

CHAPTER IV

Results and Discussions

Preparation of Crease Resistant Finishing Fabrics.

Three lots of desized, scoured, bleached and mercerized, plain woven cotton fabrics from different suppliers were used in this work. Before being finished, the existence of starch on the fabrics was tested by iodine solution. After dropping iodine solution on the fabrics, no starch on fabric was detected. This indicates that the fabrics have been pretreated completely and can be proceeded in finishing treatment.

Although three lots of fabrics were used but the physical properties of each lot of the untreated were evaluated individually as presented in Table 4.1. Mostly results of this work were, however, obtained from the lot A fabric.

Table 4.1 The physical properties of fabric.

Properties	Results		
	Lot A	Lot B	Lot C
Weight (g/m ²)	152.5	109.5	119.2
No. of yarn	20	50	45
Picks/inch	57	80	70
Ends/inch	68	150	138

Table 4.1 (Continued)

Properties	Results		
	Lot A	Lot B	Lot C
DCRA (degree)			
filling (f)	71.9	62.9	79.5
warp (w)	70.2	70.1	80.5
WCRA (degree)			
filling	68.7	-	-
warp	67.1		
Tensile strength (N) per 5 cm revelled strip			
filling	389.9	-	-
warp	454.5		
CIE Whiteness Index	50.7	78.7	76.2

Effect of Curing Temperature on Whiteness.

From the theory of pad-dry-cure process, curing of treated fabric is carried out at temperature higher than 140°C. However, at too high temperature, the desired properties of fabrics will be destroyed, so the influence of temperature on treated fabric must be studied.

The cotton fabric (lot B) was padded with 90% wet add-on in the solution of 5% citric acid and disodium hydrogenphosphate (Na_2HPO_4) with the mole ratio of 1:1. After drying at 95°C for 3 min, each of the treated fabric was cured at different temperature and time of curing then whiteness and dry crease recovery angle (DCRA) were evaluated.

Table 4.2 Whiteness of finished fabrics from treatment with 5% CA and Na_2HPO_4 over a range of different temperature of curing.

Curing Conditions		CIE Whiteness Index
150°C	60 sec	79.6
160	60	79.2
170	60	73.7
180	60	65.8
Untreated		78.7

Table 4.3 Effect of temperature and time of curing on fabric properties from treatment with 5% CA and Na_2HPO_4 .

Curing Conditions		CIE Whiteness Index		DCRA (w+f)
		Unwashed	Washed	
150°C	30 sec	81.6	81.8	128.3
	90	78.6	81.7	144.3
	180	77.9	80.6	159.5
160°C	30	81.4	82.0	150.3
	90	78.6	80.8	157.0
	180	74.3	76.8	173.5
Untreated		78.7		133.0

(10 replicates of DCRA within 5% CV)

The effect of different temperature of curing, while the period of time was fixed at 1 min, on fabric whiteness was examined, as seen in Table 4.2. The data

shows that the CIE whiteness values begin to decline at the temperature of 170° C , so the temperature higher than 160° C will be neglected. When study at two levels of temperature, 150° C and 160° C, with different time of curing, whiteness of fabrics as well as DCRA were measured (Table 4.3). Although the satisfactory value of DCRA can be gained at 160° C, 180 sec, but the fabric will loss its whiteness even after washed. Andrews and Trask-Morrell (1991) presumed that the discoloration of CA treated fabric at high temperature of curing was from dehydration to form aconitic acid and/or from trace amounts of sugars as impurities (Andrews, Welch and Trask-Morrell, 1989). An interesting data at 150° C can be obtained with curing time of 180 sec which can be comparable to the data obtained at 160° C, 90 sec, approximately 20% of resiliency has been improved whereas whiteness of fabric was quite constant, that means the faster cured can be done by increasing curing temperature and from this result, the curing condition at 160° C for 90 sec will be chosen and fixed in the next experiments.

Effect of Citric Acid (CA) with Various Catalysts on Fabric Properties.

To determine the effect of catalyst on CA treated fabric, the catalysts used were tetra-sodiumpyrophosphate decahydrate ($\text{Na}_4\text{P}_2\text{O}_7 \cdot 10\text{H}_2\text{O}$), di-sodiumhydrogen phosphate dihydrate ($\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$), sodium hypophosphite monohydrate ($\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$) and sodium dihydrogenphosphate dihydrate ($\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$). The concentration of CA and each catalyst used here is 5% (w/v). The fabrics (lot A) were padded with 90% wet add-on. The padded fabrics were dried at 95° C for 3 min and cured at 160° C for 90 sec.

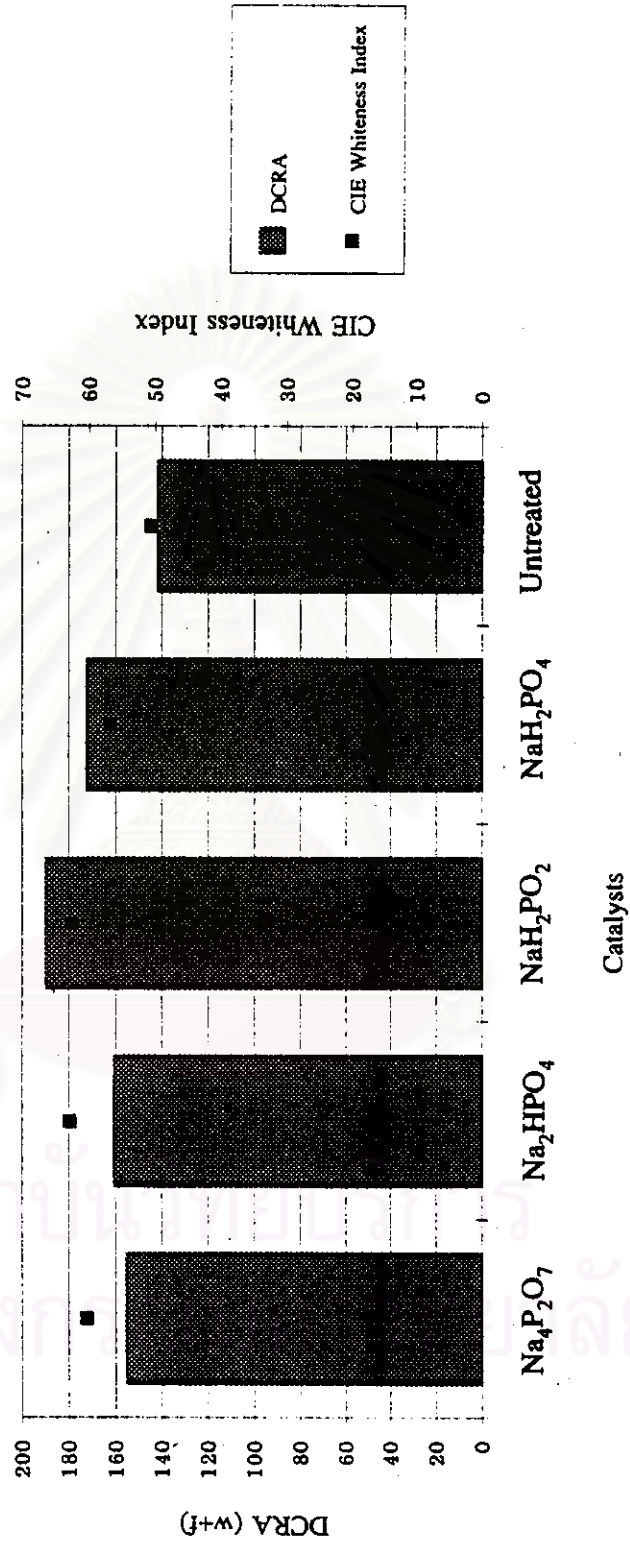
Table 4.4 Properties of fabric finished with citric acid and phosphorus-containing catalysts.

