Antisoiling of Polyester (PET) by a Novel Method of Plasma Treatments and Its Evaluation by Color Measurement

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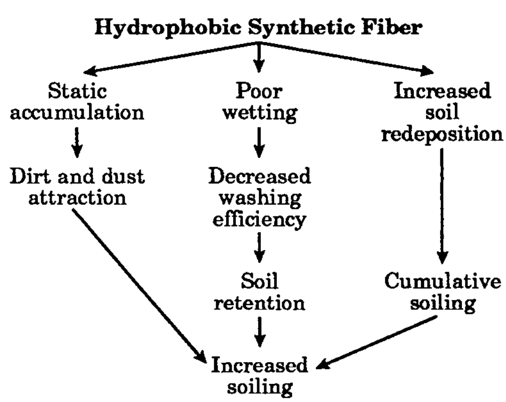
Synopsis

Direct current and radiofrequency plasma treatment of different durations were given to polyester fabric to impart soil resistance. The soil repellency increased with increasing time of treatment. The possible mechanism of soil repellency is outlined. The evaluation of soiling has been made from CIE brightness variable Y with source D_{65} using Minolta Chromameter Reflectance II. This was well in agreement with the conventional reflectance measurement method using spectrophotometer. The plasma treatment system has the advantage of energy and floor space savings and pollution-free atmosphere.

INTRODUCTION

The advent of more and more synthetic fibers in the textile field has made the problem of soiling and its evaluation more pertinent, as these hydrophobic fibers attract soil to a greater extent than natural fibers because of the development of electrostatic charges on the surface.[1] Moreover soiling was reported to increase rapidly when the moisture content of the fibers dropped below 4.0%.[2] Polyester has the least moisture regain value among the synthetic fibers studied and displays maximum soiling.[2] When an antistatic treatment was given to polyester, the soiling tendency was also found to be reduced. Thus soil release finishes are found to be of great use in the case of PET.[3] A flowchart showing how the hydrophobic nature of synthetic fiber leads to greater soiling follows.



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The mechanism of soiling entails two different types of forces of retention: (a) those dependent on some form of bond energy and (b) those dependent on mechanical entrapment. Accordingly dirt particles are retained in a fabric by mechanical and electrostatic forces and by soil bonding.[4] The soil particles also adhere to fibers by occlusion in pits and crevices on fiber surfaces,[5] entrapment in intra or interyarn spaces in the irregularities of the fiber surface, and by sorption by van der Waals or coulombic forces.[6]

Particle size of soil, textile and component fiber structure, electrostatic charge, and moisture regain are considered to be the important factors that influence soiling of textile materials.

The absorption and retention of soil results in graying, yellowing, and deterioration in whiteness and brightness of the fabric.[7] Since the appearance of soiled fabric is achromatic in nature, it is quite appropriate to use brightness value (Y) in the place of reflectance value (R) for the evaluation of soil. Therefore, in the present investigation, the brightness value Y is measured in source D_{65} with the help of Minolta Chromameter Reflectance II.[8] Furthermore, instead of the usual chemical treatment, a novel method of plasma treatments was given to polyester fabrics to impart soil resistance. The possible mechanisms involved are also outlined.

Materials

One hundred percent polyester-textured woven fabric having 99 ends/in. $\times$ 10^5 picks/in., weighing 135 g/m^2 and of size 3.8 cm^2, was used for plasma treatment and soiling purposes.

Preparation of Standard Soil

Several attempts have been made to simulate soil in a fabric artificially, however, this task was reported to be difficult because of the nature of soil and the environment.[9] Many types of artificial soil preparations have been proposed, and many of them contain carbon black due to its powerful effect on light reflectance. In the present investigation, the standard soil was prepared according to IS-5785 standard procedure. The following recipe was used:

Coconut oil	9.0 g
Distil coconut fatty acid	4.5 g
Refined mineral oil	1.9 g
Unhydrous lanolin	0.9 g
Colloidal graphite suspension (10%)	3.5 g

and carbon tetrachloride (CTC) to make the total volume 1000 mL.

