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# Fabric handle of plasma-treated cotton fabrics with flame-retardant finishing catalyzed by titanium dioxide

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## Abstract

N-methylol dimethylphosphonopropionamide flame retardant agent (FR) combined with a melamine resin crosslinking agent (CL) and a catalyst (phosphoric acid, PA) is used for enhancing the flame-retardant property of cotton fabrics. Titanium dioxide ($TiO_2$)/nano-$TiO_2$ co-catalyst is added to the FR formulation in order to enhance the crosslinking of the FR-CL-PA components. Atmospheric pressure plasma jet is used as a pre-treatment, to enhance the fabric properties by a sputtering or etching effect. The Kawabata Evaluation System for Fabrics (KES-F) was used to determine the tensile, shearing, bending, compressional and surface properties. In comparison, with the control fabric the specimens after FR treatment had worse tensile, bending, compression, surface friction and variation properties; an improvement was observed only in the shearing properties. With the aid of plasma pre-treatment, tensile and compressional properties of the FR-treated specimens were improved, while the shearing and bending, as well as the surface friction and variation properties, were affected negatively. A neutralization process could neutralize excessive acids and remove both the unfixed chemicals on the treated fabric surfaces, and the unattached metal oxide particles as well. The process improved the fabric handle in some aspects, such as tensile energy, extensibility, shear stiffness, bending rigidity, bending moment, compressional linearity, compressional energy and coefficient of friction.

**Keywords:** flame-retardant; low stress mechanical properties; plasma pre-treatment; titanium dioxide.

## 1. Introduction

Cotton is essentially a cellulose and its pyrolysis behavior has a significant influence on fabric flammability, i.e., evolution of combustible volatiles in the prelude to ignition and the subsequent fire propagation [1, 2]. Flame resistance is a critical property for textile products, to ensure the safety of consumers [3]. Based on this reason, most of the efforts in the area of textile FR finishing have focused on reducing the flammability of cotton [2–11]. However, the increasing concerns over the toxicological and environmental consequences of applying chemical processes to textile substrates have impeded the development of new chemicals for application [12]. In general, the N-methylol dimethylphosphonopropionamide flame retardant agent (FR), combined with a melamine resin crosslinking agent (CL) and a catalyst (phosphoric acid, PA) is widely used to enhance the FR property of cotton fabrics [2]. In previous studies [13, 14], it was found that, without any wet treatment, the FR-treated specimens did not ignite, and the flame was extinguished right after removal of the ignition source without the flame spreading. The use of PA as a catalyst in the finishing formulation increased the fixation of the FR and the reaction of CL on the fabric, even after multiple laundering cycles or neutralization. However, the systems are still inadequately configured to meet specific requirements, such as preserving soft handle of the treated cotton fabric. In the present study, a titanium dioxide ($TiO_2$)/nano-$TiO_2$ co-catalyst is added to the FR formulation in order to enhance the crosslinking of components of the FR-CL-PA system, i.e., FR-CL-PA-$TiO_2$ and FR-CL-PA-nano-$TiO_2$ system [13, 14]. In addition, it is known that the surface texture of the fabric also affects flammability. Fabrics with long, loose or fluffy piles ignite more readily [15]. Plasma technologies, such as atmospheric pressure plasma jet (APPJ), can help retain the inherent advantages of the substrates, while enhancing the material properties by a sputtering or etching effect, which alters the characteristics of fiber surface [16–21]. Previous research has confirmed that the etching effect on the fabric surface by plasma gas, removed weak boundary layers and organic contamination from the fiber surface, which avoided interference of bonding between fiber and FR-CL-PA-linkages, i.e., exhibited excellent FR performance [13, 14].

Understanding the relationship between the end-use and properties of the fabric is fundamental for classification, selection, and control of apparel fabric treatments. For example, handle is a critical physical property for consumers when making purchasing decisions [22]. The Kawabata Evaluation System for Fabrics (KES-F) is used frequently to measure hand-feel of fabric, which is determined by the sensory tests conducted by instrumental measurements [23]. In the present study, detailed information concerning the effect of plasma pre-treatment on the handle of cotton fabric subjected to FR treatment is evaluated.

## 2. Experimental details

### 2.1. Material

One hundred percent semi-bleached plain weave cotton fabric (warp: 58 ends/cm, yarn count 40 tex; weft: 58 picks/cm,

yarn count 38 tex; fabric weight: 175 g/m$^2$, Tai Tung Textiles Company, Hong Kong) with a size of 20 cm×20 cm was used. The FR and cellulose crosslinking agent, supplied by Huntsman Ltd., were an organic phosphorus compound (Pyrovatex CP New, FR, Huntsman Ltd., China) and melamine resin (Knittex CHN, CL, Huntsman Ltd., China), respectively. PA of analytical reagent grade, which served as a catalyst, was supplied by the Sigma-Aldrich Company, USA. Co-catalysts used were micro-$TiO_2$ (2 μm diameter) and nano-$TiO_2$ (100 nm diameter) obtained from UniChem Ltd., USA and International Laboratory Ltd., USA, respectively, both having a purity of >99.5%. The particle size of $TiO_2$ was further confirmed by the particle size analyzer (LS13320 Beckman Coulter, Beckman Coulter Inc., USA). Sodium carbonate, analytical reagent grade, was supplied by Sigma-Aldrich Co.

### 2.2. Plasma pre-treatment

Plasma pre-treatment of cotton fabric was carried out by the APPJ apparatus, Atomflo 400 Plasma controller integrated with a robot, which is manufactured by Surfx Technologies, USA. The cotton fabric was moved automatically at 10 mm/s treatment speed. The machine produced a stable discharge at atmospheric pressure, with a radio frequency of 13.56 MHz and output power of 120 W. The treatment was carried out by a rectangular nozzle mounted vertically (3 mm jet-to-substrate distance above the cotton fabric covering an active area of 50.8 mm×1 mm. Helium (30 l/min flow rate, Hong Kong Oxygen and Acetylene Co Ltd., Hong Kong) and oxygen (0.2 l/min flow rate, Hong Kong Oxygen and Acetylene Co Ltd., Hong Kong) were used as the carrier and reactive gas, respectively.

