Evaluation of the Availability of Easy Cationic Dyeable Copolyester Fibers as Electrostatic Flocking Piles

Lifang Liu,^{1,2} Longdi Cheng,^{1,2} Jianyong Yu,^{1,3} Hao Xie^{1,2}

¹The Key Lab of Textile Science and Technology, Ministry of Education, Donghua University, Shanghai 201620, People's Republic of China ²College of Textiles, Donghua University, Shanghai 201620, People's Republic of China

³Modern Textile Institute, Donghua University, Shanghai 200051, People's Republic of China

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ABSTRACT: Easy cationic dyeable copolyester (ECDP) fiber produced by the copolymerization of sulfoisophthalate acid groups and poly(ethylene glycol) with poly(ethylene terephthalate) improves the dyeability compared to regular polyester (PET) fiber; this has resulted in a growing increase in its applications. However, there is little research involving their application in electrostatic flocked fabrics. In this study, we investigated the properties of ECDP fiber, and we discuss their availability for use as electrostatic flocking piles and also compare them to PET fiber. The experimental results show that ECDP fiber possessed a lower crystallinity degree and tensile strength, better conductivity and moisture absorption capability, and higher elongation than PET fiber, mainly because of the copolymerization of sulfoisophthalate acid groups and

INTRODUCTION

Easy cationic dyeable copolyester (ECDP) fiber, a kind of polyester (PET) fiber with modified properties, is produced by the incorporation of an additive containing sulfoisophthalate acid groups (sodium salt of 5sulfoisophthalate acid) and poly(ethylene glycol) into poly(ethylene terephthalate).¹⁻⁴ It possesses the durability, wash-and-wear properties, and excellent performance of PET fiber and also a better dyeability and moisture absorption capability because of the copolymerization with sulfoisophthalate acid groups and poly(ethylene glycol).¹⁻⁹ The added advantages of ECDP fiber have promoted significant interest and increasing applications. However, there is little research involving its application in electrostatic flocking. The objective of this study was to investigate the poly(ethylene glycol). The pretreatment and electrical treatment introduced in this study significantly improved the flocking properties of ECDP and PET piles, especially ECDP piles; they achieved a more pronounced improvement because of their better affinity. With regard to the flocked fabrics, the ECDP flocked fabric had a much higher flock density and a lower weight loss in the wear-resistance test than the PET one and nearly the same good wear resistance as the commercially available polyamide one; this indicated that the ECDP fiber examined in this study could be successfully used to produce flocked fabrics. © 2010 Wiley Periodicals, Inc. J Appl Polym Sci 120: 195–201, 2011

Key words: microstructure; modification; polyesters

possibility of using ECDP fiber for flocked fabric with electrostatic flocking technology.

Electrostatic flocking technology means to flock in electrostatic field; that is, dielectric piles (very short fibers) are polarized by the electric field and aligned with it, and then, the charged piles are preferably propelled toward an adhesive coated substrate (placed on the opposite electrode) by an electrostatic field force.^{10–17} It is also a special surface finish technique with quite a high efficiency and low cost, so the flocked products are widely used in textile products, toys, wallpapers, and so on.¹⁷ When the substrate used is fabric, the flocked product thus produced can be called flocked fabric, which has long been widely used for clothes, sheets, curtains, and so on.^{12,18} There are three key factors within electrostatic flocking technology: the electrostatic field, piles, and an adhesive-coated substrate.^{10,16,17} These three components must be carefully matched to each other for a good quality fabric to be produced; among them, the piles have a particular influence on the appearance of the flocked fabric. Polyamide and rayon fibers are the most commonly used flocking piles at present. Generally speaking, PET fibers have better properties than polyamide and rayon fibers and are much cheaper because of their high yield.¹⁹ However, it is difficult for PET fibers to be used as

Correspondence to: J. Yu (yu_jianyong@163.com).

