CHAPTER IV

RESULTS AND DISCUSSION

In this present work, surface chemical, morphological and flame retardation modifications of polyester, cotton and polyester/cotton fabrics after treatment by nitrogen pulsed-plasma were investigated. The numbers of plasma shots and the concentration of diammonium hydrogen phosphate (DAP) were varied while other discharge parameters were kept constant in each experiment. All the abbreviations of the samples are present in the appendix.

4.1 Properties and Morphology of PET Fabrics

4.1.1 Surface Chemical Structure

Attenuated total reflection infrared spectroscopic (ATR/FT-IR) studies of untreated and treated PET fabrics were performed to assess any structural changes, the introduction of any new functional groups, or the alteration of existing groups on the surface of fabrics as a result of plasma modification using theta-pinch device.

Figure 4.1 shows ATR/FT-IR spectra of untreated PET fabric (spectrum a), 10 shots of nitrogen plasma treated PET fabrics (spectrum b), 20 percent of DAP treated PET fabric (spectrum c), 20 percent of DAP with 10 shots of nitrogen plasma treated PET fabrics before (spectrum d), and after washing (spectrum e). Also, Table 4.1 presents the interpretation of ATR/FT-IR spectra of these fabrics.

From these spectra it is possible that the peak corresponding to –P-O-C- may occur at 787 cm^{-1} in Figure 4.1 (d) and 798 cm^{-1} in Figure 4.1 (e). while the peak corresponding to –P-O-H peak may occur at 975 cm^{-1} in Figures 4.1 (c) and (d), and 796 cm^{-1} in Figure 4.1 (e). In addition, P=O stretching peaks may occur at 1243 cm^{-1} in Figure 4.1 (c), 1238 cm^{-1} in Figure 4.1 (d), and 1239 cm^{-1} in Figure 4.1 (e) . However, it is difficult to see these peaks because they overlap with the peaks corresponding to the functional groups of PET fabric as shown in Table 4.1.

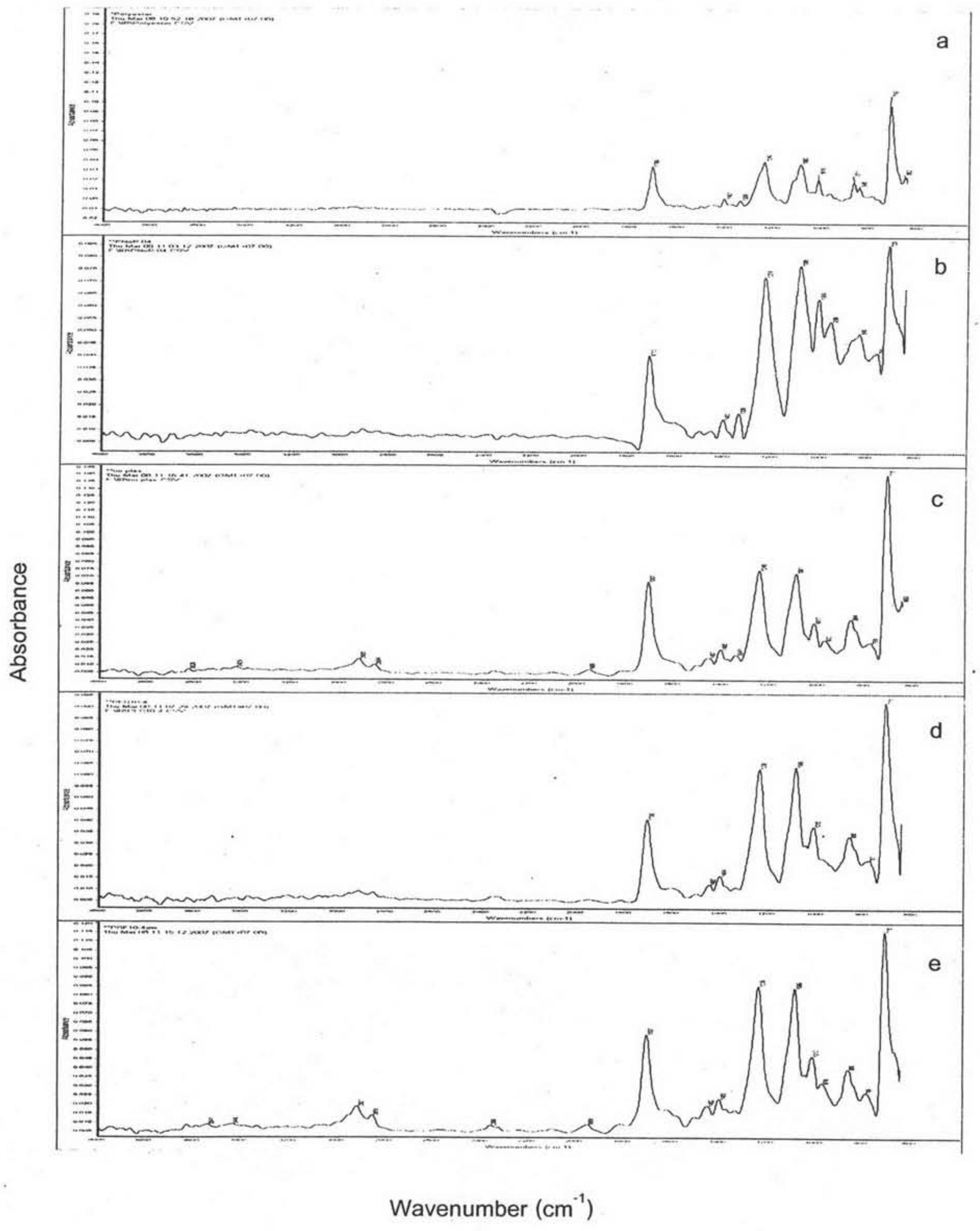

Figure 4.1 ATR/FT-IR spectra of Unt. P. (a), PNoP 10 shots (b), PNoplas 20 (c), P 10-20 before (d) and after (e) washing

Table 4.1 The interpretation of ATR/FT-IR spectra of untreated and plasma-treated PET fabrics

Wavenumber (cm^{-1})					Interpretation
Unt. P.	PNoP 10	PNoPlas 20	P 10-20 bw	P 10-20 aw	
1709	1713	1707	1708	1707	C=O stretching of the ester carbonyl group
1401	1402	1404	1450,1404	1452,1403	Skeleton vibrations of the conjugated system and related to the p-disubstituted benzene rings
1338 ,1016	1338,1009	1340,1016	1015	1017	C-H in-plane bending in substituted aromatic rings and skeleton vibrations involving C-O stretching
1240,1092	1231,1086	1243,1090	1238,1088	1239,1089	The ester C-O-C asymmetric and symmetric stretching vibrations
					-P=O (stretching)
967,845	959, 840	959	-	976	Wagging vibrations corresponding to CH_2- and oxyethylene groups
					-P-O-H
871	-	865	866	868	Out of plane C-H of p-substitued aromatic rings
-	-	-	787	798	-P-O-C-
719	720	716	716	716	Out of plane bending of the two carbonyl substituents on the aromatic ring

4.1.2 Wettability

The wettability of plasma-treated fabric samples were determined by measuring the wetting time directly after plasma treatment. As previously described in Chapter III, the wettability of untreated and plasma-treated synthetic-fiber fabrics were both investigated in horizontal areas. Figure 4.2 shows the position of a fabric sample held in chamber of theta-pinch device.

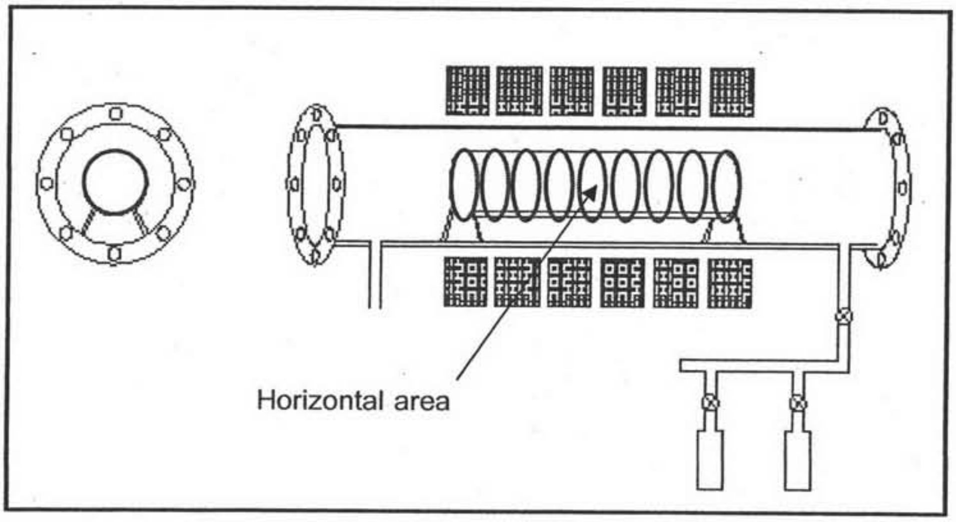

Figure 4.2 Configuration of theta-pinch device with the placement of a fabric sample

As shown in Figure 4.3, when DAP was not applied, it can be obviously seen that the wetting time of untreated PET fabric is lower than that of nitrogen plasma-treated PET fabrics. The wetting time of the plasma-treated fabrics increases until the number of plasma shots exposed on the fabric was 5 shots. This is a result of the etching effect that etched amorphous region on the fiber surface, so the crystallinity of the fabrics increased and made them more hydrophobic. At 10 shots of plasma, the wetting time of plasma-treated fabric decreases because of the functionalization that occurred during the plasma treatment [7].

For DAP-treated fabrics without plasma treatment, the results in Figure 4.3 show that the wetting time of DAP-treated fabrics slightly increases with increasing DAP concentration. The functional groups on the fabric interacted with DAP after applying DAP onto the fabrics. This made DAP-treated fabrics more hydrophobic.

Also, the wetting time of DAP-plasma-treated fabrics increases with increasing the number of plasma shots and the concentration of DAP.

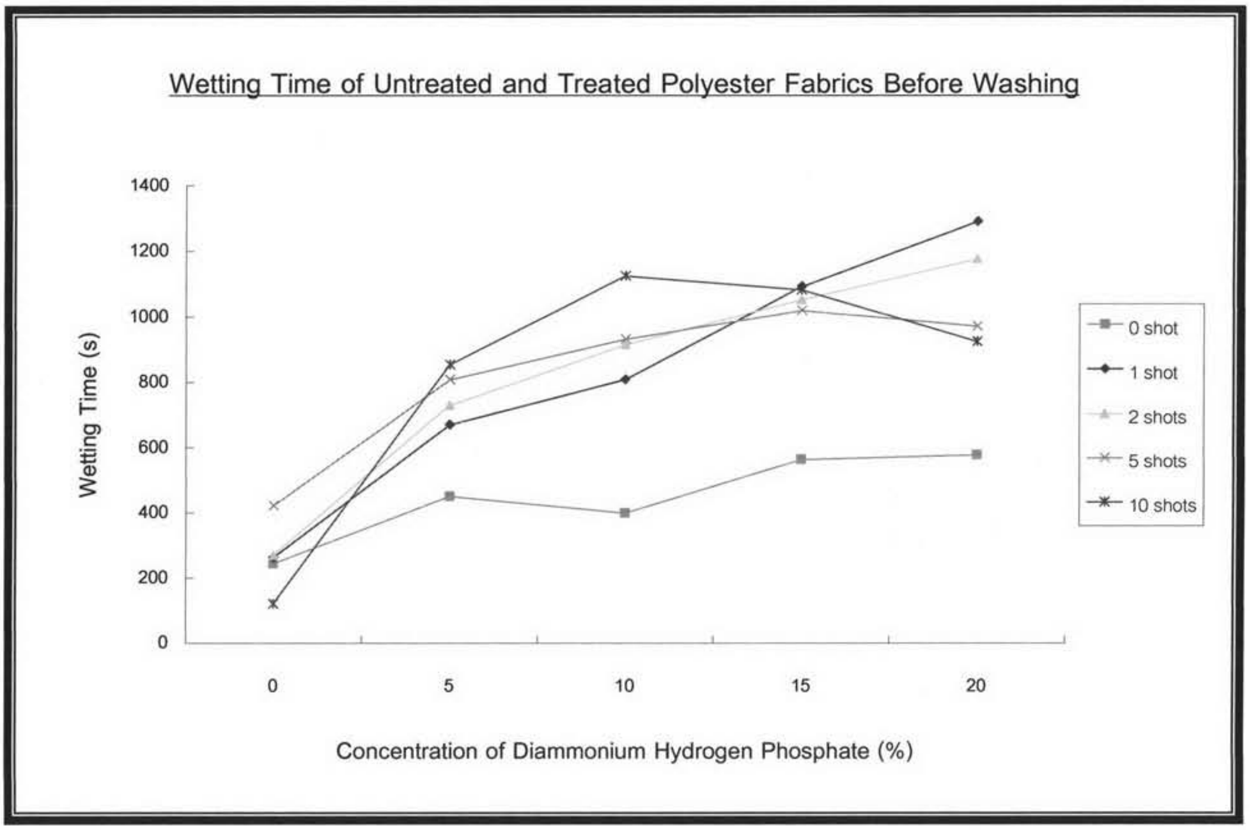

Figure 4.3 The wettability of untreated and treated PET fabrics before washing

From Figure 4.4, the results show that the suitable ranges of DAP concentration for the interaction between DAP and the fabric to occur were at 5 and 10 percent. In this range, the process could be done by increasing DAP concentration with decreasing the number of plasma shot or decreasing DAP concentration when the number of plasma shots was increased.

