

CHAPTER IV

RESULTS AND DISCUSSION

4.1 Preparation of Natural Rubber Sheet

Natural rubber latex consists of particles of rubber hydrocarbon and non-rubber constituents suspended in an aqueous phase. These natural rubber particles can be separated from the aqueous phase by electrodecantation method. The natural rubber particles can be grown to various thickness on the anode (a working electrode) yielding natural rubber sheet. Effect of voltage applied across the cell and the period of time during which a current is applied across the cell on natural rubber sheet, is clearly shown in Figure 4.1 and 4.2 respectively.

Table 4.1 Effect of voltage and time during coagulation of natural rubber

Voltage (volt)	Time of Rubber Coagulation					
	4 min		6 min		8 min	
	Weight (g/cm ²)	Thickness (mm)	Weight (g/cm ²)	Thickness (mm)	Weight (g/cm ²)	Thickness (mm)
2	0.017	0.09	0.019	0.11	0.021	0.12
3	0.077	0.46	0.103	0.62	0.107	0.68

Table 4.1 (continued)

Voltage (volt)	Time of Rubber Coagulation					
	4 min		6 min		8 min	
	Weight (g/cm ²)	Thickness (mm)	Weight (g/cm ²)	Thickness (mm)	Weight (g/cm ²)	Thickness (mm)
4	0.1157	0.58	0.159	0.81	0.22	1.03
5	0.1759	1.06	0.218	1.18	0.27	1.35
6	0.2121	1.01	0.217	1.17	0.307	1.35
7	0.1921	1.10	0.233	1.25	0.285	1.36
8	0.1912	1.25	0.278	1.35	0.264	1.52
9	0.1915	1.26	0.227	1.35	0.263	1.52
10	0.2382	1.34	0.247	1.35	0.282	1.51

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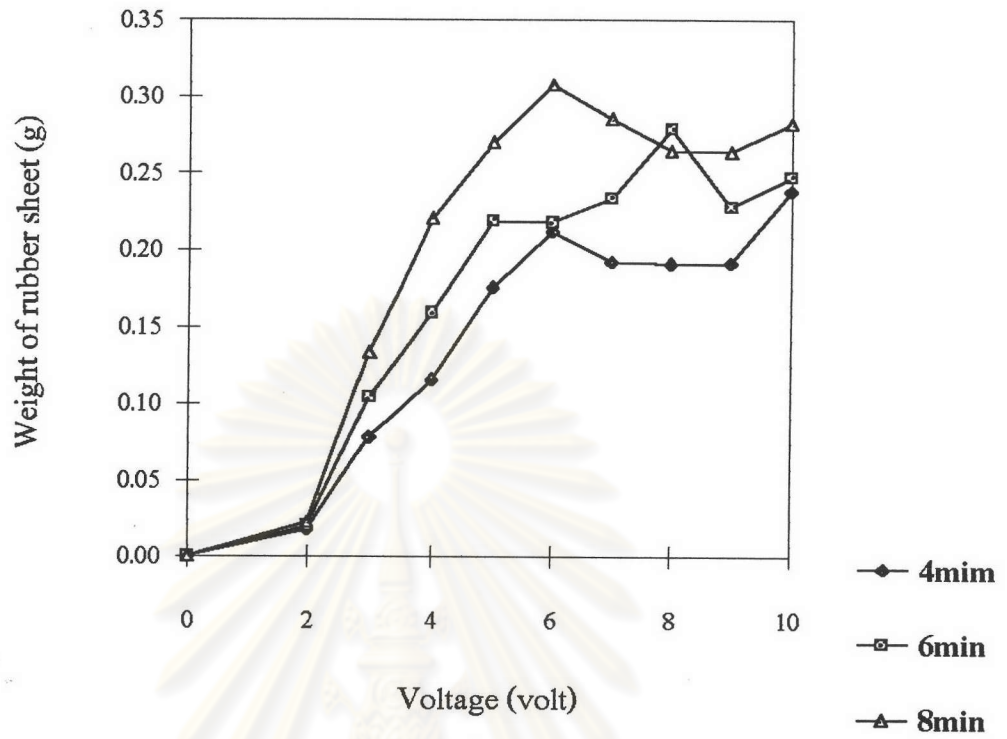


Figure 4.1 Growth of natural rubber sheet on anode

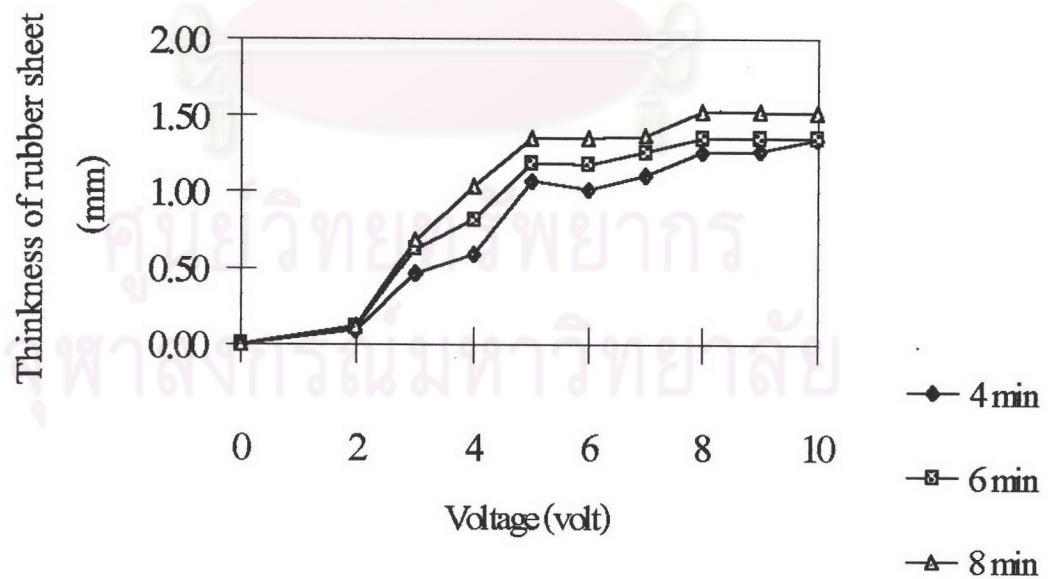


Figure 4.2 Effect of applied voltage on the thickness of natural rubber sheet (mm)

From Figure 4.1 and 4.2 weight and thickness of natural rubber sheet were sharply increased as the voltage increased when up to 6 volts. At higher voltage the increment of thickness and weight were not smooth. It was postulated that high voltage induced deposition of negatively charged rubber particles occurred rapidly on the surface of the anode and the passivation or over-resistance occurred on the anode at the same time. Therefore a voltage higher than 6 volts does not enhance the deposition. A voltage of 4 volt was chosen for the preparation of the rubber sheet in this work.

Table 4.2 Effect of voltage and time during coagulation of natural rubber.

Coagulation Time (min)	Voltage Applied			
	4 volt		6 volt	
	Weight (g/cm ²)	Thickness (mm)	Weight (g/cm ²)	Thickness (mm)
4	0.115	0.58	0.212	1.01
10	0.348	1.35	0.390	1.48
15	0.394	1.70	0.410	2.02
30	0.505	2.69	0.580	2.75
40	0.511	2.71	0.587	2.77
50	0.512	2.71	0.588	2.77
60	0.512	2.71	0.588	2.77