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Scheme code	Pretreatment		Electrical treatment					
	Amino silicone	Penetrating agent	Sodium silicate	Glacial acetic acid	Antistatic agent	Dispersant agent	Aluminum potassium sulfate	
1	0	0	3	2	0	0	4	
2	6	3	3	2	0	0	4	
3	6	3	3	2	2	0	4	
4	6	3	3	2	2	2	4	
5	6	3	3	2	2	2	2	
6	6	3	3	2	2	2	6	
7	6	3	3	2	2	2	8	
8	6	3	3	2	2	2	10	

TABLE I Treatment Schemes for the PET and ECDP Piles

flocking piles because of their lower conductivity and, more importantly, because of their hydrophobicity, which increases the difficulty in modifying their conductivity. However, ECDP fibers, which have many more polarization groups, which result from the introduction of sulfoisophthalate acid groups and poly(ethylene glycol), have the possibility of being used as flocking piles. Therefore, we investigated the properties of ECDP fibers and validated their availability as flocking piles in this study and compared them with regular PET fibers to verify their superiority.

EXPERIMENTAL

Materials

ECDP filament and PET filament (linear density = 1.11 dtex) were kindly supplied by Defulun Chemical Fiber Co., Ltd. (Shanghai, China). All of the chemicals used in this study were reagent grade and were used without further purification.

Plain cotton fabric (thickness = 0.4 mm, weight = 220 g/m²) was used as the substrate. The fabric was washed, vacuum-dried at 60°C for 4 h, conditioned at 20 \pm 2°C and 65 \pm 3% relative humidity for 24 h, and then cut to 10 \times 10 cm² and evenly coated with adhesive before use.

Modification of the ECDP and PET piles

The ECDP and PET filaments were cut to 1 mm on a ZSQ-3 cutting machine (Automatic Equipment Co., Ltd., Jiaxing, China) and were then subjected to pretreatment and electrical treatment, respectively. The pretreatment was performed to improve the affinity of the piles and was implemented by immersing the piles into the hydrophilic agent solution at 50°C for 15 min with a liquid-to-piles ratio of 20 : 1. The electrical treatment was performed to increase the electrical conductivity of the piles and, thus, to make it possible to move them in the electrostatic field. The piles after pretreatment were electrically treated by immersing them into a mixed solution at 65° C for 60 min with a liquid-topiles ratio of 30: 1. The treatment schemes for the pretreatment and electrical treatment are listed in Table I.

Electrostatic flocking

The flocking machine used in this study was a multifunctional electrostatic flock model CN250 (Jie Yi Printing Materials Co., Ltd., Dongguan, China) designed for flocked fabrics. The schematic illustration of the machine is shown in Figure 1. The flocking distance, that is, the spacing between the upper electrode and the substrate; the field strength; and the flocking time were 70 mm, 60 kV, and 10 s, respectively.

Measurement and characterization

Property analysis of the fibers

The microstructure of the PET and ECDP fibers were obtained by Fourier transform infrared (FTIR) spectra analysis conducted on an FTIR spectrophotometer (NEXUS-670, Thermo Nicolet Instrument Co., Madison, WI) and by wide-angle X-ray diffraction analysis conducted on an X-ray diffractometer (D/Max-2550 PC, Rigaku, Tokyo, Japan) with Cu K α radiation (40 kV and 300 mA) operating at a scanning rate of 10°/ min, respectively.

The tensile properties of the ECDP and PET fibers were determined by a single-fiber test conducted on a LLY-06 tensile tester (Laizhou Electronic Instrument Co., Ltd., Shandong Province, China), with clamps spaced at 100 mm and a strain rate of 100 mm/min.



Figure 1 Diagrammatic sketch of the flocking machine.

Characteristics of the piles

The piles were conditioned at $20 \pm 2^{\circ}$ C and $65 \pm 3^{\circ}$ relative humidity before testing.

Moisture regain. A predetermined amount of conditioned piles was oven-dried at 100°C to a constant weight. The moisture regain of the conditioned piles was expressed by the percentage of water in the dried piles:

$$W = \frac{G - G_1}{G_1} \times 100\%$$
 (1)

where *W* is the moisture regain of the piles (%), *G* is the weight of the conditioned piles (g), and G_1 is the weight of the dried piles (g).

Conductivity. The electrostatic behavior is of the utmost importance for the processing of flocking and, hence, for the properties of the flocked fabric. A high-resistance measuring instrument (DMB 10 conductivity meter, Maag Flockmaschinen GmbH, Gomaringen, Germany) was used to check the conductivity, which was expressed by the specific resistance in this study (Ω g/cm²).

