Application Performance and Surface Morphologies of Amino Polysiloxanes with Different Amino Values and Amino Types

Yingjun Xu,[1,2] Hong Yin,[1] Haifeng Zheng,[1] Shenfeng Yuan,[1] Zhirong Chen[1]

[1]*Department of Chemical Engineering, Zhejiang University, Hangzhou 310027, People's Republic of China*
[2]*School of Chemical and Environmental Engineering, Shanghai Institute of Technology, Shanghai 210032, People's Republic of China*

Received 12 October 2009; accepted 3 June 2010
DOI 10.1002/app.32928
Published online 30 August 2010 in Wiley Online Library (wileyonlinelibrary.com).

ABSTRACT: Amino polysiloxanes (APSs) with different amino values and amino types were synthesized and applied to cotton fabrics. Softening and smoothening properties of the fabrics treated with APSs were investigated and evaluated by measuring wrinkle recovery angles and friction coefficients, and the morphological features of the APSs adsorbed onto cellulose substrate films were characterized by atomic force microscope (AFM). The results indicate that the amino values and amino types of the APSs have a significant impact on the softening and smoothening properties of the fabrics. APSs with relatively high amino values exhibit superior smoothening property, while APSs with moderate amino values exhibit excellent softening property. Compared to the traditional softener *N*-β-aminoethyl-γ-aminopropyl polydimethylsiloxane (APS-1), the new amino type softeners γ-piperazinylpropyl polydimethylsiloxane (APS-2) and *N*-γ′-dimethylaminopropyl-γ-aminopropyl polydimethylsiloxane (APS-3) gave better fabric performance, whereas aminopropyl polydimethylsiloxane (APS-4) and *N*-cyclohexyl-γ-aminopropyl polydimethylsiloxane (APS-5) gave unsatisfactory fabric performance. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 119: 2326–2333, 2011

Key words: amino polysiloxane; fabric performance; surface morphology; amino value; amino type

INTRODUCTION

Siloxanes are special materials that impart desired mechanical performance properties of fabrics such as softness, bounciness, and antiwrinkle properties to fabrics and related materials.[1–3] Reactive siloxanes and modified functional siloxanes have been used to enhance interaction between siloxanes and textile materials and fabric durability during the washing process.[4,5] In practice, amino polysiloxanes (APS's) exhibit superior softening and mechanical properties, and are widely used in the textile industry as fabric softeners.[6–8]

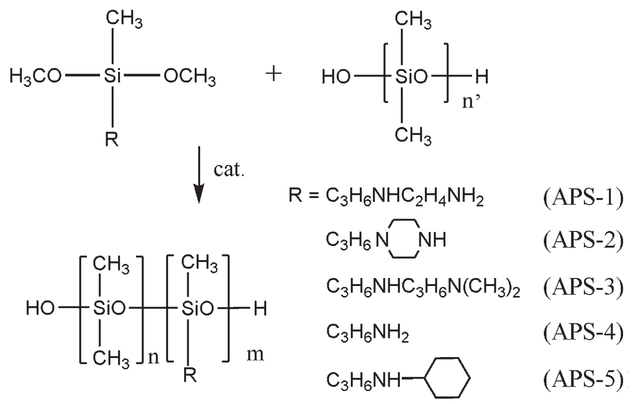
Amino values and amino types of the amino polysiloxanes have a significant impact on fabric performance. In preliminary studies, the authors used a series of advanced instruments to study the film morphology and orientation of APS on cellulose substrate, and proposed that the orientation of APS molecule was with the amino functional groups adsorbed onto the cellulose interface while the main polymer chains and the hydrophobic Si—CH_3 groups extend toward the air.[9] Film morphology and orientation can influence the tactile and other performances of polysiloxane softeners. The average length of $[(CH_3)_2Si—O]_n$ segment between two adjacent amino-hydrocarbons becomes shorter with an increase in the amino value, which leads to a smoother surface and perhaps superior softening benefit. For different amino types, the number of amino groups and different N-substituent groups can also affect the fabric handle.[10–13] However, no study has attempted to correlate the macroscopic fabric performance with microscopic film morphology, compare APSs with different amino values and amino types and explain the softening and smoothening mechanism at the molecular level. Therefore, it has important theoretical significance and practical value to study the surface morphologies, structure and fabric performance of amino polysiloxanes. To this end a series of APSs with various amino values and amino types were synthesized and applied to cotton fabrics. The fabric performance including the softening and smoothening properties were investigated and evaluated by measuring wrinkle recovery angles and friction coefficients of the fabrics treated with APSs, and the morphological features of the APSs adsorbed onto cellulose substrate films were characterized by AFM.