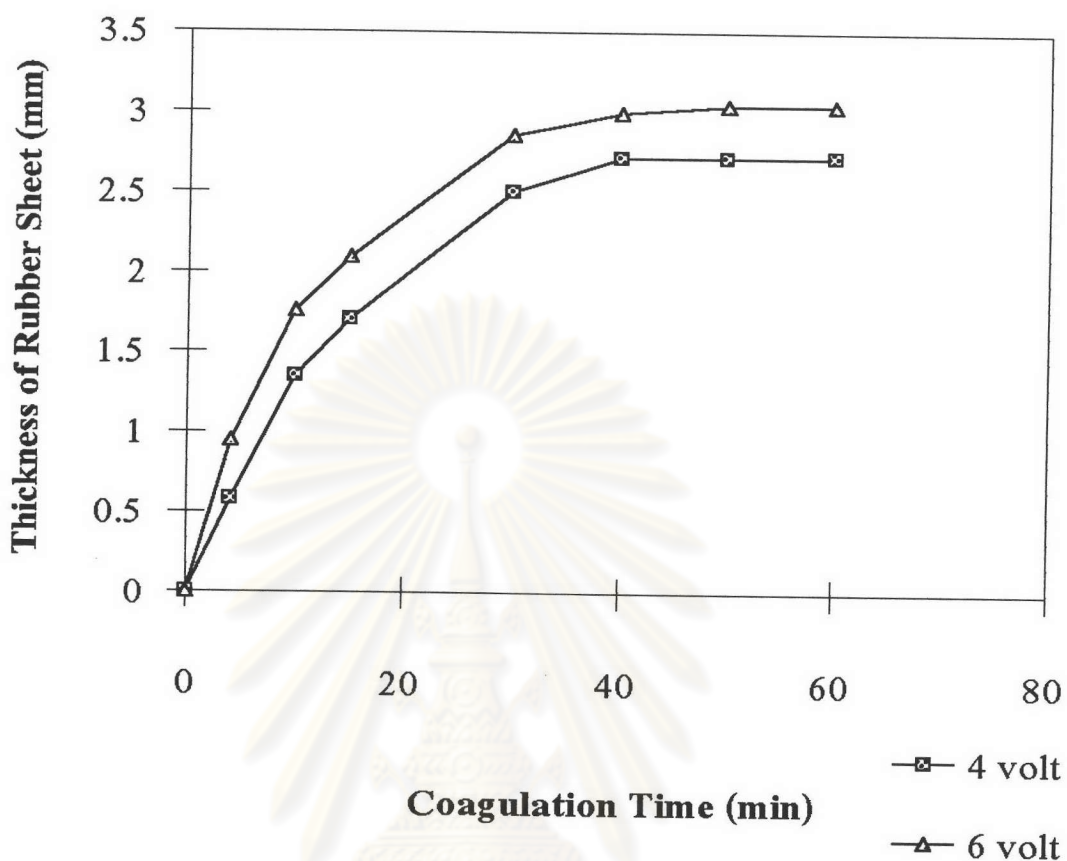


Figure 4.3 Growth of natural rubber sheet on anode at various coagulation time.

From Figure 4.3 weight and thickness of the rubber sheet increased almost constant when the coagulation time increased until 60 minute. The thickness results in the insulating of natural rubber sheet at the anode, and the current flow will be reduced across two electrodes, called “passivation” or over-resistance at the anode. So thickness of the rubber sheet gradually increased at longer time. The coagulation time of 4 and 10 minute were chosen for the preparation of natural rubber sheet in this work.

4.2 Preparation of Conductive Natural Rubber by Electrochemical Method.

Reaction conditions to prepare high electrical conductivity of conductive natural rubber were studied in order to select the optimum condition. The major parameters that influence the quality of the conductive rubber prepared with 0.05 M of pyrrole monomer and the choice for the preparation of natural rubber sheet at 4 volt for 4 min of coagulation time are as follows :

- (1) effect of concentration of the LiClO_4 solution;
- (2) effect of voltage applied;
- (3) effect of reaction time;
- (4) effect of reaction temperature;
- (5) effect of atmospheres.

A suitable solvent must be a solvent in which the pyrrole monomer and LiClO_4 is soluble but the polypyrrole on natural rubber sheet is insoluble. Additionally, the solvent must be electrochemically inert at the potential at which the pyrrole monomer is oxidized. Methanol solvent has been chosen for this work. Lithium perchlorate (LiClO_4) is a suitable electrolyte for use with methanol solvent and it can penetrate into pore of natural rubber sheet. LiClO_4 is soluble in the solvent being used and which is electrochemically

inert at the potential at which the pyrrole monomer is oxidized. It is therefore a suitable electrolyte for use in the present method.

The natural rubber sheet was prepared by the concentrated electrodecantation method (see section 3.2). It contains pores in the host polymer making it suitable as the porous medium for pyrrole to be added on such pore surface.

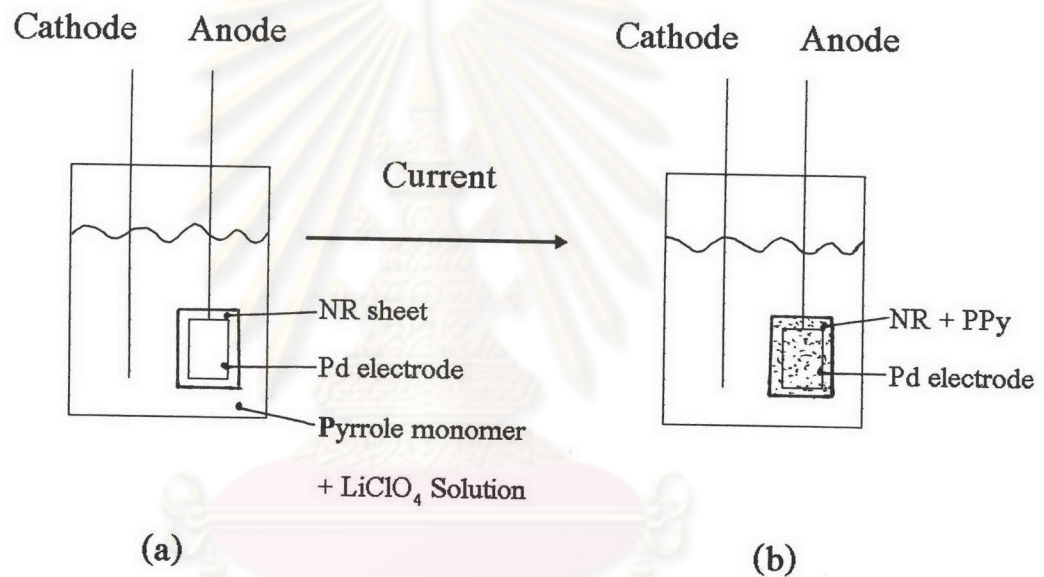


Figure 4.4 The electroplating of polypyrrole (PPy) on natural rubber (NR) sheet (anode).

In this experimental method, a porous medium of NR sheet was first obtained by the concentrated electrodecantation method (see section 3.2), and further employed as host for the conducting polymer composites. The incorporation of PPy was performed by :

i) Imbibing the host polymer (NR sheet is obtained on the positive electrode) with pyrrole solution (pyrrole monomer and electrolyte solution, see Figure 4.4 (a))

ii) When the passage of an electric current through this liquid system takes place by the transport of electrons from the cathode to the anode. During conventional electrolysis, the current through is carried through the solution by ions, as shown in Figure 4.4 (b). A black film of PPy is formed inside the porous host polymer (NR sheet) on the positive electrode (anode). Therefore the conductivity of the composite can be ascribed as the network of PPy formed inside the porous host polymer.

4.2.1 Effect of Concentration of the LiClO_4 Solution.

The polymerization reaction was carried out by the prepared NR sheet by coagulated electrodecantation method (see section 3.2) at 4 volt for 4 minute coagulation time and various LiClO_4 concentration from 0.10-4.00 molar. Table 4.3 showed the dependence of the electrical conductivity on the reaction time while the other parameters were kept constant at 25 °C for 4 h reaction time and the potential application was 4 volt in 20 ml MeOH of LiClO_4 solution.