Size of Graphite Suspension

The size of graphite suspension was measured using a projection microscope and the size varies from 5 to 12 μm.

EXPERIMENTAL

Application Procedure

The fabric is agitated in the soiled solution at room temperature for 1 min. The material to liquor ratio was kept at 1 : 100. After soiling, the fabric is gently blotted in a filter paper without pressure being applied and then dried at atmospheric conditions.

Design of Plasma Tube

A Pyrex glass tube of internal diameter 4.5 cm and length 32.5 cm was designed for treatment. Both ends of the tube were closed by removable brass flanges. Neoprene rubber O-rings were used inside the joints to provide a vacuum seal. A brass electrode of thickness 1.3 cm and the same size as the internal diameter of the tube was sealed at one end inside the tube. This serves as the positive electrode, and the brass flanges at the opposite end serve as the negative electrode. The experimental arrangement is shown in Figure 1.

dc Plasma Treatment of Fabrics

A fabric piece of 3.8 cm^2 was placed perpendicular to the electrodes at the center of the tube. The tube was sealed and then evacuated down to 0.1 torr. Evacuation was carried out for another 10 min. A Beltronix solid-state dc power supply (5 kV max.) was used to apply voltage across the tube. The voltage was slowly increased untill the glow discharge was initiated and then maintained at required voltage. Treatment was carried out for required duration of time after which glow discharge was switched off and the sample was allowed to be in vacuum for another 10 min. After this vacuum pump was turned off, the

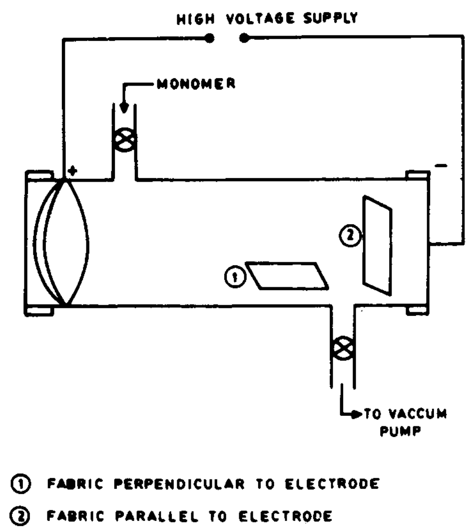


Fig. 1. Experimental setup.

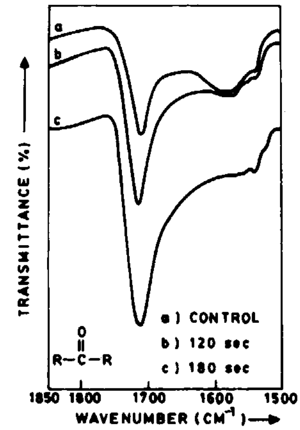


Fig. 2. ATR-IR spectra of dc plasma treated polyester fabric.

tube is filled with air slowly untill it returned to atmospheric pressure, and then the sample was removed for further study.

RF Plasma Treatment

Similar to dc plasma treatment, the fabric was kept at the center of the tube and perpendicular to the edges of the tube, which was then evacuated down to 0.1 torr. The tube was inductively coupled to a 20-MHz radio frequency generator supplied by Universal R.F. Equipment. The samples were then treated at the requisite power for the specified time periods.

Reflectance and Brightness Measurement

Reflectance of control for treated and soiled fabrics was taken over the visible range of 400–700 nm using a Data Colour Instrument. Further the CIE brightness variable (Y) of the fabric was also taken using Minolta Chromameter Reflectance II in source D_{65}.

RESULTS AND DISCUSSION

Energy can be transferred from plasma to the polymer through optical radiation, through neutral particle fluxes, and through ionic particle fluxes. The

TABLE I
Carbonyl Content of Plasma Treated Fabric from ATR-IR Spectra

Duration of treatment (s)	A_{1715}	A_{1410}	A_{1715}/A_{1410}
Control	0.097	0.042	2.31
120	0.167	0.047	3.55
180	0.240	0.063	3.81

energy transferred from the plasma is dissipated within the polymer by a variety of chemical and physical processes. These dissipation processes are the origin of the desired surface property modifications.