EXPERIMENTAL

Materials

Amino silane coupling agents, *N*-β-aminoethyl-γ-aminopropyl methyldimethoxysilane, γ-piperazinylpropyl methyldimethoxysilane, *N*-γ′-dimethylamino-

Correspondence to: H. Yin (yinh@zju.edu.cn).



Scheme 1 Synthesis route to APSs.

propyl-γ-aminopropyl methyldimethoxysilane, aminopropyl methyldimethoxysilane and *N*-cyclohexyl-γ-aminopropyl methyldimethoxysilane, all industrial grade, were purchased from Hangzhou Dadi Chemical α,ω-dihydroxypolydimethylsiloxane ($n' = 45$), industrial grade, was purchased from Dow Coming Acetic acid, isopropanol, toluene and hydrochloric acid, all A.R., were from Hangzhou Chemicals. Fatty alcohol polyoxyethylene ethers (AEO-3 and AEO-9), industrial grade, was purchased from Shangyu Huangma Chemical. The alkaline catalyst is an ethanol solution of sodium ethoxide (mass fraction 5%), prepared in our laboratory. Desized, scoured and bleached cotton cloth (84 × 64), weighing 119 g/m^2, was obtained from Hangzhou Xinfu Textile. All the chemicals and materials were used as received.

Synthesis

To a four-necked flask equipped with a nitrogen purge, a vacuum device, a stirrer and a thermometer, were added in the following order 90 g (0.027 mol) of α, ω dihydroxypolydimethylsiloxane, a proper amount of amino silane coupling agents corresponding with the desired amino value and 3.6 mL of the alkaline catalyst. The reaction mixture was slowly heated to 90°C and kept at this temperature for 5 h. At the end of the reaction, a proper amount of acetic acid was added to neutralize the sodium ethoxide and terminate the polymerization reaction. The alcohols and water were removed by vacuum distillation. A glutinous, colorless and transparent fluid, amino polysiloxane (shown in Scheme 1), was obtained with a yield of 98% (confirmed by ^{1}H-NMR measurements[14]).

Characterization

The amino values were estimated by chemical titration, of which the details are available elsewhere.[15] The average molecular weights were determined by measuring the intrinsic viscosity [η].[16] ^{1}H-NMR spectra were recorded at 25°C on an Avance DMX 500 with $CDCl_3$ as the solvent and tetramethylsilane (TMS, $\delta = 0$) as the internal standard.

Treatment of the fabrics

Amino polysiloxane softeners are usually used in textile industry in emulsions. In accordance with this, 7.5% APS emulsions (6 g APS emulsified in 4 g fatty alcohol polyoxyethylene ether and 70 g H_2O) were used as samples in the experiment to finish the fabrics. The fabrics were impregnated in a diluted aqueous bath (bath ratio, 7.5% amino silicone emulsion: $H_2O = 2 : 3$) containing a circa 3% APS emulsion sample, padded to wet pick-up at about 70% on the weight of the dry fabrics. The padded fabrics were then dried at 100°C for 10 min, cured at 120°C for 2 min and finally conditioned at 20°C and 65% relative humidity.

MEASUREMENTS

Wrinkle recovery angles

The wrinkle recovery angles were measured according to GB/T 3819-1997 (Textile fabrics determination of the recovery from creasing of a folded specimen by measuring the angle of recovery) by YG541E fabric wrinkle recovery angle tester. The samples were compressed under 10N for 5 min. The recovery time for fast recovery angles is 15 s while for slow recovery angles is 5 min. The presented result was the sum of the recovery angles of the tested fabrics in the warp and filling (W + F) directions. The result was the average of 10 measurements.

Friction coefficients

The friction coefficients were measured with a KESFB-4 fabric style instrument. The presented result was the sum of the recovery angles of the tested fabrics in the warp and filling (W + F) directions. The result was the average of six measurements.

Observation of the film morphology by AFM

Natural fibers are fixed with difficulty in AFM observation, and the cellulose substrate allows a better morphological characterization of coating on the nanoscale than the rugged surface of fabrics. We therefore used cellulose film (cellophane) as a model surface.