Since the oxidation potential of LiClO_4 in methanol solution is different at each concentration which will affect the polymerization, it should thus be recorded. This was performed by

construction of the calibration curve as shown in Figure 4.5. It is apparent that the oxidation potential increases with increasing concentration of LiClO_4 :

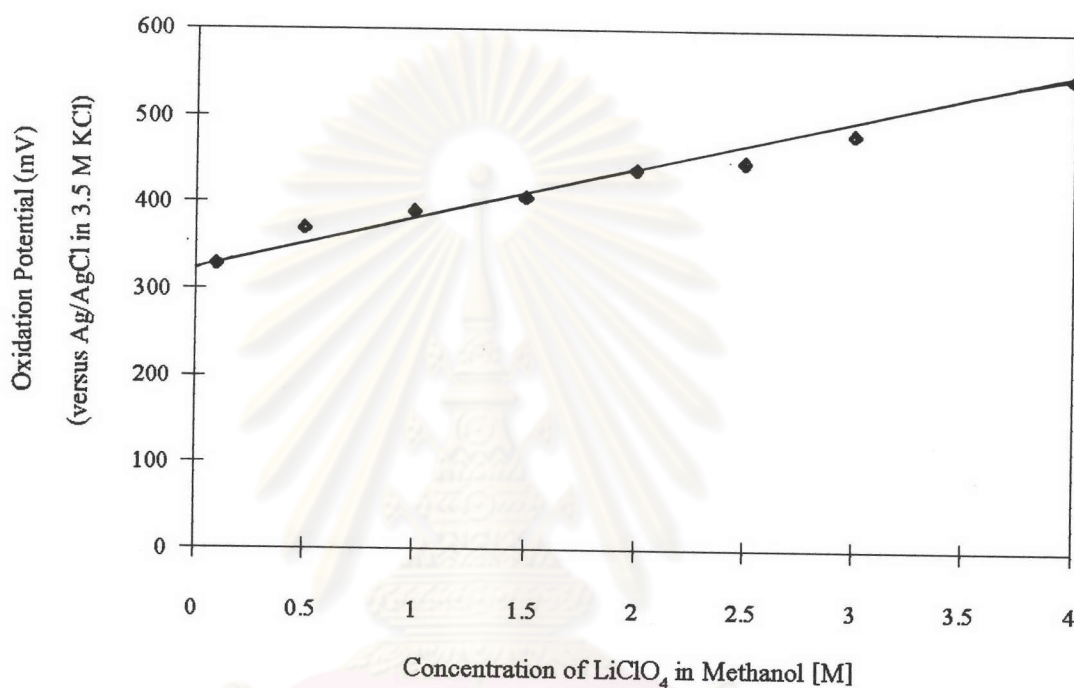


Figure 4.5 The change in oxidation potential of LiClO_4 in methanol at various concentration.

The upper limit of LiClO_4 concentration was 4.00 M because at concentration greater than 4 M LiClO_4 was not completely dissolved in methanol. The initial oxidation potential of each solution is obtained from Figure 4.5 (shown in Table 4.3).

Table 4.3 Effect of LiClO_4 concentration on conductivity of CNR.

LiClO_4 Concentration [M]	Initial Oxidation Potential (mV) (versus Ag/AgCl in 3.5 M KCl)	Product Yield (g/cm²)	Conductivity (S/cm)
0.10	329	0.118	2.916E-04
0.50	370	0.119	1.299E-03
1.00	390	0.123	2.196E-03
1.50	406	0.126	2.553E-03
2.00	438	0.127	4.201E-03
2.50	448	0.129	3.114E-03
3.00	480	0.120	2.829E-03
4.00	546	0.119	1.963E-03

*See experiment 3.3

By plotting conductivity and LiClO_4 concentration (Figure 4.6), it clearly exhibited that higher conductivity was obtained as the concentration was increased and the oxidation potential of solution was high also. However, when the concentration was higher than 2.00 M, the conductivity suddenly decreased.

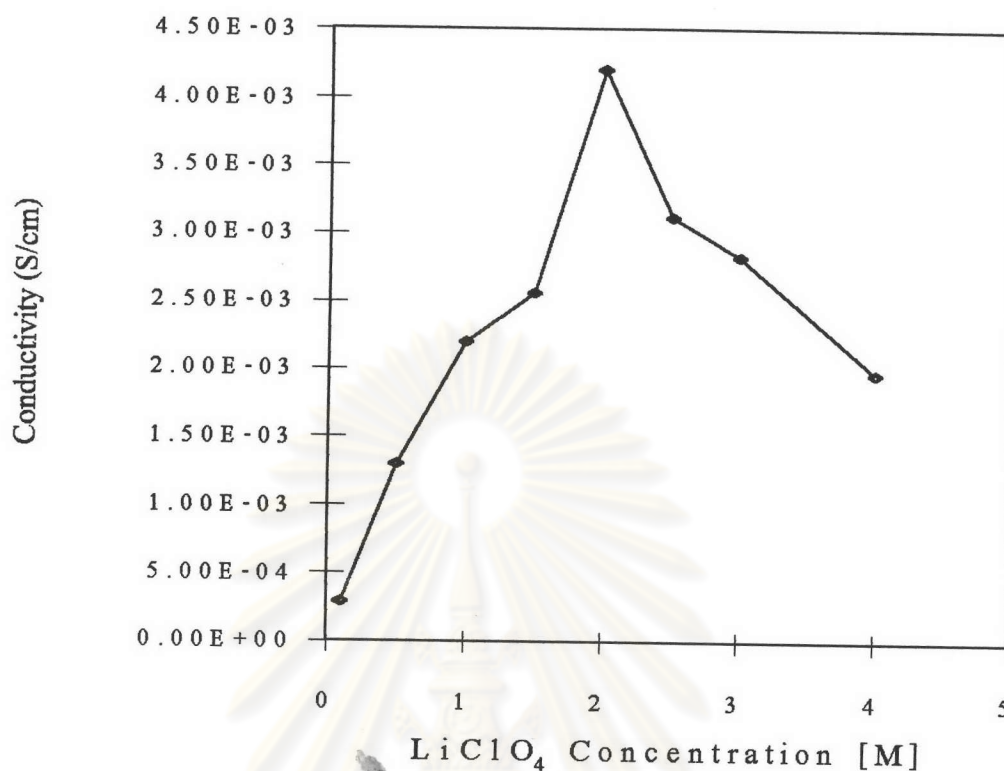


Figure 4.6 The electrical conductivity of conductive natural rubber at various concentration of LiClO₄.