Siftability. A flock siftability tester (RPG 1000, Maag Flockmaschinen GmbH, Gomaringen, Germany) was used to assess the siftability and the dispersable potential of the conditioned piles. A predetermined amount of piles (20 g in this study) was used to fill and then sift through the rotary screen cylinder (rotating at 40 rpm with a sieve gauge of 0.6 mm). The quantity of residual piles in the screen cylinder after 60 revolutions was weighed. The siftability was expressed by the percentage of residual piles:

$$\varepsilon = \frac{G_a}{G_0} \times 100\% \tag{2}$$

where ε is the siftability of the piles (%), G_a is the weight of the residual piles (g), and G_0 is the weight of the initial piles, which was 20 g in this study.

This procedure was repeated three times to obtain an average value.

Rise time. A rise time tester (SPG 1000, Maag Flockmaschinen GmbH, Gomaringen, Germany) was used to assess the piles' behavior in electrostatic field. The distance between the two electrodes was 95 mm, and the field strength was 40 kV. The conditioned piles with a weight of 2.0 g were put onto the underelectrode and then sifted onto the grounded base plate. The time that all piles required to jump from the underelectrode was measured. If no piles jumped from the underelectrode, the test was stopped after 30 s. Three tests were repeated to obtain an average value.

Flock density

Flock density was expressed by the weight of piles on the unit area of the flocked fabric, which was calculated as follows:



Figure 2 FTIR spectra of ECDP fiber and PET fiber. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

$$M = \frac{G - G_0}{A} \tag{3}$$

where *M* is the flock density (g/cm^2) , *G* is the total weight of the flocked sample (g), *G*₀ is the weight of the adhesive coated sample (g), and *A* is the area of the samples, which was 0.01 m² in this study.

Wear resistance

The wear resistance of the flocked fabric was determined by ASTM D 4966 conducted on a Martindale abrasion instrument (YG401G, Ningbo Textile Instrument Factory, Ningbo, China) and was expressed by the weight loss in weight percentage after certain rounds, that is, 4000, 6000, and 10000 rounds in this study.

RESULTS AND DISCUSSION

Characteristics of the ECDP and PET fibers

FTIR spectra

Figure 2 compares the FTIR patterns of the ECDP and PET fibers, showing that the two fibers had similar spectra, with strong bands at 1720, 1244, 1098, and 728 cm⁻¹. The results suggest that both fibers had hydroxyl groups attached to the benzene ring, mainly because of the fact that both of them were PET fibers.

However, the ECDP fiber possessed a stronger band at 755 cm⁻¹, which corresponded to the sulfoisophthalate acid groups, compared to the PET fiber. Moreover, the FTIR spectra of the ECDP fiber tended to shift to a high-frequency direction. This was explained by the fact that the sulfoisophthalate acid group, which caused electrostatic inducement,



Figure 3 X-ray diffraction patterns of ECDP fiber and PET fiber. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

was strongly electronegative. The electrostatic inducement made the electron move toward the middle of the double bonds, increasing the bond force constant and vibration frequency of the C=O bond;⁹ thus, the absorption peaks of the ECDP fiber shifted to a high-frequency direction.

X-ray diffraction

The X-ray diffraction pattern of the ECDP and PET fibers are shown in Figure 3. The ECDP fiber had similar diffraction peaks as the PET fiber. As reported by Pan,²⁰ the distance between the crystal faces of the diffraction peak and the shape of the unit cell were determined by the diffraction angle. Thus, we concluded that the additional components

of the ECDP fiber, the sulfoisophthalate acid groups and poly(ethylene glycol), did not exist in the unit cell but in the amorphous area. Furthermore, the crystallinity degrees of the ECDP and PET fibers were calculated according to Figure 3, as shown in Table II. The ECDP fiber had a crystallinity degree of 48.95%, a little lower than that of the PET fiber (51.46%). This was mainly due to the addition of sulfoisophthalate acid groups, which brought -SO₃Na groups into the ECDP fiber. The high polarity and steric hindrance effect of the sulfoisophthalate acid groups disturbed the regularity of the polymer chain molecules and the internal rotation of the molecule, so as to decrease the crystallization speed and the crystallinity degree and weaken the nucleation capacity. Therefore, the crystallinity degree of the ECDP fiber was lower; this implied that the ECDP fiber had a looser structure than the PET fiber. This was also verified by its better dyeability.^{2,3}