The cellulose films were cleaned sequentially with water, acetone, isopropanol and hexane by ultrasound. This operation was developed to remove

TABLE I
^{1}H-NMR Peak Positions of APSs with Different Amino Types

Sample	^{1}H-NMR peak positions (ppm)
APS-1	0.05 (s, Si—CH$_3$), 0.52 (t, Si—CH$_2$), 1.54 (m, CH$_2$—CH$_2$), 2.60 (t, NH—CH$_2$), 2.66 (t, NH—CH$_2$), 2.79 (t, NH—CH$_2$)
APS-2	0.07 (s, Si—CH$_3$), 0.51 (t, Si—CH$_2$), 1.53 (m, CH$_2$—CH$_2$), 2.59 (t, N—CH$_2$), 2.65 (t, N—CH$_2$), 2.78 (t, NH—CH$_2$)
APS-3	0.07 (s, Si—CH$_3$), 0.52 (t, Si—CH$_2$), 1.54 (m, CH$_2$—CH$_2$), 1.66 (m, CH$_2$—CH$_2$), 2.22 (s, N—CH$_3$), 2.31 (t, NH—CH$_2$), 2.58 (t, N—CH$_2$), 2.63 (t, NH—CH$_2$)
APS-4	0.08 (s, Si—CH$_3$), 0.52 (t, Si—CH$_2$), 1.54 (m, CH$_2$—CH$_2$), 2.66 (t, NH—CH$_2$)
APS-5	0.07 (s, Si—CH$_3$), 0.50 (t, Si—CH$_2$), 1.04 (m, CH$_2$—CH$_2$), 1.16 (m, CH$_2$—CH$_2$), 1.22 (m, CH$_2$—CH$_2$), 1.51 (m, CH$_2$—CH$_2$), 1.61 (m, CH$_2$—CH$_2$), 1.72 (m, CH$_2$—CH$_2$), 1.86 (m, CH$_2$—CH$_2$), 2.39 (m, NH—CH), 2.59 (t, NH—CH$_2$)

surface contaminants (due to processing and handling), and minimize the wrinkling while drying.[17]

The APSs were dissolved in isopropanol (0.05 wt %) and applied to the cellulose substrates by dip coating. Then the samples were dried at 100°C for 10 min, cured at 120°C for 2 min and finally conditioned at 20°C and 65% relative humidity.

Morphological features of the cellulose substrate surfaces were characterized by AFM (Nanoscope IV a Controller, Vecco) at 25°C in air with relative humidity of 48%, and all the scanning was performed in tapping mode with Pointprobe silicon cantilevers (Nanosensors, Wetzlar, Germany). The root-mean-square-roughness (Rq) of a 2 μm^2 scanned area was calculated by the Nanoscope® III software using the following equation:

$$R_q = \sqrt{\frac{\sum Z_i^2}{n}} \quad (1)$$

where Z_i are the height deviations with reference to the mean of n data points.[18]

RESULTS AND DISCUSSION

Structure characterization

Molecular structure affects fabric performance primarily, so the chemical structures of the synthesized APSs were determined through ^{1}H-NMR spectrometers, and ^{1}H-NMR peak positions were shown in Table I.

By adding the same amount of alkaline catalyst, the average molecular weights of different APSs were in the same order of magnitude, about 38,200–56,300 (determined by measuring the intrinsic viscosity [η], and the MHS equation is $[\eta]_{(\text{toluene},25^\circ\text{C})} = 2.00 \times 10^{-4}\ M^{0.66}$). The amino values were controlled by adjusting the amount of silane coupling agents and determined by chemical titration, each kind of APS including 0.2–1.0 mmol/g three or five amino values.

FABRIC PROPERTIES

Softening property

Softening property is one of the most important evaluation indices for fabric handle, and it is assessed by measuring the wrinkle recovery angle (WRA). A large wrinkle recovery angle indicates satisfactory antiwrinkle effects and softening properties. The fast and slow recovery angles of cotton fabrics untreated and treated with APSs with different amino values and amino types were estimated and shown in Figures 1 and 2.

The fast recovery angles (FRA) and slow recovery angles (SRA) for the untreated fabrics are 129.2° and 171.3°, respectively. The fabrics treated with APSs have significantly larger wrinkle recovery angles, showing that the APSs can potentially be used as fabric softeners.

Amino values and amino types of the APSs significantly impact fabric softening properties. Amino value controls the average length of $[(CH_3)_2Si—O]_n$ segment between two adjacent amino-hydrocarbon groups, and APSs with moderate amino values exhibit superior softening property. For APSs with two amino groups on one coupling agent (APS-1, APS-2 and APS-3), the moderate amino value is 0.6 mmol/g (Fig. 1). For APSs with only one amino group on one coupling agent (APS-4 and APS-5), the moderate amino value is 0.4 mmol/g (Fig. 2).