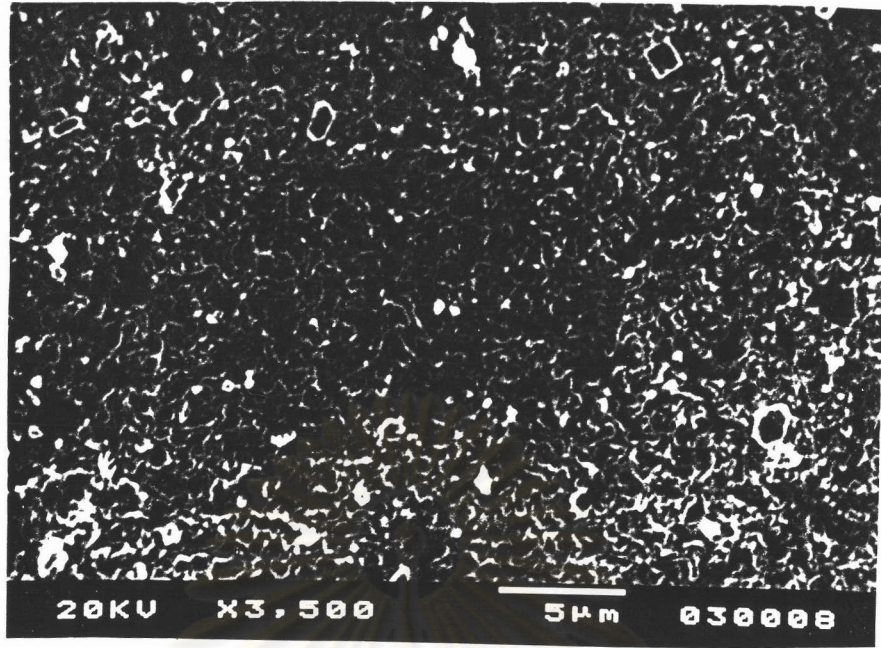
Volume of LiClO₄ Slution 20 ml

Volume of Pyrrole 0.05 ml

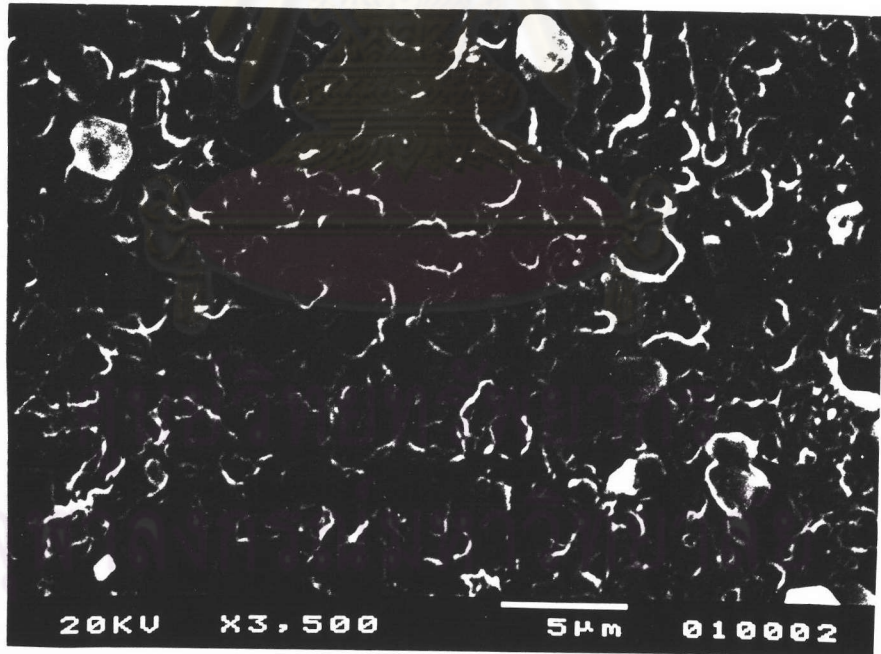
Reaction Time 4 h

Reaction Temperature 25 °C

This must be associated with the morphology of PPy onto NR sheet, as studied by SEM picture of the backsides of conductive natural rubber (CNR) sheet (the side which attached to Pd electrode) shown in Figure 4.7 (a) and (b) respectively. For the 2 M solution (Figure 4.7 (a)) CNR sheet of rather smooth surface was obtained, whereas in which 4 M solution CNR sheet with surface containing large member of particles aggregated was obtained.



(a) SEM picture of CNR prepared in magnified 3500 times at 2 M LiClO₄ solution.



(b) SEM picture of CNR prepared in magnified 3500 times at 4 M LiClO₄ solution.

Figure 4.7 Morphology of CNR sheets.

It is proposed that in 4 M LiClO₄ solution polymerization rate was high because of the oxidation potential was high such that many polymerized nuclei evolved at the same time, this led to the PPy network in the porous medium (porous in matrix of NR sheet) having aggregated structure surface resulting in low conductivity.

4.2.2 Effect of Applied Potential

NR sheet prepared at 4 volt for 4 minute of coagulation time appears to be the best condition as compared to other potential difference of 1 to 6 volt. The upper limit was 5 volt because when the voltage was greater than 5 volt the working electrode (Palladium (Pd);anode) was corroded. Table 4.4 showed the dependence of the electrical conductivity on the potential application across the cell (see experiment 3.3). While the other parameters were kept constant, i.e., 0.05 M pyrrole monomer concentration 20 ml of 2.00 M LiClO₄ solution for 4 h at 25 °C.

Table 4.4 Effect of potential application.

Voltage (volt)	Conductivity (S/cm)	Product Yield (g/cm ²)
1	0	0
2	0	0
3	2.478E-04	0.119
4	4.201E-03	0.123
5	7.314E-03	0.144

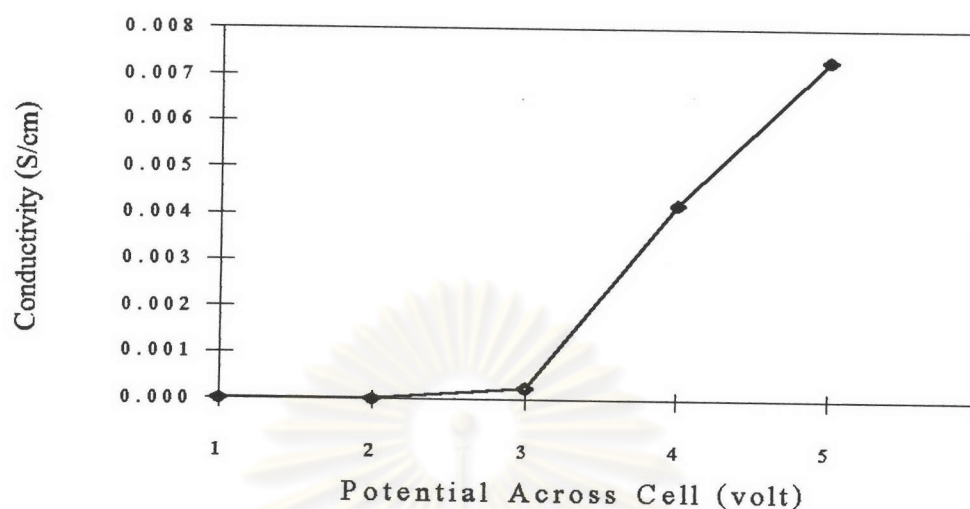


Figure 4.8 The electrical conductivity of CNR at various reaction voltage.

By plotting conductivity and reaction voltage (Figure 4.8), it clearly exhibited that higher conductivity was obtained as the voltage was increased. At a potential of 1 to 2 volt no PPy was built onto NR sheet. The conductivity of NR sheet was zero (S/cm) because at the anode coated with NR sheet the resistance was too high that reaction of pyrrole could not occur at anode. When higher voltage was applied across this electrochemical cell, PPy was obtained onto NR sheet and the conductivity of CNR was higher.

In summary, the sheet are conveniently prepared potentiostatically or galvanostatically with the voltage applied to the cell at 4 volt in order to protect the corrosion of Pd electrode, and it can be used repeatedly in other experiments.

4.2.3 Effect of Reaction Time.

The polymerization was carried out on the NR sheet prepared at 4 volt for 4 minutes of coagulation time. In each reaction 0.05 M of pyrrole monomer, 20 ml of 2.00 M LiClO₄ solution (438 mV of oxidation potential versus Ag/AgCl in 3.5 M KCl), voltage 4 volt and reaction temperature 25°C were kept constant. However, the reaction time was varied as detailed in Table 4.5

Table 4.5 Effect of reaction time on conductivity and product yields.

Reaction Time (h)	Conductivity (S/cm)	Product Yields (g/cm ²)
1	1.019E-04	0.116
2	1.161E-03	0.117
3	1.589E-03	0.120
4	4.201E-03	0.127
5	5.188E-03	0.139
6	6.503E-03	0.154
7	5.688E-03	0.161
8	5.636E-03	0.165
9	3.189E-03	0.179
10	2.300E-03	0.222

