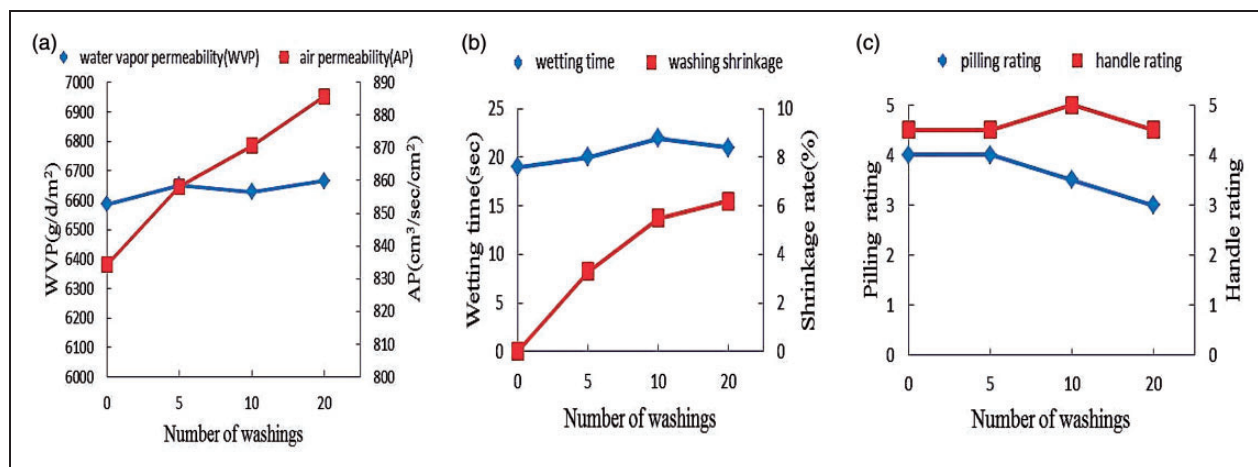


Figure 8. Scanning electron microscopy photos of treated and untreated cashmere knitted fabrics: (a) original sample (5000 \times); (b) cashmere knitted fabric treated with blocking polyether silicone oil and waterborne polyurethane (5000 \times).

Table 1. Influence of treatment agent on fabric color

Hue	Lightness difference ΔL	Coloration difference ΔC	Hue difference ΔH	Total color Difference ΔE
Light brown	-0.09D	0.20B	0.09 R	0.28
Bright red	-0.15D	0.28B	0.10 Y	0.20

**Figure 9.** Influence of number of washings on finishing properties.

(Figure 9(b)) increased, and resistance to pilling (Figure 9(c)) decreased, but the wettability (Figure 9(c)) of the treated fabric remained almost unchanged. The washing was conducted in neutral conditions. The adhesive films formed by the WPU on the surface of the cashmere fibers could not be easily washed off as there is a relatively strong gravity charge, hydrogen bonding, and van der Waals force between the cationic WPU and fibers, thus leading to relatively strong adsorbability and even adhesive force between the WPU and fibers. Therefore, the resistance to shrinkage and pilling and the washing durability of the treated fabric were excellent. With an increase in the number of washings, the amount of WPU and blocking polyether silicone oil on the fiber surface was inevitably reduced, and the blocking polyether silicone oil was gradually washed off. As the amount of blocking polyether silicone oil on the fiber surface was reduced after washing, the fabric became fluffy due to the washing effect, and thus the handle of the washed fabric slightly improved.

It was found that there are Si(2s) and Si(2p) characteristic peaks in the XPS spectrums of the cashmere fabric treated with WPU before and after washing (Figure 10(a)). However, the absorption intensity of the Si(2s) and Si(2p) characteristic peaks in the spectrum of the WPU treated fabric that was washed 10 times was reduced in intensity as opposed to those of the treated fabric that was not washed (Figure 10(b)).