In preliminary studies, the authors[9] have proposed that the orientation of APS molecule on cotton fabric is such that the amino functional groups interact with the -OH sites on the cellulose and are adsorbed onto the cotton fabric surface, while the main polymer chains and the hydrophobic Si—CH$_3$ groups extend toward the air. Chen[19] proposed that the fabric softening ability of APS depends mainly on the APS molecular configuration. APS has a flexible chain structure, with free rotation about the Si-O bond. The bending, rotating and sliding of the $[(CH_3)_2Si—O]_n$ segment impart desired mechanical performance properties such as softness, bounciness and antiwrinkle properties to fabrics.

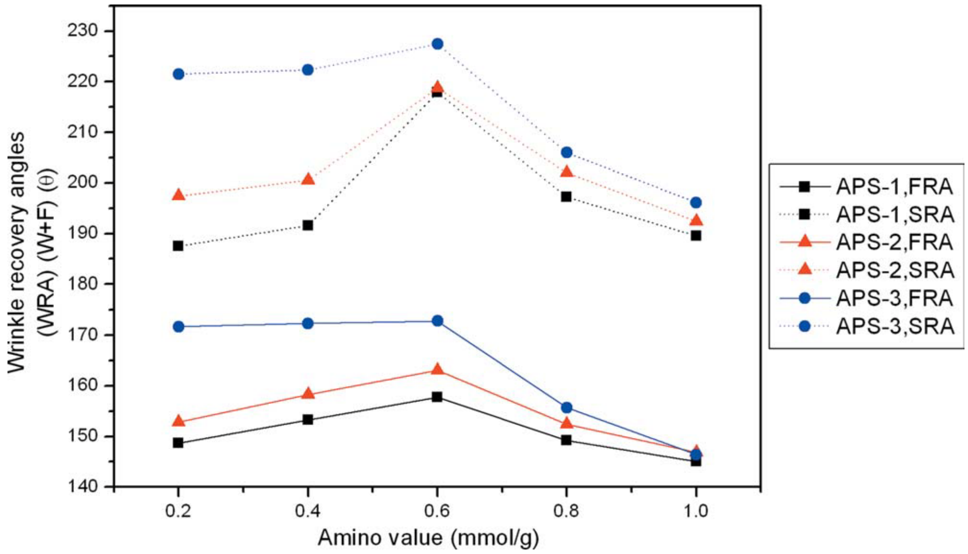


Figure 1 Wrinkle recovery angles for cotton fabrics treated with APS-1, APS-2, and APS-3 with different amino values. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Figure 3 schematically illustrates the reason why an excellent softening property needs a moderate amino value. Amino value control the average length of $[(CH_3)_2Si—O]_n$ segment between two adjacent amino-hydrocarbon groups directly, and only a $[(CH_3)_2Si—O]_n$ segment with a moderate length can bend, rotate and slide in an easy manner, and thereby soften the fabric [Fig. 3(a)]. If the amino value is excessively high, the length of the $[(CH_3)_2Si—O]_n$ segment would be too short to allow such movements, leading to inferior softening properties [Fig. 3(b)]. And if the amino value is excessively low, the length of the $[(CH_3)_2Si—O]_n$ segment would be overly long and pile up on the surface of the fabric in flocks, and the unequal distribution of the APS would also weaken its softening properties [Fig. 3(c)].

In addition, the amino value can also affect the fastness of APSs adsorbed on fabrics. With an increase in the amino value, the APSs are more firmly absorbed onto the cotton fabric and the orientation of the polymer main chains more ordered, which can lead to better softening effects.

In addition, amino types (especially the number of amino groups on one amino silane coupling agent and the nature and steric hindrance of the N-substituent groups) of the APSs also have a great impact on the fabric softening property.

The number of amino groups on one coupling agent has a significant impact. APSs with two amino