Moisture regain and conductivity

The ECDP fiber had a remarkably higher moisture regain and conductivity than the PET fiber, as shown in Table III. The ECDP fiber was produced by the incorporation of sulfoisophthalate acid groups and poly(ethylene glycol) into the PET fiber. The sulfoisophthalate acid groups and poly(ethylene glycol) had a great affinity to water molecules and could form hydrogen bonds with them; this resulted in a considerable increase in the moisture regain. Furthermore, the lower crystallinity degree and the corresponding increase in the amorphous area also contributed to the higher moisture absorption capability of the ECDP fiber. The conductivity of a textile fiber strongly depends on its microstructure, such as the amount of polar molecules, polymerization degree,

Fibe	er	20	<i>d</i> (nm)	Centroid	Height	Area (Al)	1%	Shape	Skew
PET	1	21.626	4.1058	22.454	1486 (423)	858,114 (231,411)	100.0	4.000p	-0.314
	2	25.483	3.4926	25.716	2177 (428)	365,707 (69,666)	42.6	1.813p	-0.334
	3	44.029	2.0549	45.348	132 (6)	77,453 (6,208)	9.0	4.000p	-0.464
	4	17.388	5.0958	17.167	1438 (231)	213,239 (33,285)	24.8	1.725p	0.361
	5	22.503	3.9479	22.522	1157 (352)	167,385 (50,219)	19.5	4.000p	-0.077
	6	33.502	2.7080	31.844	170 (30)	85,792 (11,095)	10.0	0.500p	0.779
		Amorpho	us peak: tota	l area = 1,767,6	690 (249,391), cry	stallinity = 51.46% (15.6)	5%), fwhm	threshold =	3.0.
ECDP	1	21.454	4.384	21.991	3415 (444)	2,090,312 (244,423)	100.0	4.000p	-0.195
	2	17.653	5.0201	17.329	3808 (203)	585,148 (30,463)	28.0	1.633p	0.498
	3	25.728	3.4598	25.780	5001 (462)	806,401 (71,570)	38.6	1.756p	-0.081
	4	22.617	3.9281	22.677	2662 (431)	357,246 (58,175)	17.1	4.000p	-0.099
	5	32.658	2.7397	32.923	192 (44)	25,677 (6310)	1.2	4.000p	-0.421
	6	44.487	2.0348	44.688	300 (14)	229,749 (21,109)	11.0	4.000p	-0.057
		Amorpho	ous peak: tota	al area $= 4,094,$	533 (263,936), cry	ystallinity = 48.95% (6.54	%), fwhm t	threshold $= 3$	3.0.

TABLE II Calculation of the Crystallinity Degree of the PET and ECDP Fibers

fwhm = full width at half-maximum. d is the interplanar crystal spacing.

TABLE III Conductivity and Moisture Regain of the PET and ECDP Fibers							
Fiber	Moisture regain (%)	Specific resistance $(\Omega \text{ g/cm}^2)$					
PET ECDP	0.49 2.31	$\begin{array}{c} 3.87 \times 10^{13} \\ 8.47 \times 10^{11} \end{array}$					

orientation degree, and crystallinity degree.¹⁹ With regard to the ECDP fiber, the higher conductivity mainly came from the sulfoisophthalate acid groups and poly(ethylene glycol) (polar molecules), lower crystallinity degree, and higher moisture absorption capability.

Tensile properties

Table IV shows the tensile properties of the PET and ECDP fibers. The ECDP fiber had a lower tensile strength and initial modulus but a higher elongation than the PET fiber. As shown in Table IV, the tensile strength and initial modulus of the ECDP fiber decreased by 13.7 and 18.2%, respectively, whereas the elongation increased by 20.5% compared to the PET fiber. This was also due to the addition of sulfoisophthalate acid groups and poly(ethylene glycol) in the ECDP fiber. As illustrated in Table II, the ECDP fiber had a lower crystallinity degree than the PET fiber; this implied that the molecules had a lower bonding force. Therefore, the tensile strength and initial modulus of the ECDP fiber decreased, and the elongation increased.