Catalyst	CIE Whiteness Index	DCRA (w+f)
5% $\text{Na}_4\text{P}_2\text{O}_7$	60.4	155.7
5% Na_2HPO_4	62.9	161.0
5% NaH_2PO_2	62.6	190.3
5% NaH_2PO_4	56.8	172.8
Untreated	50.7	142.1

(10 replicates of DCRA within 6% CV)

Table 4.4 and Figure 4.1 shows the effect of catalyst on whiteness and crease recovery property in CA finished fabrics. The catalyst that has proven most successful in esterification cross-linking is sodium hypophosphite which has the same result as those carried out by Andrews (1990) and Welch (1990). Di-sodium hydrogen phosphate produces a white fabric but poor appearance properties. Although mono-sodium dihydrogen phosphate has a tendency to produce a faint yellowing in cotton during heat curing; but it has shown to be a fairly active catalyst. From this results, it demonstrates that citric acid, as a cross-linking agent, can impart improved crease recovery property to cotton fabric especially when sodium hypophosphite is used as a catalyst. However, the optimum concentration of citric acid and sodium hypophosphite is still an interesting case needed to be determined further. Sodium dihydrogen phosphate, another interesting catalyst, is also being studied the possibility for using with sodium hypophosphite as a mixed catalyst.

Figure 4.1 CIE whiteness index and DCRA of fabric finished with CA and phosphorus-containing catalysts.



Effect of Ratio of CA/NaH₂PO₂ on Fabric Properties.

To determine the suitable ratio of CA/NaH₂PO₂, the fabric (lot A) were treated with CA/NaH₂PO₂ in the ratio of 1 : 1.0, 1 : 1.5, 1 : 2.0, and 1 : 2.5 by the same procedures as the previous experiment. The concentration of CA used in this test were separated into two levels, 5% and 10% respectively.

Table 4.5 Fabric properties from treatments with 5% CA and sodium hypophosphite over a range of CA/NaH₂PO₂ ratios.

CA/NaH ₂ PO ₂ Ratio	CIE Whiteness Index	DCRA (w+f)
1 : 1	57.3	184.2
1 : 1.5	58.8	185.6
1 : 2.0	58.7	186.1
1 : 2.5	60.2	184.0
Untreated	50.7	142.1

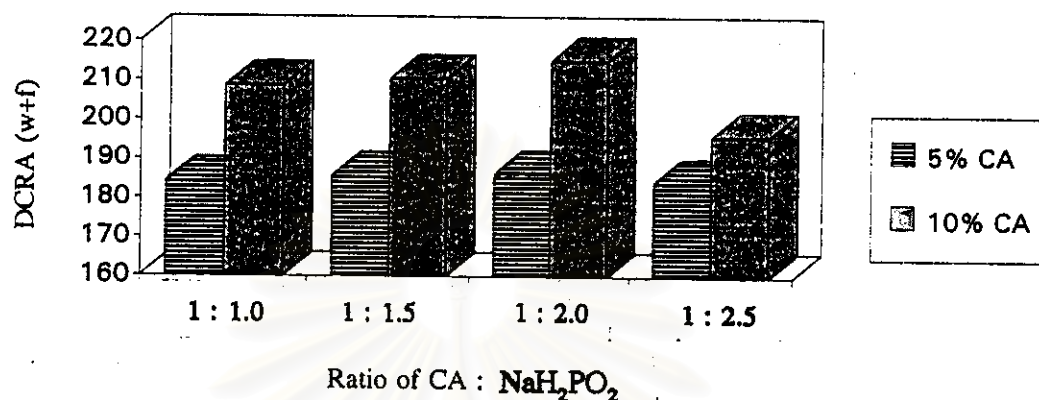
(10 replicates of DCRA within 5% CV)

Table 4.6 Fabric properties from treatments with 10% CA and sodium hypophosphite over a range of CA/NaH₂PO₂ ratios.

CA/NaH ₂ PO ₂ Ratio	CIE Whiteness Index	DCRA (w+f)
1 : 1.0	48.8	208.7
1 : 1.5	53.7	210.5
1 : 2.0	55.1	215.1
1 : 2.5	58.2	195.9
Untreated	50.7	142.1

(10 replicates of DCRA within 5% CV)

Figure 4.2 Effect of various ratios of CA/ NaH_2PO_2 on DCRA.



At 5% CA, Table 4.5 as well as Figure 4.2 shows a slight increase in resiliency and there is no obvious difference result between the different ratios. As shown in Table 4.6, when the higher concentration of citric acid was used, the additional amount of CA causes the fabric to get yellowish which can be reduced by increasing the amount of NaH_2PO_2 . It can be seen that when the acid to catalyst ratio was increased, fabric appearance was accordingly improved. For both levels of citric acid, it was found that the balance of DCRA (51% improvement) and whiteness appeared to be at a ratio of 1: 2 and dropped off at 1: 2.5.

From the work studied by Andrews (1990), by treatments with 7% CA, the best properties of fabric appeared to be at 1 : 1.5 for CA/sodium hypophosphite system. In his work, by curing at 180°C for 90 sec, the whiteness of washed fabric was dropped from 87 to 56 with 51% DCRA improved and 49% loss of breaking strength.

Effect of Concentration of CA on Fabric Properties.

To find out a suitable concentration of citric acid, sodium hypophosphite was used as a catalyst at a mole ratio of 1: 2 . The effects of the concentration variations of citric acid on dry and wet crease recovery, breaking strength as well as the whiteness of the finished fabric (lot A) are summarized in Table 4.7.

Table 4.7 Effect of concentrations of citric acid on fabric properties.

% CA (w/v)	CIE Whiteness Index	CRA(w+f)		Breaking Strength (N)		% Strength loss (w+f)
		Dry	Wet	filling	warp	
5	59.1	186.7	185.1	258.0	332.3	30.09
7	59.1	208.1	204.8	258.7	327.3	30.60
9	57.2	209.4	208.6	255.0	333.0	30.36
11	56.5	212.1	211.3	249.6	337.5	30.47
Untreated	50.7	142.1	135.8	389.9	454.5	-

(10 replicates of DCRA within 5% CV, 5 replicates of breaking strength within 7% CV)

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Figure 4.3 Effect of citric acid concentration on crease recovery property.

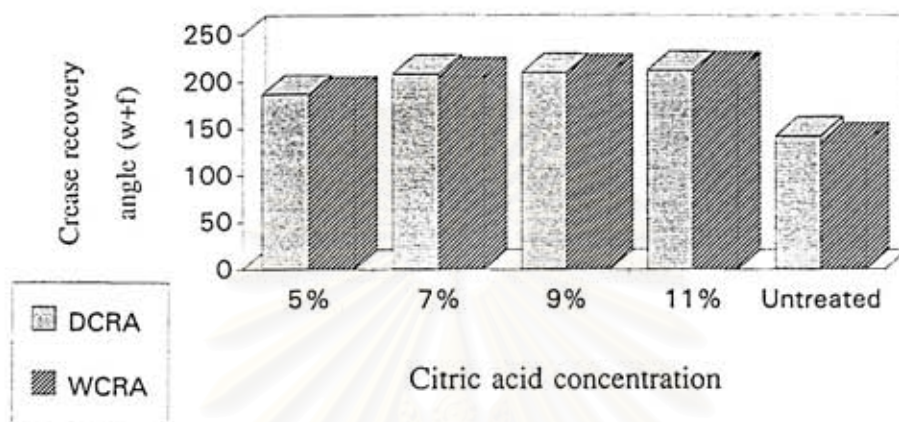
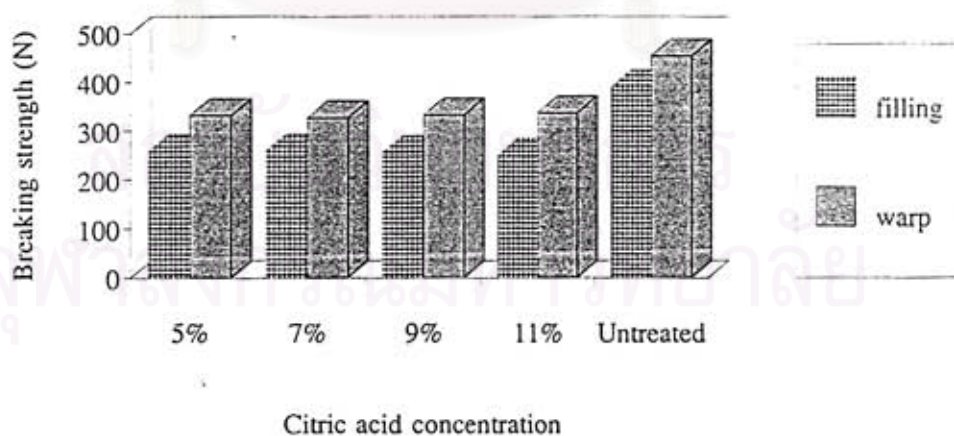


Figure 4.4 Effect of citric acid concentration of fabric strength.