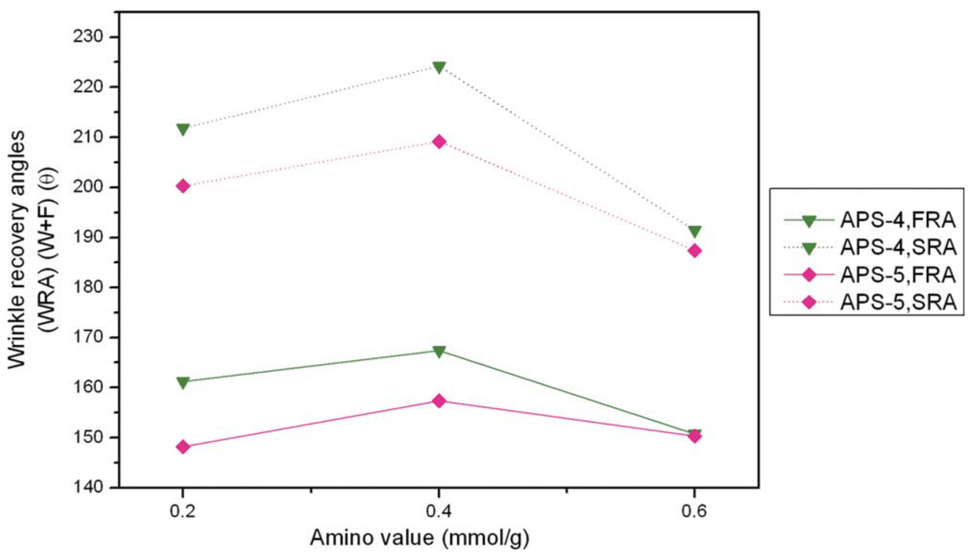


Figure 2 Wrinkle recovery angles for cotton fabrics treated with APS-4 and APS-5 with different amino values. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

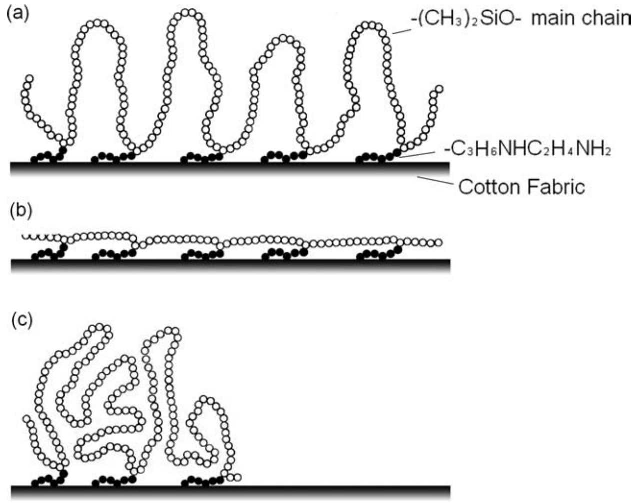


Figure 3 Proposed schematic model for the orientation of APS adsorbed on cotton fabric for (a) moderate amino value, (b) high amino value, and (c) low amino value.

groups on one coupling agent can adsorb on the cotton fabric more firmly than those with just one amino group. Better adsorption can promote an orderly orientation of the main polymer chains, leading to better softening effects. But with the same amino value (especially relatively low amino value such as 0.2 mmol/g and 0.4 mmol/g), the APSs with one amino group on one coupling agent have more equal amino distribution, leading to moderate $[(CH_3)_2Si{-}O]_n$ segment lengths and thereby better softening effects.

The substituents on N also have a great impact on the fabric softening property, mainly via the inductive effect and protonation mechanisms. As alkyl groups are electron-donating, the electron density on the nitrogen atom becomes larger with an increase in the number of alkyl groups, and the amino functional groups can form hydrogen bonds with the hydroxyl groups on cotton fabric more easily. Protonation rates of primary, secondary and tertiary amino groups are also different owing to this same inductive effect. Tertiary amino groups get more easily protonated, carry positive charges, and preferentially adsorb onto the negatively charged cotton fabric surface. Therefore, the order of the ability of amino groups to adsorb onto cotton fabrics is, from strongest to weakest: tertiary amino, secondary amino, primary amino. The more ordered the orientation of the $(CH_3)_2Si{-}O$ main chain, the better the softening effect. Thus according to the corresponding references and experimental data,[20,21] the order of fabric handle imparted by APSs with different amino types, from most to least satisfactory, is: tertiary amino, secondary amino, primary amino. As shown in Figure 1, the wrinkle recovery angles of fabric treated with APS-2 and APS-3 are slightly larger than the ones treated with APS-1, which was owing to the secondary amino and tertiary amino on γ-piperazinylpropyl group and *N*-γ′-dimethylaminopropyl-γ-aminopropyl group.

The steric hindrance of substituent groups also affects the fabric handle. For APS-5, the amino group cannot adsorb onto cotton fabric easily because of the steric hindrance of cyclohexyl, therefore the softening effect decreases. In addition, the hydrophobic cyclohexyl extend toward the air, which has an effect on the bending and sliding of the $[(CH_3)_2\text{-}Si{-}O]_n$ segment, and lead to a coarse fabric handle. As shown in Figure 2, the wrinkle recovery angles of fabric treated with APS-5 are significantly smaller than the ones treated with APS-4, which was due to the *N*-cyclohexyl-γ-aminopropyl that greatly increased the steric hindrance.