### 2.3. Two-bath pad-dry-cure flame-retardant treatment

Plasma pre-treated cotton fabric samples were treated by a two-bath method using different compositions of finishing agents, as shown in Table 1. In the first bath, the fabrics were dipped and padded with FR, i.e., FR-CL or FR-CL-PA, until a wet pick up of 80% was achieved at 25°C. The fabrics were then dried at 110°C for 5 min. In the second bath, dipping and padding processes of fabrics with 80% wet pick up were performed using a $TiO_2$/nano-$TiO_2$ solution dispersed in 10% Matexil DN-VL (a dispersing agent to disperse metal oxide particles in water). Subsequently, the padded fabrics were dried at 110°C for 5 min, followed by curing at 170°C for 1 min. After curing, the treated specimens were neutralized with 30 g/l sodium carbonate for 15 or 30 min at 50°C for comparison. After neutralization, the specimens were rinsed in water at 50°C. Finally, the fabrics were conditioned at 21±1°C and 65±5% RH for 24 h prior to any further treatment.

### 2.4. Kawabata evaluation system for fabrics (KES-F)

KES-F (Kato Tech Co., Ltd., Japan) was used for testing surface stress and other mechanical properties related to physical and tactile comfort. The system was able to test fabric stiffness, thickness, extensibility, appearance retention, surface smoothness and bulkiness. The KES system comprises five specialized instruments for testing tensile strength, shearing strength, bending, compression, and surface friction and variation. Test specimens with a size of 200 mm×200 mm were examined in a standard environment. All specimens were conditioned under standard conditions for 24 h prior to all measurements.

**2.4.1. Tensile properties** The tensile properties of test specimens were evaluated by a Tensile and Shearing Tester, FB1-A instrument of KES-F. Tensile properties, including textile resilience and textile strain, which are related to the ability to maintain fabric shape, and the extensibility or stretchability of the fabrics, as well as the tensile energy, were measured. The samples were mounted on the machine which automatically measured the tensile property. The tensile properties were recorded with an average of six measurements taken along the warp and weft directions.

**2.4.2. Shearing properties** The shearing properties of test specimens were also evaluated by the Tensile and Shearing Tester, FB1-A instrument of KES-F. Shearing properties are composed of shear stiffness, hysteresis of shear force at 0.5° of shear angle and hysteresis of shear force at 5° of shear angle. Shear stiffness is related to the softness handle of the fabric. The samples were mounted on the machine, which automatically measured the shearing property in both the warp and weft direction. The shearing properties were recorded with an average of six measurements taken along the warp and weft directions.

**2.4.3. Bending properties** Pure Bending Tester, FB2 instrument of KES-F, measures the bending property of test specimens. The properties include bending rigidity and hysteresis of bending moment, which indicate the softness of

**Table 1** Flame-retardant treatment conditions.

| Sample symbol | Pyrovatex CP New | Knittex CHN | Phosphoric acid (85%) | Micro-titanium dioxide | Nano-titanium dioxide |
|---|---|---|---|---|---|
| F1 | 40% | 5% | – | – | – |
| F2 | 40% | 5% | 2.5% | – | – |
| F4 | 40% | 5% | 2.5% | 0.2% | – |
| F6 | 40% | 5% | 2.5% | 0.4% | – |
| F24 | 40% | 5% | 2.5% | – | 0.2% |
| F26 | 40% | 5% | 2.5% | – | 0.4% |

*Concentration percentage measured based on weight of volume.

a fabric and the resilience to bending or wrinkle resistance, respectively. Three samples were mounted on the machine subsequently to measure the bending property of the test specimens in both warp and weft directions. Readings were recorded with an average of six measurements.

**2.4.4. Compressional properties** The compression property of test specimens was evaluated by Automatic Compression Tester, FB3-A instrument of KES-F. In the compression test, compression properties such as compressional linearity, compression energy and compressional resilience were measured. A sample was mounted on the machine and the procedure started when the sensor was scanning a certain part of the fabric surface under a constant speed. Three distinct points were measured automatically by the machine. All measurements were repeated for three distinct points on the fabrics and averaged with an analytical tolerance limited to 5%.

**2.4.5. Surface properties** Automatic Surface Tester, FB4 instrument of KES-F, measures surface properties, including the coefficient of friction and geometrical roughness. The data obtained from this machine have a good correlation with human hand fingers. Two samples were measured in both the weft and warp directions. The samples were mounted on the machine and the procedure started when the sensor was scanning a certain part of the fabric surface under constant speed. All measurements were repeated for three distinct points on the fabrics in both the warp and weft directions and averaged with analytical tolerance limited to 5%.

## 3. Results and discussion

The mechanical strength of cotton specimens has been studied in previous research [13, 14]. The reduction of mechanical strength of FR-treated specimens was mainly attributed to the strong acidity of the finishing bath and high temperature curing, which tendered the fabric strength. Once the fabrics were pre-treated by plasma gas, the mechanical strength of the specimens increased slightly because of the enhancement of inter-yarn and inter-fiber friction. The $TiO_2$ or nano-$TiO_2$ co-catalyst might compensate for the reduction in tensile and tearing strength caused by FR agents. On the other hand, texture denotes what people feel when they touch an object. It refers to the properties and sensations felt when touching the external surface of the object. Handle measurement is based on the idea of replacing the conventional subjective judgment of the physical properties of objects with objective data. In the present paper, KES-F was used to determine the properties of tensile, shearing, bending, compression, and surface friction and variation. The results, as shown in Table 2, obtained for the cotton fabrics subjected to plasma pre-treatment and/or flame-retardant treatment in the presence of $TiO_2$/nano-$TiO_2$, are discussed in the following sections.

### 3.1. Tensile properties

Apparel usually undergoes small extension and relaxation during wear. Hence, the study of tensile properties of fabrics, including tensile energy (WT), tensile resilience (RT) and extensibility (EMT), is becoming increasingly important. In general, fabrics with higher WT, RT and EMT values possess better tensile strength. The results of these tensile properties are presented in Table 2. WT and EMT were highly correlated, as shown in Table 2; values for the control specimen were 15.10 gf cm/cm$^2$ and 9.14%, respectively, showing a significant drop after FR treatment. When the fabric was treated with FR-CL (F1 specimen), the WT and EMT values decreased, because the fabric was tendered by acid at low pH during treatment, i.e., pH 5, resulting in poor tensile strength and toughness. In addition, a further decrease in WT and EMT values was also observed in the F2 specimen. A PA catalyst was added to the treatment to enhance the FR performance of the treated fabrics, while the extremely low pH, i.e., pH 1–2, severely damaged the specimens.