The results illustrate that there is no different in tensile strength over the range of 5-11% citric acid. The increasing amount of citric acid made the whiteness of fabrics decrease, but still higher than the untreated. At 7% CA the wrinkle recovery shows a remarkable, 46.4%, DCRA improvement from 142.1 (w+f) of the original to 208.1. When the concentration of citric acid was higher than 7%, however, a significant increase in crease recovery was not achieved so 7% citric acid was found to be the most suitable concentration. This result corresponds with those proposed by Andrews, Welch and Trask-Morrell (1989). In those works, however, with CA and NaH_2PO_2 in 1 : 1 formula weight ratio by curing at 180°C for 90 sec, the strength of fabrics were seriously lost by 59%.

Effect of Curing Temperature on Properties of Fabrics Finished with 7% CA.

To study the relationship among whiteness, resiliency, breaking strength and curing temperature of citric acid finished cotton fabrics (lot A), the experiment was carried out by using 7% CA and sodium hypophosphite with the ratio of 1: 2 and drying at 95°C for 3 min. The properties of fabrics with various temperatures and times of curing are shown in Table 4.8.

The influence of temperature and time of curing on the fabrics properties are shown in Table 4.8 and Figure 4.5. At each level of temperature, whiteness index and fabric strength were decreased by longer time of curing, in contrast, the finishing agents were more effective in improving resiliency of the fabrics. When fabrics were cured at 170°C for 50 sec, the superior DCRA was achieved whereas the decrement of fabric strength was not significantly differ from those of curing at lower temperature, however, this too short time of curing causes more reduction in

Table 4.8 Curing conditions and properties of finished cotton fabric.

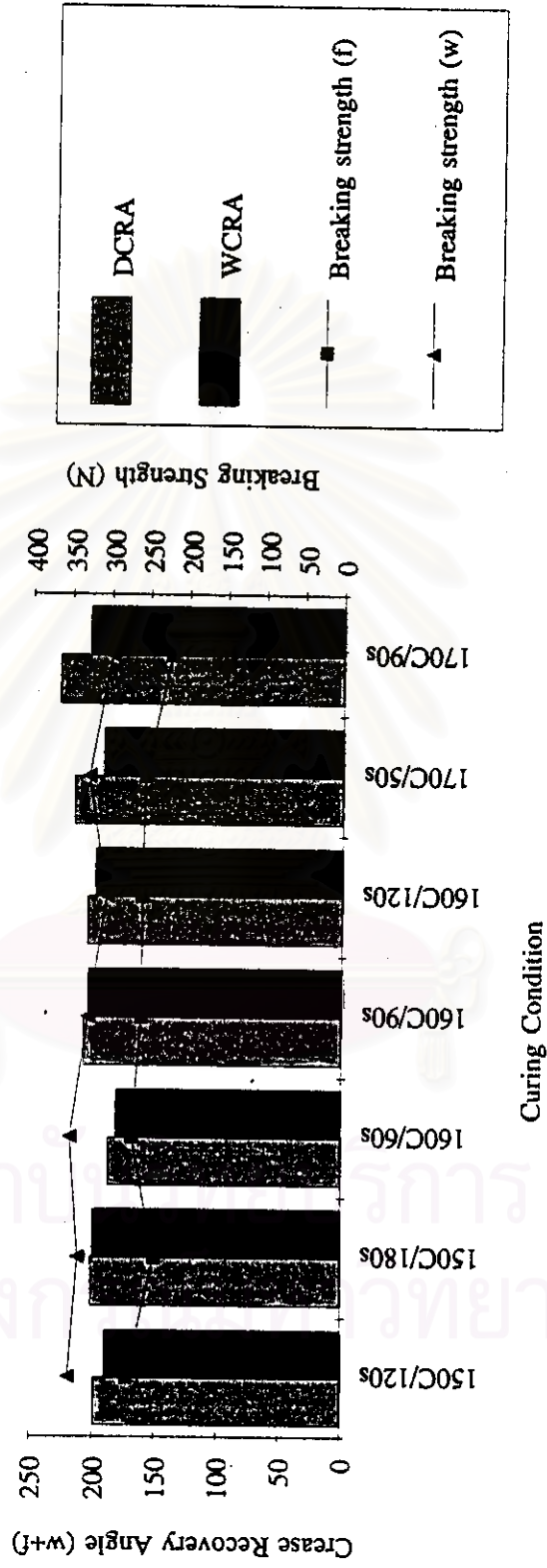
Cure Temp. (C°)	Cure Time (sec)	CIE Whiteness Index	CRA (w+f)		Breaking Strength (N)		% Strength loss (w+f)
			Dry	Wet	filling	warp	
150	120	63.4	198.9	190.3	276.9	351.3	25.60
	180	61.0	201.6	200.0	240.6	339.1	31.35
160	60	61.0	188.1	181.8	270.2	349.1	26.66
	120	56.0	205.5	199.0	258.5	304.5	33.32
170	50	55.2	216.5	193.5	254.9	328.0	30.97
	90	46.0	228.6	204.8	222.1	299.4	38.24
Untreated	-	50.7	142.1	135.8	389.9	454.5	-
160	90	59.1	208.1	204.8	258.7	327.3	30.60

(10 replicates in CRA within 5% CV, 5 replicates of breaking strength within 7% CV)

wet crease recovery property. The best improvement of crease recovery appears to be obtained from curing at 170°C for 90 sec, however, the fabrics started to get yellowish and the fabric strength was seriously deteriorated by 38%.

By comparison with the previous experiment data of the fabric cured at 160°C for 90 sec in the same procedure, the better balance between whiteness retention and physical properties can be accomplished.

Figure 4.5 Effect of curing condition on properties of fabrics finished with 7% CA.



Effect of Repeated Laundering on DCRA of Fabrics.

The durability of crosslinks on laundering was determined from dry crease recovery of fabrics. The cotton fabrics (lot A) were treated with 7% CA and sodium hypophosphite was used as a catalyst at a ratio of 1 : 2. After drying at 95°C for 3 min and curing at 160°C for 90 sec, the finished fabrics were washed by launder-o-meter. DCRA of fabrics were measured after one cycle as well as 5 and 10 replication of laundering (See procedures in the appendix.).

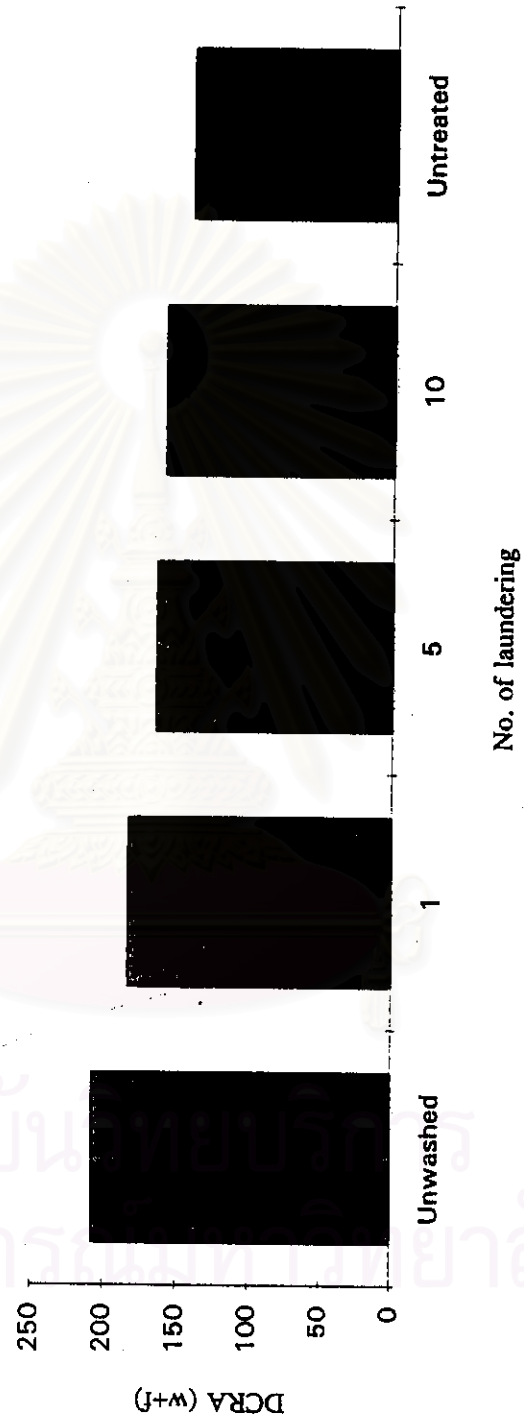
Table 4.9 Crease recovery property of treated fabric after repeated laundering.