Smoothening property

Smoothening property is another important evaluation indexes for fabric handle, and it is assessed by measuring the friction coefficient. A small friction coefficient indicates satisfactory smoothening property. The friction coefficients of cotton fabrics untreated and treated with APSs with different amino values and amino types were estimated and shown in Figure 4.

The friction coefficient for the untreated fabrics is 0.276. The fabrics treated with APSs have significantly small friction coefficients, showing that the APSs exhibit excellent smoothening property.

With the same amino value, the APSs with one amino group on one coupling agent have a relatively short $[(CH_3)_2Si{-}O]_n$ segment, so the smoothening property brought by the sliding of $[(CH_3)_2Si{-}O]_n$ segment is reduced, and the friction coefficients

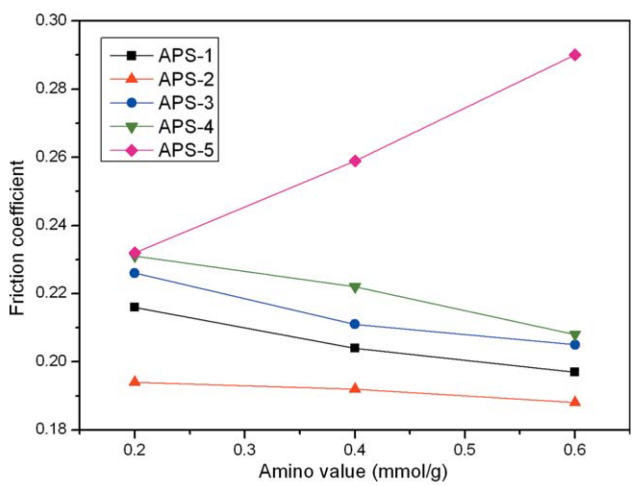


Figure 4 Friction coefficients for cotton fabrics treated with APSs with different amino values. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