Previous investigations have proved that $TiO_2$/nano-$TiO_2$ in the FR-CL-PA treatment could improve the effectiveness of FR treatment [13, 14]. The results showed that the addition of 0.2–0.4% $TiO_2$ or the nano-$TiO_2$ co-catalyst might compensate the reduction in tensile breaking strength caused by the finishing agents. This was probably due to the fact that the metal oxide co-catalytic effect dominated over the acid hydrolysis process [14].

WT is the energy required for extending a fabric of a specific length without damaging it. The value indicates the toughness of the fabric, which reflects the mobility of the garment under deformation [24–26]. On the other hand, EMT is determined as the percentage extension at the maximum applied load of 500 gf/cm of the specimen's width, which has a good correlation with the fabric handle. As demonstrated in Table 2, the WT and EMT values of F4, F6, F24 and F26 specimens were slightly lower when compared with the F2 specimen. The results revealed that the FR-CL-PA-$TiO_2$ and FR-CL-PA-nano-$TiO_2$ treated specimens required less energy to extend the fabric without breaking the specimen and even with poor stretch. The diminished values of WT and EMT might be attributed to the second padding process of $TiO_2$/nano-$TiO_2$ solution, as well as drying at 110°C for 5 min. Finishing was an extremely complex subject, as there were a large number of changes in fabric properties during a sequence of finishing processes [27, 28]. Some finishing operations, comprising the use of pressure and heat, might deplete the fabric strength. After comparing with the F2 specimen, it was concluded that FR-CL-PA-$TiO_2$ and FR-CL-PA-nano-$TiO_2$ treated specimens needed less tensile energy to stretch the fabric to a certain extent, but they required higher force to damage the fabric by stretching.

Moreover, the WT and EMT values of the fabric were slightly enhanced to 7.3% and 4.6%, respectively, by the plasma treatment. Plasma treatment could not alter the fabric structure, as it only etched the fabric surface [29–31]. The roughening effect created a larger contact area within the fibers and yarns microscopically, resulting in the enhancement of inter-yarn and inter-fiber friction [29, 31, 32]. The increased WT values were probably due to the greater cohesive force developed during extension. On the other hand, the increased EMT values might be due to the rise of interaction force between fibers and yarns, leading to a slight reduction