Surface Property Modifications

The surface modification brought about by plasma imparts hydrophibility to the fabrics by oxidation reactions that produce oxygen-containing functional groups (carbonyl C=O), which are attached to the polymer surface. Further oxidation depends on the time of treatment. Due to this plasma, treated fabrics showed more resistance to soiling compared to control, and it increases with time of treatment.

The rotating and vibrating groups of molecules in the polymer will absorb energy at their characteristic frequencies, which lie in the infrared spectrum range of the electromagnetic spectrum. Plasma treatment is confined to the surface layers of the polymer without much effecting the bulk properties.[10,11] Therefore, it was decided to use ATR-IR technique to evaluate the surface changes on the polyester. The ATR-IR spectrum in Figure 2 clearly shows the growth of the 1715 band for oxygen-containing functional groups (C=O) with time treatment. The ratio of absorbance at 1715 cm^{-1} to the absorbance at 1410 cm^{-1} was calculated for different duration treated fabrics, keeping 1410 cm^{-1} band as standard. The chosen standard band should be such that it should not undergo any change of intensity on thermal treatment and crystallization. The spectra of PET showed that the intensity of the 1410 cm^{-1} band is rather insensitive to heating. Hence, the band at 1410 cm^{-1} was chosen as the internal standard for ATR measurements. This band is assigned to COH end group and has been shown to be insensitive to crystallization.[12]

This value increases with time of treatment and is tabulated in Table I.

Evaluation of Soil

The reflectance curve of control, dc and RF plasma treated for different durations, and soiled fabrics were taken over the visible range of 400–700 nm

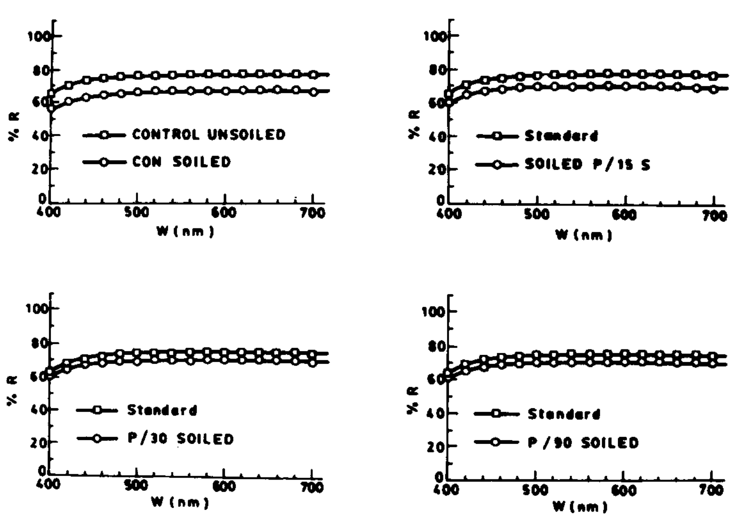


Fig. 3. Reflectance curve of dc plasma treated and soiled fabrics.

