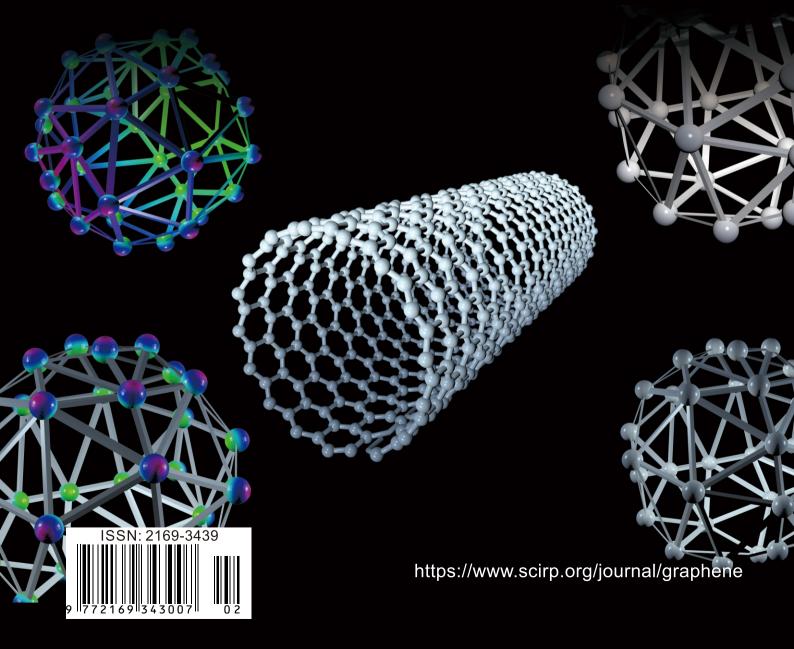
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Preparation and Application of Anionic and Cationic Waterborne Polyurethanes and Graphene-Cellulose Nanocrystal as an Antistatic Agent for Cashmere

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Abstract

The main purpose of this research work is to improve anti-static properties of Cashmere fabric by introducing application comprising anti-static agent by foaming which was made with cationic waterborne polyurethane and graphene-CNC. Cashmere fabric was cut into 10 pieces of sample cloth of 5 cm * 5 cm size, washed with acetone solution, and then dried in an oven at 60 °C. Three forms of waterborne polyurethanes such as two forms of Cationic waterborne polyurethane (CWPU) and a form of Anionic waterborne polyurethane (AWPU) were synthesized. Cellulose nanocrystalline (CNC)/graphite powder solution with the ratio of 0.5/1, 1/1, 2/1 was prepared by ultrasonic probe stripping method, and the concentration of graphite powder was ensured to be 1 mg/ml. The fabric was treated with anionic and cationic WPUs foaming solution until the weight gain reached 2.5 - 3.5 wt%. After drying, the elastic cloth was foamed with graphene solution until the graphite content of the cloth was close to 10%, 20%, 40%, 60% respectively, and then dried for reserving. Characterization properties of pure graphite powder, pure CNC and graphene solution with different proportions of three components were tested by Fourier transform infrared spectrometer (FTIR), X-ray diffraction (XRD), Thermalgravitimetric analysis (TGA) and scanning electron microscopy (SEM). Take the original cloth, only WPU treated cloth and four clothes with different graphite content for the fabric performance test.

Keywords

Cashmere Fabric, Anionic and Cationic Waterborne Polyurethane, Graphene, Cellulose Nanocrystal, Anti-Static Property

[#]Co-first authors.