**Table 2** Different properties of the treated fabric as measured by the KES-F system.

| Sample | Neutralization (min) | Tensile | | | Shearing | | | Bending | | Compression | | | | | Surface | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | WT | RT | EMT | G | 2HG | 2HG5 | B | 2HB | Tm | To | LC | WC | RC | MIU | SMD |
| Control | 0 | 15.100 | 38.590 | 9.140 | 2.810 | 5.380 | 7.840 | 0.103 | 0.113 | 0.637 | 0.975 | 0.357 | 0.301 | 36.403 | 0.202 | 5.483 |
| F1 | 0 | 9.865 | 59.289 | 5.609 | 3.498 | 4.693 | 7.470 | 0.119 | 0.162 | 0.498 | 0.724 | 0.291 | 0.187 | 32.067 | 0.238 | 6.010 |
| F2 | 0 | 9.251 | 62.230 | 5.431 | 4.013 | 3.585 | 6.760 | 0.138 | 0.178 | 0.487 | 0.703 | 0.293 | 0.158 | 36.087 | 0.243 | 7.170 |
| F4 | 0 | 8.203 | 58.277 | 4.687 | 4.158 | 5.375 | 9.138 | 0.155 | 0.188 | 0.539 | 0.850 | 0.324 | 0.267 | 35.793 | 0.274 | 5.610 |
| F6 | 0 | 8.243 | 59.916 | 4.706 | 4.290 | 5.423 | 9.283 | 0.164 | 0.192 | 0.545 | 0.847 | 0.315 | 0.257 | 35.000 | 0.281 | 5.690 |
| F24 | 0 | 8.180 | 54.172 | 4.071 | 7.013 | 10.415 | 19.015 | 0.172 | 0.197 | 0.545 | 0.876 | 0.301 | 0.248 | 36.927 | 0.280 | 4.589 |
| F26 | 0 | 8.366 | 54.697 | 4.215 | 8.133 | 12.520 | 19.723 | 0.188 | 0.210 | 0.544 | 0.868 | 0.312 | 0.252 | 35.937 | 0.302 | 4.186 |
| Plasma only | 0 | 16.200 | 42.350 | 9.560 | 2.910 | 6.783 | 9.410 | 0.114 | 0.146 | 0.524 | 0.841 | 0.297 | 0.236 | 35.370 | 0.242 | 5.954 |
| Plasma-F1 | 0 | 9.641 | 60.118 | 6.544 | 3.876 | 6.644 | 9.203 | 0.122 | 0.186 | 0.507 | 0.798 | 0.308 | 0.235 | 31.793 | 0.284 | 6.025 |
| Plasma-F2 | 0 | 8.629 | 65.390 | 5.905 | 4.298 | 6.020 | 8.530 | 0.148 | 0.191 | 0.502 | 0.789 | 0.319 | 0.228 | 35.177 | 0.278 | 6.564 |
| Plasma-F4 | 0 | 8.494 | 53.402 | 4.529 | 4.786 | 6.853 | 8.084 | 0.152 | 0.167 | 0.542 | 0.809 | 0.337 | 0.258 | 35.573 | 0.295 | 5.493 |
| Plasma-F6 | 0 | 8.191 | 53.792 | 4.261 | 5.231 | 8.418 | 9.186 | 0.158 | 0.168 | 0.530 | 0.824 | 0.341 | 0.250 | 34.197 | 0.294 | 4.788 |
| Plasma-F24 | 0 | 8.351 | 55.344 | 4.162 | 7.660 | 11.073 | 18.850 | 0.168 | 0.181 | 0.550 | 0.856 | 0.333 | 0.285 | 34.223 | 0.315 | 6.183 |
| Plasma-F26 | 0 | 8.066 | 55.079 | 4.861 | 8.708 | 14.373 | 19.620 | 0.188 | 0.185 | 0.544 | 0.846 | 0.334 | 0.268 | 35.880 | 0.319 | 5.473 |
| F1 | 15 | 13.578 | 46.955 | 8.020 | 2.821 | 5.492 | 8.517 | 0.106 | 0.117 | 0.581 | 1.040 | 0.341 | 0.391 | 30.763 | 0.194 | 4.948 |
| F2 | 15 | 10.112 | 53.935 | 5.953 | 3.059 | 7.415 | 9.156 | 0.133 | 0.142 | 0.571 | 0.949 | 0.377 | 0.384 | 37.120 | 0.182 | 4.936 |
| F4 | 15 | 10.303 | 50.708 | 5.770 | 3.258 | 8.200 | 9.686 | 0.151 | 0.151 | 0.571 | 0.993 | 0.360 | 0.380 | 33.440 | 0.211 | 6.063 |
| F6 | 15 | 10.877 | 49.488 | 6.175 | 3.721 | 7.983 | 9.002 | 0.141 | 0.152 | 0.583 | 0.959 | 0.365 | 0.343 | 34.440 | 0.188 | 5.733 |
| F24 | 15 | 10.350 | 43.445 | 6.298 | 5.553 | 12.568 | 22.423 | 0.154 | 0.171 | 0.584 | 0.998 | 0.353 | 0.364 | 35.663 | 0.184 | 5.999 |
| F26 | 15 | 10.000 | 43.503 | 6.050 | 5.805 | 12.978 | 23.083 | 0.140 | 0.170 | 0.579 | 0.928 | 0.368 | 0.320 | 33.717 | 0.193 | 6.078 |
| Plasma-F1 | 15 | 13.486 | 43.823 | 7.984 | 2.831 | 5.759 | 8.654 | 0.086 | 0.101 | 0.596 | 0.978 | 0.362 | 0.344 | 34.720 | 0.255 | 6.060 |
| Plasma-F2 | 15 | 9.467 | 53.186 | 6.826 | 3.491 | 6.700 | 9.766 | 0.104 | 0.112 | 0.583 | 0.994 | 0.374 | 0.385 | 36.650 | 0.240 | 5.015 |
| Plasma-F4 | 15 | 10.079 | 51.771 | 5.902 | 3.588 | 8.552 | 9.738 | 0.116 | 0.139 | 0.573 | 0.910 | 0.366 | 0.308 | 35.780 | 0.254 | 5.871 |
| Plasma-F6 | 15 | 9.975 | 51.040 | 5.770 | 3.889 | 8.405 | 9.615 | 0.122 | 0.149 | 0.574 | 0.908 | 0.364 | 0.303 | 35.127 | 0.219 | 4.906 |
| Plasma-F24 | 15 | 10.041 | 51.692 | 5.966 | 3.668 | 7.703 | 9.883 | 0.111 | 0.123 | 0.565 | 0.949 | 0.340 | 0.326 | 36.080 | 0.247 | 4.949 |
| Plasma-F26 | 15 | 10.015 | 51.256 | 5.920 | 3.845 | 7.590 | 9.325 | 0.118 | 0.134 | 0.568 | 0.967 | 0.348 | 0.327 | 37.273 | 0.267 | 5.145 |
| F1 | 30 | 13.575 | 46.088 | 8.214 | 3.107 | 5.573 | 8.519 | 0.101 | 0.110 | 0.602 | 1.055 | 0.393 | 0.444 | 31.480 | 0.231 | 5.608 |
| F2 | 30 | 10.503 | 54.628 | 6.418 | 3.230 | 7.347 | 9.406 | 0.135 | 0.147 | 0.566 | 0.996 | 0.347 | 0.391 | 35.277 | 0.225 | 4.726 |
| F4 | 30 | 10.902 | 49.274 | 6.157 | 3.463 | 7.520 | 9.567 | 0.142 | 0.152 | 0.555 | 0.958 | 0.346 | 0.349 | 36.113 | 0.230 | 5.996 |
| F6 | 30 | 10.975 | 50.260 | 6.162 | 3.741 | 7.971 | 9.346 | 0.148 | 0.170 | 0.578 | 0.918 | 0.377 | 0.321 | 36.627 | 0.212 | 5.691 |
| F24 | 30 | 10.538 | 44.858 | 6.238 | 5.403 | 12.103 | 21.820 | 0.143 | 0.165 | 0.576 | 0.941 | 0.328 | 0.298 | 34.720 | 0.200 | 6.518 |
| F26 | 30 | 10.950 | 44.483 | 6.173 | 5.395 | 12.528 | 21.935 | 0.144 | 0.169 | 0.579 | 0.996 | 0.342 | 0.355 | 35.263 | 0.198 | 6.111 |
| Plasma-F1 | 30 | 13.550 | 43.145 | 8.028 | 2.951 | 6.052 | 8.948 | 0.091 | 0.104 | 0.601 | 0.988 | 0.391 | 0.378 | 32.380 | 0.220 | 6.001 |
| Plasma-F2 | 30 | 9.628 | 52.599 | 6.795 | 3.506 | 6.971 | 9.446 | 0.106 | 0.110 | 0.591 | 1.081 | 0.346 | 0.422 | 35.553 | 0.220 | 5.768 |
| Plasma-F4 | 30 | 10.316 | 48.731 | 5.813 | 3.791 | 8.041 | 9.937 | 0.128 | 0.150 | 0.573 | 0.889 | 0.370 | 0.291 | 36.050 | 0.230 | 4.685 |
| Plasma-F6 | 30 | 10.275 | 48.229 | 6.050 | 3.690 | 7.878 | 9.930 | 0.119 | 0.130 | 0.562 | 0.933 | 0.350 | 0.324 | 35.457 | 0.207 | 5.598 |
| Plasma-F24 | 30 | 10.428 | 51.835 | 6.231 | 3.503 | 7.508 | 10.145 | 0.114 | 0.137 | 0.579 | 1.050 | 0.342 | 0.361 | 34.640 | 0.262 | 5.026 |
| Plasma-F26 | 30 | 10.642 | 51.175 | 6.473 | 3.485 | 7.753 | 10.283 | 0.111 | 0.125 | 0.579 | 1.014 | 0.347 | 0.378 | 34.780 | 0.271 | 5.565 |

in relative movement of fibers and yarns during extension. However, when the plasma pre-treated specimen was subjected to the FR treatment, the positive effect in WT and EMT values was negligible. The FR treatment used water as the processing medium, reducing the inter-yarn and inter-fiber friction.

Neutralization of the FR-treated specimens was essential to remove the unfixed acid on the fabric surface. Table 2 shows that the WT values of the FR-treated specimens and plasma-FR-treated specimens, increased by the range of 9.3–37.6% and 9.7–39.9%, respectively, after 15 min of neutralization with 30 g/l sodium carbonate at 50°C. Similarly, Table 2 shows that the EMT values of the FR-treated and plasma-FR-treated specimens increased by the range of 9.6–54.7% and 15.6–43.4%, respectively, after 15 min of neutralization. The enhancement of both WT and EMT values became even greater if 30 min of neutralization was carried out. The result thus proved that the acidic FR finishing did weaken the fabrics.