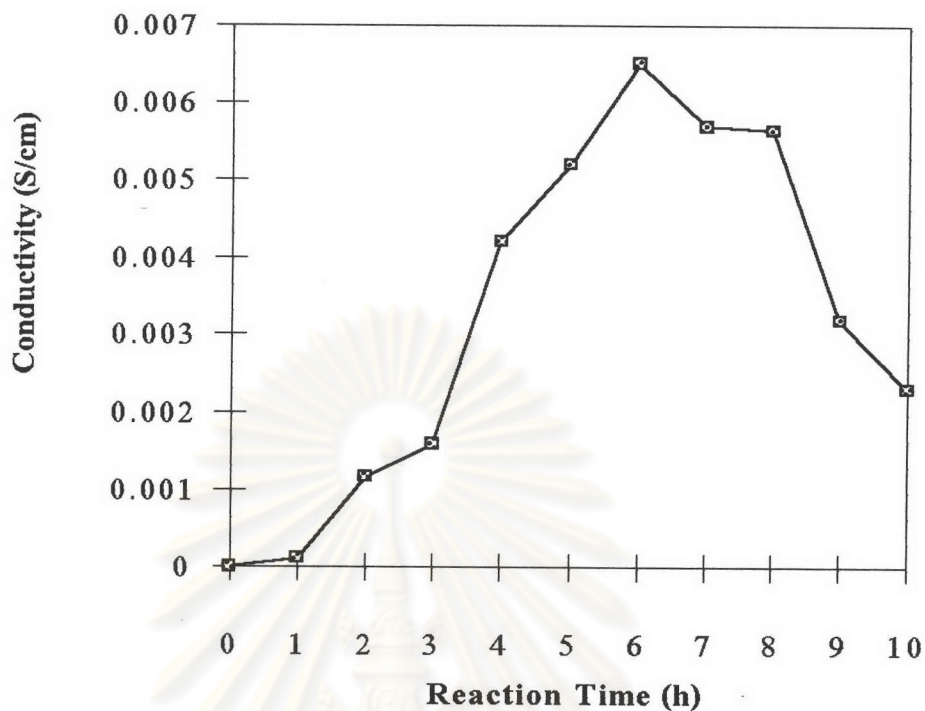


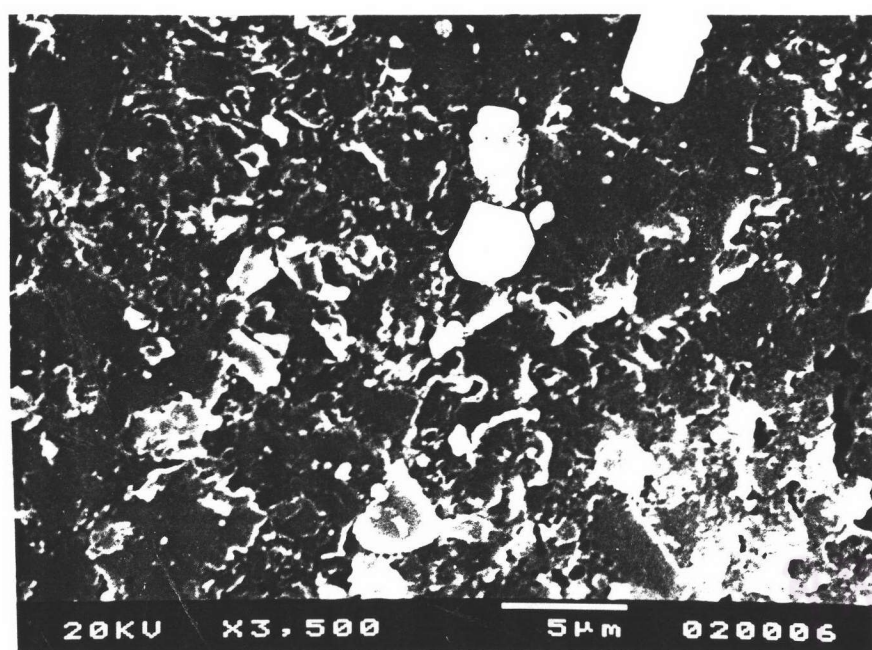
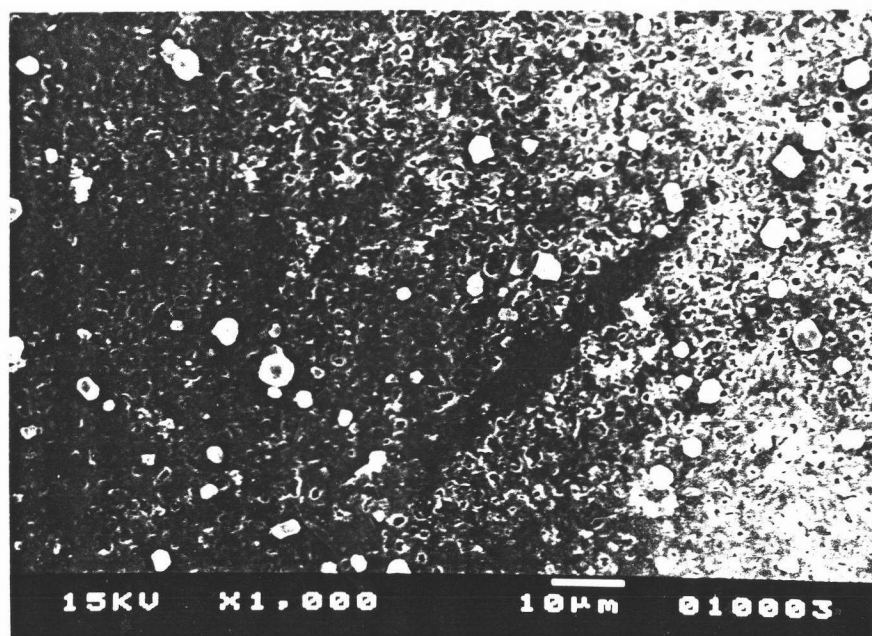
Figure 4.9 The electrical conductivity of conductive natural rubber at various reaction time.

From plotting conductivity and reaction time (Figure 4.9), it clearly exhibited that maximum electrical conductivity is obtained at 6 h. At shorter polymerization time (below 6 h) incorporation of pyrrole onto NR sheet was not complete thus this resulted in the shorter conjugate chain length and low product yields. At longer polymerization time, higher product yields was obtained but with low conductivity. This is confirmed by SEM picture of CNR sheet for 6 h and 10 h of polymerization time (t_p) shown in Figure 4.10.

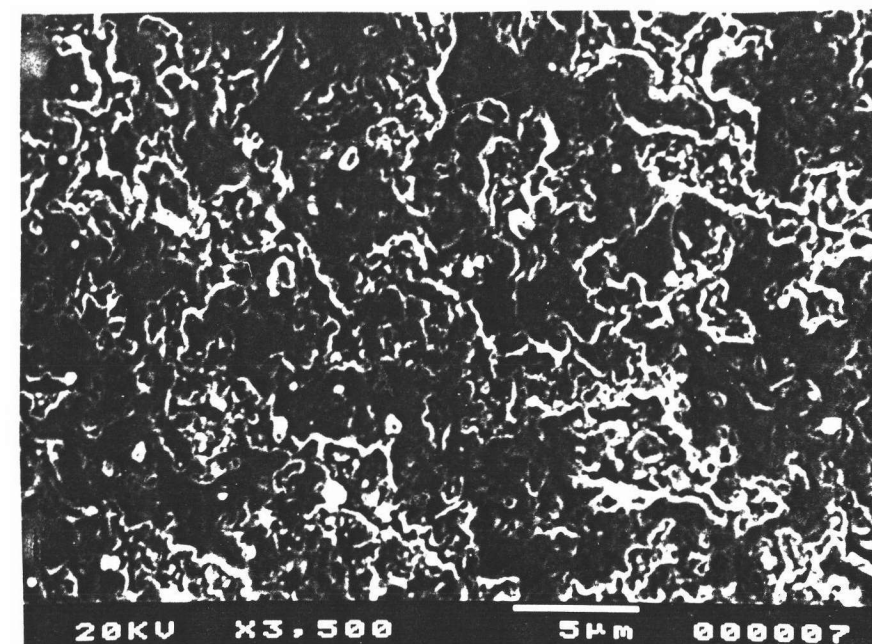
For $t_p = 6$ hr, the backside of CNR sheet was rather smooth, but for $t_p = 10$ hr the backside of surface was aggregated in its structure. It is proposed that when the NR sheet was dipped into pyrrole monomer (Figure 4.4(b)) PPy did not only wet NR sheet but some of them dissolved into NR sheet. When the polymerization process for a long time, PPy was continuously synthesized and deeply penetrated into NR sheet to form an aggregated structure as shown in Figure 4.10 (a). This is a reason why PPy attached very well with NR sheet (see section 4.2.4, SEM picture cross section) and formed a strong composite film.



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(a) $t_p = 10$ h (SEM picture magnified 1000 and 3500 times)



(b) $t_p = 6$ h (SEM picture magnified 3500 times)

Figure 4.10 The morphology of the backside of CNR (the side which attached to Pd electrode) sheet when (a) $t_p = 10$ h (b) $t_p = 6$ h.

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4.2.4 Effect of Reaction Temperature

CNR sheet was prepared at various reaction temperatures from 0 - 60°C. Table 4.6 shows the dependence of electrical conductivity on the reaction temperature while the other parameters were kept constant, i.e., the reaction time of 6 h at 25°C, 4 volt of applied potential across this cell, 20 ml of 2.00 M LiClO₄ solution, 0.05 M of pyrrole monomer and NR sheet prepared by 4 volt 4 minutes of coagulation time.

Table 4.6 Effect of reaction temperature.