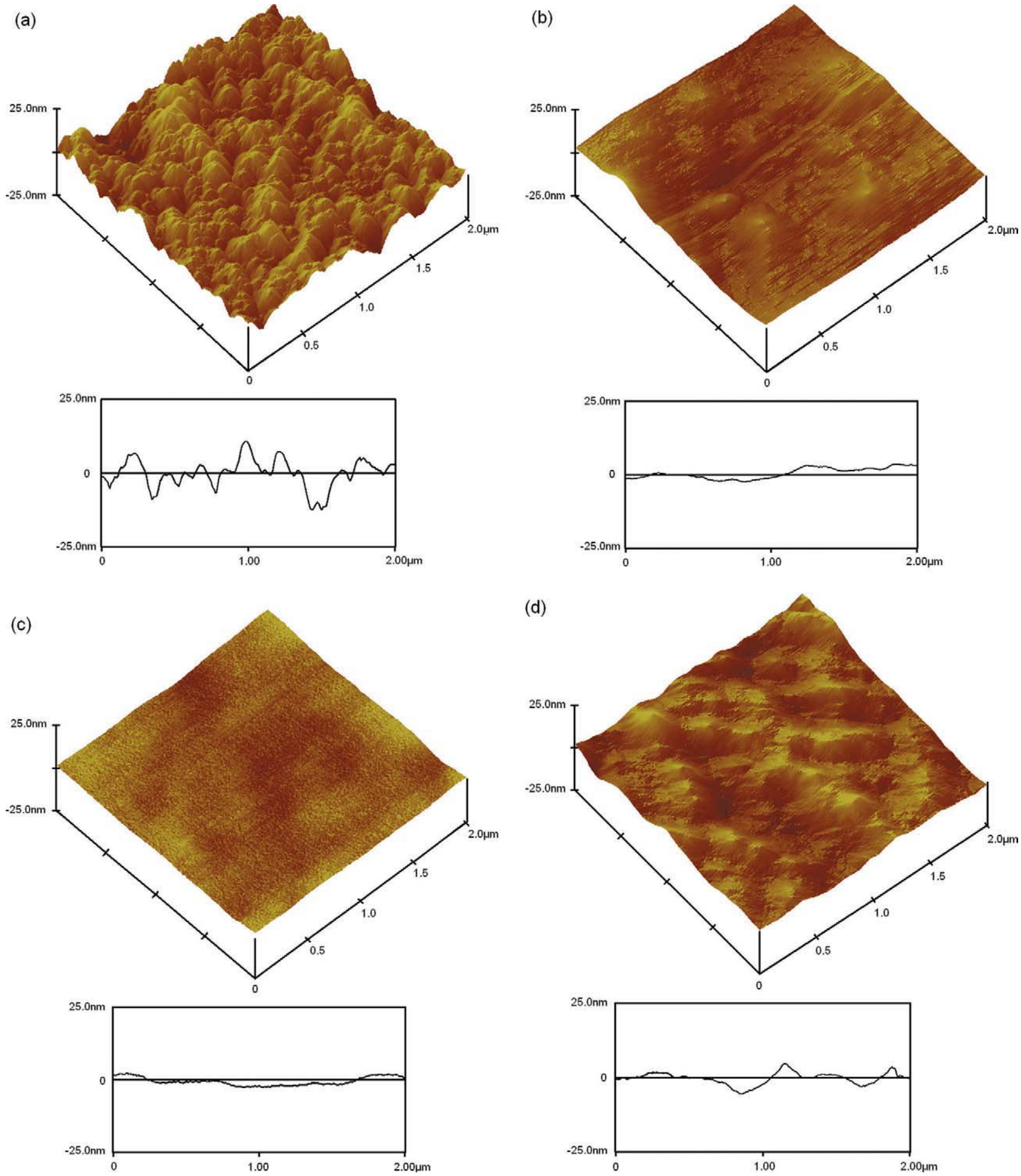


Figure 5 Three-dimensional AFM images and height profiles for cellulose substrates untreated and treated with APSs with amino value 0.4 mmol/g: (a) untreated; (b) treated with APS-1; (c) treated with APS-2; (d) treated with APS-3; (e) treated with APS-4; (f) treated with APS-5. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

increased. As shown in Figure 4, the friction coefficients of fabric treated with APS-4 and APS-5 are slightly larger than the ones treated with APS-1, APS-2, and APS-3.

Amino value and the substituents on N are the other two significant factors on friction coefficients. A low amino value leads to a relatively long $[(CH_3)_2Si{-}O]_n$ segment, and the surface roughness and smoothening property are greatly affected by the airwards-oriented main polymer chains. As shown in Figure 5, with an increase in the amino value, the friction coefficients decrease (except the