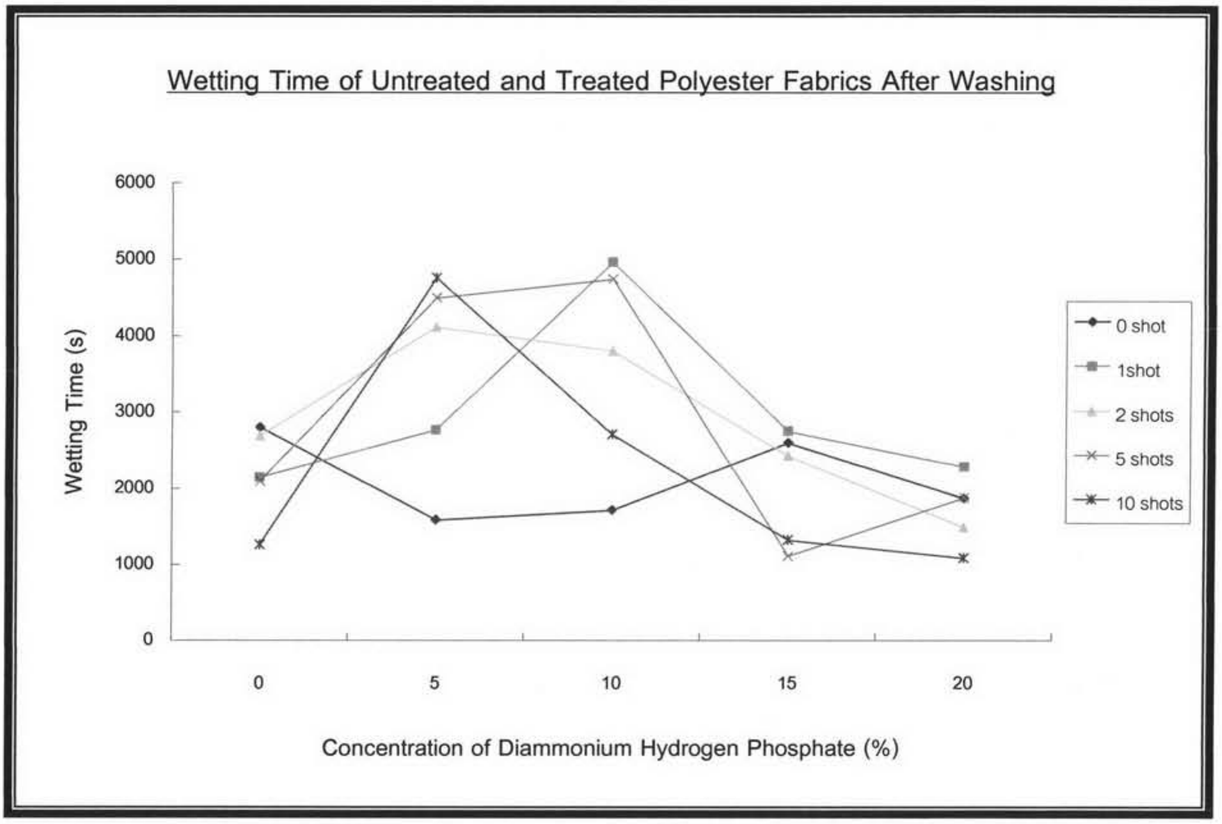

Figure 4.4 The wettability of untreated and treated PET fabrics after washing

4.1.3 Morphological Analysis

Surface morphological changes on PET fabrics were observed by scanning electron microscopy (SEM). As shown in Figure 4.5 (a) the original surface of untreated PET fabric is smooth. When they were exposed to 5 shots and 10 shots of nitrogen plasma (Figures 4.5 (b) and (c)), their surfaces were etched, resulting in surface roughness and the breaking of the fiber. These results can be attributed to the etching process or ablation effect caused by the bombardment of nitrogen plasma reactive species on the fabric surface. When the fabrics were dipped in the 15 and 20 percent of DAP solution (Figure 4.5 (d) and (e)), they were attached with DAP on their surface after drying.

The changes in the surface morphology of the fabrics observed after the plasma treatment can be explained by the localized degradation of polymer at the surface layer. The degradation seems to be the effect of the interaction of plasma with a fabric surface. This process leads to an almost completed breakdown of relatively small numbers of molecules at the surface into low molecular components which eventually vaporize in the low pressure system.

The bombardment of plasma species can be enhanced by increasing the number of plasma shots. Therefore, it can be concluded that the changes in surface morphology of the plasma-treated fabrics depend on the number of plasma shots.

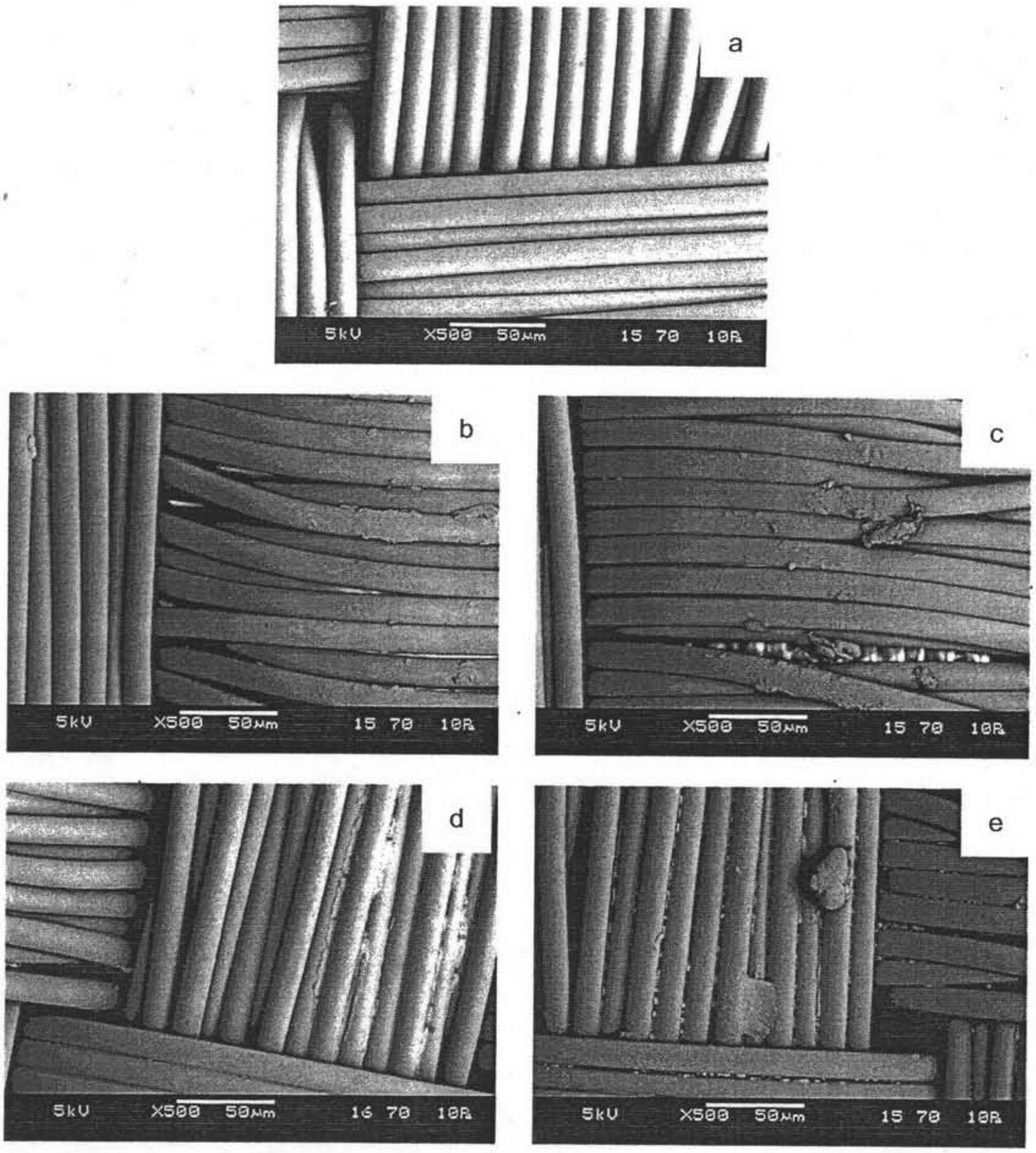

Figure 4.5 SEM photographs at X500 of Unt. P. fabric (a), PNoP 5 fabric (b), PNoP 10 (c), PNoPlas 15 (d), and PNoPlas 20 (e)

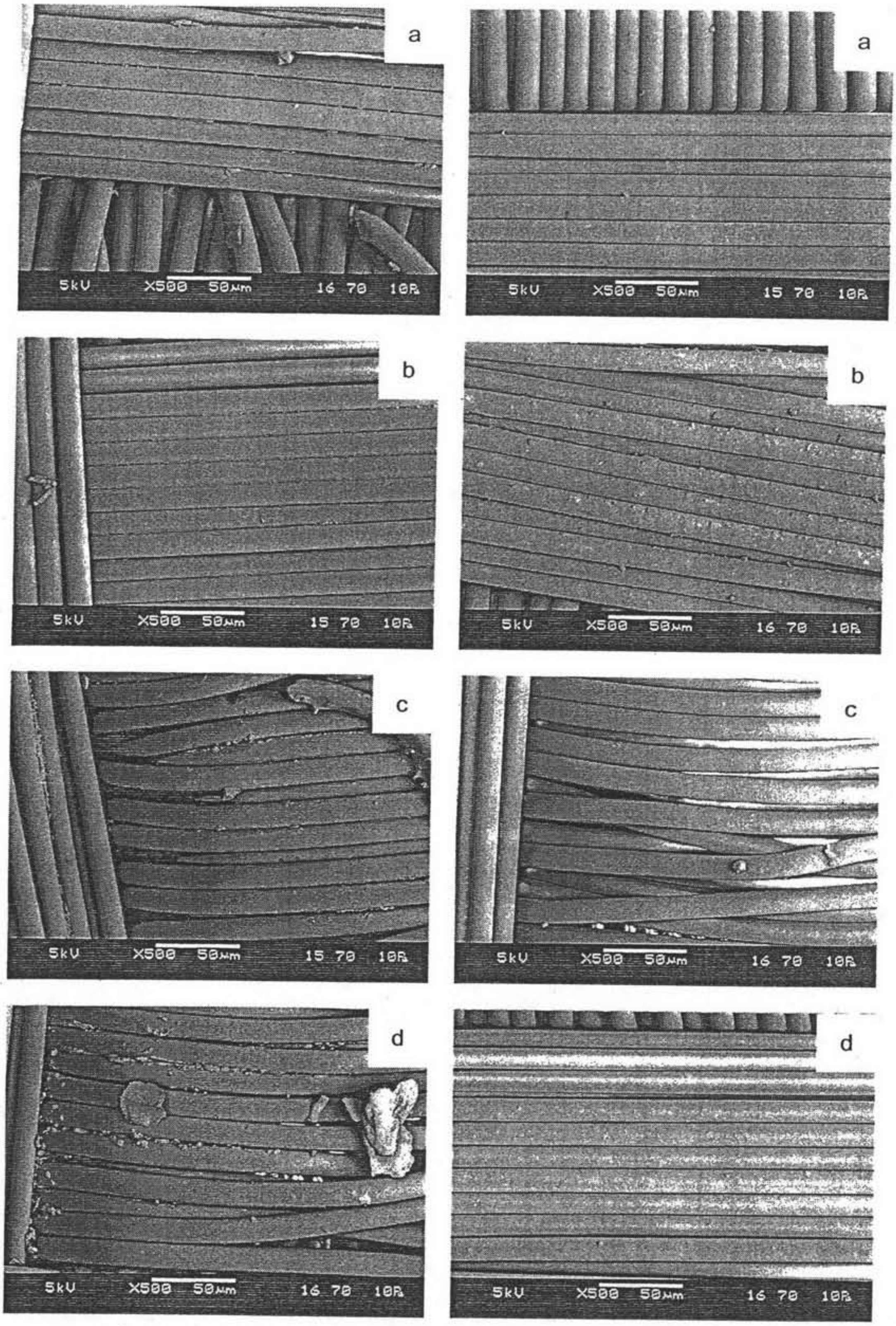

Figure 4.6 SEM photographs at X500 of P 5-15 (a), P 10-15 (b), P 5-20 (c), and P10-20 (d) before washing (left) and after washing (right)

As shown in Figure 4.6 the treated PET fabrics were covered with small amount of DAP after washing, on the right, compared with the treated fabrics before washing. This was result of the removing of DAP that did not react with the fabrics surface during washing process.

However, these photographs show that the surface of the fabrics were bound with DAP during nitrogen plasma treatment because of the change in surface roughness of treated PET fabric.

4.1.4 Whiteness and Yellowness

Table 4.2 present the whiteness and yellowness of untreated and treated PET fabrics before washing. Most of treated fabrics are yellower than untreated one. The change of whiteness and yellowness was the result of DAP application and plasma treatment. The yellowness of the plasma treated fabric increases with increasing the number of plasma shot because higher degradation of the fabric occurs when it is subjected to higher degree of plasma exposure [83].

Table 4.2 The whiteness and yellowness of untreated and treated PET fabrics before washing

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
Unt. P	76.43	76.38	N/A	-	-	-	-
Unt. P*	77.56	77.47	N/A	-	-	-	-
Unt. P**	75.77	75.74	N/A	-	-	-	-
PNoP 1	73.70	73.75	0.43	-2.73	-2.63	N/A	0.55
PNoP 2	73.25	73.31	0.57	-3.18	-3.07	N/A	0.65
PNoP 5	73.78	73.81	0.33	-2.65	-2.57	N/A	0.51
PNoP 10*	67.79	67.99	2.88	-9.77	-9.48	N/A	1.86
PNoPlas 5	76.95	76.88	N/A	0.52	0.50	N/A	0.20
PNoplas 10	77.88	77.78	N/A	1.45	1.40	N/A	0.37
PNoPlas 15*	73.66	73.73	0.75	-3.90	-3.74	N/A	0.75
PNoPlas 20	77.25	77.17	N/A	0.82	0.79	N/A	0.16
P 1-5**	74.39	74.45	0.49	-1.38	0.49	N/A	0.59
P 2-5**	73.85	73.94	0.69	-1.92	0.69	N/A	0.83
P 5-5**	72.37	72.51	1.19	-3.40	1.19	N/A	0.88
P 10-5**	70.17	70.36	1.99	-5.60	1.99	N/A	1.89
P 1-10**	76.89	76.85	N/A	1.12	N/A	N/A	0.52

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
P 10-10**	72.16	72.28	1.17	-3.61	1.17	N/A	0.83
P 1-15**	76.31	76.29	N/A	0.54	N/A	N/A	0.48
P 2-15**	75.13	75.15	0.01	-0.64	1.01	N/A	0.39
P 5-15**	76.01	76.02	N/A	0.24	N/A	N/A	0.56
P 10-15**	73.31	73.42	0.74	-2.46	0.74	N/A	0.76
P 1-20**	75.32	75.38	0.37	-0.45	0.37	N/A	0.76
P 2-20**	75.19	75.23	0.21	-0.58	0.21	N/A	0.60
P 5-20**	71.35	71.51	1.53	-4.42	1.53	N/A	1.02
P 10-20**	68.28	68.49	2.62	-7.49	2.62	N/A	1.56

* compared with Unt. P.*

** compared with Unt. P.**

Table 4.3 presents the whiteness and yellowness of untreated and treated PET fabrics after washing. The treated fabrics are still yellower than the untreated one. In addition, the yellowness increases with increasing the number of plasma shot while the concentration of DAP does not affect this property.