1. Introduction

The utilization of traditional solvent that is based on Polyurethane (PU) has regularly been being deterred, because of firm environmental and governmental influences [1]. Textiles and other spheres such as adhesives, coatings, construction materials, inks as well as biological materials could be fields in which Waterborne polyurethane (WPU) might be utilized as a kind of multifunctional environmentally friendly substance due to its excellent properties. In terms of the outlook of material technology, the electrical properties, acoustic, optical properties as well as static and dynamic mechanical features are considered as essential properties and the performances of PU plastic are chemical, biological and corrosion resistance, adhesive properties. In spite of being a substantial number of excellent advantages of WPU, there might be found several disadvantages of WPUs which were recorded and published in some researches. As an example, it can be shown that if compared to a traditional solvent which is based on polyurethanes, a feebler drying rate is observed on WPUs because of the high secretive heat and evaporation of water. Furthermore, the linear hydrophilic groups that WPUs contain in their chain instigate to require too much drying time and higher energy utilization for WPUs to reach expected mechanical power. In the case of meeting the time efficiency and economic profit requirements, this could not be useful [2].

Hence, research nowadays is mainly focused on the preparation of WPUs with a high solid content, which is of growing interest both in the field of scientific research and in industrial applications [3]-[7]. As an important category of WPUs, cationic waterborne polyurethanes show excellent adhesion to anionic substrates, such as glass and leather [8]. For that reason, Cashmere is a soft wool fabric woven from the hair of the Cashmere which can be applied to an anionic substrate. Cashmere fibers are extremely soft and silky to the touch and weigh practically nothing. When even compared to Merino, Cashmere can be found softer and fine-drawn, and it is more delicate than other types of animal wool. Besides, Cashmere is especially useful in terms of using it for accessories and curtains. In many cases of specifications of wool fabrics, Cashmere can be admitted as the king of the fiber and treasure, owing to its pros and cons [9]. When comes to advantages of Cashmere, it can be counted that increasing the deep sleep which illustrates in experiments that cashmere material could enhance about more than 25 percentage of the individuals deep sleep time as well it can maintain heart rate slowly and steady. Additionally, moisture absorption and it keeps dry, warm in cold weather since cashmere special molecule structure can absorb the water vapor more than 35% into the hollow structure which cannot feel wettability and can remove quickly. Also, it presents no less than 40% natural elastic, after pressing, it has been observed that it could recover approximately 95% of initial ply. Moreover, as advantages of cashmere, fluffy, softness, natural fire-resistant, dust preventive are considered and cashmere has 17 kinds of abundant amino acid which provide the cashmere the feature of closing to the skin. Although here several pros have been mentioned, there some cons of cashmere which are caused problems to fabricate products form it because cashmere is wool, it is a hydrophilic fiber, and wool fabrics frequently are highly charged caused by the firm friction between the wool scales. To solve this issue of cashmere fabric, anti-static finishing is required [10]. The importance of optimal performance is uniform fabric penetration while applying anti-static finishes to fabrics. In the finish formulation, the utilization of wetting agents is recommended, Cationic anti-static agents fit for exhaust processes [11]. The low add-on values (mostly about one or a few percents on the weight of fiber) enable spray and foam applications. Padding leads to lesser effects than foam does application with an identical add-on. The higher concentrations of anti-static agents on the fiber surface by the foam application can be the actual factor for this. Some of the potential side effects of the use of anti-static finishes include wearing comfort (no clinging and pleasant skin contact caused by hydrophilicity). To improve the anti-static ability of Cashmere fabric, graphene has been widely used over the last decade due to possessing excellent thermal, mechanical and electrical properties and high surface area. Graphene, a primary constructing block of graphite, possessing a one-atom-thick sheet of hexagonally comprised sp2-hybridized carbon atoms [12]. There have a lamellar structure and stacked jointly by van der Waals forces in the graphitic layers. Extraordinary electronic properties were anticipated to have in these individual layers. Graphene has extraordinary electrical and thermal conductivities, ultra-high theoretic specific surface area (2630 m²/g) and high mechanical strength. These excellent properties make graphene a promising candidate for energy storage devices, composites, and electronics [13]. High specific surface area and electrical conductivity with mechanical strength make this material a promising choice for energy storage devices. For those aspects, some researchers have investigated experiments regarding graphene based on cellulose nanocrystal (CNC) which has hydroxyl group (OH) presenting hydrophilicity as to improve electrical conductivity. Anti-static agents can be prepared by making of three forms of WPUs such as an anionic waterborne polyurethane and two cationic waterborne polyurethanes and graphene-based on cellulose nanocrystal (CNC) to apply that solution to coat on the surface of Cashmere fabric through a foam finishing. Here, the duty of WPUs is to serve as adhesives and to cover the graphene microparticles so that graphene microparticles are durable for several times laundarying, simultaneously WPUs can reduce pilling properties of Cashmere which is the most undesired.