Characteristics of the ECDP and PET piles

Effects of pretreatment

Table V lists the main characteristics of the ECDP and PET piles after different modifications in term

TABLE IVTensile Properties of the PET and ECDP Fibers

Fiber	Tensile strength	Initial modulus	Elongation
	(cN/dtex)	(cN/dtex)	(%)
PET	3.72	3.23	16.11
ECDP	3.21	2.64	19.41

of flocking properties. The ECDP and PET piles both showed increases in the moisture regain, conductivity, and siftability after treatment with 4 wt % aluminum potassium sulfate, 3 wt % sodium silicate, and 2 wt % glacial acetic acid (treatment scheme 1). However, the rise time, which is an important parameter in evaluating the moving behavior of piles in an electrostatic field, was difficult to measure because no piles jumped from the underelectrode. The possibility of a pile moving in an electrostatic field depends directly on its conductivity.^{16–18} To a certain extent, only piles with better conductivity have the possibility of jumping from the underelectrode. Thus, for the rise time, it made sense only for the piles after further treatments.

After they underwent pretreatment with amino silicone and penetrating agent (treatment scheme 2), the ECDP and PET piles showed an obvious improvement in their flocking properties. This was mainly because amino silicone, with the addition of a penetrating agent, improved the hydrophilicity of the piles and so enhanced their capability to absorb the electrical treatment agents. As shown in Table V, compared to the piles treated with scheme 1, the PET and ECDP piles treated with scheme 2 had obvious increases in their moisture regain and siftability; moreover, they shown an even more significant increase in conductivity by two orders of magnitude. After the treatment, the ECDP piles had a rise time of 27 s; this implied that they could move in the electrostatic field.

 TABLE V

 Characteristics of the PET and ECDP Piles After Different Treatments

Scheme	Moisture regain (%)		Specific resistance (Ω g/cm ²)		Siftability (%)		Rise time (s)	
code	PET	ECDP	PET	ECDP	PET	ECDP	PET	ECDP
0	0.49	2.31	3.87×10^{13}	8.47×10^{11}	96.4	91.2	_a	_a
1	0.57	2.71	2.63×10^{13}	6.12×10^{11}	92.8	88.7	_ ^a	_a
2	0.98	4.80	5.76×10^{11}	1.97×10^{9}	83.5	74.6	_ ^a	27
3	1.12	5.36	1.08×10^{10}	9.24×10^{7}	62.7	49.1	_b	20
4	1.22	5.89	6.74×10^{9}	1.56×10^{7}	50.4	34.2	_b	15
5	1.19	5.78	3.25×10^{11}	9.98×10^{8}	56.1	43.6	_ ^a	26
6	1.26	6.45	1.56×10^{9}	5.89×10^{6}	47.5	32.3	27	8
7	1.23	6.45	1.59×10^{9}	5.96×10^{6}	50.3	33.5	27	8
8	1.23	6.44	3.48×10^{9}	9.88×10^{6}	56.4	37.2	29	9

^a There were no detectable piles moving.

^b Piles could move but could not jump.

Effects of the antistatic agent and dispersant agent

For the PET and ECDP piles treated with treatment schemes 3 and 4, they each showed a considerable improvement in their siftability; this was undoubtedly because of the addition of the antistatic agent and dispersant agent. Furthermore, their moisture regain and conductivity were also enhanced because the treatment agents could easily penetrate into the piles. For the PET piles after modification, they seemed to move but still could not jump for lack of enough energy. However, for the ECDP piles, they jumped quickly and straightly toward the upper electrode, and all of them jumped off within 20 s for treatment scheme 3 and within 15 s for treatment scheme 4.

Effects of the aluminum potassium sulfate concentration

By comparing the piles treated with treatment schemes 4-8 with various concentrations of aluminum potassium sulfate, we found that the conductivity of the PET and ECDP piles were enhanced with increasing aluminum potassium sulfate from 2 to 6 wt %; this resulted in quickly jumping piles. Then, the conductivity decreased slightly when the concentration reached 8 and 10 wt %. This was chiefly due to the fact that aluminum potassium sulfate in increasing concentrations introduced more metal ions onto the surface of the piles; this resulted in an obvious increase in the conductivity. However, the piles had limited ability to absorb the metal ions; thus, aluminum potassium sulfate at too high a concentration caused a decrease in the conductivity because of adhesion and uneven distribution. Consequently, the rise time decreased with increasing conductivity. However, the siftability and moisture regain did not change too much. This was mainly because aluminum potassium sulfate mainly influenced the electrical properties of the piles by introducing metal ions (Al⁺ and K⁺) onto the surfaces of the piles and, thus, shortened the rise time. However, it had less effect on the siftability and moisture regain.