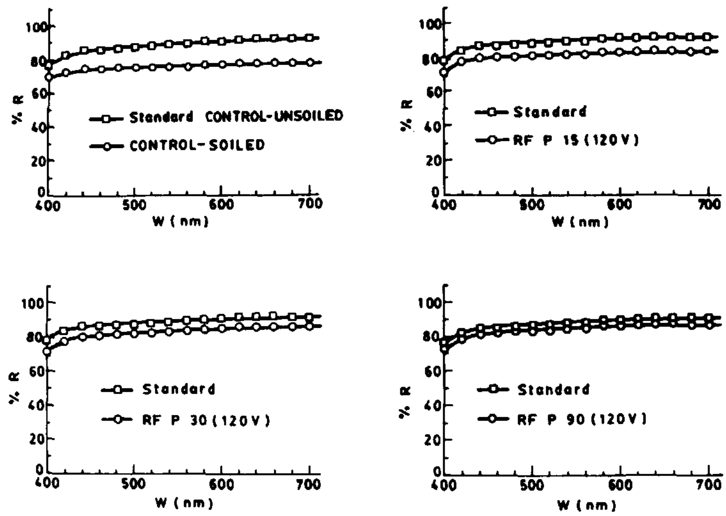


Fig. 4. Reflectance curve of RF plasma treated and soiled fabrics.

using Data Colour Instrument in source D_{65}. The reflectance curves are shown in Figures 3 and 4. The plasma treated (dc and RF treated) and soiled fabrics showed an upward trend in the reflectance curve with time of treatment. It reveals that the plasma treated fabrics have less soiling compared to untreated fabrics. The percentage soiling and soil repellency of treated fabrics were calculated from the reflectance values using the following formula:

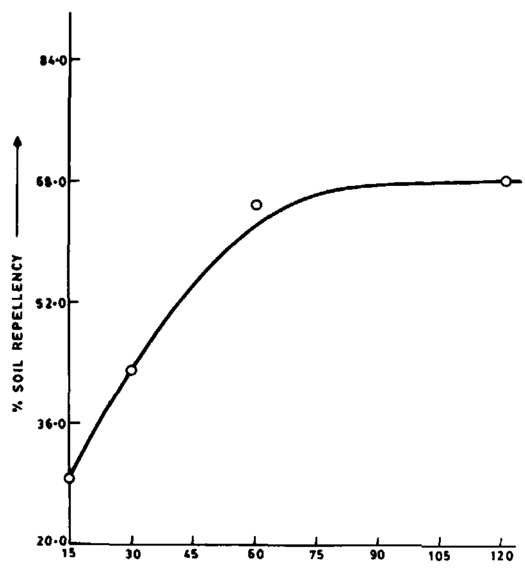


Fig. 5. Percentage soil repellency of dc plasma treated fabric at different durations (calculated from reflectance value R).

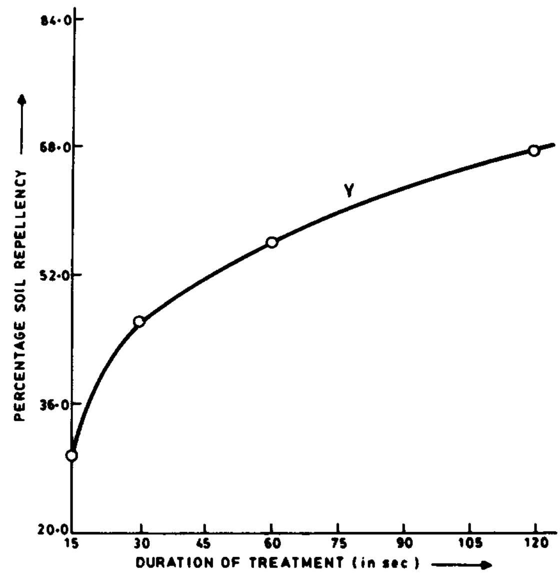


Fig. 6. Percentage soil repellency of dc plasma treated fabric at different durations (calculated from brightness variable Y).

$$\text{Percentage soiling } \frac{R_0 - R_s}{R_0} \times 100$$

where R_0 is the reflectance maximum of the unsoiled fabrics. R_s is the reflectance of the soiled fabrics.