The cost was high for the moisture evaporation of chemical reagents in the conventional fabric, finishing, however, the foam dyeing and finishing technology can save more energy in the drying process. Foam finishing is a kind of dying and finishing technology with great energy-saving effect because foam as a medium carrier of dyes was applied to substitute for part of the water. The processing effect can reduce 65% - 75% compared with the conventional water consumption. Besides, multifunctional textiles can be produced through the foam finishing technology to meet the higher demand for market consump-

tion. It is a good prospect for the application that the foam finishing technology is applied to Cashmere fabric finishing, including anti-pilling and anti-static [14]-[18].

In this current study, anti-static properties of Cashmere fabric was enhanced by casting film solution, which was prepared by mixing three types of WPUs and graphene-CNC with some concentrations of ratio for each sample, on the surface of it. The results obtained from tests showed that the anti-static properties of fabric were improved when compared with examples.

2. Experimental

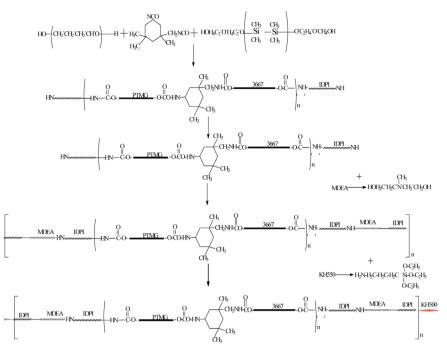
2.1. Materials

Cashmere was purchased from Beta Textiles Co., Limited (BTEXCO). Isophorone diisocyanate (IPDI) and Methyl diethanolamine (MDEA), dibutyltin dilaurate (DBTDL), *y*-Aminopropyl Triethoxysilane (KH550) chemical pure were obtained from Shanghai Jingchun Reagent Co., Ltd., and Aladdin Reagent (Shanghai) Co., Ltd; polyether triol (ZC330) industrial products Guangzhou Jinwang Chemical Co., Ltd.; N220, DMPA, Ethylenediamine (EA), Triethylamine (TEA) analytical purity China Pharmaceutical Group Chemical Reagent Co., Ltd. Graphene-CNC was derived by reaction of Graphite and cellulose nanocrystal withholding cold water temperature and centrifugal force to remove particles' residues. Anionic and cationic waterborne polyurethanes were synthesized by reacting reagents following their recipe. In synthesizing PTMG and PEG were dried and degassed under 110°C and other materials were used as received.

2.2. Preparation of Cationic Waterborne Polyurethanes with MDEA Volume Ratio 20% and 13%

Two types of cationic waterborne polyurethanes were synthesized by following recipes shown in **Table 1** and **Table 2** as well as the chemical structure of Cationic Waterborne Polyurethanes was shown in **Scheme 1**.

The first step in the synthesis of the cationic WPUs was to introduce PTMG 2000 and 3667 which were vacuumed to remove water in them under 110° C and 0.01 Mpa for 120 minutes with Isophorone diisocyanate (IDPI) which has functional group isocyanate R–N=C=O which can react with chemicals when it is effected as a second step is done under 85°C for 150 minutes. The first prepolymer I was synthesized based on IDPI and PTMG. Then MDEA was reacted to build the second prepolymer for the final product to make it ready. A 250 mL round-bottomed, four-necked flask equipped with a nitrogen inlet, a condenser with a drying tube, a mechanical stirrer and a thermometer was used for the reaction. After being kept two chemicals at the required time, a drop of DBTDL was added as a catalyst and simultaneously by repeating six times of adding, MDEA was dropped at 40°C during the 30 minutes, then with the same repetition and the duration of time, NEP was added at 40°C - 50°C. In the next step,



Cationic Waterborne Polyurethanes

Scheme 1. The chemical structure of Cationic Waterbonre polyurethanes.