No. of Laundering	DCRA (w+f)	%Loss in DCRA
Unwashed	208.1	-
1	183.7	11.72
5	165.1	20.66
10	159.5	23.35

(10 replicates within 5% CV)

The percentages of loss in DCRA after one cycle of laundering as shown in Table 4.9 and Figure 4.6 indicate that there were the excess chemical, that did not proceed cross-linking reaction, deposited on the surface of and in between the fibers, thereby glueing the fibers together and giving a sized effect (Mark, Wooding and Atlas, 1971) which can be dissolved out in subsequent laundering. In ecological viewpoint, this result has suggested the consumer for the fact that the toxicity from chemicals during the textile processes left on the fabrics is invisible and this is the reason that why the textile products must be laundered first before being used.

Figure 4.6 Effect of repeated laundering on DCRA.



After 10 cycles of laundering, 76% of crease recovery property still remains in the finished fabric. This can be proven that the use of citric acid as cross-linking agent can impart permanent crease resistant finishing for cotton fabric.

Effect of Various Ratios of $\text{NaH}_2\text{PO}_2/\text{NaH}_2\text{PO}_4$ as Mixed Catalysts on Fabrics Properties.

Because sodium hypophosphite is among the more expensive phosphorus salts, catalyst mixture of this salt with less costly sodium dihydrogenphosphate was used in citric acid finishing of cotton fabric. Ratios of sodium hypophosphite with sodium dihydrogenphosphate were ranged from 20 : 80 to 80 : 20 (mole ratios). The fabrics (lot A) were treated in the same way as the previous experiments.

Table 4.10 Effective treatment with 7% citric acid and mixtures of sodium hypophosphite/sodium dihydrogenphosphate as catalyst.

Catalyst ratio	CIE Whiteness Index	CRA (w+f)	
		Dry	Wet
20 : 80	56.1	197.1	191.4
40 : 60	57.4	201.0	193.9
50 : 50	58.6	202.9	194.9
60 : 40	59.6	204.2	200.7
80 : 20	59.8	205.2	204.9
Untreated	50.7	142.1	135.8

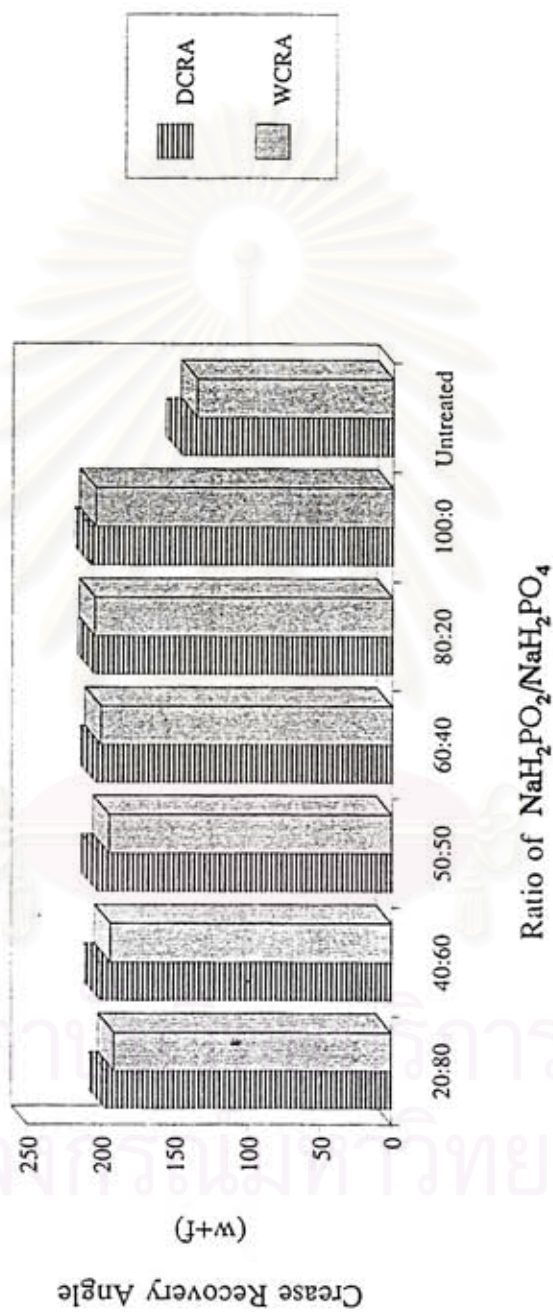
(10 replicates of CRA within 5% CV)

The results of effective treatments from catalyst mixtures of sodium hypophosphite and monobasic sodium phosphate are presented in Table 4.10. When the more sodium dihydrogenphosphate was used, the whiteness index was little adversely affected. At a minimum ratio of 20 : 80 hypophosphite/phosphate, 38.7% of DCRA improvement from the controlled can be obtained. By increasing the amount of hypophosphite, the crease recovery property was slightly increased. That means, when monobasic sodium phosphate was used on the mixtures, less hypophosphite was needed to achieve resiliency.

By using 50/50 of sodium dihydrogen phosphate/sodium hypophosphite as catalyst, with 7% citric acid and curing at 170°C for 90 sec, Andrews (1990) found that whiteness of washed fabric was decreased from 87 to 53 with 42.9% loss in breaking strength, however, the DCRA was improved by 45.8% which was 3% higher than the value obtained from this experiment.

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Figure 4.7 Effect of various ratios of $\text{NaH}_2\text{PO}_2/\text{NaH}_2\text{PO}_4$ as mixed catalysts on CRA of treated fabrics.



Effect of Various Concentrations of Fixapret COC on Fabric Properties.

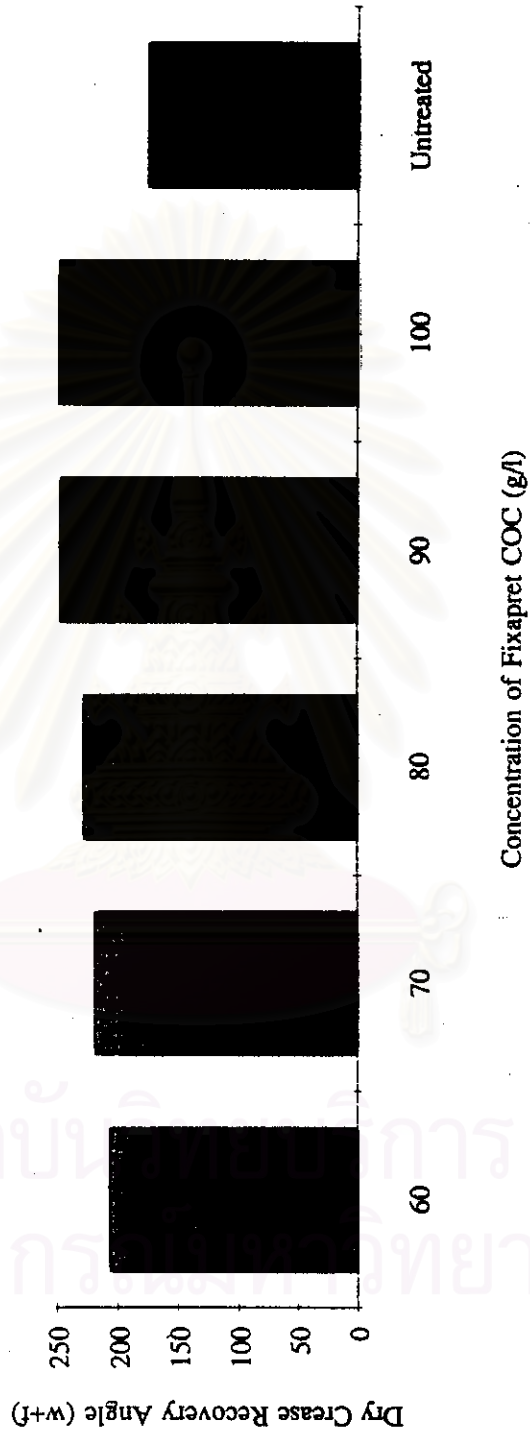
To determine the optimum concentration of Fixapret COC used for cotton fabric finishing, Condensol FB was used as a catalyst in this experiment and its concentration was fixed at 30 g/l. The fabrics (lot C) were padded with two dips and two nips at about 90% wet add-on. The Fixapret COC concentrations were varied from 60 g/l to 100 g/l. The padded fabrics were dried at 95°C for 3 min and cured at 160°C for 90 sec.