In addition, Table 2 shows that the RT value of the control sample (38.59%), was the lowest, indicating that there was an obvious increase in the RT value after the FR treatment. In general, CL functioned as a binder to form a linkage between the treated cotton cellulose, and also acted as a nitrogen provider to enhance the FR performance of the treated cotton fabrics [4, 33]. Consequently, more FR molecules could adhere to cotton through the CL crosslinking bridges [2, 31]. It was known that the cross-linked structure imparted elasticity to the fabric [34]. The results showed that the finishing agents decreased both the WT and EMT values, but increased the RT values, contributing to the reduction of tensile strength and a slight improvement of elastic properties. Moreover, the RT values of the F4, F6, F24 and F26 specimens were slightly reduced when compared with the F2 specimen, as shown in

Table 2. The decrease in RT values was due to the second padding with $TiO_2$/nano-$TiO_2$ solution followed by a drying process.

The RT values were increased by 9.7% after plasma treatment, as shown in Table 2. This was probably due to the removal of fuzz and tangled fibrils, giving the fabric a greater ability to recover from stretching. When the plasma pre-treated specimen was subjected to the FR treatment, surface fibrils re-developed during the wet padding process. Although neutralization of the FR-treated specimens helped the removal of the unfixed acid present on the fabric surface, it still facilitated the formation of tangled fibrils during wet processing. Wet treatment could swell the fibers, resulting in fabric shrinkage. In general, cotton is chemically linked by many hydroxyl groups and structural units of the cellulose orients into three different regions, namely the crystalline region, the amorphous region and an intermediate region between them. The chains in amorphous and intermediate regions are temporarily held together by weak hydrogen bonds and are easily broken [35]. The Neutralization process broke the hydrogen bonds formed between the fibers when in the swollen state, and allowed the yarn to collapse inward, thereby reducing yarn diameter and facilitating shrinkage [36, 37]. Hence, it became difficult for the fabric to recover to its original shape after shrinkage, as well as after the release of the applied tensile stress, i.e., the drop of RT value as shown in Table 2. In addition, Table 2 shows that the flame-retardant-treated specimens and plasma-flame-retardant-treated specimens had similar RT values after 15 min and 30 min of neutralization at 50°C.

### 3.2. Shearing properties

Shear is a significant determinant of the handle and drape of fabrics. A study of the shearing properties of a fabric includes the shear stiffness (G) and shear stress at 0.5° (2HG) and 5° (2HG5). In theory, fabrics with a low G, 2HG and 2HG5 values, have superior shearing properties. G is defined as the ease with which fibers slide against each other, resulting in a pliable or stiff structure. Table 2 indicates that the G value of the control specimen was the lowest one, i.e., 2.81 gf/cm degree, which would increase slightly after plasma treatment. An increase in G value indicated the enhancement of the subjective stiffness of the fabric. This primarily depended on yarn interaction, i.e., an increase in yarn interaction would normally increase shear rigidity, and was perhaps related to the smoothing effect [26, 38]. The results shown in Table 2 indicated the greatly increased inter-yarn friction in the plasma-treated fabrics, as well as the increased number of fiber contacts at yarn crossover points, generating inter-yarn pressure. Moreover, Table 2 illustrates that there was a sudden increase in the G values of the F1 specimen after the FR treatment of cotton fabrics. It was evident that the cross-linked structure and acidic treatment make the test specimens stiffer.

Previous morphological studies have shown that metal oxide particles are attached to cotton fabric during the padding process, but they are unevenly distributed on the fiber surface with various sizes [13, 14]. The results also showed that the fabric surface was somewhat rough and uneven, with a certain degree of agglomeration of particles. This could possibly be attributed to the surface attraction between small particles [13, 14]. Table 2 demonstrates that the enhancement of G values was obvious when the $TiO_2$/nano-$TiO_2$ particles were added in the treatment. These particles could promote the crosslinking reaction, thereby causing more severe rigidity. As shown in Table 2, the effectiveness of FR treatment on the cotton specimens increased in the order of 0.2% $TiO_2$, 0.4% $TiO_2$, 0.2% nano-$TiO_2$ and 0.4% nano-$TiO_2$. The results revealed that the co-catalytic effect in the crosslinking reaction was enhanced with respect to the increase in the concentration of metal oxides and decrease in the size of metal oxides. Therefore, it was proved that the selection of catalyst for the FR finishing formulation apparently played an important role, not only influencing the binding of chemicals to cotton for effective flame-retardancy [13], but also affecting the shear rigidity of fabric.

Furthermore, Table 2 shows that the rigidity effect was even worse for the plasma pre-treated cotton specimens with FR treatment, i.e., further increases in G values. This phenomenon was attributed to the fact that the etching effect on the fabric surface was caused by the plasma pre-treatment which led to a roughening of the fabric surface. As a result, the etched fabric provided a new pathway for the finishing agent to enter into the fibers, leading to more effective FR treatment. Furthermore, the increased wettability of cotton fibers might also facilitate the absorption of FR chemicals. The neutralization process helped to minimize the shear rigid effect by removing excessive acid from the fabrics. In addition, the wet process might remove the unattached metal oxides present on the fabric surface, resulting in decreased surface friction, as well as making the fabric less resistant to shear stress. Table 2 clearly shows that the G values of the FR-treated specimens, and plasma-FR-treated specimens, decreased to the range of 13.3–28.6% and 18.8–55.8%, respectively, after 15 min of neutralization with 30 g/l sodium carbonate at 50°C. A remarkable decrease in the G values of the plasma-F24 and plasma-F26 specimens was observed after neutralization. In general, the G value highly depends on the mobility of cross yarns at the intersection point. It is thus related to the fabric weave, yarn diameter and fabric surface characteristics, and perhaps associated with the smoothing effect [24, 26, 38]. The plasma-etched fabric surface is usually covered with very small nano-$TiO_2$ particles. When the excess nano-$TiO_2$ particles were washed out by the neutralization process, the fabric became smooth again. A softer and smooth material was thus less resistant to shearing, i.e., the G value decreased. Table 2 also shows that the decrease in G values was similar to that following 15 min and 30 min of neutralization.