Raw material name	mol	g	Mass ratio %	Quantity
PTMG	0.07	140	13.30%	11.67
3667	0.07	154	44.34	12.83
IDPI	2.1	466.81	44.34	38.90
DBTL			1	
MDEA	1.8	214.49	20.37	17.87
KH550	0.35	77.49	7.36	6.46
Bromabutane	1.8	246.64		20.55
Acetic acid				21.45

Table 1. CationWPU (MEDA Mass ratio 20%).

Table 2. Ca	ation WPU	(MEDA	Mass ratio	13%).
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Raw material name	mol	g	Mass ratio %	Quantity
PTMG	0.2	400	30.10	28.57
3667	0.08	176	13.25	12.57
IDPI	2.1	466.81	35.13	33.34
DBTL			1	
MDEA	1.45	172.78	13	12.34
KH550	0.45	99.63	7.5	6.46
Bromabutane	1.45	198.68		7.12
Acetic acid				14.81
Butanediol	0.15	13.52	1.02	0.97

KH550 was added and mixed for 10 minutes and as a catalyst DBTDL was introduced. Then, acetic acid as a neutralizing agent was not added into the reaction until the mixture was slowly cooled to 40°C. The neutralization reaction proceeded at the same temperature for 30 min. When the synthesis of CWPU with MDEA volume ratio 13% was done, 1,4-butanediol (BDO 1,4) was used as a different reagent.

2.3. Preparation of Graphene-Based on Cellulose Nanocrystal

Graphene solution was prepared with graphite oxide and cellulose nanocrystal to make at three types of ratio (graphite = 0.5 g): (CNC = 1 g), (graphite = 1 g): (CNC = 1 g), (graphite = 2 g): (CNC = 1 g). The volume of solution was 100 ml including graphite oxide, CNC and water and it was kept at cold water temperature for 90 minutes by mixing with stirring instrument and then the solution was held on centrifugal force instrument for removing residue of particles at speed of 4000 for 2 minutes.

2.4. Application of Anti-Static Agent for Coating Foam on the Surface of Cashmere

Firstly, required size of Cashmere fabric was cut and prepared to test for anti-static properties. In order to prepare the anti-static agent, each of three WPUs and graphene-CNC was weighted by ratio. For cationic waterborne polyurethanes, vesicant Sodium dodecyl sulfate (K12) which is to make the solution to become a bubble, graphene-CNC solution that was made in advance, sodium alginate and water at the bulk of 50 ml were used, while for anionic waterborne polyurethane, the anti-static solution was made of AWPU and further chemicals. Ready anti-static agents and fabric were tested by FP020 Foam system (Shanghai Yuhui Chemical Co., Ltd.) and results were recorded.

2.5. Characterization of WPUs, Graphene and Anti-Static Property of Cashmere

1) A latex film was prepared for further testing by casting the emulsion on a Teflon plate and drying at ambient temperature for 7 days and then in a vacuum system at 50°C for 2 days to characterize three forms of waterborne polyure-thanes such as two cationic and anionic polyurethanes by three characterizations such as film resistance to water as well as film resistance to Acid. Characterizations of three types of WPUs are film resistance to water and film resistance to acid. These two tests were examined during the 3 days which are from an initial to the final time. The results are illustrated in **Table 3** and **Table 4**. Polyurethane and two forms of cationic waterborne polyurethanes which for film resistances to water and acid were found as expected.