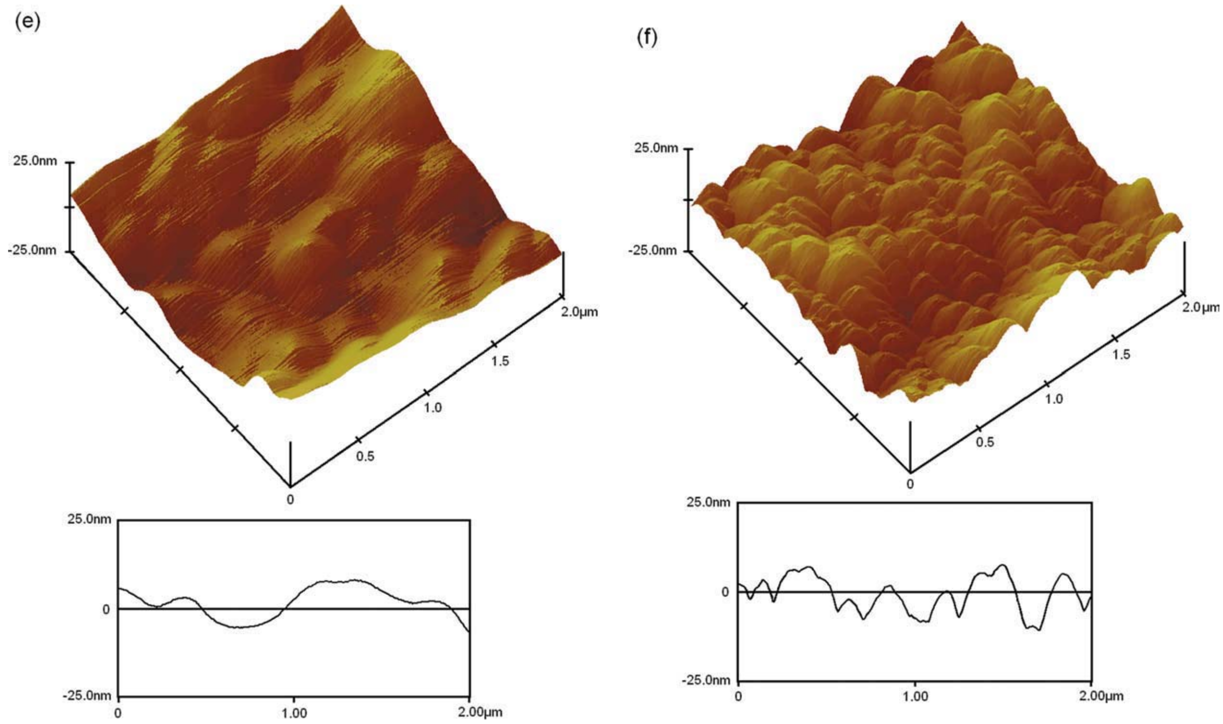


Figure 5 (*Continued from the previous page*).

APS-5). For APS-5, the hydrophobic cyclohexyl, with high steric hindrance, extend toward the air and greatly increase the surface roughness, so the friction coefficient is significantly larger than other samples and increases significantly with an increase in the amino value. And for APS-3, two hydrophobic methyls on the tertiary amino extend toward the air and increase the surface roughness of the fabric, so the friction coefficient of the fabric treated with APS-3 is slightly larger than the ones treated with APS-1 and APS-2.

Film morphology

AFM is a powerful instrument in observing film morphology of polymer and their blends. It also provides a way to observe the film morphology of functional polysiloxane on molecular scale. Therefore, AFM was utilized in our research to investigate the precise morphology of APS films with various amino types on the cellulose substrate, and the results are shown in Figure 5 and Table II.

Untreated cellulose substrates have rough surface, and the average roughness (expressed by the root-mean-square-roughness, Rq.) reaches 4.771 nm, this is consistent with previously reported AFM results for cellulose.[22,23] Comparing Figure 5(a) with (b) ~ Figure 5(f), it is discovered that APSs have formed films on the cellulose substrate and minimized the grooves in different degree.

APSs with different amino values and amino types have various film morphologies. In preliminary studies, the authors[9] have revealed the correlations between the amino value and film morphology, and proposed that the length of $[(CH_3)_2Si{-}O]_n$ segment between two adjacent amino-hydrocarbons becomes shorter with the increase in the amino value, which leads to a smoother surface and therefore superior smoothening property (Fig. 4).

TABLE II
Root-Mean-Square-Roughness (Rq) of Cellulose Substrates Untreated and Treated with APSs with Different Amino Types

Sample	Img. Rq (nm)[a]	Box Rq (nm)[b]
Untreated cellulose substrates	5.936	4.771
Cellulose substrates treated with APS		
APS-1	3.517	1.177
APS-2	1.414	0.578
APS-3	3.670	1.966
APS-4	11.793	2.369
APS-5	11.319	4.064

[a] The cellulose substrates were wrinkled when treated in APS solution, thus the Img. Rq values of substrates treated by APS were sometimes higher than the untreated substrates.
[b] The Box Rq values were chosen from 1 µm^2 smooth area.

The amino types also influence the film morphologies. As shown in Figure 4, the substrates treated with APSs with two amino groups on one coupling agent (APS-1, APS-2, and APS-3) have relatively smooth surfaces, while the average roughness of the substrates treated with APSs with one amino group on a coupling agent (APS-4 and APS-5) are dramatically larger. This is probably because the APSs with one amino group on one coupling agent cannot adsorb firmly onto the cotton and the main polymer chains are not trimly oriented (with some main chains overlapped with each other). The surface inhomogeneity of the silicone film increased the surface roughness. For APS-5, the cyclohexyls with high steric hindrance greatly increase the surface roughness [Fig. 5(f)]. For APS-3, the methyls also slightly increase the surface roughness [Fig. 5(d)].

CONCLUSIONS

Amino polysiloxanes (APSs) provide excellent softening and smoothening benefit for textiles, and amino values and amino types of APSs significantly affect the performance of the finished fabric. Amino value controls the average length of $[(CH_3)_2Si{-}O]_n$ segment between two adjacent amino-hydrocarbon groups and thereby affects the bending, rotating and sliding of the main polymer chain. High amino values result in superior smoothening property while moderate amino values result in better softening property. For different amino types, the fabric handle is affected by the number of amino groups on one amino silane coupling agent, the substituent on N and the steric hindrance of the substituent. Compared with the traditional softener APS-1, the new type softeners APS-2 and APS-3 give better fabric performance, whereas APS-4 and APS-5 give unsatisfactory fabric performance.

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