Apart from G, both 2HG and 2HG5 also determine the shearing properties of fabrics. The 2HG and 2HG5 values of the control specimen were 5.38 gf/cm and 7.84 gf/cm, respectively, as presented in Table 2. These values slightly decreased for the F1 specimen after the FR-CL treatment, due to the formation of CL crosslinking bridges which imparted an elastic property [34]. Better 2HG and 2HG5 values were even obtained in the F2 specimen because of the effect of

the PA catalyst. However, when $TiO_2$ particles were added to the treatment, some of the residing $TiO_2$ particles between the fibers or adhering to the fibers would obstruct the fabrics from reverting to the original state after shear stress was applied at both 0.5° and 5°. As shown in Table 2, the 2HG and 2HG5 values of the F24 and F26 specimens were significantly increased after the addition of nano-$TiO_2$ particles in the FR treatment. The nano-$TiO_2$ particles filled up the space between the fibers, due to their small size, although some agglomerated to form large particles with irregular shapes. In addition, some of the nano-$TiO_2$ particles still remained on the fabric surface, while others partitioned the fabrics, thereby obstructing the restoration of fabric to its original state after the application of shear stress. The greater the number of particles between the fibers, the worse the recovering ability of the fabric, i.e., the 2HG and 2HG5 values of the F26 specimen were higher than those of the F24 specimen. Table 2 also indicates that the 2HG and 2HG5 values of the plasma-treated cotton fabrics were increased slightly when compared with the control specimen, which was probably due to the increased inter-yarn friction and number of fiber contacts. These two values were further augmented when the plasma pre-treated specimens were subjected to FR treatment.

Neutralization minimized the shear rigid effect by removing excess acid from the fabrics and consequently decreased the recovering ability of fabric. The result was highly correlated with RT. Neutralization of the FR-treated specimens resulted in fabric shrinkage, which made the fabric more difficult to restore to its original shape after releasing the applied tensile stress and shear stress. However, in the catalyzed FR-CL-PA treatment, the mobility of cellulose macromolecules was limited by CL [39]. As a result, there was only a slight decrease in RT and a mild increase in both 2HG and 2HG5, as shown in Table 2. In addition, Table 2 also demonstrates that the FR-treated and plasma-FR-treated specimens showed similar RT, 2HG and 2HG5 values after 15 min or 30 min of neutralization at 50°C. Nevertheless, a significant loss of the 2HG and 2HG5 values was observed in the plasma-F24 and plasma-F26 specimens, as presented in Table 2. Since the neutralization process could shrink the fabrics, both the plasma-F24 and plasma-F26 specimens had inferior recovering abilities. However, the plasma-etched fabric surface was filled up with only very small nano-$TiO_2$ particles, which were considered to be the dominant cause for lower 2HG and 2HG5 values. There was still room for movement in the amorphous regions and the ability to restore the original shape after the shear stress was removed. Therefore, the plasma-F24 and plasma-F26 specimens showed results similar to the plasma-F4 and plasma-F6 specimen after neutralization.

### 3.3. Bending properties

Bending properties of a fabric include bending rigidity (B) and bending moment (2HB). Both B and 2HB depend on the bending resistance properties of, and the friction between, fibers and yarns, as well as the fabric structure, which may enhance dramatically when the fabric thickness increases [40]. In general, the fabric with low B and 2HB values has excellent bending properties. Changes in the B values were similar to the changes in G values after treatment with different formulations of FR treatment, when comparing the results shown in Table 2. Table 2 indicates that: (i) the B value of the control specimen increased originally from 0.103 gf $cm^2$/cm to 11.0% after plasma treatment, (ii) there was an obvious increase in the B value of the F1 specimen and a further increased B value of the F2 specimen, when compared with the control fabric, (iii) the B values were further increased in the ascending order of 0.2% $TiO_2$, 0.4% $TiO_2$, 0.2% nano-$TiO_2$ and 0.4% nano-$TiO_2$, when $TiO_2$/nano-$TiO_2$ particles were added to the treatment formulation, when compared with the F2 specimens, and (iv) the rigid effect of the plasma pre-treated specimens subjected to FR treatment was even worse. The results obtained were consistent with the G values. When compared, the pattern of the B and G values of specimens after neutralization for 15 min and 30 min was similar, as illustrated in Table 2. The neutralization process helped to minimize both the shear and bending rigid effects by removing excessive acid and unattached metal oxides from the fabric surface, i.e., a diminution in the B and G values. A softer and smooth material was thus obtained from the neutralization of the FR-treated specimens.

Although 2HG, 2HG5 and 2HB refer to the recovering ability of a material, 2HB still shows quite a different pattern when compared with 2HG or 2HG5, as shown in Table 2. This might be attributed to different force or momentum applied to deform the material. Shear stress is defined as a stress applied parallel or tangential to a force of a fabric as opposed to a normal stress that is applied perpendicular. By contrast, bending characterizes the behavior of a fabric subjected to an external load which is applied perpendicular to a longitudinal axis of the fabric.

The 2HB value of the control specimen shown in Table 2 was 0.11 gf.cm/cm and was slightly increased after FR treatment. It was assumed that the CL imparted an elastic property to the fabrics. However, the low pH reaction medium weakened the fabrics, leading to a poor recovering ability upon bending at the curvatures from 2.5 $cm^{-1}$ to -2.5 $cm^{-1}$. Therefore, the 2HB value of the F2 specimen was larger than the F1 specimen. In addition, the $TiO_2$/nano-$TiO_2$ particles present between the fibers, or on the fiber surface, impeded the restoration of fabric to its original state after being bent. In addition, Table 2 indicates that the 2HB values of all the plasma-treated cotton fabrics were greatly increased as compared with the control specimen, probably contributed to by the increased inter-yarn friction, the number of fiber contacts at yarn crossover points and inter-yarn pressure. By contrast, the 2HB values of the plasma pre-treated specimens with FR treatment were also increased accordingly.