Table 4.11 Properties of cotton fabrics treated with various concentrations of Fixapret COC.

Fixapret COC concentration (g/l)	CIE Whiteness Index		DCRA (w+f)	Formaldehyde content (ppm)	
	Unwashed	Washed	Unwashed	Unwashed	Washed
60	71.4	71.1	206.8	48.4	13.9
70	72.6	69.6	216.2	54.6	15.0
80	73.2	72.1	228.5	59.1	16.0
90	72.6	72.5	247.8	63.7	17.8
100	73.0	72.0	248.6	69.9	17.8
Untreated	76.2	-	160.0	-	-

(10 replicates of DCRA within 5% CV)

Figure 4.8 Effect of various concentrations of Fixapret COC on DCRA.



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Figure 4.9 Effect of various concentration of Fixapret COC on formaldehyde content.

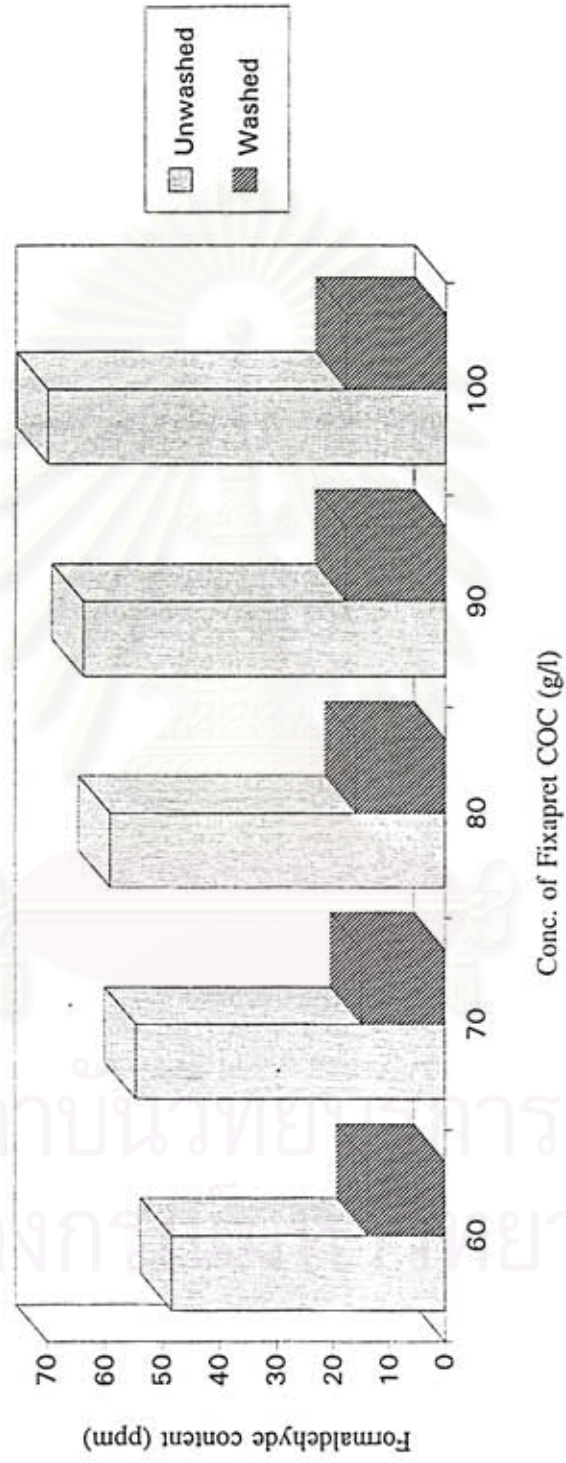


Table 4.11 shows that there is no effect of Fixapret COC concentration between 60-100 g/l on whiteness of the treated fabrics, however it was slightly dropped from those of the untreated. The improvement of DCRA is proportional to the increment of the resin concentration by giving the superior performance at 90 g/l Fixapret COC, approximately 55% of DCRA was improved from the untreated, however, there is no more improvement at higher concentration (Figure 4.8). The formaldehyde content of the finished fabrics has been determined according to Japanese test method law 112-1973. The results suggest that free formaldehyde left on the treated fabric is still rather high. Washing procedures can reduce this problem by dissolving out free formaldehyde for 70-75% (Figure 4.9). As can be seen that the formaldehyde contents after washed are not significantly differ from each level of Fixapret COC concentrations, finishing fabric at 90 g/l of Fixapret COC resin is seem to be the most effective procedure.

In the case of cost comparison between the use of Fixapret COC and citric acid, when Fixapret COC was used at 70g/l, the DCRA can be obtained at 35% but from the previous experiment, 46% improvement of resiliency was accomplished with 7% citric acid system. From the fact that their costs are rather at equal, so the use of citric acid as cross-linking agent can reduce the finishes expense.

Effect of Various Ratios of Fixapret COC/CA as Mixed Resins on Fabric Properties.

From the result of the previous experiment, Fixapret COC has shown its high efficiency in crease resistant finishing, so it is interesting to study its possibility to be used with citric acid as mixed resins in order to reduce the amount of Fixapret COC used that means to reduce the formaldehyde content. The experiment was

taken place by using citric acid substituted to Fixapret COC in the ratios of 20 : 80 to 80 : 20 (volume ratio). For CA solution, the concentration of CA was 7% and used sodium hypophosphite as a catalyst with the mole ratio of 1 : 2 . For Fixapret COC solution, the concentration of Fixapret COC was 90 g/l and using Condensol FB as a catalyst with the weight ratio of 3 : 1 (Fixapret COC : Condensol FB). The fabrics used in this experiment were lot A.

Table 4.12 Fabric properties from treatment with various ratios of Fixapret COC/CA.

Resin ratio FixapretCOC : CA	CIE Whiteness Index	DCRA (w+f)	Breaking Strength (N)		Formaldehyde Content (ppm)	
			filling	warp	Unwashed	Washed
0 : 100	59.1	208.1	258.7	327.3	0	0
20 : 80	62.4	189.7	269.0	337.4	30.6	8.4
40 : 60	64.0	199.7	259.9	325.7	57.7	9.0
50 : 50	63.7	201.1	257.8	314.6	66.8	10.4
60 : 40	61.8	201.4	243.3	303.6	75.8	11.8
80 : 20	64.9	215.8	135.0	248.0	81.4	13.9
100 : 0	65.7	217.7	133.1	239.7	88.4	15.3
Untreated	50.7	142.1	389.9	454.5	-	-

(10 replicates of DCRA within 5% CV, 5 replicates of breaking within 7% CV)

From Table 4.12 it shows that, in mixed resin system, although the increment of DCRA can be obtained by increasing the portion of Fixapret COC but the fabrics strength were adversely affected. The best performance of DCRA is obtained at the ratio of 80 : 20 (Fixapret COC : CA). However, the breaking

strength data suggests that even though 52% of DCRA was improved, but 65% of fabric strength was destroyed (Figure 4.11).