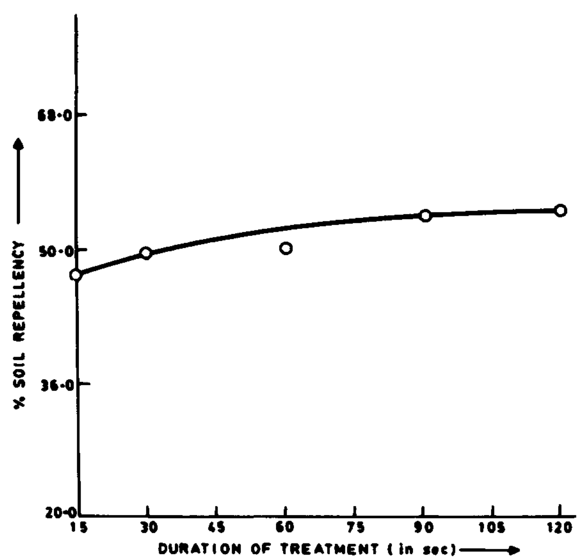


Fig. 7. Percentage soil repellency of RF plasma treated fabric at different durations (calculated from reflectance value R).

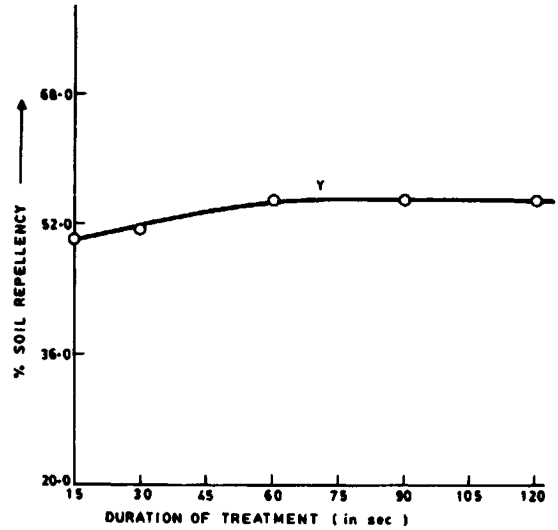


Fig. 8. Percentage soil repellency of RF plasma treated fabric at different durations (calculated from CIE brightness variable Y).

$$\% \text{ Soil repellency} = \frac{\begin{Bmatrix}\text{amount of} \\ \text{soil in control}\end{Bmatrix} - \begin{Bmatrix}\text{amount of soil in} \\ \text{treated fabric}\end{Bmatrix}}{\{\text{amount of soil in control}\}}$$

Further in the present investigation, the percentage of soil and soil repellency were calculated using CIE brightness variable Y instead of reflectance in the

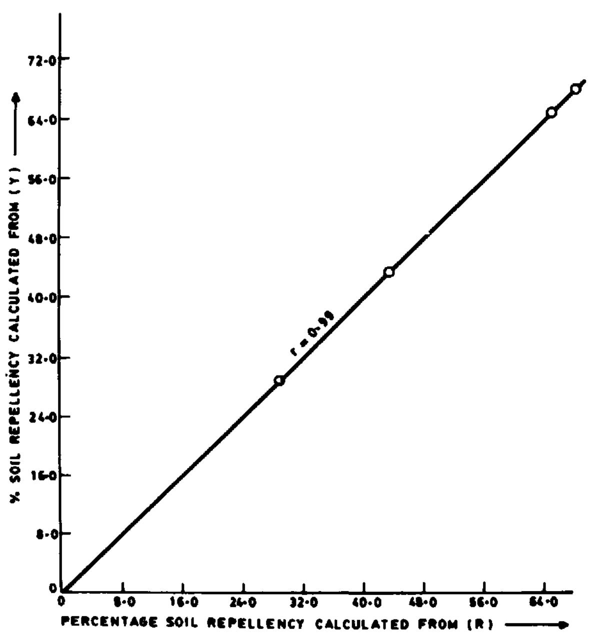


Fig. 9. Correlation between soil repellency calculated from reflectance value (R) and from CIE brightness variable Y for dc plasma treated fabric.