Table 4.3 The whiteness and yellowness of untreated and treated PET fabrics after washing

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
Unt. P	76.64	76.58	N/A	-	-	-	-
Unt. P*	76.43	76.38	N/A	-	-	-	-
Unt. P.**	77.56	77.47	N/A	-	-	-	-
PNoP 1*	73.70	73.75	0.43	-2.73	-2.63	N/A	0.55
PNoP 2*	73.25	73.31	0.57	-3.18	-3.07	N/A	0.65
PNoP 5*	73.78	73.81	0.33	-2.65	-2.57	N/A	0.51
PNoP 10**	67.79	67.99	2.88	-9.77	-9.48	N/A	1.86
PNoPlas 5	77.58	77.52	N/A	0.94	0.94	N/A	0.53
PNoplas 10	77.36	77.31	N/A	0.72	0.73	N/A	0.43
PNoPlas 15	70.10	70.34	2.04	-6.54	-6.24	N/A	1.42
PNoPlas 20	61.50	62.15	5.56	-15.14	-14.43	N/A	3.44
P 1-5	67.01	67.31	3.16	-9.63	-9.27	N/A	1.98
P 2-5	60.54	60.54	5.90	-16.10	-16.04	N/A	3.65
P 5-5	65.08	65.08	3.60	-11.56	-11.50	N/A	2.24
P 10-5	65.10	65.10	3.95	-11.54	-11.48	N/A	2.34
P 1-10	69.24	69.50	2.35	-7.40	-7.08	N/A	1.57
P 2-10	69.17	69.46	2.45	-7.47	-7.12	N/A	1.63

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
P 5-10	66.88	67.20	3.27	-9.76	-9.38	N/A	2.05
P 10-10	64.03	64.29	4.12	-12.61	-12.29	N/A	2.50
P 1-15	68.08	68.44	2.91	-8.56	-8.14	N/A	1.93
P 2-15	68.78	69.08	2.58	-7.86	-7.50	N/A	1.73
P 5-15	69.00	69.27	2.47	-7.64	-7.31	N/A	1.63
P 10-15	64.27	64.62	4.21	-12.37	-11.96	N/A	2.54
P 1-20	65.95	66.18	3.38	-10.69	-10.40	N/A	2.09
P 2-20	67.08	67.38	3.12	-9.56	-9.20	N/A	1.99
P 5-20	67.10	67.48	3.28	-9.54	-9.10	N/A	2.13
P 10-20	67.92	68.29	3.07	-8.72	-8.29	N/A	1.97

* compared with Unt. P.*

** compared with Unt. P.**

Thus, the plasma treatment is an effective process for generated phosphate groups on the PET fabric surface but nevertheless it does not fit for the textile products that their whiteness is concerned.

4.1.5 Flammability

In flammability test, it can be seen that the untreated and treated PET fabrics burnt and melted when they were set on fire. During combustion, the molten PET fabrics accompanying with the flame drop to the bottom of the tester as shown in Figure 4.7.

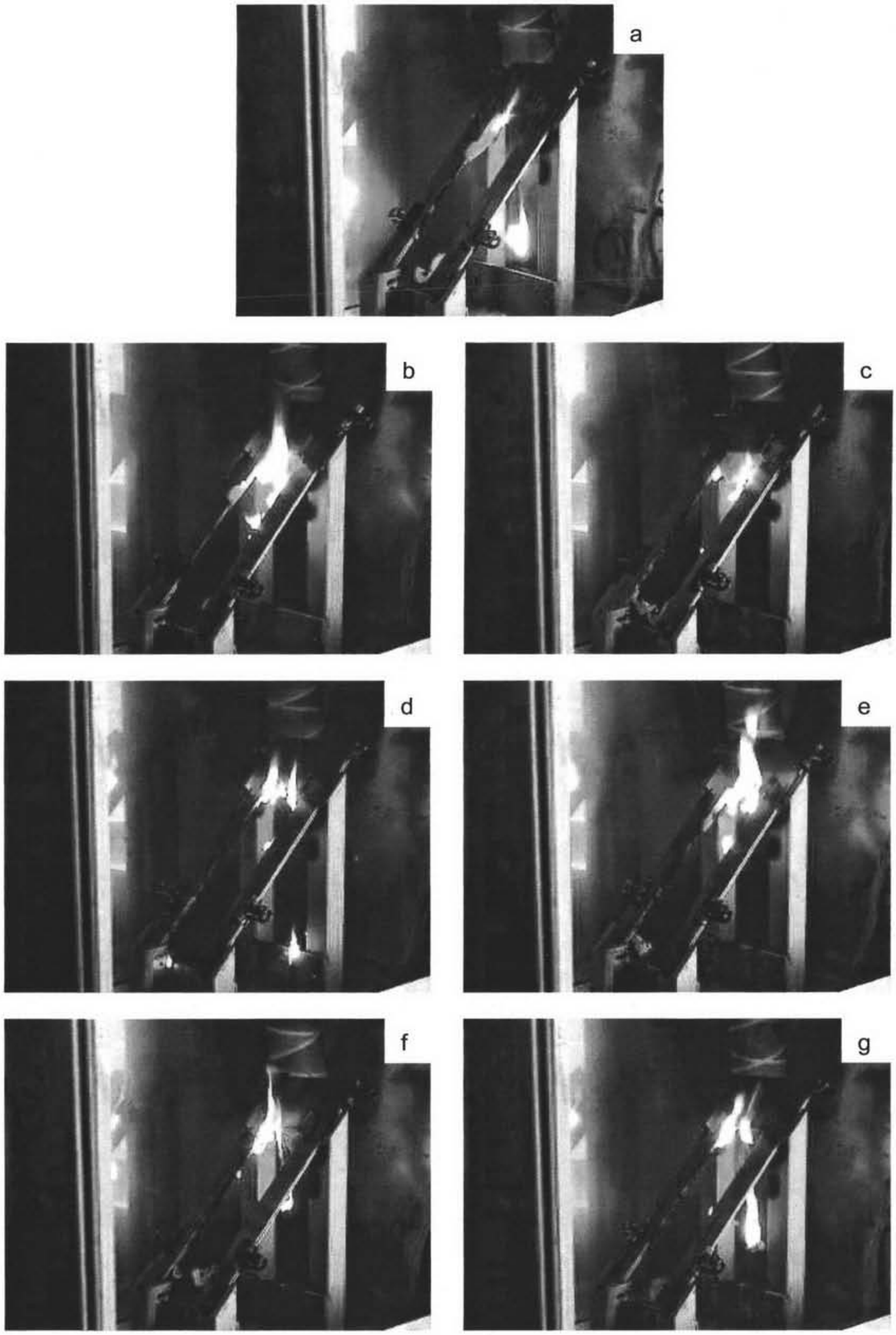

Figure 4.7 45 degree flammability test of untreated PET fabric (a), PNoP 2(b), PNoPlas 5(c), P 1-5 (d), P 2-5 (e), P 5-5 (f), and P 10-5 (g)

Flame spread rates of the untreated and treated PET fabrics at 31°C and 52 percent of humidity are shown in Figure 4.8. The flame spread rates of all treated PET fabrics are higher than that of the untreated PET fabric because the etching effect that occurred during plasma treatment made the fabrics easier for combustion.

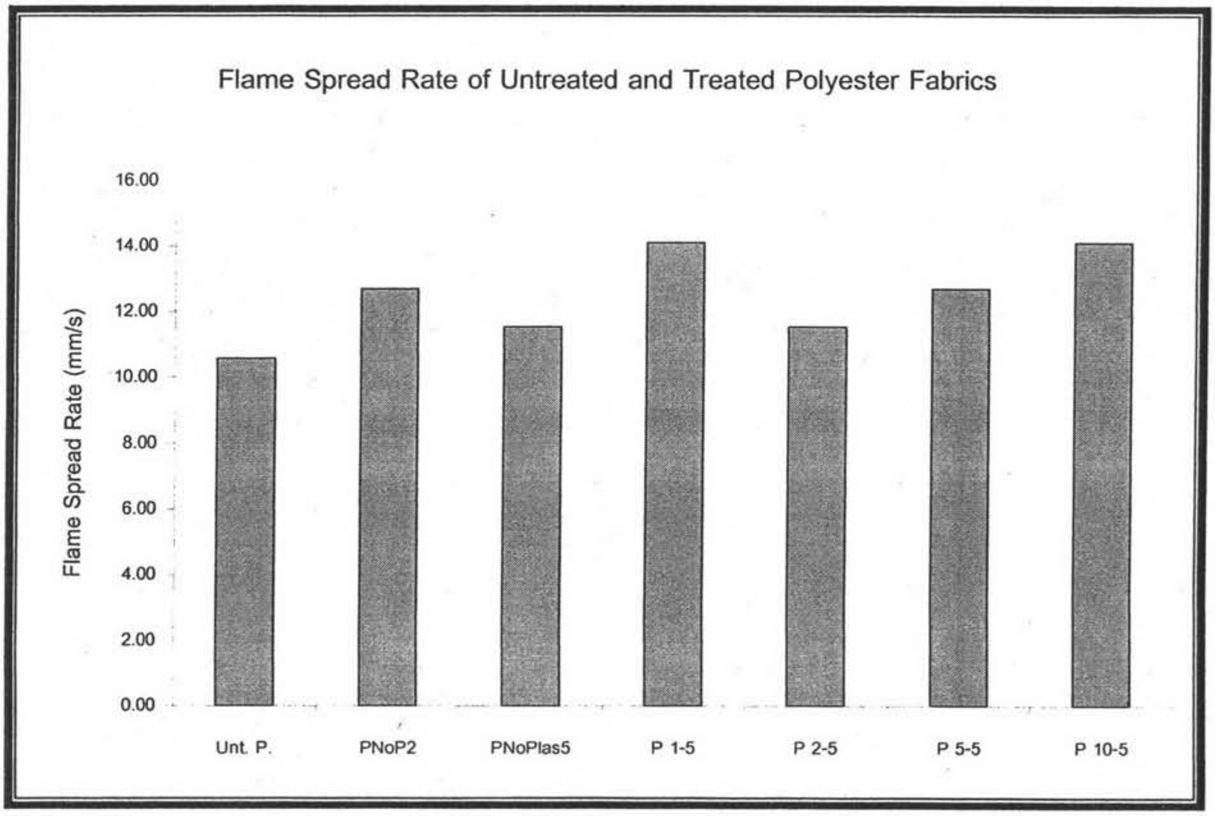

Figure 4.8 Flame spread rates of untreated and treated PET fabrics

However, when ignition time is considered, it is found that the ignition time of the treated fabrics are higher than the untreated one as shown in Table 4.4, and increases with the concentration of DAP.

Table 4.4 The ignition time of untreated and treated PET fabrics after washing

Fabrics	Ignition Time (s)
Unt. PET	1
P 5-15	2
P 5-20	2
P 10-15	3
P 10-20	3

4.2 Properties and Morphology Cotton Fabrics

4.2.1 Surface Chemical Structure

The ATR/FT-IR studies of untreated and treated cotton fabrics were performed to assess any structural changes or the alteration of existing groups on the surface of fabrics as a result of plasma modification using theta-pinch device.

Figure 4.9 shows ATR/FT-IR spectra of untreated cotton fabric (spectrum a), 1 shots of nitrogen plasma treated cotton fabrics (spectrum b), 15 percent of DAP treated cotton fabric (spectrum c), and 15 percent of DAP with 1 shots of nitrogen plasma treated cotton fabrics (d). Also, Table 4.5 presents the interpretation of ATR-FTIR spectra of these fabrics.

For treated cotton fabric after washing, the new peak at 825 cm^{-1} in Figure 4.9 (e) attributed to –P-O-C- occurred. This suggests that DAP interacted with the fabrics surface during plasma treatment. The disappearance of the peak at 1743 cm^{-1} on the treated fabrics after washing indicates that some substance having ester functional groups was removed from the fabric after washing. This suggests that it did not interact with the fabric during plasma treatment.