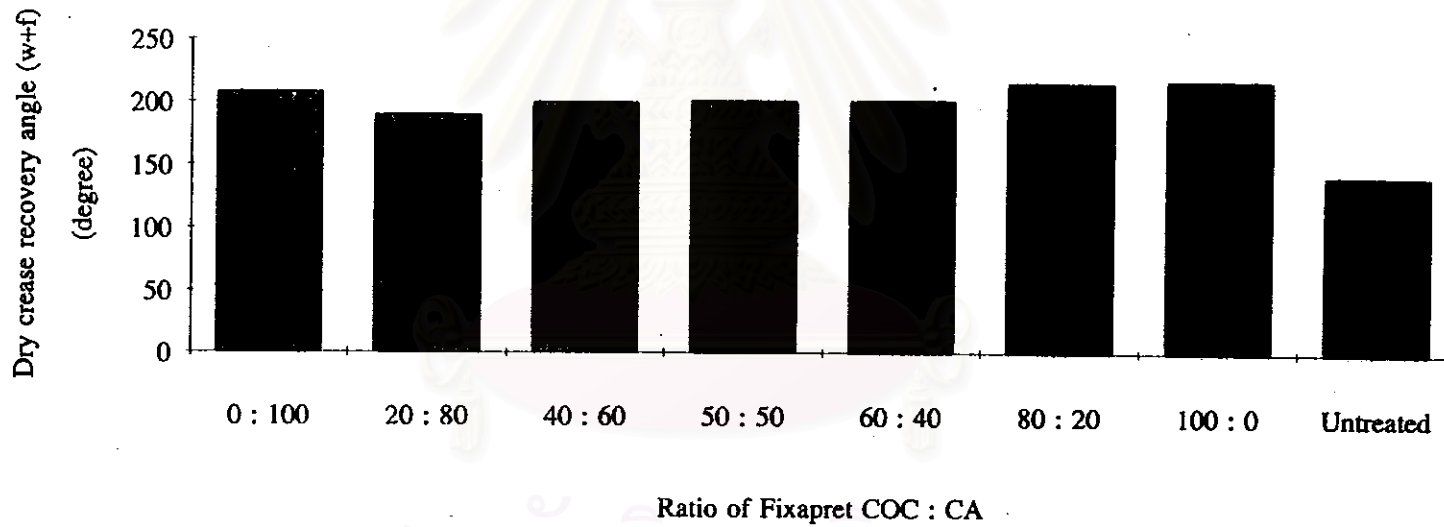
The mixed resins at a ratio of 40 : 60 can impart 67% strength retention of treated fabric which was quite similar to those obtained from treating with pure 7% CA but the DCRA result of mixed resin system shows that its efficiency to cross-link is still lower, although free formaldehyde on washed fabric is in satisfactory level (Figure 4.12).

Even though, in this experiment, the improvement of fabric properties was not succeeded, but the use of pure citric acid was proven that it can provide several advantages not only retentive whiteness and better balance of physical properties, but also impart no risk from toxicity of formaldehyde.



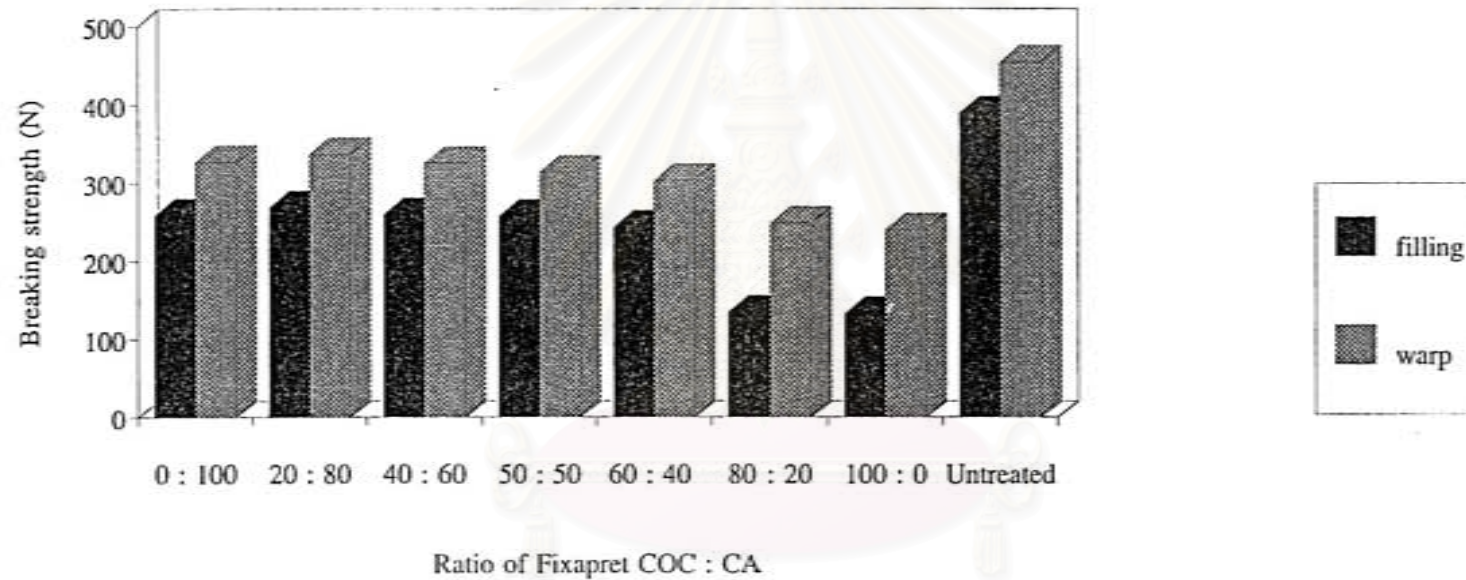
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Figure 4.10 Effect of various ratios of Fixapret COC/CA on DCRA of fabrics.



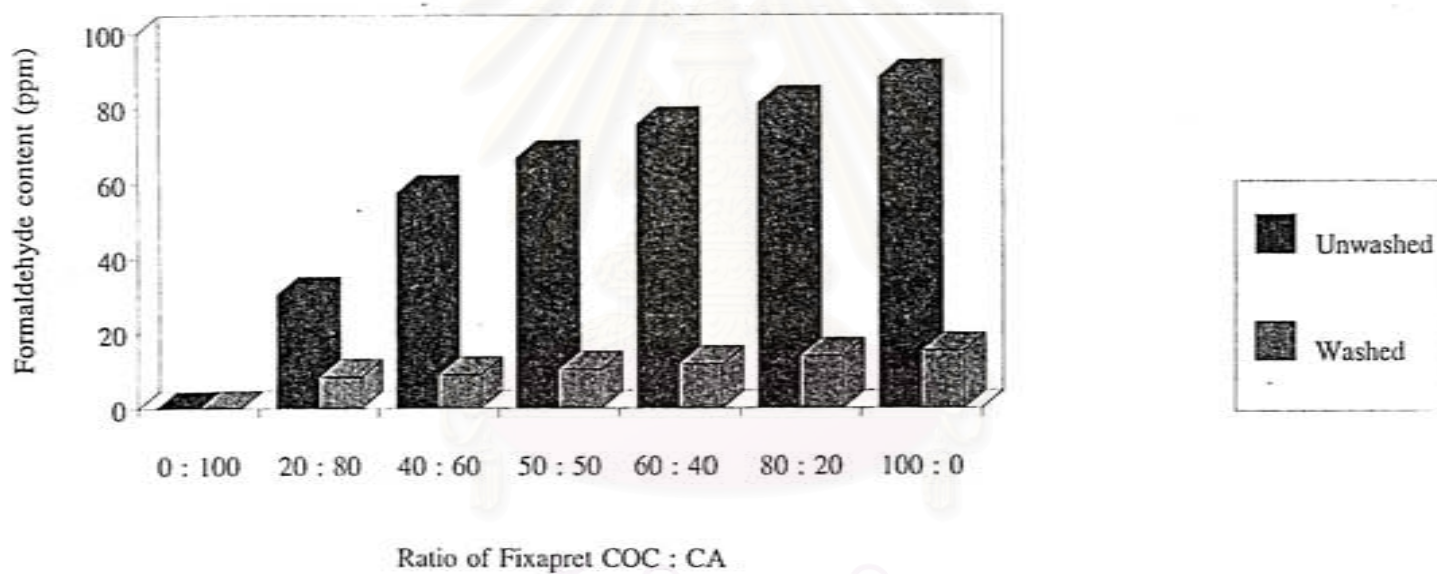
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Figure 4.11 Effect of various ratios of Fixapret COC/CA on fabric strength.



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Figure 4.12 Effect of various ratios of Fixapret COC/CA on formaldehyde content.