2) The graphite and graphene-cellulose nanocrystal was tested by three analyzers of Nicolet iS5 Fourier Transform Infrared spectrometer (FTIR Thermo Scientific Company USA), TGA and XDR. The FTIR spectra infrared tester was

Time	PU 3667 + 2348	PU 3030	PUA 2348	PUA 3667 + 2348	PUA 3030
0 hour	0.090	0.130	0.180	0.210	0.170
3 hour	0.130	0.160	0.160	0.240	0.130
8 hour	0.102	0.143	0.168	0.322	0.272
1 day	0.110	0.145	0.179	0.334	0.267
2 day	0.115	0.146	0.175	0.327	0.259
3 day	0.113	0.144	0.174	0.327	0.260

Table 3. Film resistance to water.

Table 4. Film resistance to acid.

Time	PU 2348	PU 3667 + 2348	PU 3030	PUA 2348	PUA 3667 + 2348	PUA 3030
0 hour	0.169	0.124	0.161	0.109	0.180	0.168
3 hour	0.231	0.160	0.180	0.153	0.240	0.210
8 hour	0.228	0.163	0.187	0.156	0.249	0.226
1 day	0.220	0.170	0.200	0.160	0.260	0.240
2 day	0.221	0.182	0.202	0.165	0.264	0.238
3 day	0.225	0.197	0.203	0.174	0.268	0.235

used to analyze sample at the resolution of 3 cm^{-1} , scanning from 4000 cm^{-1} to 500 cm^{-1} . The analysis was carried out at ambient temperature.

3) The anti-static property of Cashmere fabric was tested by FY342E-II Fabric inductive electrostatic meter. Before testing samples, the each of samples was prepared through coating anti-static agents with foam method on the surface of the fabric and the coated samples were dried by Vacuum Drying Chamber (ShanghaiJinghong Co., Ltd.). Results taken by Fabric inductive electrostatic meter were recorded. After testing samples for an electrostatic property, samples were observed to check how the graphene-CNC fixed on the surface of Cashmere fabric by S-4800 Emission Scanning Electron Microscope (Hitachi Japan) and figures were recorded and presented as good results.

3. Results and Discussions

The FTIR analysis: The FTIR spectra test was conducted to determine the chemical compositions on Graphite, Cellulose Nano-Crystal and three forms of the Graphene by three ratios ssuch as (graphite = 0.5): (CNC = 1); (graphite = 1): (CNC = 1); (graphite = 2): (CNC = 1). The result of the CNC taken by FTIR spectra shows that The FT-IR spectra for cellulose nanocrystal, CNC is shown in **Figure 1**; the peak at 1605 cm⁻¹ corresponds to C=C stretching vibrations of CNC. The cellulose nanocrystal illustrates OH stretching 3400 cm⁻¹ and C-H stretching 2850 cm⁻¹ corresponding to the aliphatic moieties. The strong absorption peak at 1058 cm⁻¹ ought to be the stretching vibration of the C-O-C bond in cellulose nanocrystal. C–O and C=O bond of Cellulose hit 1058 cm⁻¹ and 1725 cm⁻¹

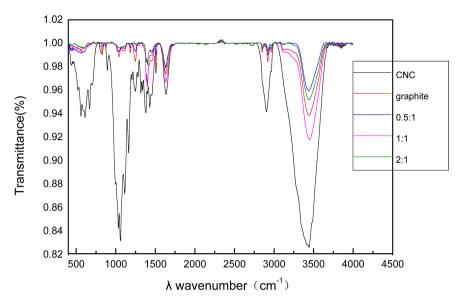


Figure 1. The FTIR spectra of CNC, Graphite and Graphene (0.5:1; 1:1; 2:1).

as it is shown in **Figure 1**. These characteristic peaks show on CNC that Cellulose nanocrystal was truly introduced in the Graphite to prepare Graphene. It can be seen from the figure that the absorption peak of about 3400 cm⁻¹ belongs to the stretching vibration of –OH, while the surface of CNC without graphene modification contains a large number of hydroxyl groups, which makes it difficult to disperse uniformly in ordinary solvents, and the amount of CNC hydroxyl groups after modification decreases, indicating that the dispersion of CNC in water can be improved.