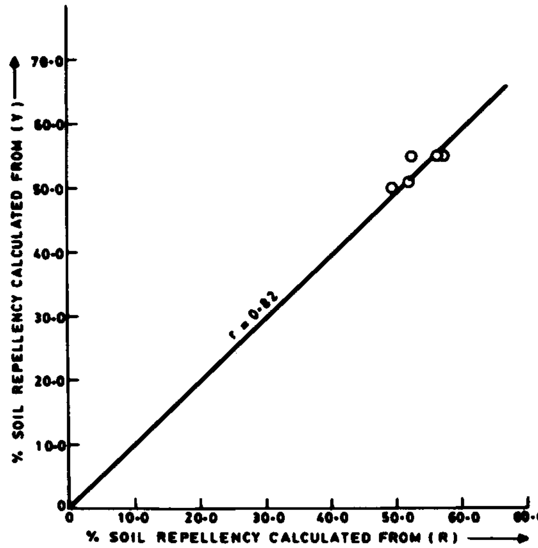


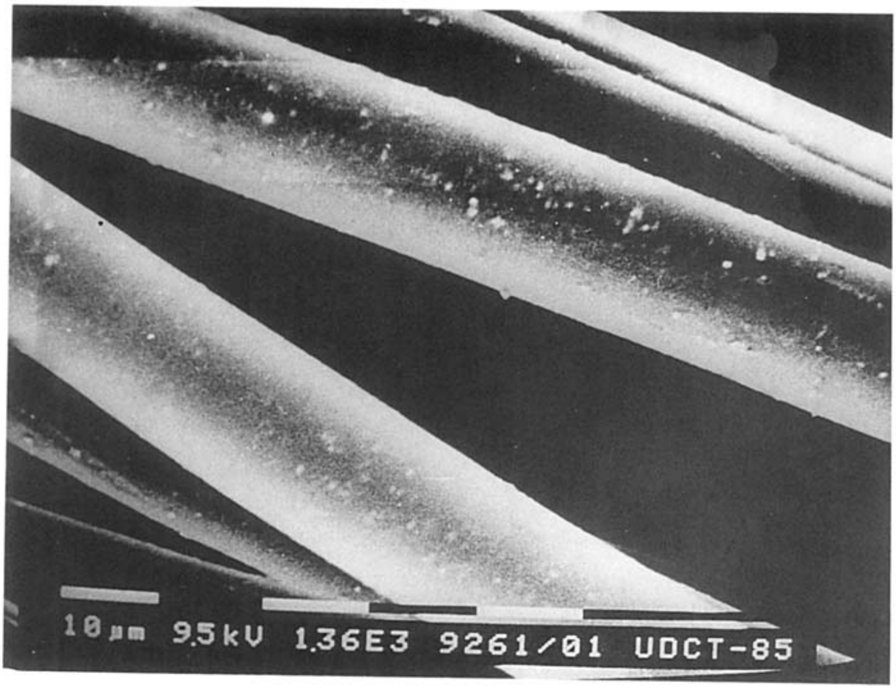
Fig. 10. Correlation between soil repellency calculated from reflectancy value (R) and from CIE brightness variable (Y) for RF plasma treated fabric.

preceding formula with the help of Minolta Chromameter Reflectance II. Since the soil deposited on the fabric is achromatic in nature, it will affect the brightness of the fabric. The soil repellency were calculated for dc and RF plasma treated and soiled fabrics.

The soil repellency of dc plasma treated and soiled fabrics for different durations (15, 30, 60, and 120 s) were calculated separately using reflectance value (R) and CIE brightness variable Y and is shown in Figures 5 and 6. The figures clearly show that the soil repellency increases slowly with time of treatment and then reaches a steady state at higher durations of treatment. Similarly, soil repellency was calculated for RF plasma treated and soiled fabrics for the specified durations (15, 30, 60, 90, and 120 s) and is shown in Figures 7 and 8.