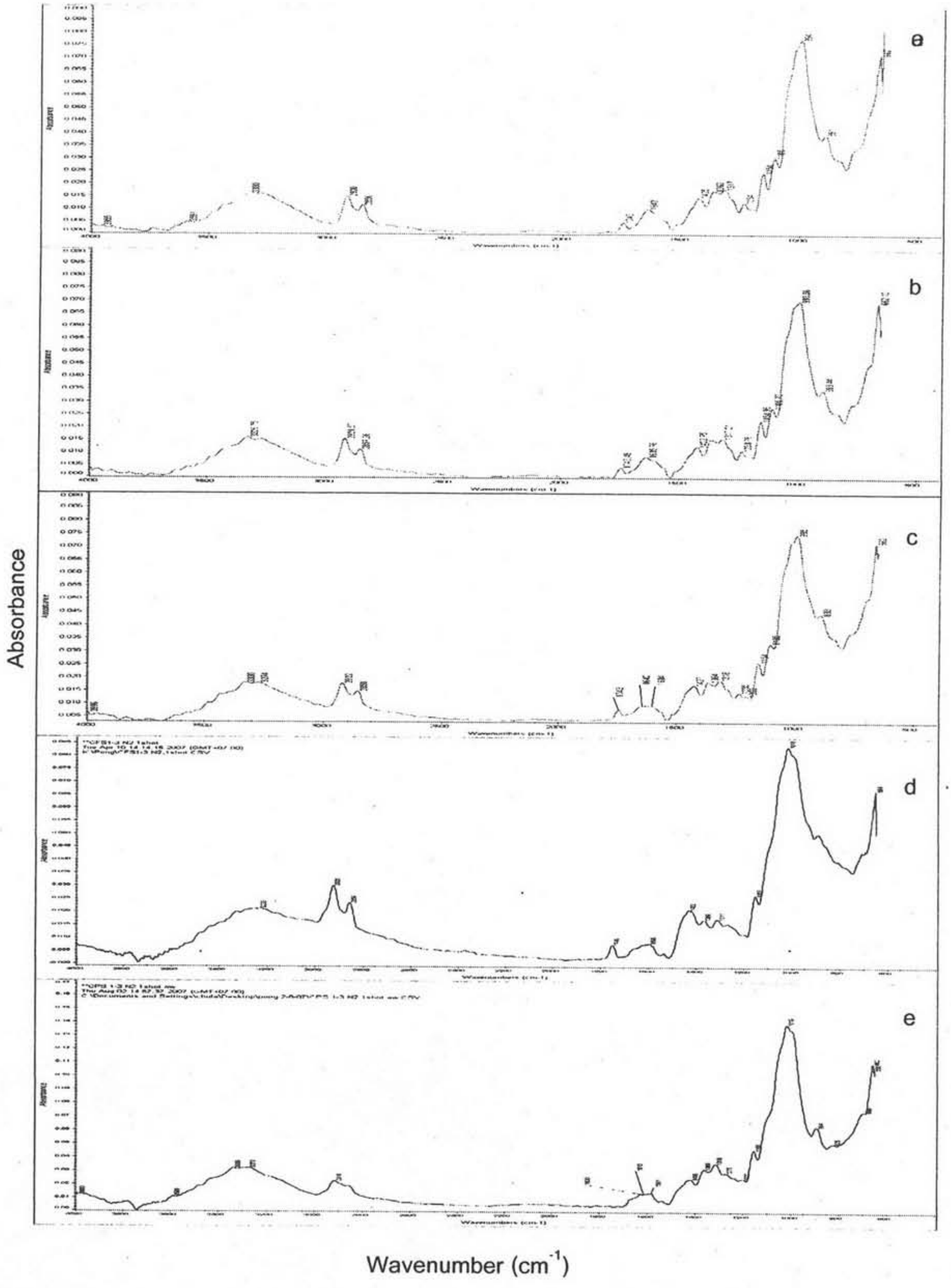

Figure 4.9 ATR/FT-IR spectra of Unt. C. (a), CNoP 1 (b), CNoplas 15 (c), and C 1-15 before (d) and after (e) washing

Table 4.5 The interpretation of ATR/FT-IR spectra of untreated and plasma-treated cotton fabrics

Wavenumber (cm-1)					Interpretation
Unt. C.	CNoP 1	CNoPlas 15	C 1-3 bw	C 1-3 aw	
3330	3330	3330	3330	3330	O-H stretching : intermolecular hydrogen bonding
2920	2920	2922	2926	2916	C-H stretching of $-CH_2$
1425	1426	1427	1418	1432	C-H bending of $-CH_2$
1317,1202	1317,1202	1318,1302	1311	1316,1202	O-H in plane bending
1154	1154	1154	1153	1155	Anti-symmetrical bridge C-O-C stretching
1105	1105	1105	-	-	C-O stretching of secondary alcohol
-	-	-	-	825	-P-O-C-
662	662	662	654	662	O-H out-of-plane bending

4.2.2 Wettability

The wettability of plasma-treated fabric samples treated in this research was determined by measuring the wetting time directly after plasma treatment.

As shown in Figure 4.10, it can be obviously seen that the wetting time of untreated cotton fabric is lower than that of nitrogen plasma-treated cotton fabrics. This may be the results of the etching effect of amorphous region and the forming of new functional groups on the fabric surface.

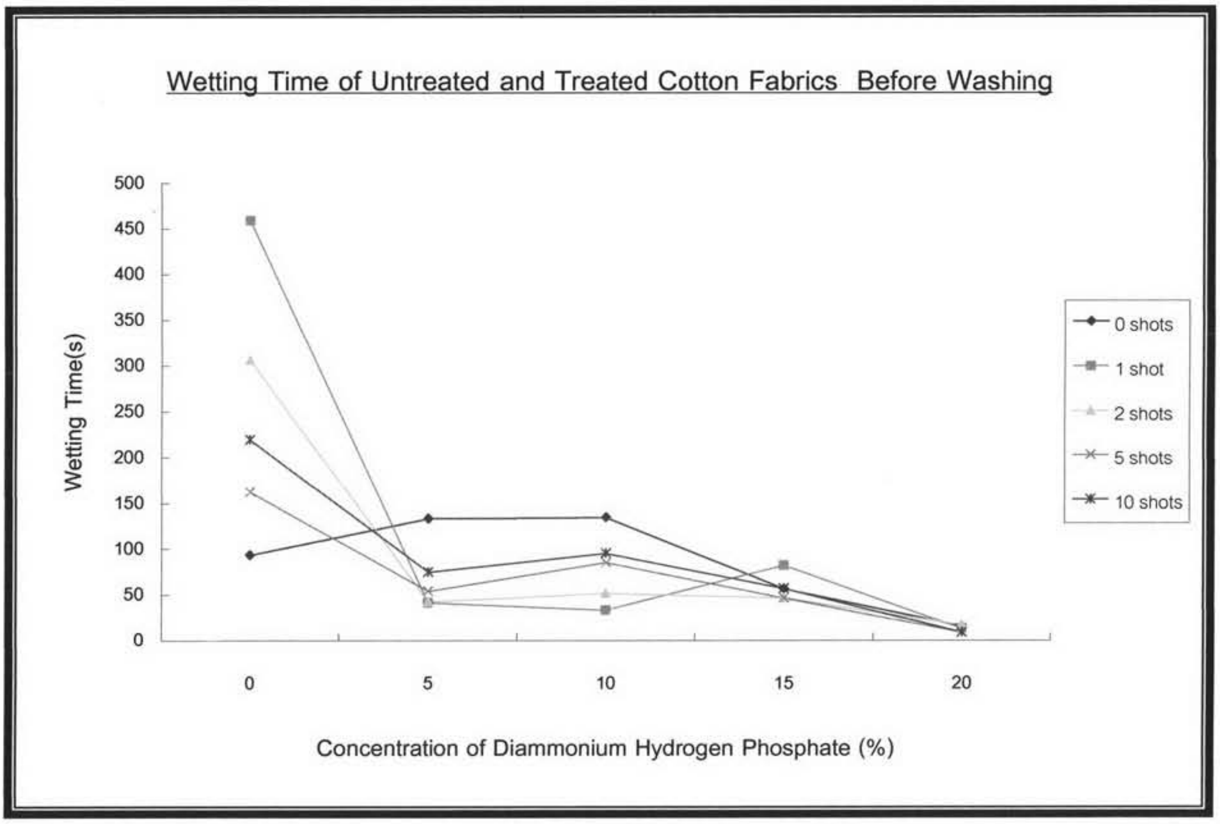

Figure 4.10 The wettability of untreated and treated cotton fabrics before washing

After washing process, the wetting time of the treated fabric changes as shown in Figure 4.11. This might be the result of the removing of DAP that did not interact with the fabric surface.

In the case of non DAP-treated fabrics, the results in Figure 4.11 show that untreated cotton fabric has lower wetting time than nitrogen plasma-treated cotton fabrics because the new functional groups generated on the fabrics are less hydrophilic than hydroxyl group.

With increasing DAP concentration, the results show that the wetting time of the treated fabrics increases as shown in Figure 4.11. This indicates that higher amount of DAP interacted with the fabric surface, and made it more hydrophobic.

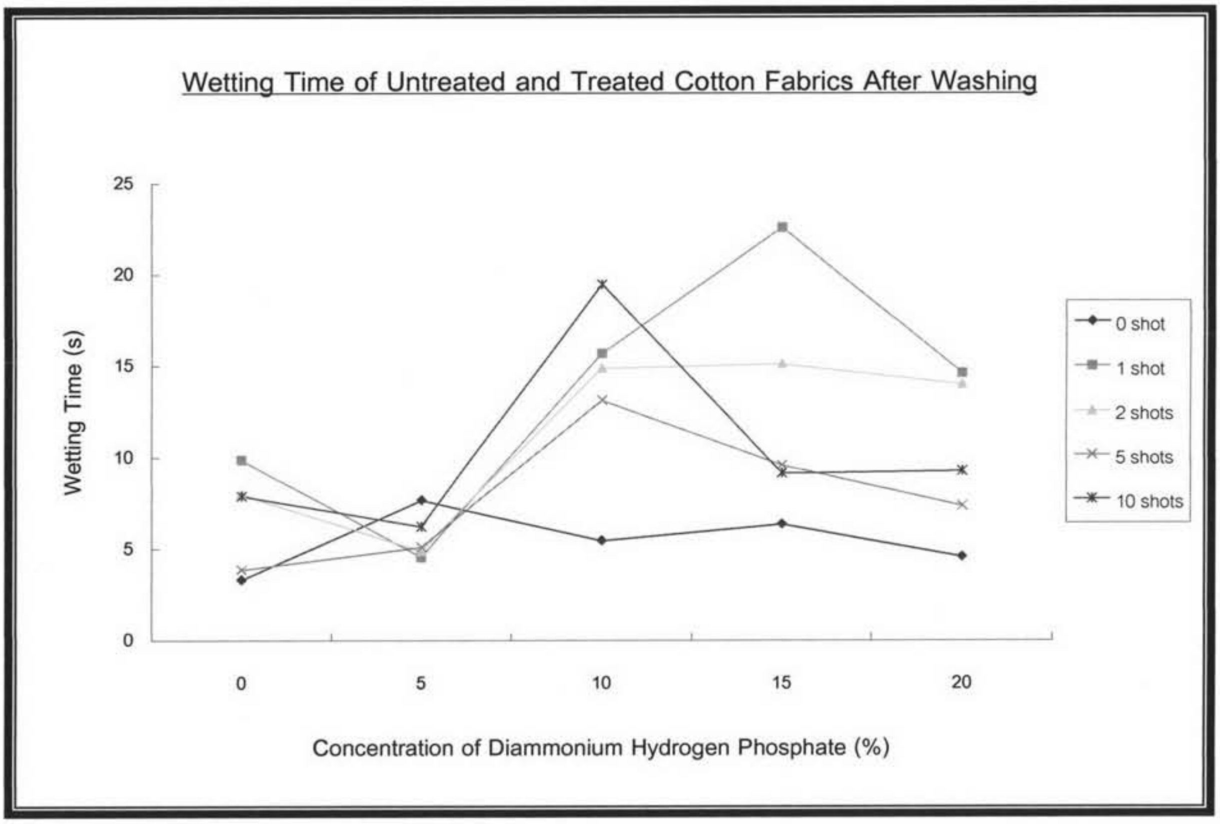

Figure 4.11 The wettability of untreated and treated cotton fabrics after washing

Moreover, these results suggest that the fabric surface may be coated by some substance containing ester functional groups because the wetting time of the untreated cotton fabric after washing decreases to 3 seconds.

Table 4.6 The wetting time of treated cotton fabric before washing from additional investigation

Fabric	Previous Investigation		Additional Investigation	
	X	SD	X	SD
CNoP 10	220	103	12	3
C 10-10	96	57	16	10
C 1-15	82	60	8	2

Table 4.7 The wetting time of treated cotton fabric after washing from additional investigation

Fabric	Previous Investigation		Additional Investigation	
	X	SD	X	SD
CNoP 10	8	3	6	2
C 10-10	19	6	8	3
C 1-15	23	8	5	2

From additional investigation by washing cotton fabrics before DAP and plasma treatment, it was found that this ester substance did not react during plasma treatment since the wetting time of plasma-treated fabric after washing from this additional experiment (6 ± 2) as shown in Table 4.7 is comparable to that from the previous experiment (8 ± 3). However, the results also suggest that this ester substance has positive effect for adhesion between DAP and cotton fabric surface since the wetting times of DAP-plasma-treated fabrics from previous experiment are higher than those from additional experiment.

4.2.3 Morphological Analysis

Surface morphological changes of cotton fabrics were observed by SEM. The original surface of untreated cotton was showed in Figures 4.12 (a). When they were exposed to 5 shots and 10 shots of nitrogen plasma (Figure 4.12 (b) and (c)), their surface were etched, resulting in surface roughness and the breaking of the fiber. These results can be attributed to etching process or ablation effect caused by the bombardment of nitrogen plasma reactive species on the fabric surface. When the fabrics were dipped in 15 percent and 20 percent of DAP solution (Figure 4.12 (d) and (e)), with DAP appears on their surface after drying.

The changes in the surface morphology of the fabrics observed after the plasma treatment can be explained by the localized degradation of polymer at the surface layer.

The degradation seems to be the effect of the interaction of plasma with a fabric surface. This process leads to an almost completed breakdown of relatively small numbers of molecules at the surface into low molecular components which eventually vaporize in the low pressure system.

The bombardment of plasma species can be enhanced by increasing the number of plasma shots. Therefore, it can be concluded that the changes in surface morphology of the plasma-treated fabrics depend on the number of plasma shots.