It was previously suggested that the neutralization of the FR-treated specimens would develop tangled fibrils, apart from causing fabric shrinkage during the wet processing (it was difficult for the fabrics to recover to their original shape after the applied shear stress was increased). Nevertheless, an opposite result of 2HB values was observed, as presented in Table 2, i.e., the 2HB values of both the neutralized FR-treated specimens and the neutralized plasma-FR-treated specimens

decreased obviously. In general, after minimizing the problem of low pH in specimens, caused by neutralization, such a result was highly related to the force or momentum applied. A small tangential movement of a material which occurred after the application of shear stress, might cause the entanglement of fuzzy fibrils, resulting in an irreversible recovery. On the other hand, bending deformed the fabric perpendicular to the longitudinal axis of the fabric. The fuzzy or tangled surface fibrils were pulled outward and the natural fiber crimp provided an elastic characteristic to restore fibers to their original shape. Therefore, the overall 2HB values decreased proportionally after neutralization for 15 min and 30 min, as shown in Table 2. When comparing the plasma-F4 and plasma-F6 specimens with the plasma-F24 and plasma F26 specimens after neutralization, it was found that the 2HB values of those specimens treated with nano-$TiO_2$ were lower than those treated with $TiO_2$. This was probably due to the fact that the smaller particles of nano-$TiO_2$ were able to fill up the gaps present in the plasma-etched fabric surface, while the larger particles of $TiO_2$ were removed during neutralization. There was less obstruction to hinder the fabrics from recovering after bending, but it was not the case when $TiO_2$ was used.

### 3.4. Compression properties

Compression properties of the cotton specimens, such as fabric thickness with a pressure of 0.5 gf/cm$^2$ ($T_o$) and 50 gf/cm$^2$ ($T_m$), compressional linearity (LC), compressional energy (WC) and compressional resilience (RC) were measured at three distinct points of the specimens. Fabric thickness always changes upon any physical or chemical treatment, and is measured as $T_o$ and $T_m$, representing the surface and intrinsic thickness, respectively. Table 2 indicates that the $T_o$ and $T_m$ of the control fabric were 0.98 mm and 0.64 mm, respectively, but they subsequently decreased after FR treatment. In the case of F1 and F2 specimens, they were dipped and padded with the FR agents, i.e., FR-CL or FR-CL-PA. Both the compression in the padding process and the natural fabric shrinkage in a wet condition competed with each other, resulting in changing the fabric thickness. The results showed that there was a reduction in the fabric surface and intrinsic thicknesses of the F1 and F2 specimens within the range of 21.8–27.9%. It was believed that the main contribution to the reduction was the pressure generated between the two padding rollers. The F4, F6, F24 and F26 specimens were dipped and padded twice with the FR agents, and then with the metal oxide solutions. It was believed that there would be a further reduction in the fabric thickness due to double compression applied to the fabrics during padding. The fabric shrinkage dominated over the pressure applied to the fabrics, thus the $T_o$ and $T_m$ of the F4, F6, F24 and F26 specimens were higher than those of the F1 and F2 specimens, but still lower than those of the control specimen. Moreover, the etching effect of plasma pre-treatment removed the fuzzy fibrils present on the fabric surface resulting in a decrease of $T_o$ and $T_m$. It was obvious that the $T_o$ and $T_m$ of the plasma-F1 and plasma-F2 specimens were reduced further. The fabric shrinkage of the plasma-FR-CL-PA-treated specimens undergoing the second padding process of metal oxide solutions dominated over the etching effect generated from plasma pre-treatment and the compression effect created by padding, resulting in the formation of dense fabric with higher $T_o$ and $T_m$ values.

Table 2 also shows that there was a significant increase in the $T_o$ and $T_m$ of the neutralized FR-treated specimens and plasma-FR-treated specimens. By contrast, neutralization produced tangled fibrils during wet processing and at the same time, fibers swelled, resulting in fabric shrinkage. As shown in Table 2, the $T_o$ of all neutralized specimens increased inconsistently due to the random formation of fuzzy fibrils, causing an uneven bulkier fabric. However, when the sensory device was pressed on the bulked surface at 50 gf/cm$^2$, the $T_m$ values of all neutralized specimens increased consistently, to a range of 0.56–0.60 mm, as shown in Table 2. This could be due to the fact that wet treatment of fabric might lead to fabric shrinkage, thereby enhancing the intrinsic thickness of fabric. The results further confirmed the inferences about the drop of RT and enhancement of 2HG and 2HG5 as mentioned in Section 3.1 and 3.2, respectively. Furthermore, Table 2 demonstrates that both the FR-treated specimens and the plasma-FR-treated specimens showed similar $T_m$ values after 15 min or 30 min of neutralization at 50°C.

Apart from measuring the fabric thickness, the machine also measures the LC, WC and RC of fabrics. It was suggested that fabric with good compression properties usually possessed higher LC, WC and RC values. In general, LC determines the compressibility along with the change in fabric thickness, while WC represents the fluffy feeling of a fabric. Table 2 shows that the LC value of the control fabric was 0.357. However, after the FR treatment, there was a downward tendency of the LC values, especially when no $TiO_2$/nano-$TiO_2$ particles were added to the treatment. By contrast, Table 2 demonstrates that the WC value of the control fabric was 0.301 gf.cm/cm$^2$, but it was also reduced after FR treatment, especially without the presence of a $TiO_2$/nano-$TiO_2$ co-catalyst in the treatment recipe. It was obvious that both the dipping and padding processes could change the fabric thickness, as well as the compressibility of the specimens. There was a drop in the LC and WC values of both the F1 and F2 specimens in a range of 18.0–18.4% and 37.8–47.5%, respectively, as shown in Table 2. This was attributed to the fact that the F1 and F2 specimens were compressed following squeezing between two rolling padders, which required higher energy for compressing a dense fabric, leading to a poor compressibility property. Besides, the dipping and padding of F4, F6, F24 and F26 specimens twice would create fuzzy fibrils on the fabric surface. It was believed that the fluffy fabric could act as a sponge-like structure with a high compressibility. However, the LC and WC values of these specimens were still lower than those of the control specimen as the padding process so applied twice still compressed the fabrics.

Moreover, the plasma surface modification pre-treatment could remove fuzzy fibrils on the fabric surface resulting in lower LC and WC values. However, the effect was overcome by the post-dipping and post-padding processes, as the surface-raising of cotton fabric during the post-treatment could provide a highly compressible structure. In addition,

Table 2 shows that there was a significant increase in the compressibility of the neutralized FR-treated and plasma-FR-treated specimens. The inconsistent enhancement of the LC and WC values might be indicative of random development of the tangled fibrils after neutralization. Table 2 also demonstrates that both the FR-treated specimens and the plasma-FR-treated specimens showed an inconsistent enhancement of LC and WC values after 15 min and 30 min of neutralization.