TABLE II
Percentage Soil Repellency Calculated from Reflectance Value (R) and from CIE Brightness Variable (Y)

	DC plasma treatment			RF plasma treatment		
Duration of treatment (s)	Percentage soil repellency calculated from R	Percentage soil repellency calculated from Y	Correlation coefficient r	Percentage soil repellency calculated from R	Percentage soil repellency calculated from Y	Correlation coefficient r
15	28.85	29.41		48.88	50.00	
30	43.50	46.06		51.67	50.95	
60	64.61	56.86	0.97	52.09	54.89	0.82
90	—	—		55.97	54.89	
120	68.15	67.66		56.64	54.89	



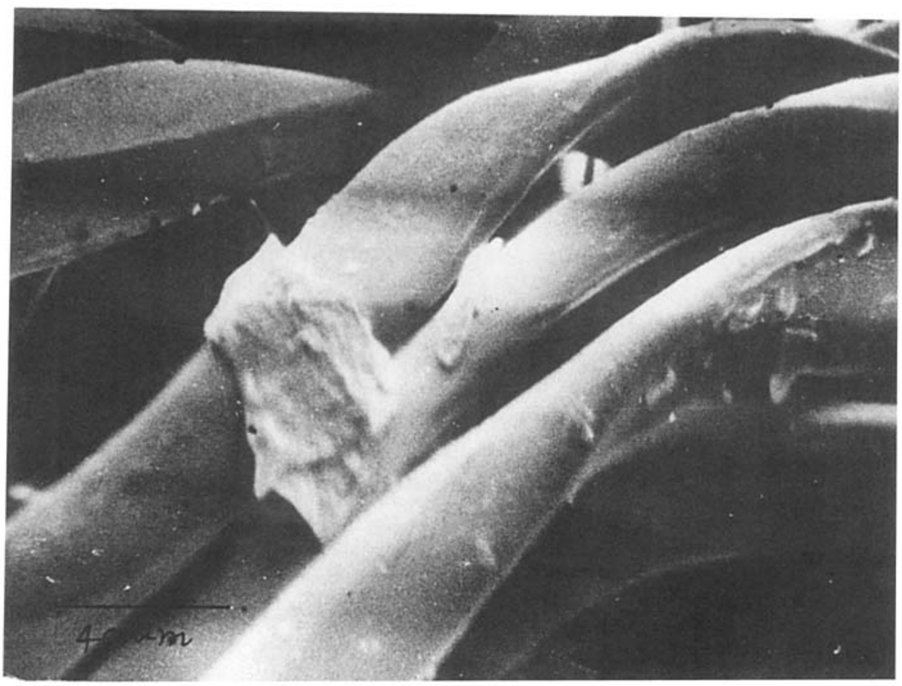
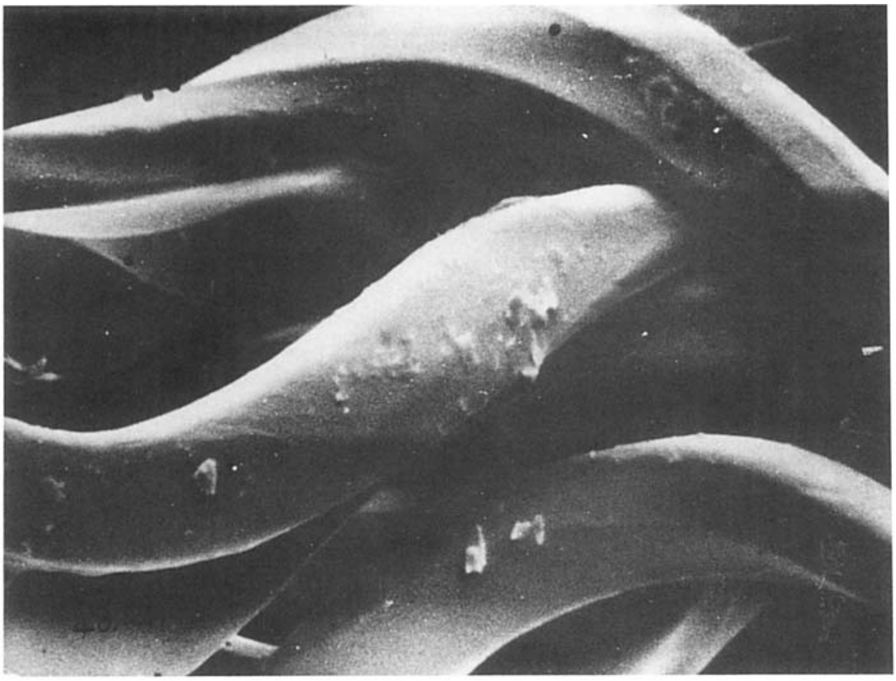


Fig. 11(A). Control unsoiled. Fig. 11(B). Control soiled.