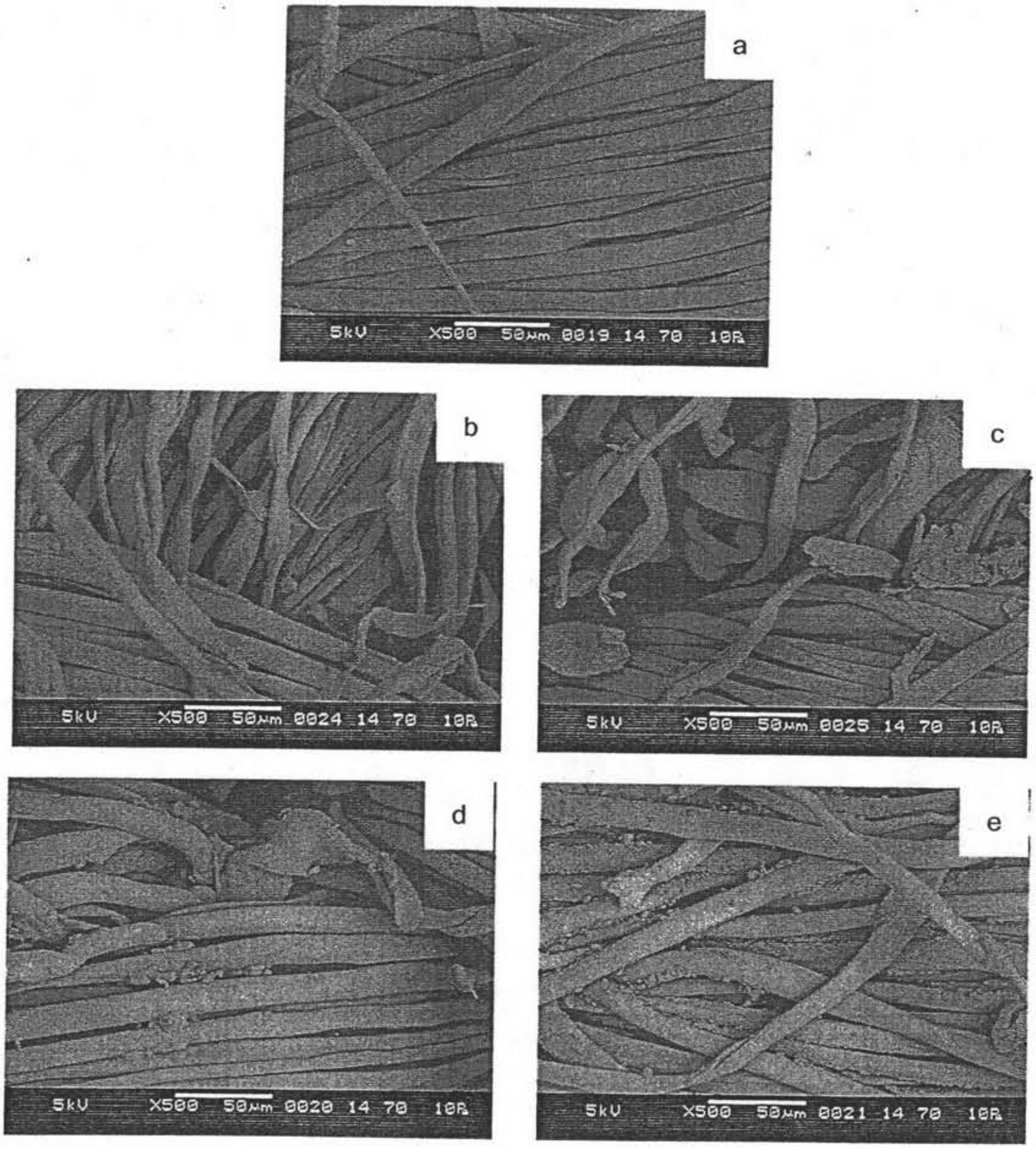

Figure 4.12 SEM photographs at X500 of Unt. C. (a), CNoP 5 (b), CNoP 10 (c) ,CNoPlas 15 (d), and CNoPlas 20 (e)

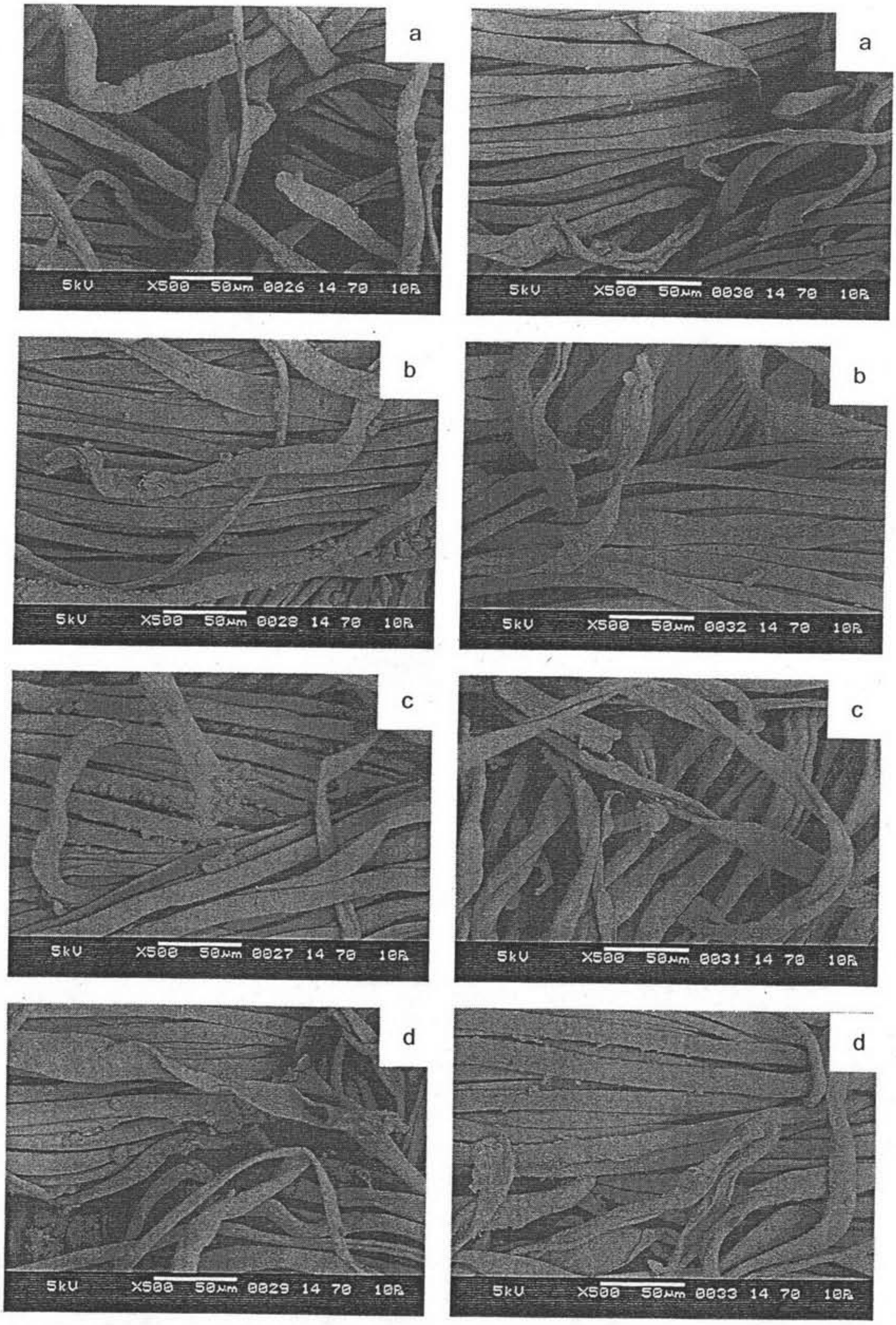

Figure 4.13 SEM photographs at X500 of C 5-15 (a), C 10-15 (b), P 5-20 (c), and C10-20 (d) before washing (left) and after washing (right) (con't)

From the Figure 4.13, it was found that DAP still contacted with the fabrics surface after washing process by the same token with the treated PET fabrics.

4.2.4 Whiteness and Yellowness

Table 4.8 presents the whiteness and yellowness of untreated and treated cotton fabrics before washing. Most of treated fabrics are yellower than untreated cotton fabric accepted for the fabrics that treated with DAP without nitrogen plasma. The changes of whiteness and yellowness were the results of DAP and the plasma treatment. The yellowness of the treated fabric increases with increasing the number of plasma shot and the concentration of DAP. The yellowness of the treated fabric increases with increasing the number of plasma shot because higher degradation of the fabric occurs when it is subjected to higher degree of plasma exposure [83].

Table 4.9 presents the whiteness and yellowness of untreated and treated cotton fabrics after washing. The treated fabrics are still yellower than the untreated one. In addition, the yellowness increases only with increasing the number of plasma shot while the concentration of DAP does not affect this property.

Table 4.8 The whiteness and yellowness of untreated and treated cotton fabrics before washing

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
Unt. C.	52.34	55.17	11.11	-	-	-	-
Unt. C.*	50.46	53.56	11.80	-	-	-	-
CNoP 1	51.08	54.45	11.67	-1.26	-0.72	0.56	0.69
CNoP 2	51.98	55.28	11.34	-0.36	0.11	0.23	0.59
CNoP 5	46.09	49.39	13.27	-6.25	-5.78	2.16	1.35

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
CNoP 10	48.18	51.46	12.58	-4.16	-3.71	1.47	0.97
CNoPlas 5	56.08	59.65	10.03	3.74	4.48	-1.08	0.99
CNoplas 10	54.76	59.64	10.20	2.42	4.47	-0.91	1.09
CNoPlas 15	56.19	58.41	10.70	3.85	3.24	-0.41	1.10
CNoPlas 20	56.37	59.66	10.30	4.03	4.49	-0.81	1.18
C 1-5	42.25	45.06	14.91	-10.09	-10.11	3.80	1.99
C 2-5	30.74	31.45	19.06	-21.60	-23.72	7.95	4.53
C 5-5	25.02	25.13	21.31	-27.32	-30.04	10.20	5.70
C 10-5	23.25	23.23	22.01	-29.09	-31.94	10.90	6.05
C 1-2*	44.51	47.15	13.97	-5.95	-6.41	2.17	1.29
C 2-2*	36.32	38.29	16.95	-14.14	-15.27	5.15	2.94
C 5-2*	27.79	28.27	20.22	-22.67	-25.29	8.42	4.84
C 10-2*	14.47	11.57	25.61	-35.99	-41.99	13.81	6.98
C 1-3*	38.53	40.81	16.20	-11.93	-12.75	4.40	2.24
C 2-3*	34.63	36.28	17.66	-15.83	-17.28	5.86	2.97
C 5-3*	24.31	23.38	21.78	-26.15	-30.18	9.98	5.02
C 10-3*	23.93	23.77	21.83	-26.53	-29.79	10.03	5.07
C 1-4*	43.94	46.66	14.25	-6.52	-6.90	2.45	1.39

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
C 2-4*	44.29	46.88	14.15	-6.17	-6.68	2.35	1.41
C 5-4*	35.39	37.40	17.41	-15.07	-16.16	5.61	3.13
C 10-4*	22.03	20.92	22.51	-28.43	-32.64	10.71	6.24

* compared with Unt. C.*

Table 4.9 The whiteness and yellowness of untreated and treated cotton fabrics after washing

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
Unt. C.	51.80	54.55	11.23	-	-	-	-
Unt. C.*	52.38	55.85	11.31	-	-	-	-
Unt. C.**	52.27	55.10	11.13	-	-	-	-
Unt. C.***	52.34	55.17	11.11	-	-	-	-
CNoP 1***	51.08	54.45	11.67	-1.26	-0.72	0.56	0.69
CNoP 2***	51.98	55.28	11.34	-0.36	0.11	0.23	0.59
CNoP 5***	46.09	49.39	13.27	-6.25	-5.78	2.16	1.35
CNoP 10***	48.18	51.46	12.58	-4.16	-3.71	1.47	0.97
CNoPlas 1	53.63	56.93	10.93	1.83	2.38	-0.30	0.74
CNoplas 2	52.92	56.30	11.23	1.12	1.75	0.00	0.75

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
CNoPlas 3*	54.52	57.75	10.61	2.14	1.90	-0.70	0.52
CNoPlas 4*	52.74	56.02	11.21	0.36	0.17	-0.10	0.27
C 1-5	47.07	50.12	13.20	-4.73	-4.43	1.97	1.00
C 2-5	42.54	45.00	14.75	-9.26	-9.55	3.52	1.87
C 5-5	40.28	42.81	15.62	-11.52	-11.74	4.39	2.31
C 10-5	40.08	42.59	15.67	-11.72	-11.96	4.44	2.34
C 1-2**	52.25	54.62	11.60	-0.02	-0.48	0.47	1.18
C 2-2**	49.69	52.13	12.44	-2.58	-2.97	1.31	1.11
C 5-2**	41.26	42.85	15.28	-11.01	-12.25	4.15	2.61
C 10-2**	33.14	33.30	18.33	-19.13	-21.80	7.20	4.44
C 1-3**	46.49	48.58	13.57	-5.78	-6.52	2.44	1.76
C 2-3**	44.31	46.30	14.25	-7.96	-8.80	3.12	1.99
C 5-3**	40.14	41.42	15.69	-12.13	-13.68	4.56	2.96
C 10-3**	36.57	37.63	17.10	-15.70	-17.47	5.97	3.57
C 1-4**	46.69	48.89	13.51	-5.58	-6.21	2.38	1.64
C 2-4**	45.04	46.98	14.22	-7.23	-8.12	3.09	2.15
C 5-4**	44.75	46.78	14.22	-7.52	-8.32	3.09	2.00
C 10-4**	35.21	35.57	17.68	-17.06	-19.53	6.55	4.17

* compared with Unt. C.*

** compared with Unt. C.**

*** compared with Unt. C.***

4.2.5 Flammability

In flammability test, it can be seen that after untreated and treated cotton fabrics were set on fire, the char formation occurs while the flame continues spreading as shown in Figure 4.14.

Flame spread rates of the untreated and treated cotton fabrics at 31°C and 52 percent of humidity are shown in Figure 4.15. The flame spread rates of all treated cotton fabrics are higher than that of the untreated cotton fabric. This would be cause of etching effect that occurred during plasma treatment process made the fabrics easier to combustion.