Reaction Temperature (°c)	Conductivity (S/cm)	Product Yields (g/cm ²)
0	3.475E-04	0.117
10	3.682E-03	0.118
25	6.503E-03	0.154
55	6.612E-03	0.154
60	8.522E-03	0.154

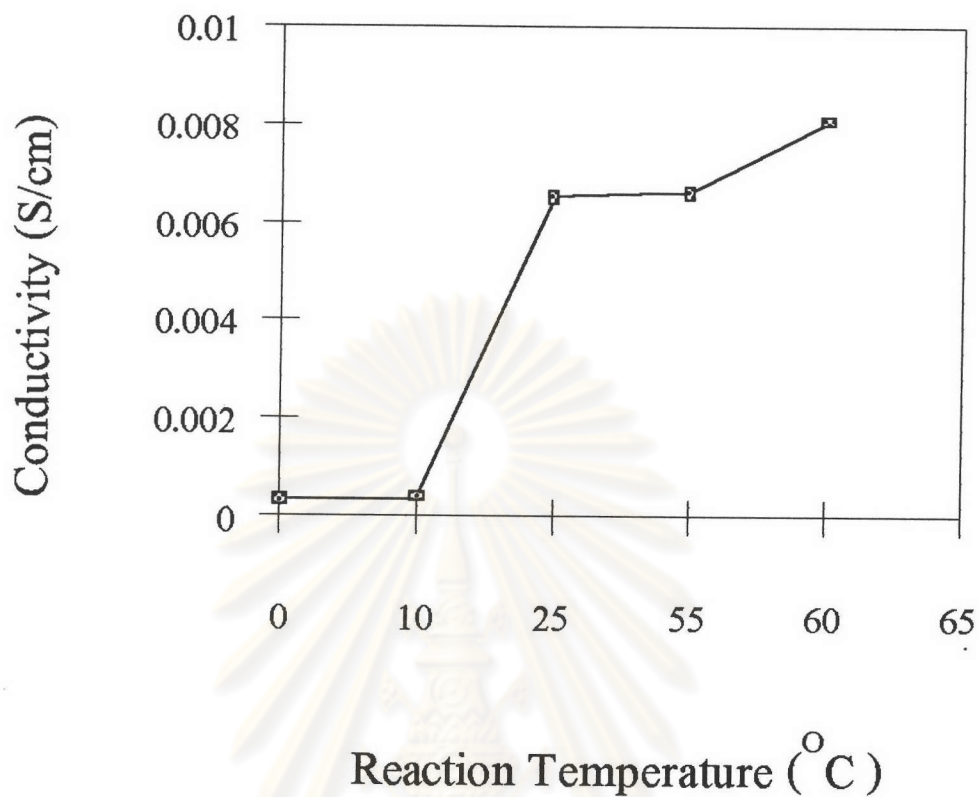
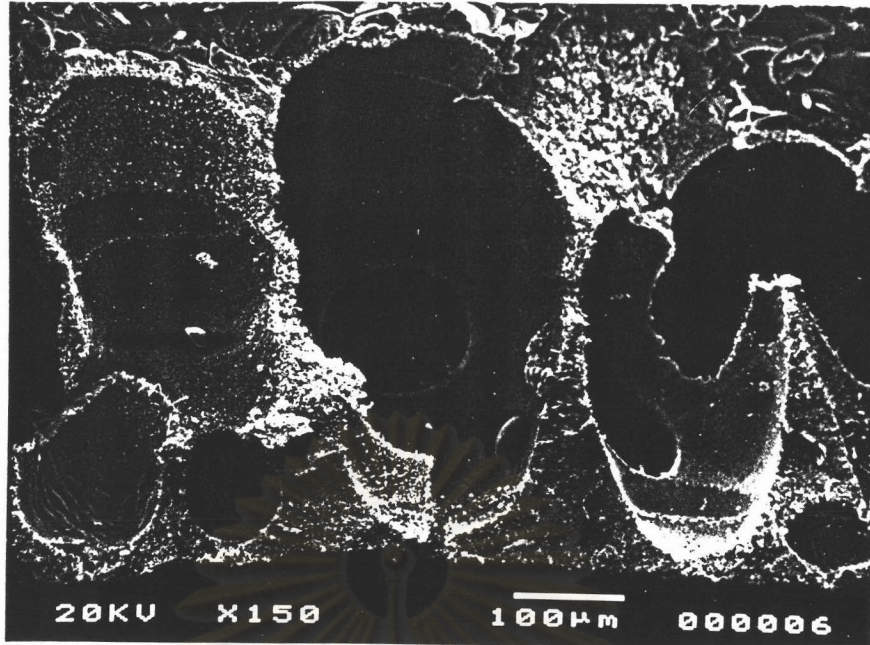
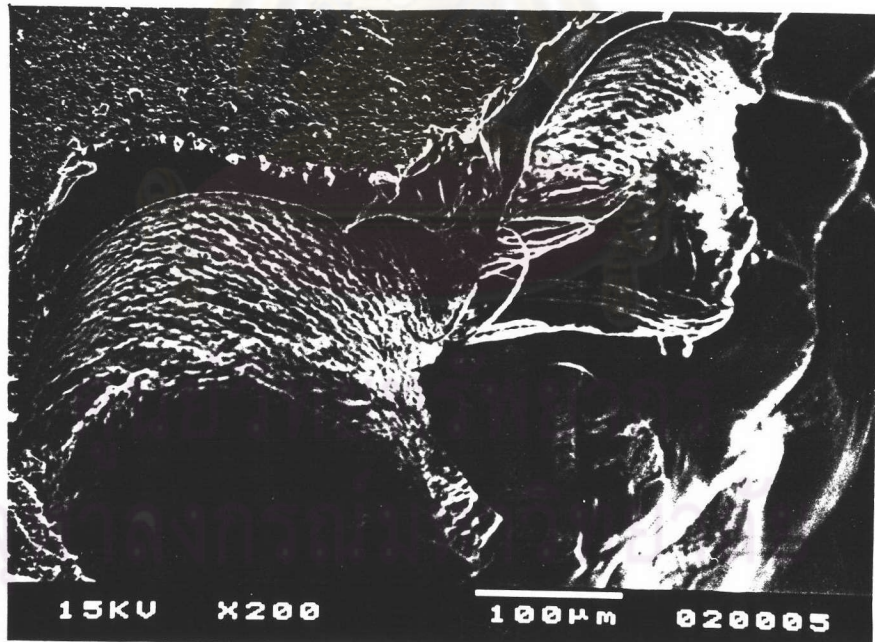


Figure 4.11 The electrical conductivity of conductive natural rubber prepared at various reaction temperature.

The result from Figure 4.11 suggested that CNR sheet was also prepared at temperature below 25 °C. It was let to the product with low electrical conductivity of 10^{-4} S/cm and very low yield. It may be explained that absorption of pyrrole monomer into NR sheet is retarded because NR sheet can be shrunk at low temperature, resulting in low product yield of CNR.



(a) 60 °C of polymerization temperature.



(b) 25 °C of polymerization temperature.

Figure 4.12 SEM of CNR for polymerization temperature at 60 °C and 25 °C.

At higher temperature, higher yield of CNR sheet with higher conductivity was obtained with a maximum conductivity of 8.5×10^{-3} S/cm at 60°C . However, NR can be degraded at 60°C . At higher temperature, the conductivity obtained is higher but the physical properties of product is inferior i.e., low flexibility and higher brittleness.