According to the area of the fitted peak, the Si content of the unwashed treated fabric was 8.01%, while that of the treated fabric that was washed 10 times was 5.12%; therefore, the resistance to washing of the cashmere fabric treated with WPU is excellent, and this is in accordance with the results shown in Figure 9.

Conclusions

1. Blocking polyether silicone oil is obtained through a reaction between amino-terminated polyether and epoxide-terminated polyether silicones, and used as a soft finishing agent for cashmere knitted fabric. WPU can be used as a finishing agent when PTMG is used as the soft segment, and blocking polyether silicone oil as the modifying agent and chain extender, and then used as a resistance to pilling and shrinkage finishing agent. The proportion of the cationic unit (MDEA) and blocking polyether silicone oil in the WPU has an important influence on the handle, resistance to pilling, and felting shrinkage of the treated fabric. Optimal finishing properties when the WPU is used as a finishing agent can be obtained when there is 8.0 wt% of the cationic monomer (MDEA) and 20 wt% of the blocking polyether silicone oil.
2. Cashmere knitted fabric has been treated with both a cationic WPU and blocking polyether silicone oil in

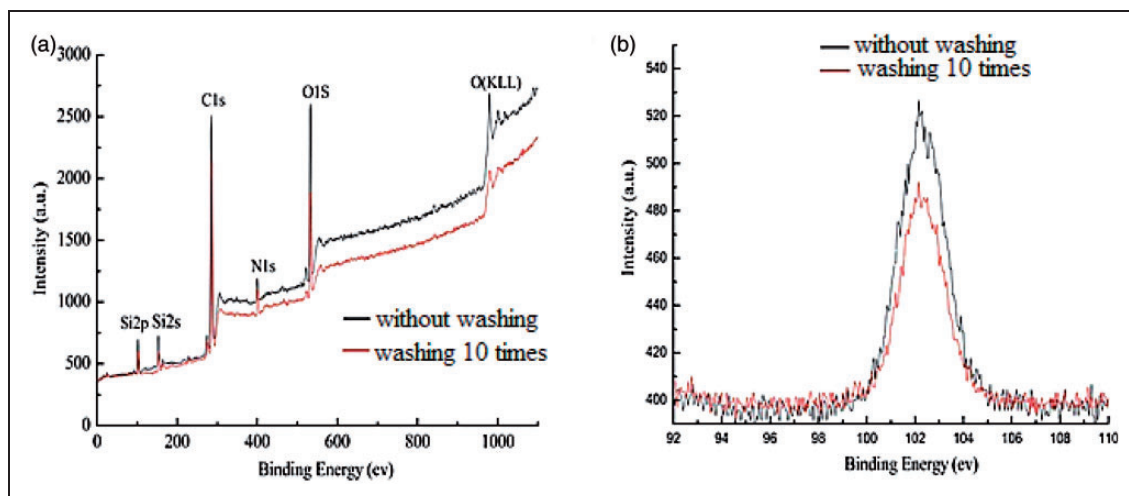


Figure 10. X-ray photoelectron spectroscopy (XPS) spectrums of surface elements of cashmere knitted fabric treated with both waterborne polyurethane (WPU) and blocking polyether silicone oil: (a) XPS spectrum of surface elements of cashmere knitted fabric treated with both WPU and blocking polyether silicone oil without washing, and WPU and blocking polyether silicone oil treated fabric after washing 10 times; (b) Si spectrum of cashmere knitted fabric treated with both WPU and blocking polyether silicone oil without washing, and WPU and blocking polyether silicone oil treated fabric after washing 10 times.

a bath, and a tough film with integrity is observed to be wrapped around the fiber surface formed by the WPU and blocking polyether silicone oil. This improves the resistance to pilling from 2–3 to 4 and reduces the washing shrinkage rate from 11.2% to 3.3%. An ideal amount of resistance to pilling and shrinkage of the treated fabric is obtained. The handle is good, the color shade remains almost unchanged, and hydrophilicity and water vapor and air permeabilities are excellent. Overall, the fabric has an ideal durability in terms of the finishing properties.

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