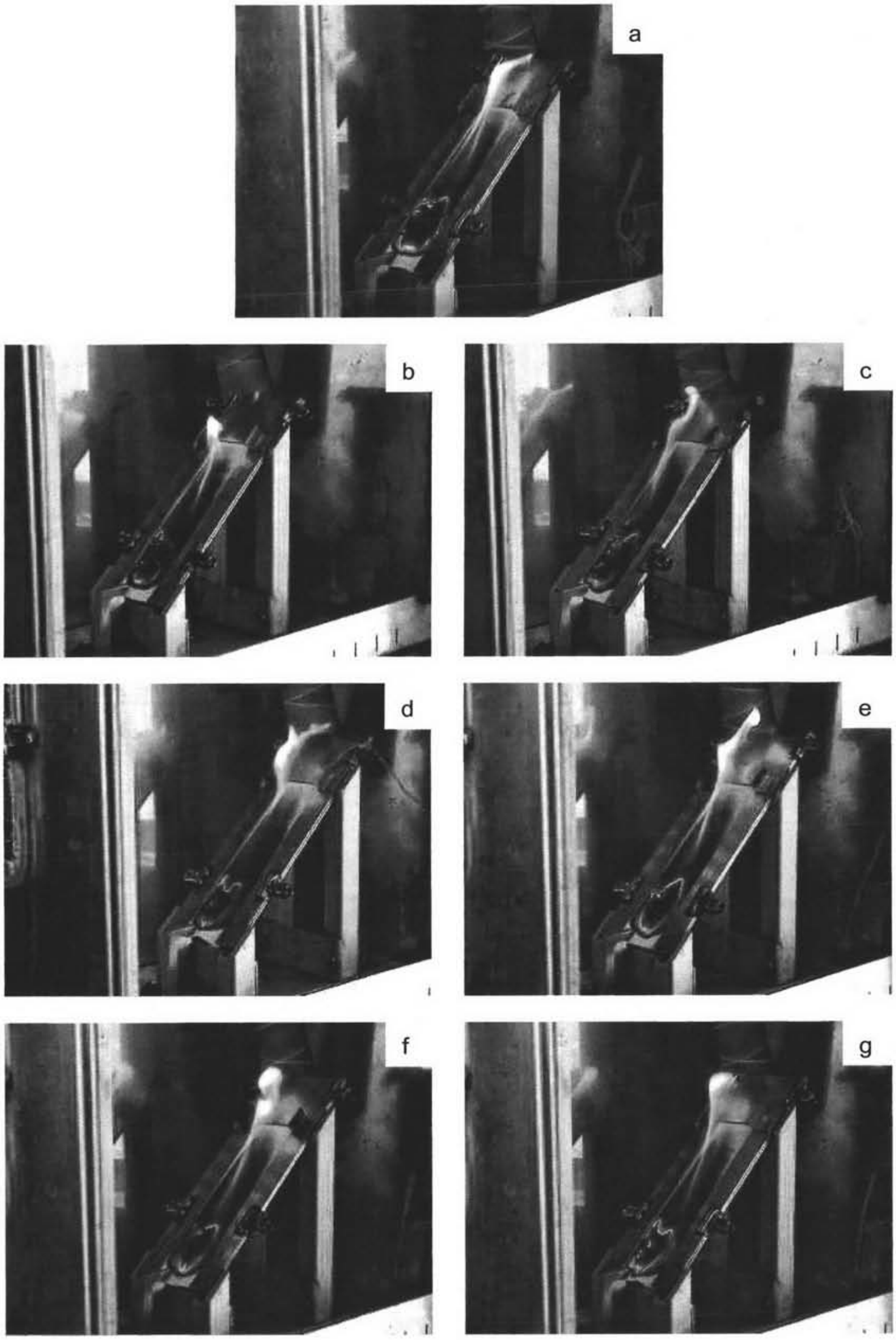

Figure 4.14 45 degree flammability test of untreated cotton fabric (a), CNoP 1(b), CNoPlas 15(c), C 1-15 (d), C 2-15 (e), C 5-15 (f), and C 10-15 (g)

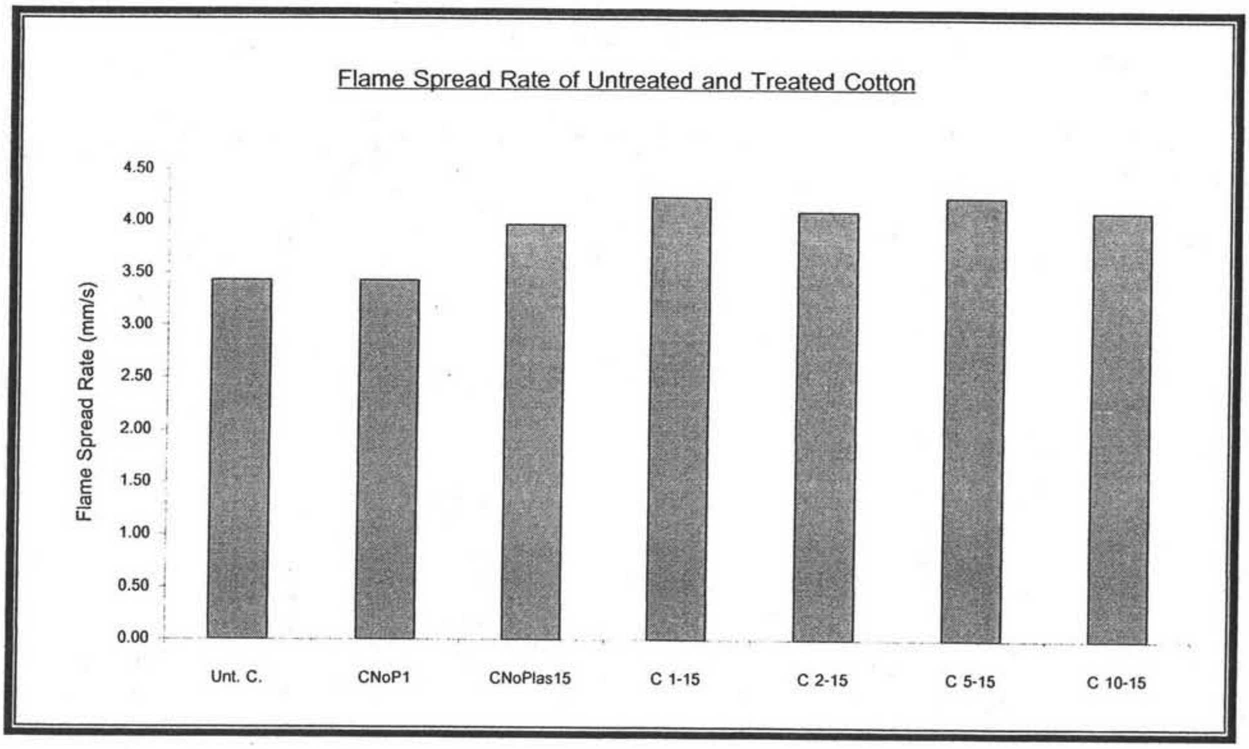

Figure 4.15 Flame spread rates of untreated and treated cotton fabrics

4.3 Properties and Morphology of Polyester/Cotton Blended Fabrics

4.3.1 Surface Chemical Structure

The ATR/FT-IR studies of untreated and treated T/C fabrics were showed in Figure 4.16 shows ATR/FT-IR spectra of untreated T/C fabric (spectrum a), 10 shots of nitrogen plasma treated cotton fabrics (spectrum b), 5 percent of DAP treated T/C fabric (spectrum c), and 5 percent of DAP with 10 shots of nitrogen plasma treated T/C fabrics (d). Also, Table 4.10 presents the interpretation of ATR/FT-IR spectra of these fabrics.

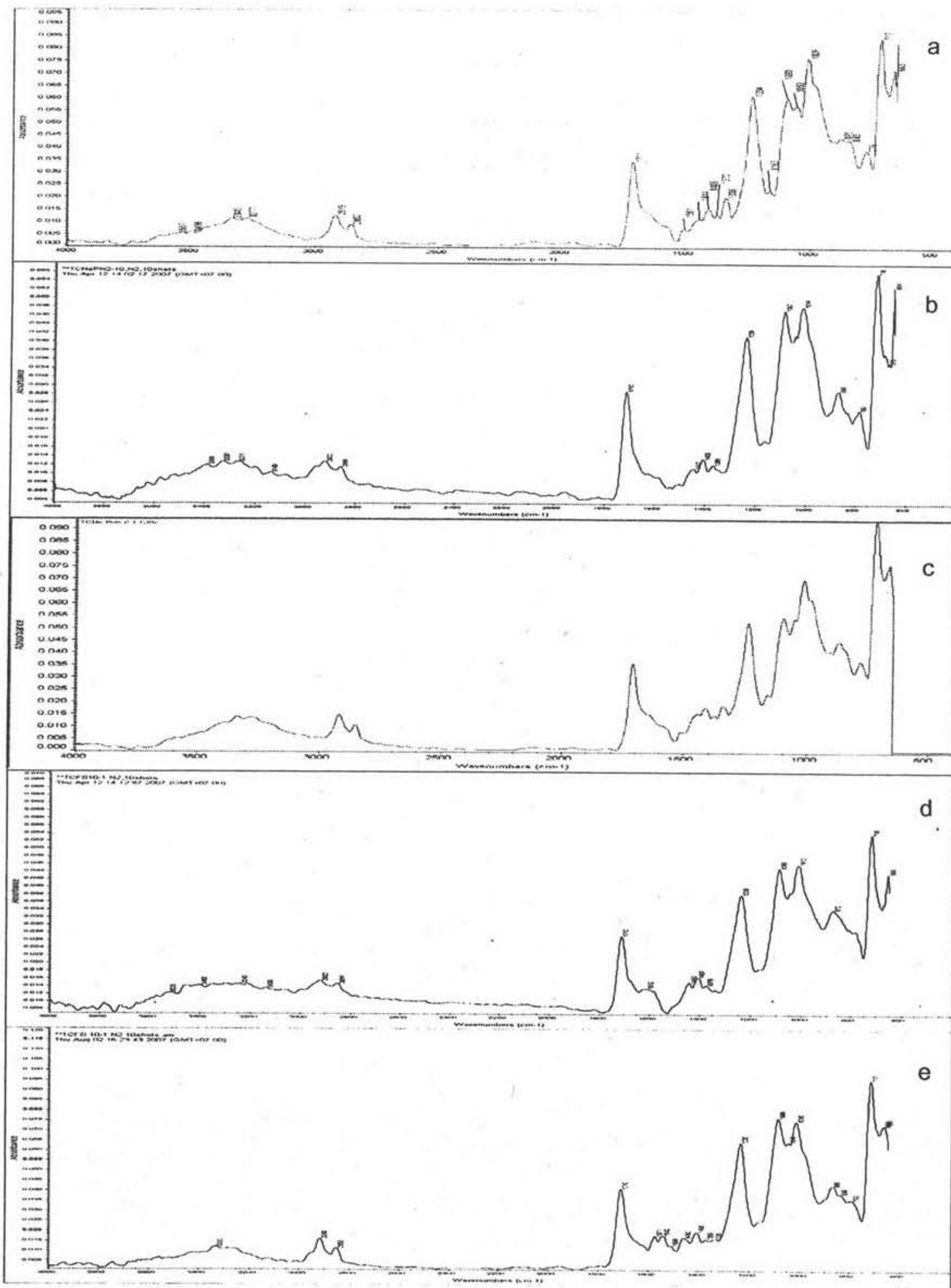

Figure 4.16 ATR/FT-IR spectra of Unt. TC. (a), TCNoP 10 (b), TCNoplas 5 (c), and TC 10-20 before (d) and after (e) washing

From these spectra, it is possible that the peak corresponding to –P-O-C- may occur at 793 cm^{-1} in Figure 4.16 (e). While the peak corresponding to –P-O-H peak may occur at 796 cm^{-1} in Figure 4.16 (e). In addition, P=O stretching peaks may occur at 1243 cm^{-1} in Figure 4.16 (c), 1238 cm^{-1} in Figure 4.16 (d), and 1239 cm^{-1} in Figure 4.16 (e) like the treated PET fabrics. Moreover, the peak at 1600 cm^{-1} attributed to C=N stretching of imine group was occurred on the fabrics that treated with DAP and plasma [7]. This shows that the fabrics are interact with DAP during the plasma treatment.

Table 4.10 The interpretation of ATR/FT-IR spectra of untreated and plasma-treated T/C fabrics

Wavenumber (cm^{-1})					Interpretation
Unt. TC.	TCNoP 10	TCNoplas 5	TC10-5 bw	TC10-5 aw	
3330	3330	3330	3330	3330	O-H stretching : intermolecular hydrogen bonding
2920	2920	2922	2926	2916	C-H stretching of -CH_2
1709	1713	1707	1708	1707	C=O stretching of the ester carbonyl group
1425	1426	1427	1418	1432	C-H bending of –CH_2
1401	1402	1404	1450,1404	1452,1403	Skeleton vibrations of the conjugated system and related to the p-disubstituted benzene rings
1338 ,1016	1338,1009	1340,1016	1015	1017	C-H in-plane bending in substituted aromatic rings and skeleton vibrations in-volving C-O stretching
1317,1202	1317,1202	1318,1302	1311	1316,1202	O-H in plane bending
1154	1154	1154	1153	1155	Anti-symmetrical bridge C-O-C stretching

Table 4.10 The interpretation of ATR/FT-IR spectra of untreated and plasma-treated T/C fabrics (con't)

Wavenumber (cm^{-1})					Interpretation
Unt. TC.	TCNoP 10	TCNoplas 5	TC10-5 bw	TC10-5 aw	
1240,1092	1231,1086	1243,1090	1238,1088	1239,1089	The ester C-O-C asymmetric and symmetric stretching vibrations -P=O (stretching)
1105	1105	1105	-	-	C-O stretching of secondary alcohol
967,845	959, 840	959	-	976	Wagging vibrations corresponding to CH_2- and oxyethylene groups
871	-	865	866	868	Out of plane C-H of p-substitued aromatic rings
-	-	-	-	793	P-O-C
719	720	716	716	716	Out of plane bending of the two carbonyl substituents on the aromatic ring
662	662	662	654	662	O-H out-of-plane bending

4.3.2 Wettability

The wettability of plasma-treated fabric samples treated in this research was determined by measuring the wetting time directly after plasma treatment.

As shown in Figure 4.17, it can be seen that the wetting time of untreated T/C fabric is lower than that of nitrogen plasma-treated T/C fabrics. After nitrogen plasma treatments, the results show that the wetting time increases. Additionally, the wetting time of nitrogen plasma-treated T/C fabrics apparently decrease with increasing number of plasma shots and the concentration of DAP. Exposing T/C fabrics to nitrogen plasma caused the incorporation of nitrogen-containing groups; especially, imine and phosphate groups.