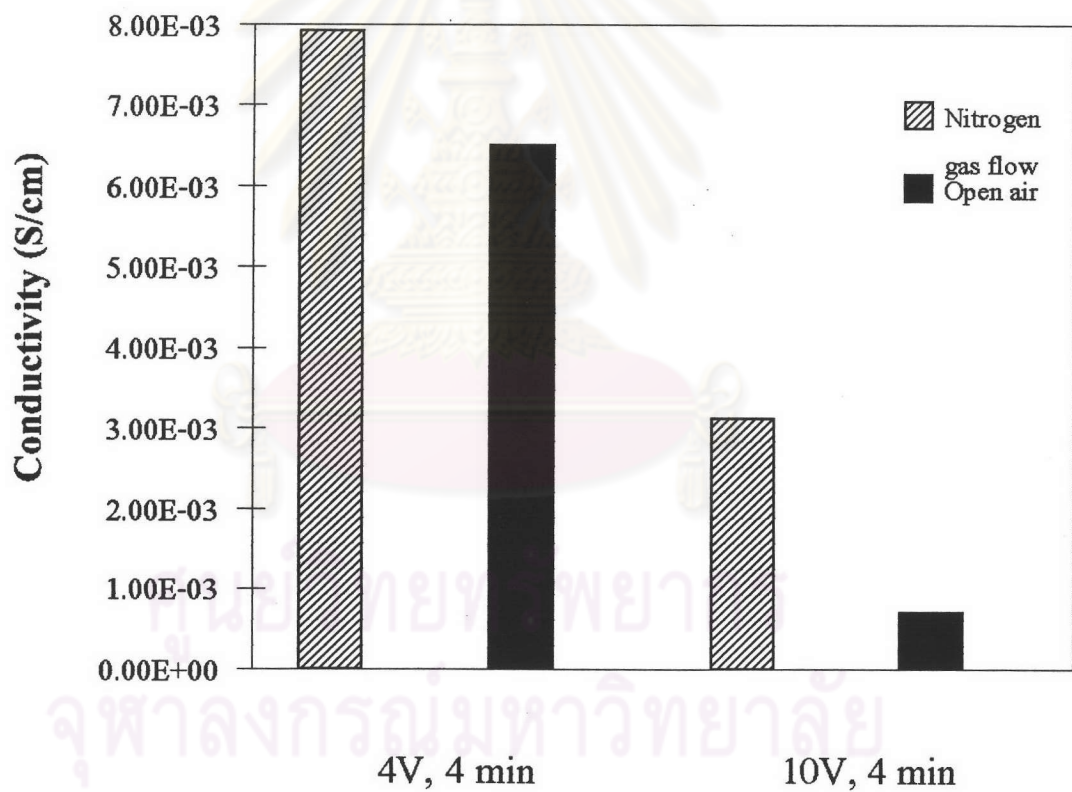
For polymerization temperature of 60°C (Figure 4.12 (a)) the CNR surface was rather smooth and with better distribution of the PPy on the internal interface of NR sheet, hence high connectivity of polypyrrole network and therefore higher conductivity. For polymerization temperature of 25°C , the cross section surface (Figure 4.12 (b)) was rough leading to lower electrical conductivity, However, it is not brittle because the fracture surface is PPy phases the filament inside that was NR which contributes to the flexibility of CNR together.

4.2.5 Effect of Reaction Atmospheres

To determine the effect of reaction atmosphere, two experiments were carried out. The first was the preparation of CNR where the atmosphere in the reactor was flowed with nitrogen. The second was carried out in the open air. All other parameters were kept the same, i.e., 0.05 M of pyrrole monomer, 20 ml of 2.00 M LiClO_4 solution for 6 h at 25°C and 4 volt applied across this electrochemical cell. Detailed results are shown in Table 4.7.

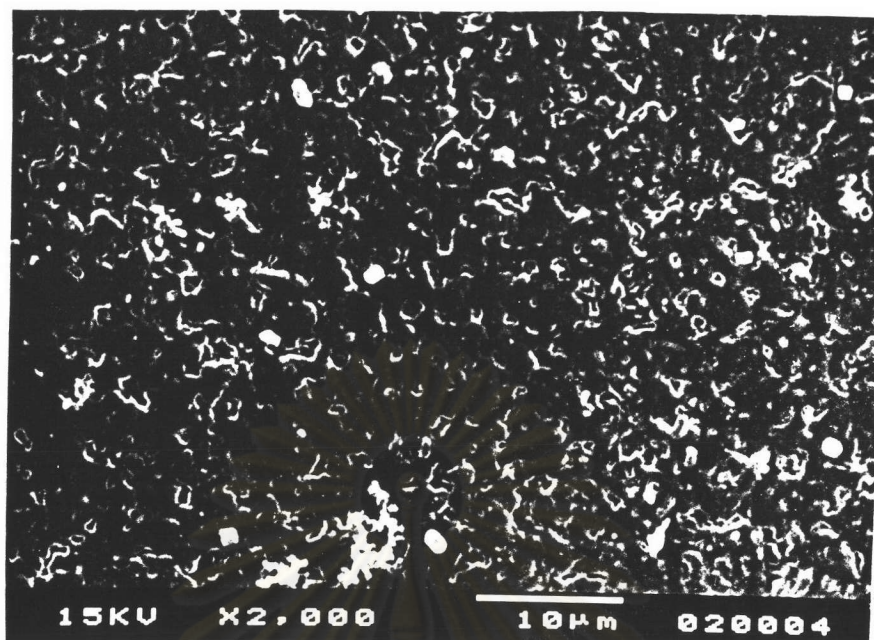
Table 4.7 Effect of various atmospheres.

Coagulation Time (4 mitnutes)	Conductivity (S/cm)	
Applied voltage (volt)	Nitrogen Gas	Open Air
4	7.916E-03	6.503E-03
10	3.117E-03	7.001E-04

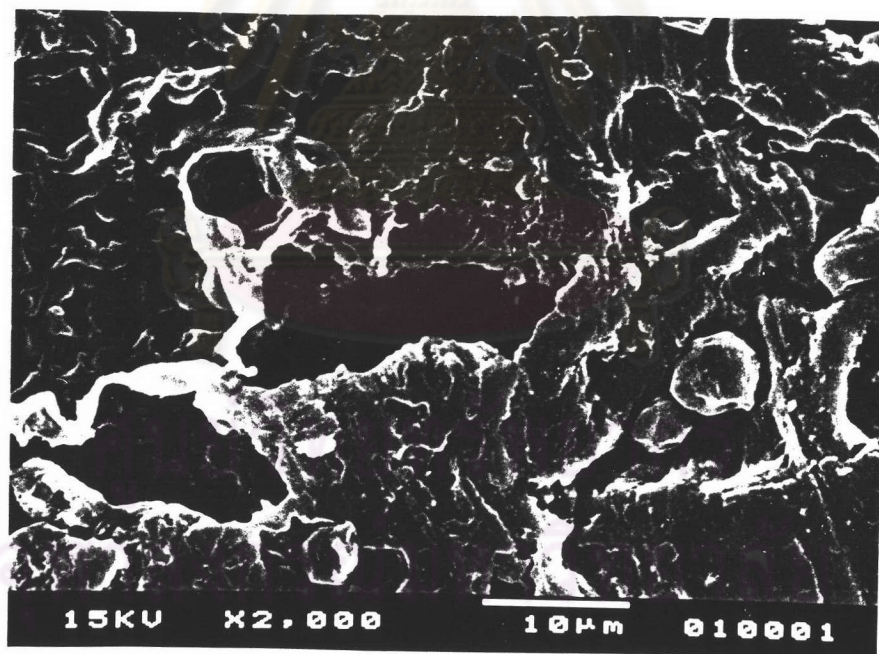
**Figure 4.13** The electrical conductivity of conductive natural rubber at various atmospheres.

As can be from Figure 4.13, it was found that the conductivity of CNR sheets prepared in electrochemical cell under nitrogen gas were higher than that prepared in open air. It is quite possible that the electrochemical prepared polypyrrole inside the porous host polymer (NR) is electroactive and can be switched between the neutral or nonconducting state to the oxidized an conducting state. The oxidation reaction is chemically reversible and repeated reactions lead to the loss of electroactivity [19]. Because of the low oxidation potential of this polymer it is very sensitive to the oxygen in the air. Therefore the preparations of CNR must be performed in the absence of oxygen. The choice of nitrogen gas flow into this electrochemical cell is also an important consideration with this reaction. Oxygen is a powerful inhibitor [4] and it reacts with radicals to form the relatively unreactive peroxy radical that reacts with itself or another propagating radical by coupling and disproportionation reactions to form inactive products. Thus the reaction was in oxygen, shorter conjugated chain polymer may be obtained and lead to low electrical conductivity.

Patil, Heeger and Wudl [1988] studied the optical properties of polypyrrole found that the electrical conductivity of polypyrrole reduced as $< 10^{-2}$ S/cm when PPy exposed to oxygen [27].



(a)



(b)

Figure 4.14 SEM of porous medium (in NR sheet) and composites (CNR sheet). (a) 4 volt for 4 min of coagulation time

(b) 10 volt for 4 min of coagulation time

From SEM examination (Figure 4.14), samples prepared at 4 volt show smaller pores than samples prepared at 10 volt. The internal surface areas of samples prepared at 4 volt are higher allowable more PPy to incorporate thus higher conductivity. This is due to the higher capillary rise and larger surface area of the porous medium can lead to a better distribution of the pyrrole and hence to a higher connectivity in the polypyrrole network. The connectivity among the polypyrrole films located on the surface of interconnected pores, rather than the polypyrrole powder accumulate in the void, contributes to conductivity.