Generally, the FTIR signals of Graphite was identified as a following; C-Cl signal bonds arranged around 600 cm⁻¹, aromatic ring C=C bonds showed at peak 1600. The peak of absorbance was 2750 cm⁻¹ and 1650 cm⁻¹ signal is due to internal CH respectively. The strong and long signals indicating that they are more in particular bonds in tat regions or radiations. Because the mass of the atom increases, usually the wavenumber will decrease. As shown in Figure 1. When it is at aromatic signal 1600 which is due to beneze ring the conguregation goes up. These upwards and downwards were observed on graphene forms. As an example when compared to graphene 0.5:1 ratio with the graphenes 1:1; 2:1 ratio, it can be contemplated that the absorbance percentages rose up substantially in accordance with increasing of ratios of the graphene which means that the amount of light energy that passes through the samples increased. As an example, OH, C=O and C-O bonds that made up around the peak of 3400 cm⁻¹, 1780 cm^{-1} and 1087 cm^{-1} on graphene 0.5:1 ratio comprised 0.96%, 0.98% and 0.99% of transmittance while that transmittance percent was 0.92%, 0.97 and 0.98 at the same peak of 3400 cm⁻¹ on graphene 1:1 ratio. The identical parameters were recorded with other chemical compounds when graphene 2:1 ratio was compared with two ratios (0.5:1 and 1:1).

The XRD analysis: While testing samples of Cellulose nanocrystal (CNC), graphite as well as three forms of graphene by ratios (graphite = 0.5): (CNC = 1);

(graphite = 1): (CNC = 1); (graphite = 2): (CNC = 1) by X-ray diffraction (XRD) analyzer, the following results were obtained and shown in **Figure 2**. The peak at 21.7 degrees was elucidated as relatively weak regarding the crystallinity, according to the line of cellulose nanocrystal (CNC). When reviewed three samples of Graphene such as (graphite = 0.5): (CNC = 1); (graphite = 1): (CNC = 1); (graphite = 2): (CNC = 1), three different the points were recorded 25.5 degrees. It can be seen that graphite has a strong diffraction peak at 26.8 degrees and the peak is high and sharp which indicates the crystallinity of this sample is very high. The intensity of diffraction peak goes down slightly with the increase of CNC ratio which indicates that the more CNC content, the greater the degree of peeling of graphite powder, thus reducing the degree of crystallization of graphite powder was shown from the three lines of graphene' samples which were presented in **Figure 2**.

The TGA analysis: When Thermalgravimetric analysis (TGA) was carried out, the following indications which were shown in Figure 3 were gained. They are explained as a following:

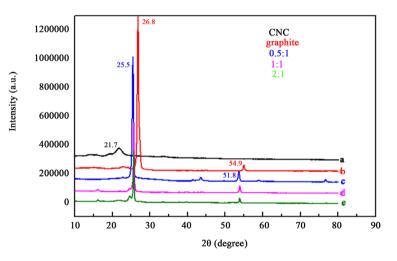


Figure 2. X-ray diffraction of CNC, graphite, graphene (0.5:1, 1:1, 2:1).

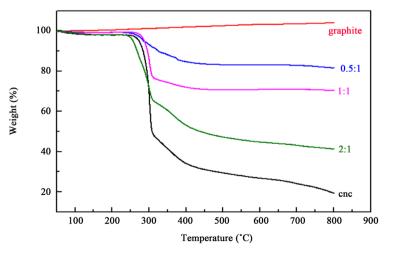


Figure 3. TGA of CNC, graphite and graphene (0.5:1, 1:1, 2:1).

If the curve of graphite is examined to identify the indices relating to Y-axis (weight %) and X-axis (Temperature °C), it can be understood that when the temperature hits 800°C, losing the mass on the pure graphite powder is not observed. Because of inaccurate baseline and inaccurate instrument precision, the mere upward trend may occur.