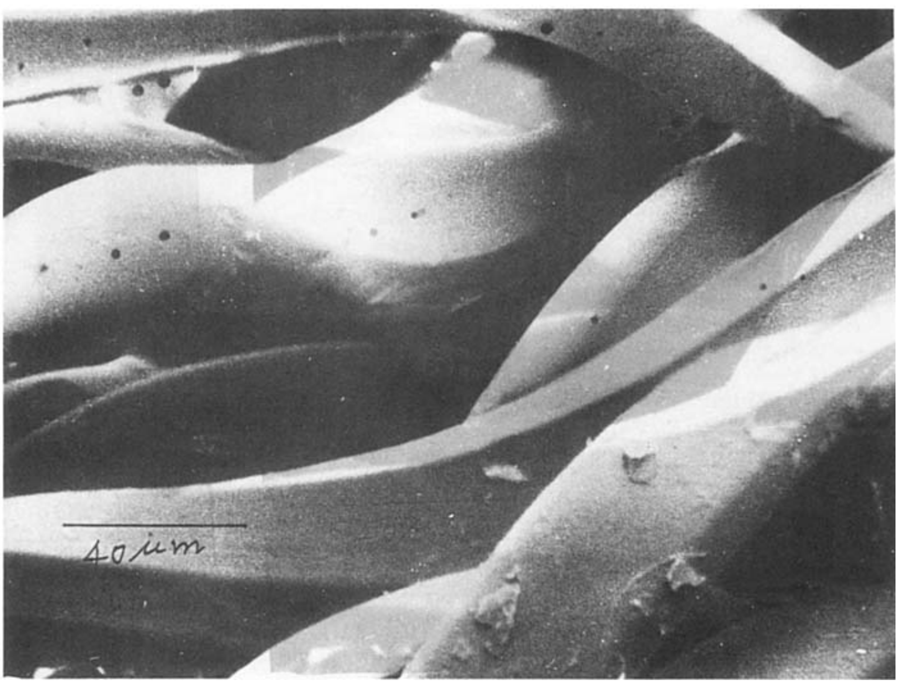


Fig. 11(C). DC plasma treated for 15 s and soiled. Fig. 11(D). DC plasma treated for 120 s and soiled.

From the figures it will be clearly seen that in the beginning, for very small duration of treatment, there is an increase in soil repellency; however, for higher durations there is not much change in soil repellency.

The percentage soil repellency calculated from reflectance value (R) and from CIE brightness variable (Y) is having very good correlation and is shown in Figures 9 and 10 and the values obtained are tabulated in Table II.

Electron Microscopic Study of Soil Deposition

Observation of soil deposition on control and plasma treated polyester fabrics was made using a scanning electron microscope.

Figures 11A–D are the electron micrographs of unsoiled, control soiled and plasma treated for different durations, and then soiled, fabrics. From the micrographs it has been clearly seen that a substantial quantity of soil has been accumulated on control fabrics compared to treated fabrics. Further, the soil deposition is less for the fabric treated for longer in plasma.

CONCLUSIONS

1. The evaluation of soil made from CIE brightness variable Y using Tristimulus colorimeter is quick compared to reflectance measurement using spectrophotometer, since one has to record the reflectance curve over 400–700 nm for finding the reflectance maximum.
2. The plasma treated fabrics showed an upward trend toward soil repellency.
3. The dc plasma treatment of very short durations have more influence on the resistance to soil compared to RF plasma.

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