As illustrated in Table IV, the PET and ECDP piles treated with treatment scheme 6 had the best flocking properties. Moreover, a comparison of ECDP piles with PET piles treated with the same scheme showed that the ECDP piles achieved a more pronounced improvement, which was mainly due to their better affinity, which came from the existence of the sulfoisophthalate acid groups and poly(ethylene glycol).

ECDP and PET flocked fabrics

The ECDP and PET piles treated with scheme 6 were used to produce flocked fabrics. In contrast, a com-

 TABLE VI

 Characteristic of Flocked Fabrics with Different Piles

		Weight loss (%)				
Piles	Flock density (g/m ²)	4000 rounds	6000 rounds	10,000 rounds		
PET	8.7	10.2	15.4	17.6		
ECDP	13.2	0	0	5.1		
Polyamide	13.0	0	0	4.8		

mercially available polyamide flocked fabric with a similar flock density was also introduced. Table VI tabulates the testing results of the flock density and wear resistance. The ECDP flocked fabric had a significantly greater flock density than the PET one; this was explained by the fact that the ECDP piles had a better performance in the electrostatic field. The ECDP piles moved toward the substrate quickly and straightly and fixed vertically on the substrate; this led to a higher flock density. The PET piles had some difficulty penetrating the adhesive for lack of enough energy; this resulted from their lower conductivity. Therefore, perhaps some of the PET piles could have dropped down by gravity and been adhered onto the surface of the substrate, but they were weakly bonded with the substrate. As shown in Table VI with regard to the results of the wear-resistance tests, the PET flocked fabric had a weight loss up to 10.2% at 4000 rounds, which was much higher compared to the weight losses of 5.1 and 4.8% at 10,000 rounds for the ECDP and polyamide flocked fabrics, respectively. Table VI also shows that the ECDP flocked fabric nearly had the same good wear resistance as the commercially available polyamide flocking fabric; this indicated that the ECDP fiber discussed in this study could be successfully used for flocking piles.

CONCLUSIONS

ECDP and PET fibers were compared experimentally with regard to their microstructure and characteristics and were used to produce flocked fabrics in this study. The experimental results show that the ECDP fiber had similar FTIR spectra as the PET fiber but possessed a stronger band at 755 cm⁻¹, which corresponded to the sulfoisophthalate acid groups. Moreover, the FTIR spectra of the ECDP fiber tended to shift to a high-frequency direction. The result of Xray analysis shows that the ECDP fiber had a lower crystallinity degree than the PET fiber. Furthermore, the ECDP fiber had a better conductivity and moisture absorption capability, a lower tensile strength and initial modulus, but a higher elongation than the PET fiber; this was mainly because of the copolymerization of the sulfoisophthalate acid groups and poly(ethylene glycol).

ECDP and PET piles were subjected to pretreatment and electrical treatment to improve their flocking properties. Both the ECDP and PET piles after the treatments had a much better conductivity and moisture absorption capability, especially the ECDP piles: they achieved a more pronounced improvement in flocking properties because of their better affinity. Pretreatment was essential for improving the absorbability for electrical treatment agents; and the antistatic agent and dispersant agent endowed the piles with a higher conductivity and dispersability. As for aluminum potassium sulfate, which was the primary agent for improving the conductivity of the piles through the introduction of metal ions onto the surfaces of the piles, its concentration undoubtedly had a strong effect on the flocking properties. The experimental results show that the piles obtained the best conductivity at 6 wt % aluminum potassium sulfate.

With regard to the flocked fabric, the ECDP flocked fabric had a much higher flock density and lower weight loss in the wear-resistance test than the PET one. Moreover, the ECDP flocked fabric nearly had the same good wear resistance as the commercially available polyamide flocking fabric. This indicated that the ECDP fiber discussed in this study could be successfully used as flocking piles.

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