RC refers to the percentage of energy recovery, but it is totally different from RT, 2HG and 2HG5, as well as 2HB, due to distinct deformation directions and the force applied. RC measures the percentage of energy recovery from the lateral compression deformation, indicating the recoverability of a fabric after the compression force is removed. The RC value of the control specimen was 36.40%, but this decreased after FR treatment, as reflected particularly by the case of the F1 specimen. Although CL functioned as a binder to form a linkage between the treated cotton cellulose and the cross-linked structure, in order to impart an elastic property [34], there was an obvious reduction in the RC values of the F1 specimen. This was probably due to the fact that CL could not effectively form linkages with cotton in the absence of a PA catalyst. The situation became even worse when the FR-CL-treated specimen demonstrated a less spongy-like fabric, due to the attack of acid during the reaction. Hence, the compressional recovering ability of the FR-CL-treated specimen was reduced. On the other hand, the effectiveness of the FR-CL reaction in forming a cross-linked structure was further enhanced by the catalytic reaction. On balancing the attack of acid, there was an enhancement of the recoverability of fabric when the PA catalyst and $TiO_2$/nano-$TiO_2$ co-catalysts were added to the finishing recipes, as shown in Table 2.

Table 2 also shows that the plasma pre-treatment slightly decreased the RC values, implying that the compressibility of the plasma pre-treated fabrics was lower than that of the plasma untreated fabrics. It was evident that the sponge-like structure of the fabric surfaces was removed completely by the plasma pre-treatment. Owing to the absence of the fuzzy fibrils, the recoverability of the fabric decreased slightly after compression. Table 2 illustrates that there was a slight decrease in the recoverability of the plasma pre-treated specimens with FR treatment, when compared with the plasma untreated specimen.

In general, the increase in thickness of the neutralized specimens should create a fuller handle, as illustrated in Table 2. However, neutralization for both 15 min and 30 min of duration did not change the RC values of the specimens with different treatment compositions. RC usually measures the recoverability of fabric from lateral compression deformation. Nevertheless, only an individual or a small group of fibers could resist the compressional force regardless of the fiber network. This phenomenon reflected that the weakening of fibers upon neutralization at 50°C could overcome the fuller handle obtained in changing the RC values. Hence, there was no obvious change in the RC values after neutralization.

### 3.5. Surface friction and variation

Surface properties, including coefficient of friction (MIU) and geometrical roughness (SMD) of the plasma-treated specimens, are shown in Table 2. Surface characteristics of a fabric influence the handle, comfort and aesthetic properties [24]. Generally speaking, fabrics with low MIU and SMD values have better surface properties. As illustrated in Table 2, there was a treatment increase in the MIU value of the control sample after plasma pre-treatment and FR treatment. In comparison with the control specimen, the increase in MIU values of the F1 and F2 specimens increased from 18.2% to 20.6%, respectively. After the FR-CL or FR-CL-PA treatment of cotton fabric, the attack of strong acidic chemicals did roughen the fabric surface, thereby enhancing the surface friction. The ratio of the force required to slide the surfaces to the force perpendicular to the surface, was further increased when the rough metal oxide particles were added in the FR treatment. The previous morphological studies showed that the metal oxide particles attached to the cotton fabric were unevenly distributed on the fiber surface, with a great variation of particle size [13, 14]. The greater the number of metal oxide particles present on the fabric surface, the rougher the fabrics would be, as reflected by the MIU values of F6>F4 and F26>F24. When compared, the MIU values of the F24 and F26 specimens were slightly higher than those of the F4 and F6 specimens. These results might be attributed to a certain degree of agglomeration of nano-particles, resulting in higher surface attraction between smaller particles. It was obvious that the MIU values increased significantly after the fabric was treated by plasma gas. This was mainly due to the etching effect caused by the bombardment of plasma on cotton specimens [38]. The MIU values of the plasma pre-treated specimens with different FR treatments were also enhanced accordingly.

As mentioned previously, the neutralization process neutralized excess acids and removed unfixed chemicals and unattached metal oxide particles on the fabrics surfaces. The removal of these chemicals or particles decreased the surface friction of the fabric, i.e., lower MIU values. The surface friction of the neutralized specimens was determined mainly by the existence of surface fibrils. Table 2 also shows that the MIU values of those specimens subjected to neutralization for 15 min and 30 min were similar, depending on the development of tangled fibrils during processing.

Table 2 shows the SMD values of different plasma pre-treated specimens, with or without FR treatment, representing the fabric surface evenness. In comparison with the control specimen (SMD=5.48 μm), it was obvious that the SMD values of other specimens increased slightly after FR treatment (SMD=6.01 μm) and plasma treatment (SMD=5.95 μm). The roughened fabric surface of the FR-treated specimen was attributed, mainly, to the development of fuzzy fibrils, due to the tendering of fibers after FR treatment in acidic conditions and the rubbing of fibers in wet processing. On the other hand, the etching effect caused by plasma treatment on cotton specimens, roughened the fabric surface [38]. However, the progressive wet treatment, i.e., the second padding process and

neutralization, continuously produced the fuzzy and tangled fibrils to different extents, causing the SMD values to become trendless, as shown in Table 2. One of the reasons was that all of these measurements were conducted only at three distinct points on the fabrics. Hence, it was noted that the change in SMD values depended highly on the fuzzy and tangled fibrils being developed, while their extent varied.

## 4. Conclusion

KES-F was used to determine the properties of tensile, shearing, bending, compression and surface friction and variation of cotton fabrics undergoing plasma pre-treatment, FR treatment, or both, in the presence of $TiO_2$/nano-$TiO_2$ particles. In general, fabrics with: (i) high tensile properties (WT, RT and EMT), (ii) low shearing properties (G, 2HG and 2HG5), (iii) low bending properties (B and 2HB), (iv) high compressional properties (LC, WC and RC), and (v) low surface friction and variation properties (MIU and SMD), possess excellent fabric handle. In comparison with the control fabric, the specimens after FR treatment had worse tensile, bending, compression and surface friction and properties varied only in respect of improvement in the shearing properties. With the aid of plasma pre-treatment, the tensile and compressional properties of the FR-treated specimens were improved, while the shearing, bending and surface friction properties showed negative effects. In addition, the neutralization process could help neutralize excess acids and remove the unfixed chemicals and metal oxide particles from treated fabric surfaces. The neutralization process improved the fabric handle in some aspects such as WT, EMT, G, B, 2HB, LC, WC and MIU.

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