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Result of FT-IR Analysis.

Fourier transform infrared spectroscopy was used to characterize the ester cross-linkages in the treated cotton fabrics. A Nicolet Impact 400D FT-IR spectrometer was used for all the FT-IR measurements. Resolution was set at 4 cm^{-1} and the number of scans was 32. The fabrics were impregnated with solutions containing citric acid as a cross-linking agent and sodium hypophosphite as a catalyst. The spectra of treated fabrics finished with different conditions were obtained.

1. Effect of Citric Acid Concentrations.

The effect of citric acid concentrations on the ester cross-linking of cotton were examined. The cotton fabrics were treated in solutions containing 5%, 7%, 9%, and 11% CA, respectively, and sodium hypophosphite as a catalyst. A constant mole ratio of $\text{CA}/\text{NaH}_2\text{PO}_2$ was used at 1 : 2. The impregnated fabrics were dried at 95°C for 3 min, cured at 160°C for 90 sec, and passed washing procedure.

As shown in Figure 4.13, the ester carbonyl band at 1730 cm^{-1} (O'Connor, 1972), which was not observed on untreated fabric, was obviously presented on finished fabric. To get more clear picture the expansion of interscan between $2000\text{-}1200\text{ cm}^{-1}$ had been done as seen in Figure 4.14. The 1374 cm^{-1} and 1431 cm^{-1} bands are associated with the bending vibration mode of hydrocarbon structures in cellulose molecules (Morris, Andrews and Catalano, 1994). It is observed that the ester carbonyl band intensity increase as the acid/catalyst concentrations are increased.

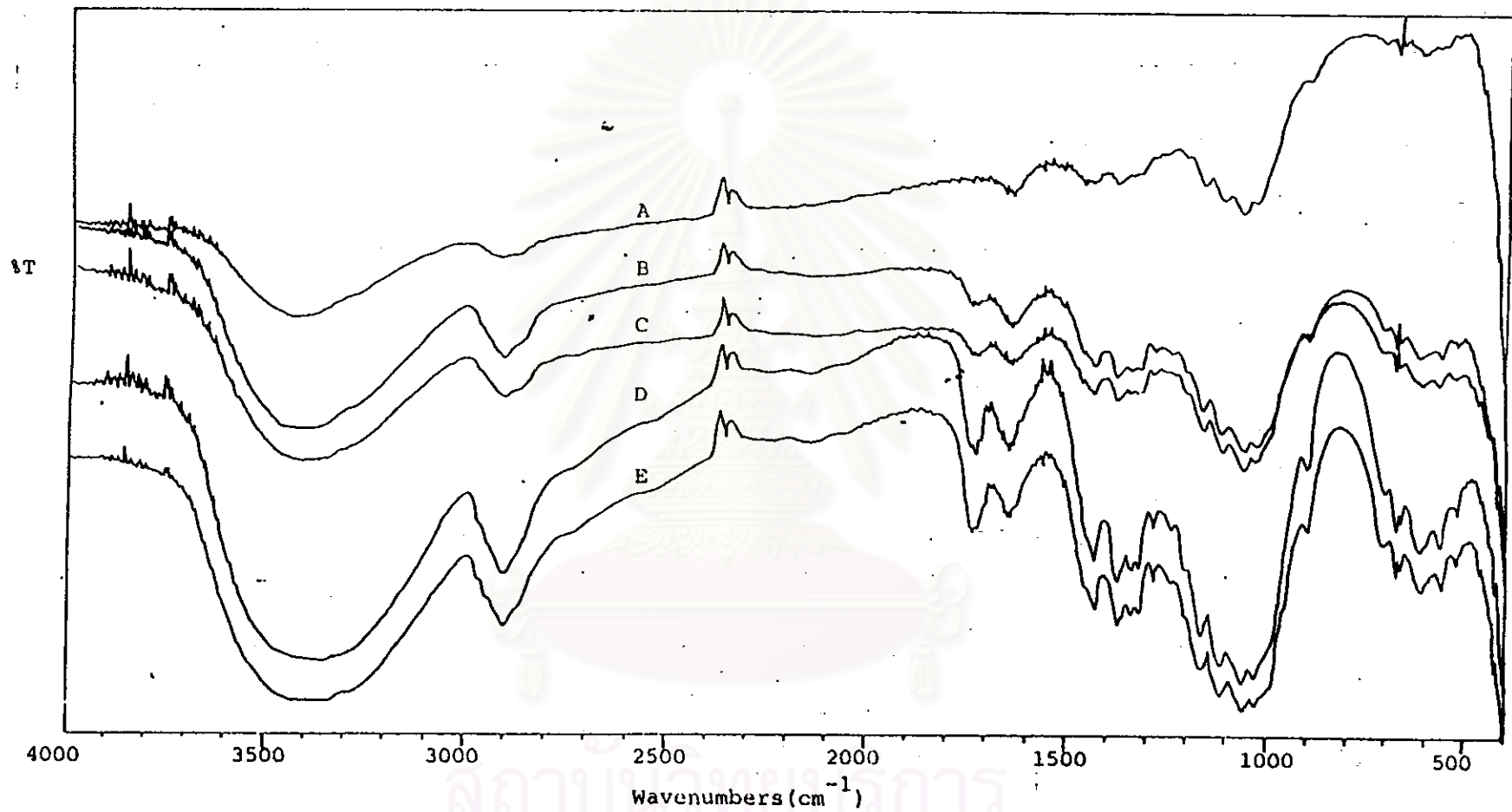


Figure 4.13 FT-IR of untreated cotton fabric and of cotton fabric treated with different levels of citric acid (scanning at 4000-400 cm⁻¹). A = Untreated, B = 5% CA, C = 7% CA, D = 9% CA, E = 11% CA