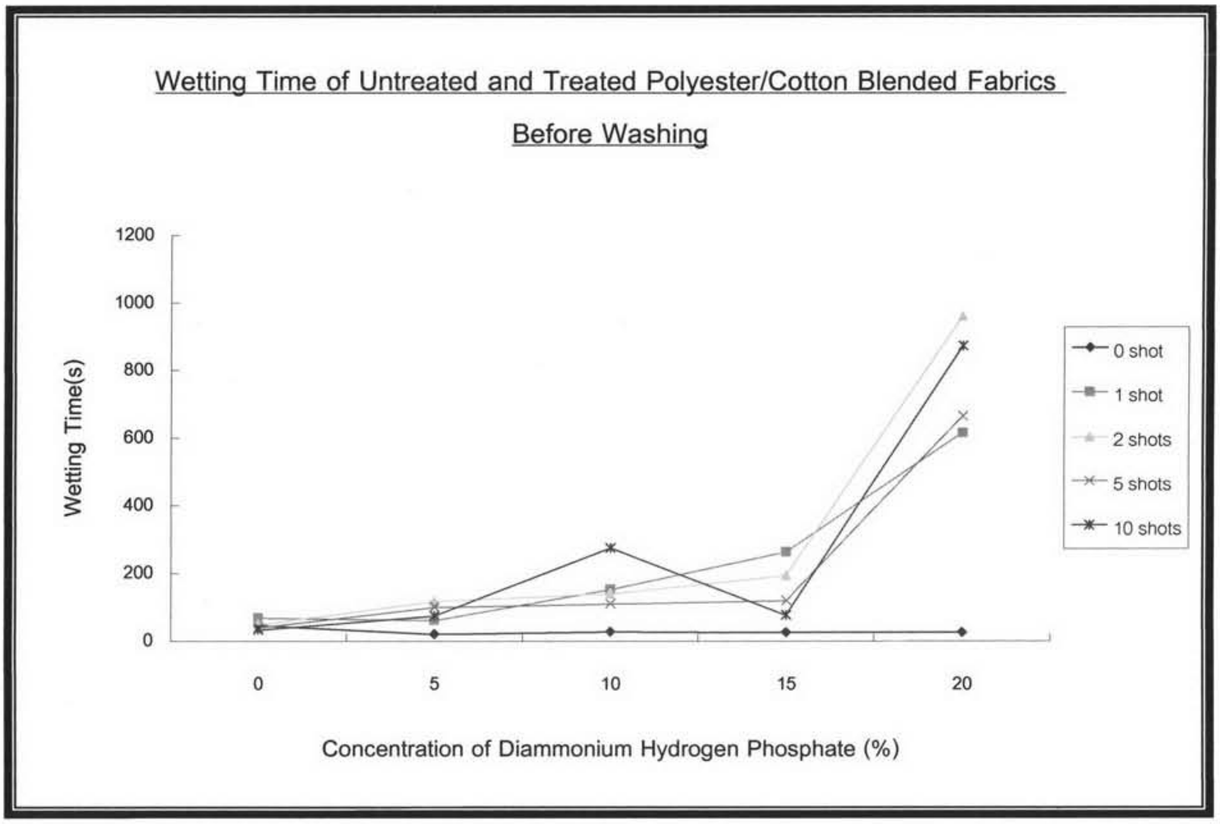

Figure 4.17 The wettability of untreated and treated T/C blended fabrics before washing

After washing process, the wetting time of the treated T/C fabrics are changed. This may be the result of the removing of DAP that do not interact with the fabric surface. The results show that the suitable concentration of DAP was 5 percent. Moreover, it is found that the wetting time of treated fabrics are increased with increasing the number of plasma shot as showed in Figure 4.18. This demonstrated that the fabrics surface are more interact with DAP when number of plasma shot increases.

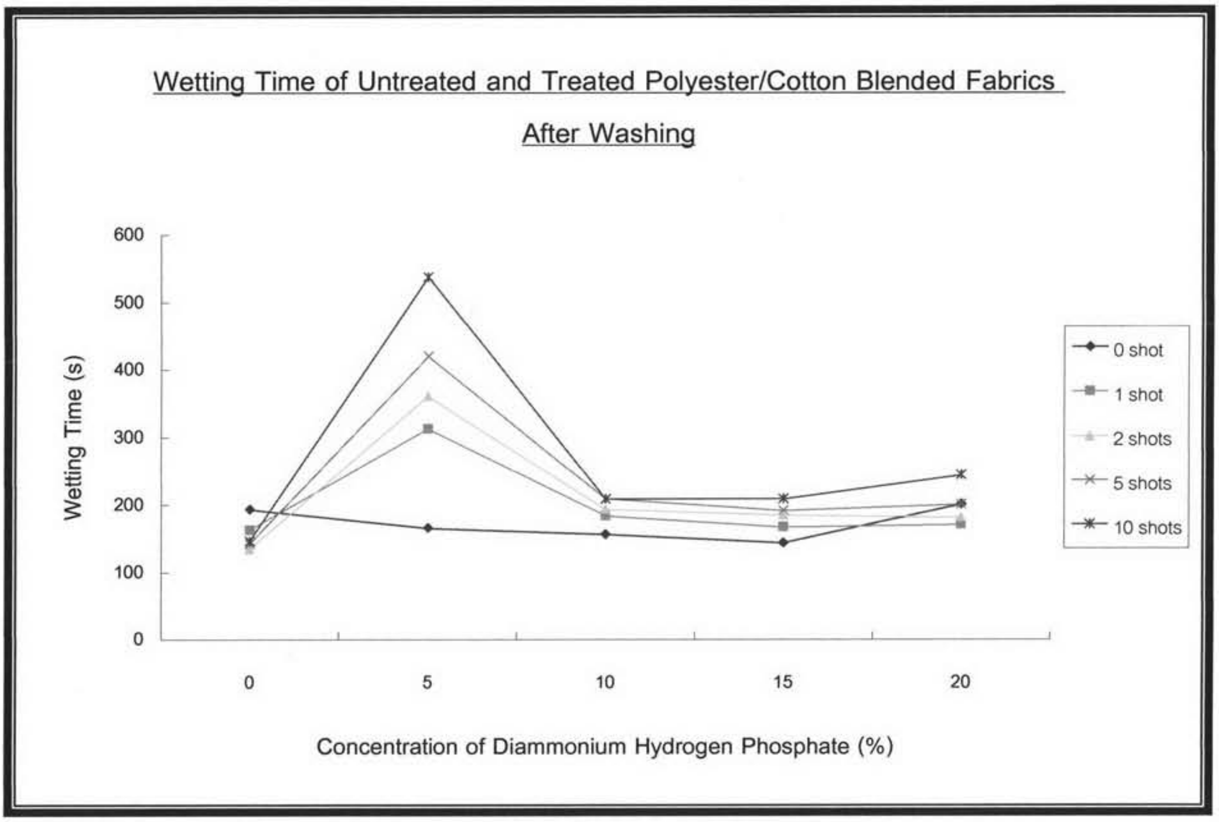

Figure 4.18 The wettability of untreated and treated T/C blended fabrics after washing

4.3.2 Morphological Analysis

Surface morphological changes on T/C fabrics were observed by SEM. Figures 4.19 (a) the original surface of untreated T/C is smooth. When they were exposed to 5 shots and 10 shots of nitrogen plasma (Figure 4.19 (b) and (c)), their surface were etched, resulting in surface roughness, the breaking and the melting of the fiber. These results can be attributed to etching process or ablation effect caused by the bombardment of nitrogen plasma reactive species on the fabric surface. When the fabrics were dipped in the 15 percent and 20 percent DAP solution (Figure 4.19 (d) and (e)), they were attached with DAP on their surface after drying.

The change of T/C fabric morphology is the same as PET fabric and cotton fabric.

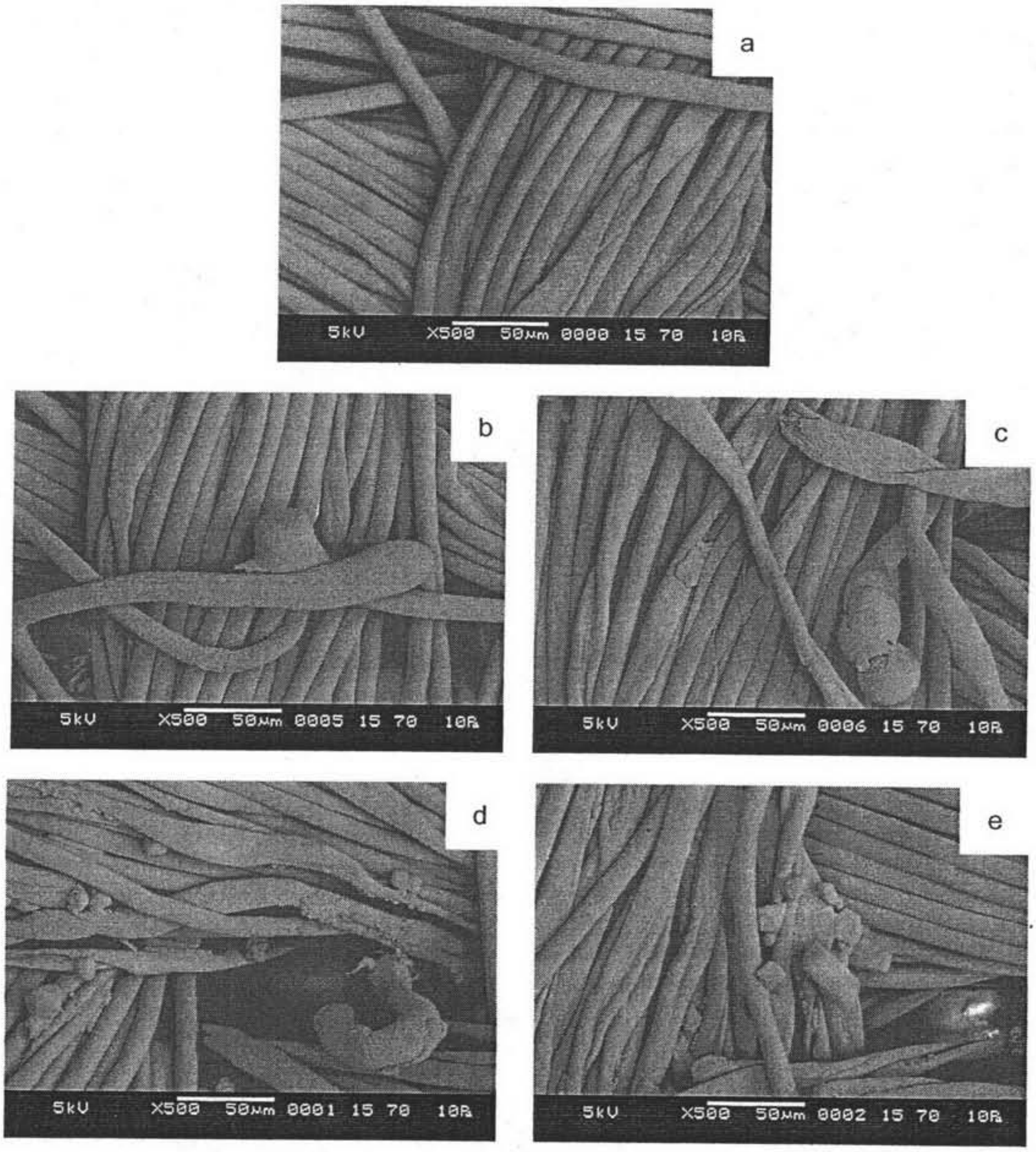

Figure 4.19 SEM photographs at X500 of Unt. TC. fabric (a), TCNoP 5 fabric (b), TCNoP 10 (c), TCNoPlas 15 (d), and TCNoPlas 20 (e)

The changes in the surface morphology of the fabrics observed after the plasma treatment can be explained by the localized degradation of polymer at the surface layer. The degradation seems to be the effect of the interaction of plasma with a polymer surface. This process leads to an almost completed breakdown of relatively small numbers of molecules at the surface into low molecular components which eventually vaporize in the low pressure system [7].

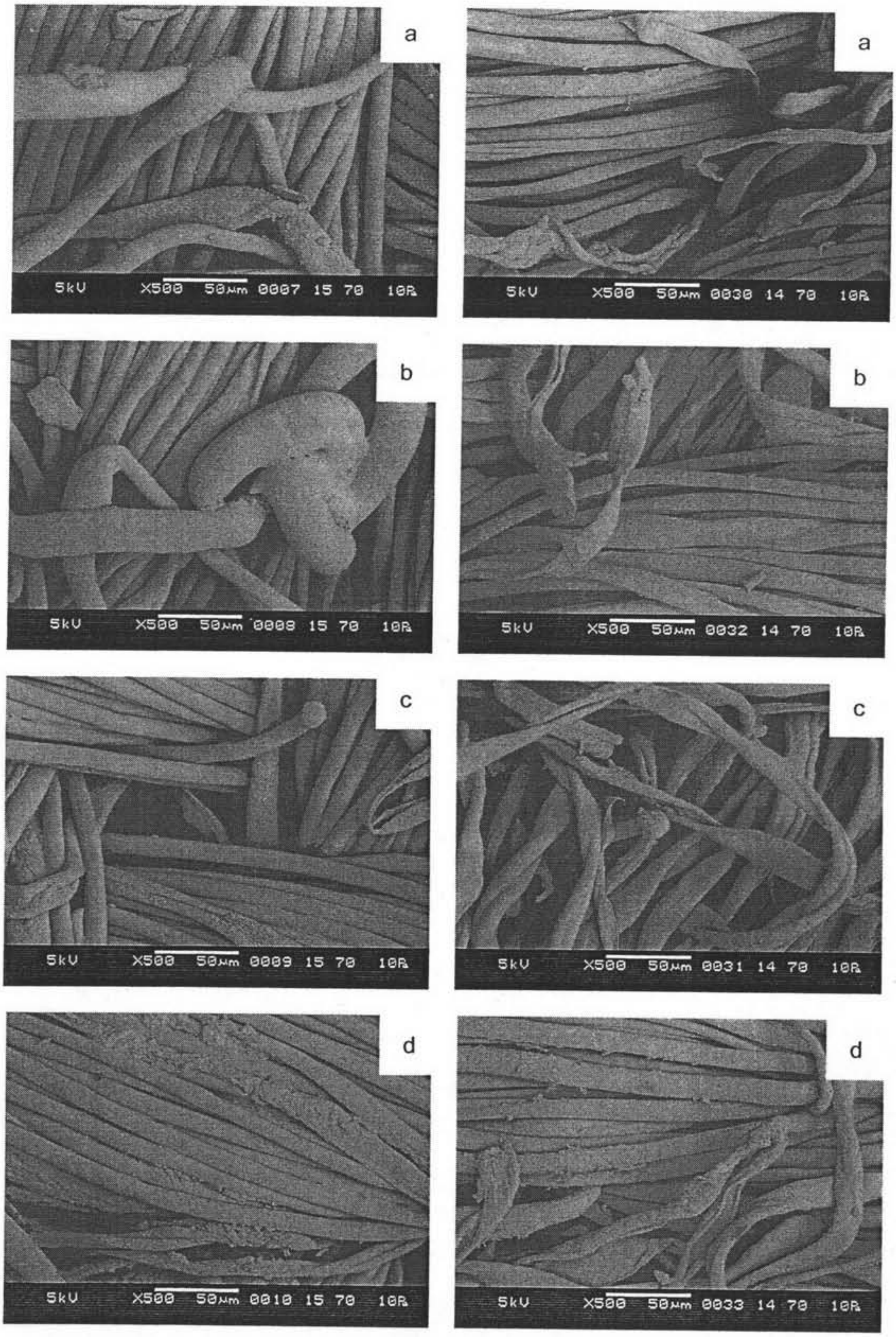

Figure 4.20 SEM photographs at X500 of TC 5-15 (a), TC 10-15 (b), TC 5-20 (c), and TC 10-20 (d) before washing (left) and after washing (right)

These photographs show that the surface of the fabrics are interact with DAP during plasma treatment because of the change in surface roughness of treated T/C fabric.