According to the curve of cellulose nanocrystal (CNC), the curve can be shown that due to the surface of Cellulose nanocrystal (CNC) which contains a great share of hydroxyl groups, there was a drammatic increase by 80.7% in the weight loss rate, when the temperature reached as same as the temperature of the pure graphite powder.

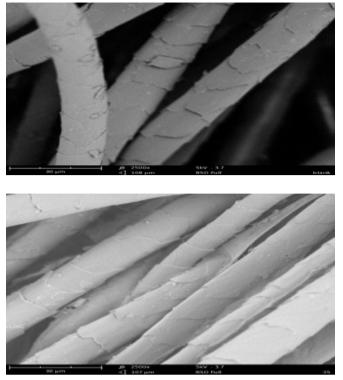
The weightlessness rates of CNC/graphite powder solutions with three proportions of 0.5:1, 1:1, and 2:1 were 18.4%, 29.6% and 58.8% respectively, indicating that with the increase of CNC dosage, the more hydroxyl groups were added, and the weightlessness rates increased with the increase of CNC concentration.

The SEM analysis: The Scanning electron microscopy shows the images of Cashmere fabric which was treated by grapheme (0.5:1, 1:1, 2:1) in Figure 4. The microscopic images were taken by the magnification of $\times 2500$ and 5 kV, 30 µm. it is clear that when the images of untreated Cashmere fabric are observed, it can be that Cashmere fabric has roughness on its surface before coating the graphene. While the samples of GO which were made of three different ratios of Graphite were coating by the foam method, the surface roughness of Cashmere fabric dispersed as shown the images. Additionally, the sample of fabric coated with graphene 2:1 ratio showed a better film on the surface than the other two samples of Cashmere and anti-static properties as well. It can be assumed that when the surface has less roughness and if the graphene film occupies well on the surface of the fabric, the Fabric can be provided with the optimum improvements such as anti-static as we expected. As an example, Figures 1-4 were presented as a following.

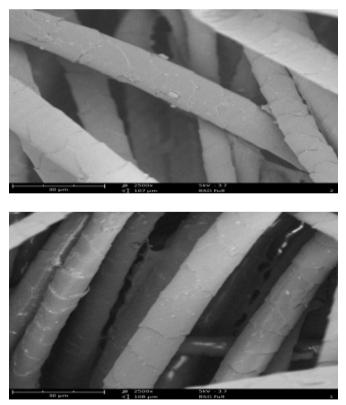
4. Conclusions

The results of the analysis we have taken lead to the following conclusion. It was observed that the graphene can be fixed to the surface of fabric well by introducing waterborne polyurethanes (WPU) and it is not easy to fall off. When compared with the foam treated fabric, the direct coating can save more raw materials and the coating is more uniform.

The fabrics treated with graphene show good antistatic and the concentration of graphene will affect the conductivity. The higher the concentration makes the conductivity better. The CNC is more used to peel graphite powder, the better the peeling effect and the better the graphene performance. However, with the increase of CNC intercalation, the conductivity of graphene will be affected to some extent. Due to the low concentration of graphene and poor moisture absorption of fabrics, some raw materials will be lost during the foam treatment process. Therefore, it is hoped that a drying device can be installed below the



Untreated Cashmere fabric Cashmere coated with graphene (graphite = 0.5: CNC = 1)



Cashmere coated with grapheme (graphite = 1: CNC = 1) Cashmere coated with graphene (graphite = 2: CNC = 1)

Figure 4. The scanning electron microscopy images of Cashmere fabric coated with grapheme.

coating machine to directly evaporate the water in the solution so that the graphene can be coated on the fabric 100 percent.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

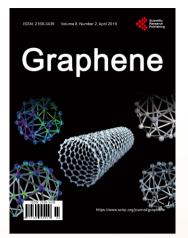
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