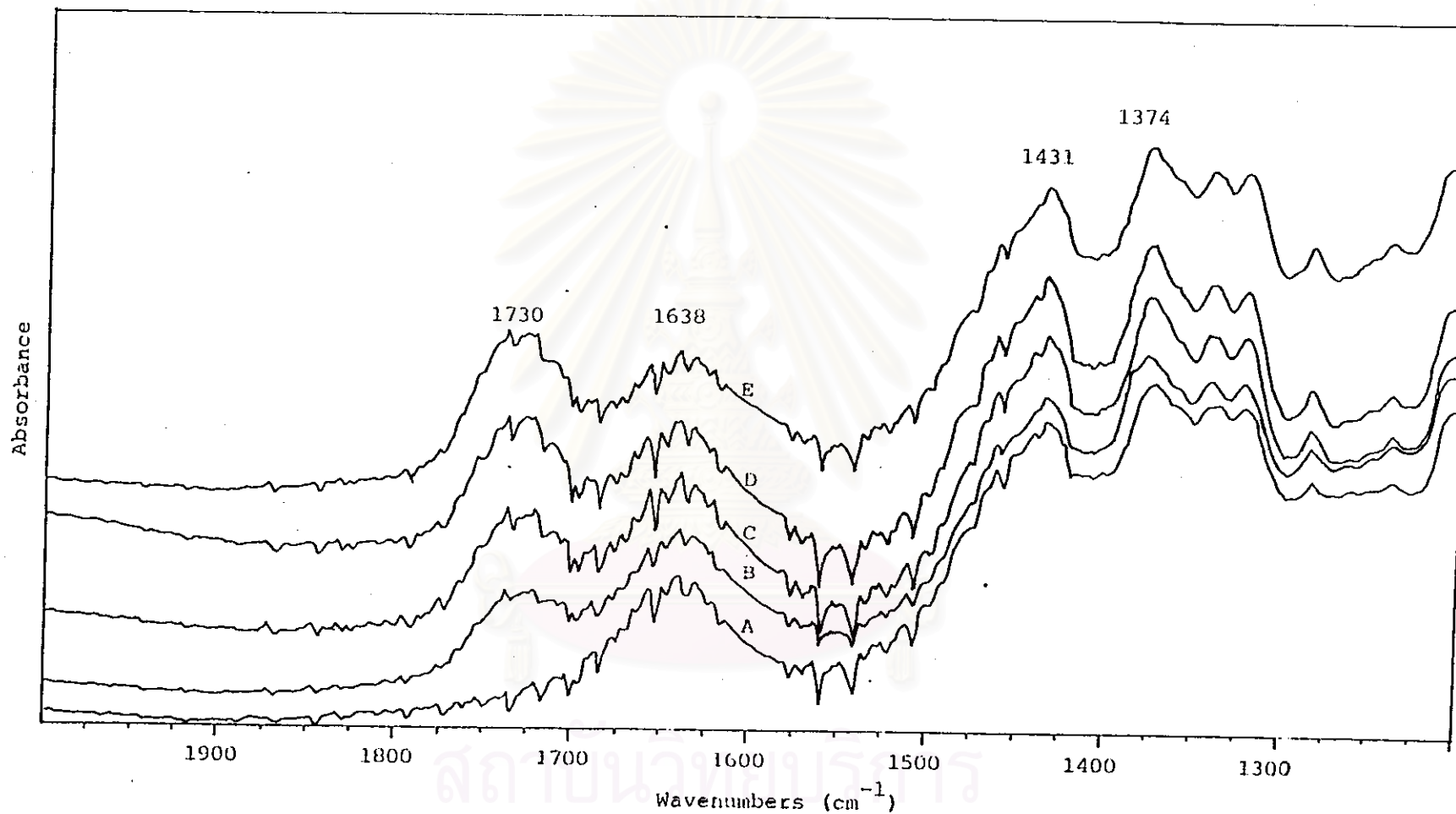


Figure 4.14 FT-IR spectra of untreated cotton fabric and of cotton fabrics treated with different levels of citric acid.

A = Untreated, B = 5% CA, C = 7% CA, D = 9% CA, E = 11% CA

2. Characterization of the Intermolecular Ester Cross-Linking of Cotton Fabrics.

From the fact that after esterification occurs between polycarboxylic acid and cellulose molecules, the carbonyls in the fabric can present in three forms; intermolecular ester linkage, carboxyl, and carboxylate (Figure 4.15), the intensity of carbonyl band at 1730 cm^{-1} will comprise of the overlapping between ester carbonyl and carboxyl bands.

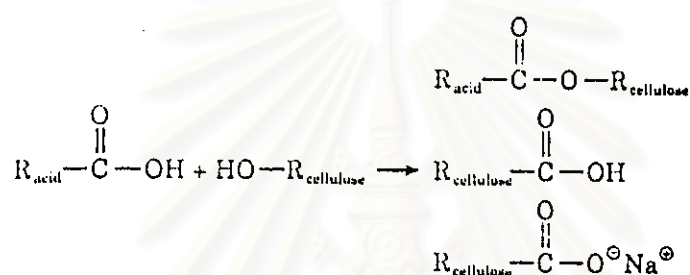


Figure 4.15 Three forms of product exist in the treatment of finished fabrics (Yang and Andrews, 1991).

To prove this, the cured fabric finished with 7% CA and sodium hypophosphite was first impregnated in distilled water with stirring for 5 min at room temperature, then treated in a 0.1 N NaOH solution for 5 min, and rinsed with distilled water again. Because sodium hydroxide converts carboxyl to carboxylate, an increase in the 1583 cm^{-1} band intensity and a decrease in the 1730 cm^{-1} band intensity are seen in Figure 4.16.

After treatment with 0.1 N hydrochloric acid in the same procedure, the carboxylate was all converted to carboxyl, as a result, the 1583 cm^{-1} band disappears completely. This can be concluded that the 1583 cm^{-1} band is due to the

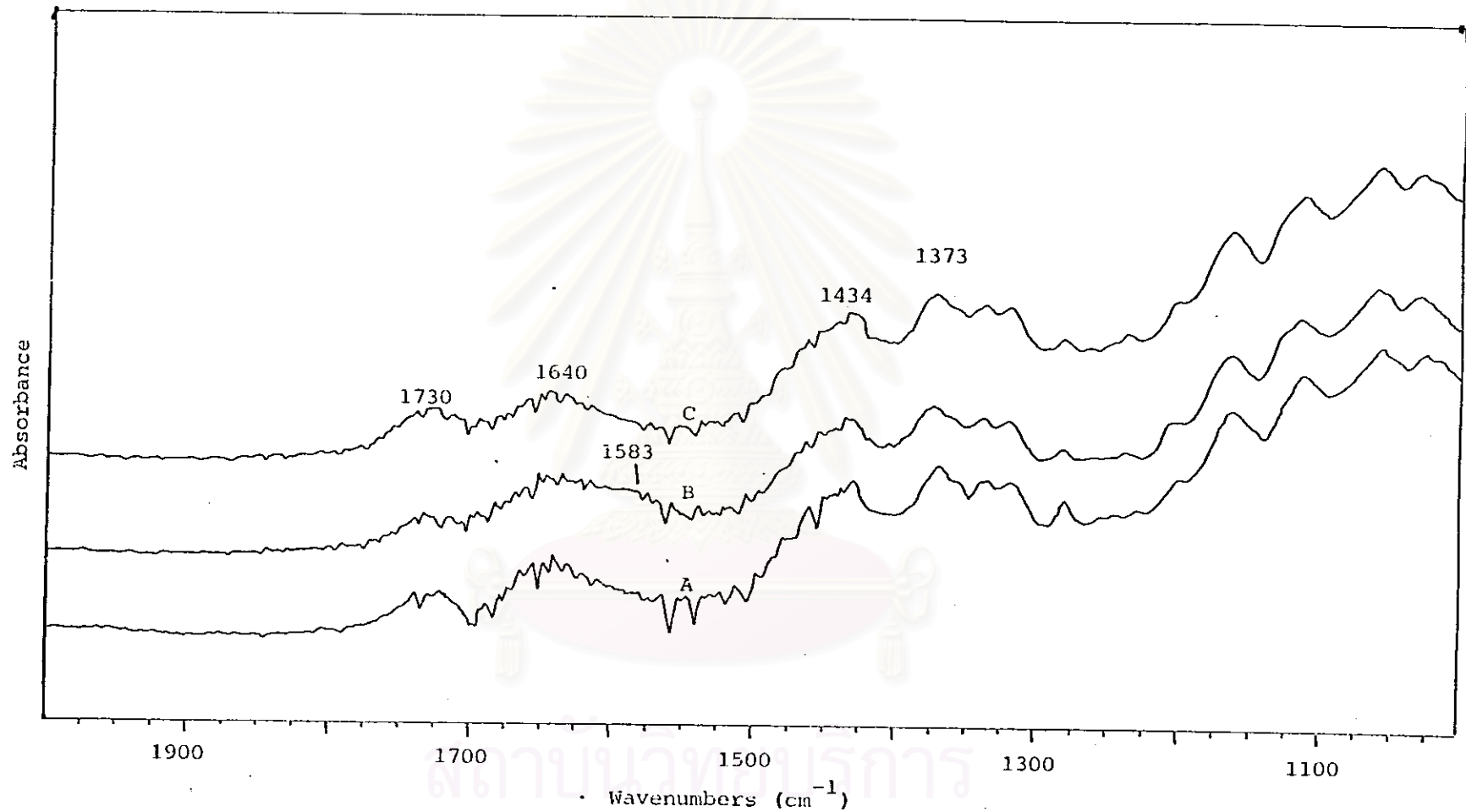


Figure 4.16 Infrared spectra of cotton fabrics finished with CA and NaH₂PO₂ and cured at 160°C for 1.5 min.

A = no treatment, B = treated with 0.1 N NaOH solution, C = treated with 0.1 N HCl solution

carbonyl of carboxylate and that the carboxyl and ester carbonyl bands overlap at 1730 cm^{-1} . With the capability to separate the carboxyls band by converting to carboxylate with a dilute NaOH solution, the total quantity of the ester groups in the finished fabrics can be determined by measuring the 1730 cm^{-1} band intensity. From the fact that the ester groups occurring in the finished fabrics is not only the ester crosslinkages, but also the ester groups between cotton cellulose and singly bonded acid molecules, so using the 1730 cm^{-1} ester carbonyl band alone is not enough to represent the effectiveness of the acid as a cross-linking agent in cotton fabric. The carbonyl band intensity ratio is, however, useful to evaluate the effectiveness of the acid. An increase in the carbonyl band intensity ratio (ester/carboxylate) indicates an increase in the average number of ester groups formed per each acid molecule (Yang and Andrews, 1991).



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