4.3.4 Whiteness and Yellowness

Table 4.11 presents the whiteness and yellowness of untreated and treated T/C fabrics before washing. The most of treated fabrics are yellower than untreated T/C fabric accepted the fabrics that treated with DAP without nitrogen plasma. The change of whiteness and yellowness is the result of DAP application and plasma treatment. The yellowness of the treated fabric was increased with the increasing of the number of plasma shot because higher degradation of the fabric occurs when it is subjected to higher degree of plasma exposure [83].

Table 4.11 The whiteness and yellowness of untreated and treated T/C fabrics before washing

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
Untreated	73.12	74.09	2.99	-	-	-	-
Untreated*	74.86	75.68	2.42	-	-	-	-
TCNoP 1	71.27	72.38	3.56	-1.85	-1.71	0.57	0.35
TCNoP 2	70.26	71.47	3.95	-2.86	-2.62	0.96	0.54
TCNoP 5	66.79	68.24	5.08	-6.33	-5.85	2.09	1.19
TCNoP 10	70.97	71.96	3.45	-2.15	-2.13	0.46	2.50
TCNoPlas 5	75.64	76.39	2.13	2.52	2.30	-0.86	0.46
TCNoplas 10	74.16	74.90	2.40	1.04	0.81	-0.59	0.44
TCNoPlas 15	74.52	75.20	2.17	1.40	1.11	-0.82	0.57

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
TCNoPlas 20	73.22	73.75	2.10	0.10	-0.34	-0.89	1.18
TC 1-5*	71.60	72.66	3.48	-3.26	-3.02	1.06	0.58
TC 2-5	63.71	65.20	6.15	-9.41	-8.89	3.16	1.72
TC 5-5*	62.32	63.84	6.59	-12.54	-11.84	4.17	2.29
TC 10-5*	52.77	54.30	9.89	-22.09	-21.38	7.47	4.12
TC 1-10*	71.09	72.18	3.61	-3.77	-3.50	1.19	0.68
TC 2-10*	70.70	71.77	3.68	-4.16	-3.91	1.26	0.75
TC 5-10*	63.33	64.78	6.18	-11.53	-10.90	3.76	2.11
TC 2-20*	67.85	69.07	4.57	-7.01	-6.61	2.15	1.27
TC 5-20*	66.59	67.89	5.05	-8.27	-7.79	2.63	1.50
TC 10-20*	59.05	60.52	7.59	-15.81	-15.16	5.17	2.91

* compared with Unt. TC.*

After washing process, the treated fabrics still have more yellowness than the untreated one. In addition, the yellowness increases only with increasing the number of plasma shot while the concentration of DAP does not affect this property as presented in Table 4.12.

Table 4.12 The whiteness and yellowness of untreated and treated T/C fabrics after washing

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
Untreated	73.00	73.92	2.98	-	-	-	-
Untreated*	76.25	76.96	1.98	-	-	-	-
Untreated**	73.12	74.09	2.99	-	-	-	-
TCNoP 1**	71.27	72.38	3.56	-1.85	-1.71	0.57	0.35
TCNoP 2**	70.26	71.47	3.95	-2.86	-2.62	0.96	0.54
TCNoP 5**	66.79	68.24	5.08	-6.33	-5.85	2.09	1.19
TCNoP 10**	70.97	71.96	3.45	-2.15	-2.13	0.46	2.50
TCNoPlas 5*	73.13	74.19	3.14	-3.12	-2.77	1.16	0.62
TCNoplas 10*	70.62	71.87	3.92	-5.63	-5.09	1.94	1.06
TCNoPlas 15	74.21	75.14	2.76	1.21	1.22	-0.22	0.32
TCNoPlas 20	73.89	74.83	2.82	0.89	0.91	-0.16	0.25
TC 1-5	73.90	74.88	2.90	0.90	0.96	-0.08	0.33
TC 2-5	65.55	67.04	5.62	-7.45	-6.88	2.64	1.40
TC 5-5	63.03	64.67	6.51	-9.97	-9.25	3.53	1.88
TC 10-5	54.65	56.05	9.17	-18.35	-17.87	6.19	3.42
TC 1-10	72.73	73.58	2.88	-0.27	-0.34	-0.10	0.22

Fabric	Whiteness		Yellowness	D Whiteness		D Yellowness	DE*
	ASTM	CIE	E313	ASTM	CIE	E313	
TC 2-10	72.02	72.95	3.16	-0.98	-0.97	0.18	0.23
TC 5-10	67.76	68.88	4.48	-5.24	-5.04	1.50	0.98
TC 10-10	65.26	66.50	5.36	-7.74	-7.42	2.38	1.42
TC 1-15	73.74	74.51	2.54	0.74	0.59	-0.44	0.28
TC 2-15	70.81	71.82	3.55	-2.19	-2.10	0.57	0.42
TC 5-15	69.68	70.70	3.87	-3.32	-3.22	0.89	0.65
TC 10-15	65.70	66.94	5.23	-7.30	-6.98	2.25	1.34
TC 1-20	71.32	72.29	3.40	-1.68	-1.63	0.42	0.34
TC 2-20	70.14	71.18	3.79	-2.86	-2.74	0.81	0.54
TC 5-20	67.45	68.51	4.51	-5.55	-5.41	1.53	1.11
TC 10-20	62.42	63.77	6.41	-10.58	-10.15	3.43	1.95

* compared with Unt. TC.*

** compared with Unt. TC.**

4.3.5 Flammability

In flammability test, it can be seen that after untreated and treated T/C fabrics were set on fire, the char formation occurs while the flame continues spreading and the molten fabrics accompanying with the flame drop to the bottom of the tester as shown in Figure 4.21.

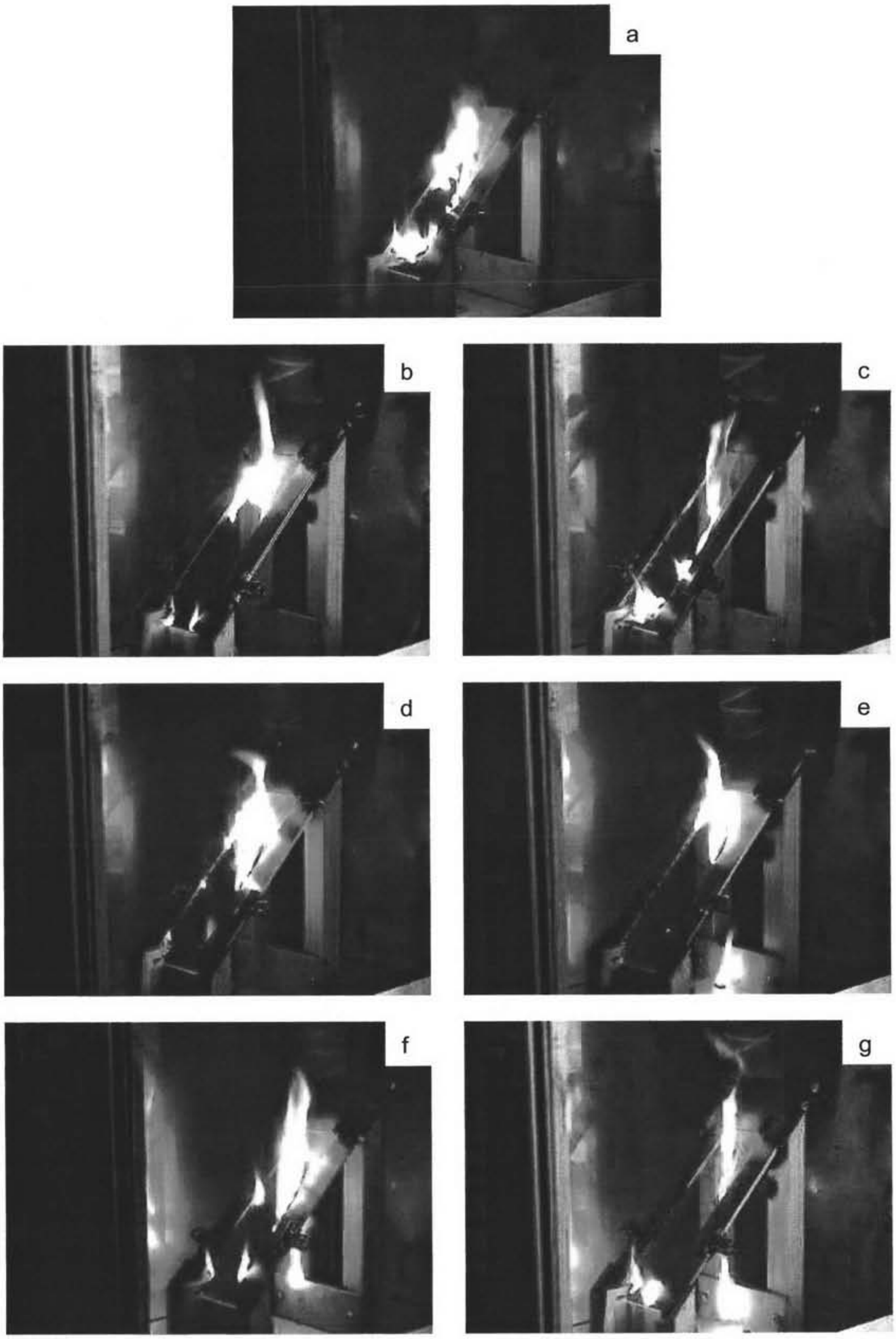

Figure 4.21 45 degree flammability test of untreated TC fabric (a), TCNoP 10 (b), TCNoPlas 5 (c), TC 1-5 (d), TC 2-5 (e), TC 5-5 (f), and TC 10-5 (g)

Figure 4.22 shows the flame spread rates of the untreated and treated T/C fabrics at 31°C and 52 percent of humidity. The flame spread rates of all treated T/C fabrics are lower than the untreated T/C fabric. This means all treated T/C fabrics have lower flammability than the untreated one. The flame spread rate decreases with increasing the number of plasma shot from 0 to 2 shots that may be the result of the interaction between DAP and fabric surface. Since the number of plasma shot is 5 shots, the flame spread rates return to high value again. This may be the result of the etching effect that etched the fabric surface.

So the plasma treatment with DAP is an effective process for flame retardant finished the T/C fabric surface at 2 shots with 5 percent of DAP.

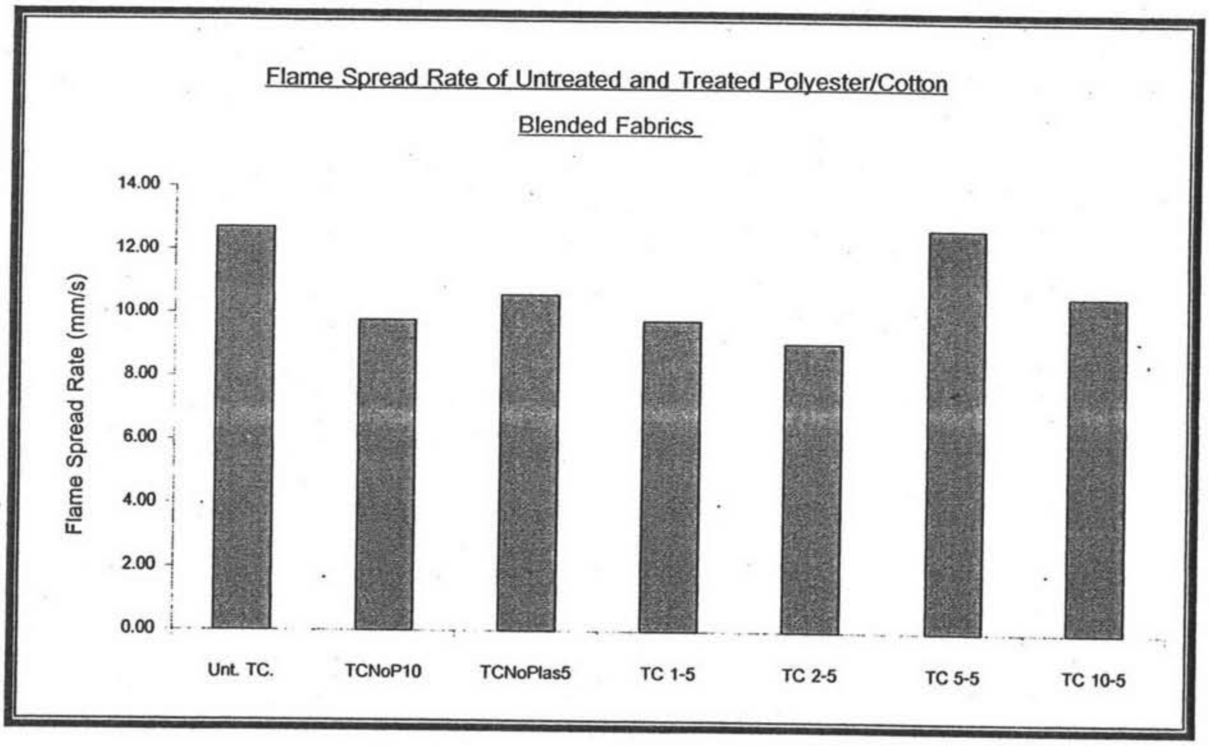

Figure 4.22 Flame spread rates of untreated and treated T/C fabrics