4.2.6 Time-Decay Conductivity of CNR Sheet

After storing CNR sheets in open air and dry air (in desiccator) at room temperature for a period of time and repeatedly measured their conductivities, it was found that conductivity decreased gradually with time until it reached zero.

Table 4.8 Time - decay conductivity of CNR sheet.

Days	Conductivity (S/cm)	
	Dry Air	Open Air
1	4.194E-03	4.200E-03
5	2.650E-03	2.141E-03
15	1.360E-03	3.734E-06

Table 4.8 (continued)

Days	Conductivity (S/cm)	
	Dry Air	Open Air
30	3.400E-04	1.331E-08
60	2.918E-05	0
90	1.870E-06	0
120	7.040E-08	0
150	0	0

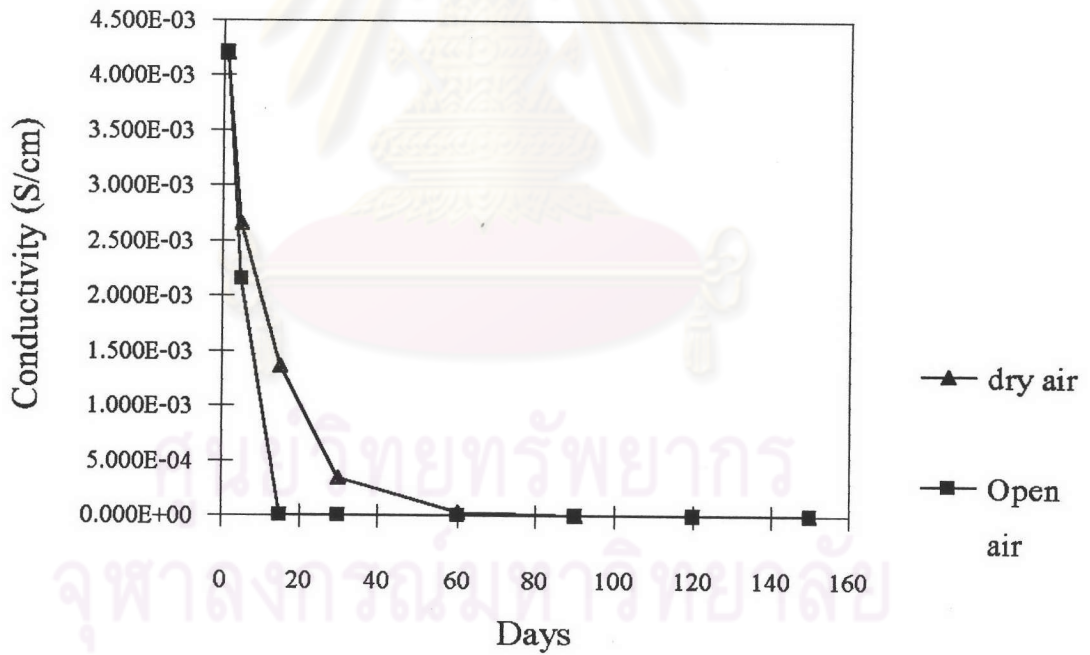


Figure 4.15 Time - decay conductivity of CNR sheet.

From Figure 4.15 it was found that the conductivities of CNR sheets which stored in desiccator (dry air) were higher than that stored

in open air. It is quite possible that for the polymer stored under open air circulation the charge decay is accelerated. This can be explained that the moisture in open air interferes with the interaction force between polymer chain of polypyrrole in porous medium of NR sheet with doping anion. Thus the conductivity was reduced. Thanawadee (1993) studied FTIR spectra of PPy disc stored in dry air in desiccator at room temperature for one month and found no significant change in FTIR spectra. However, in open air there are absorption band at $3000 - 3500 \text{ cm}^{-1}$ which indicated OH covalent bond in the polymer chains.

However, FTIR spectrum of CNR sheet stored in open air did not show any significant change compared to those of CNR sheet in dry air (Figure 4.16).

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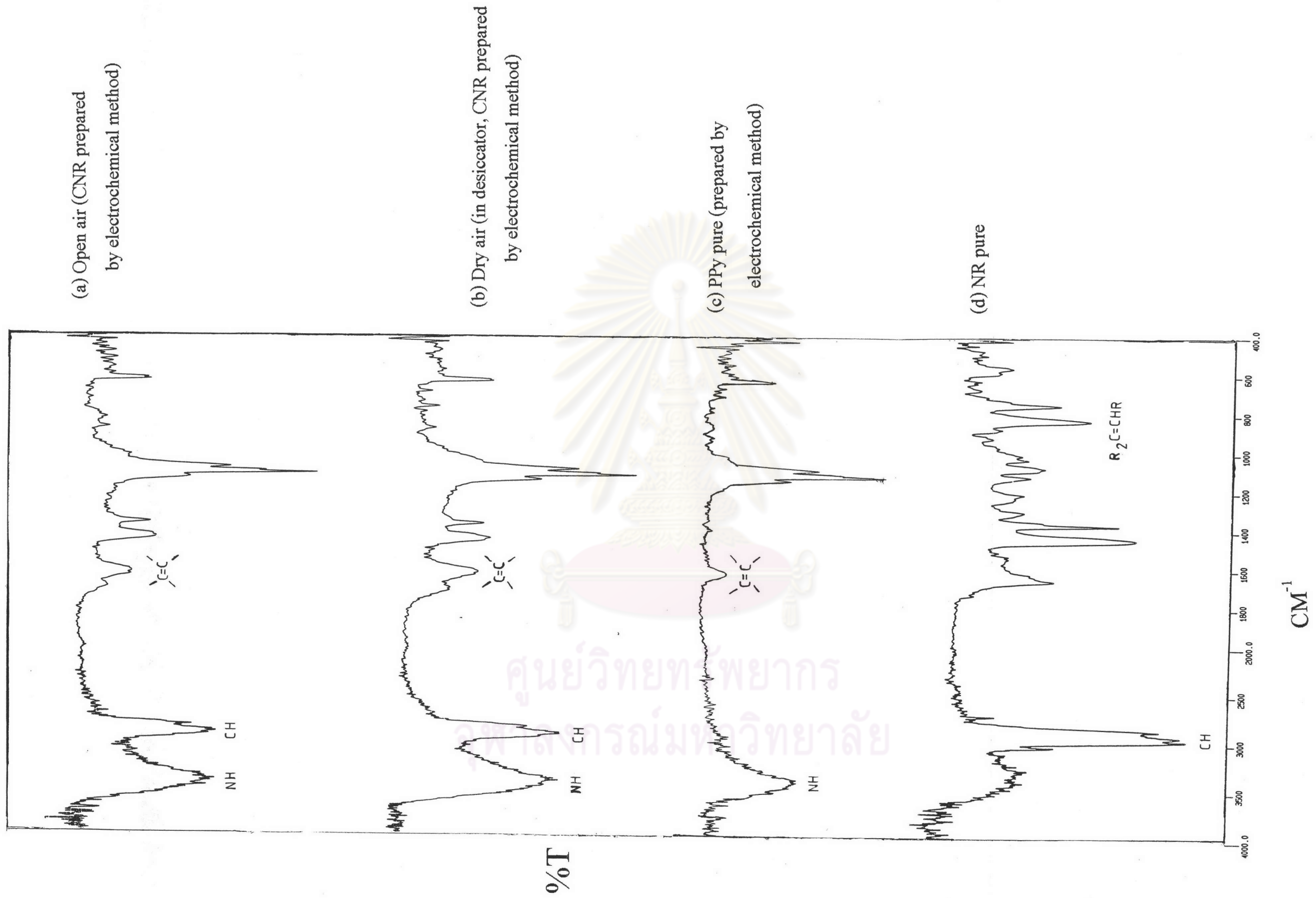


Figure 4.16 FTIR spectrum of conductive natural rubber sheet stored in open air and dry air (in desiccator).

4.2.7 Effect of Acid and Base Solution on the Conductivity of CNR Sheet.

Experimental results on the treatment of the CNR sheet, initial conductivity of $7.1 \times 10^{-03} \text{ Scm}^{-1}$, with various concentration of H_2SO_4 and NaOH from 0.25 M to 1.00 M at room temperature for 7 days, were shown in Table 4.9.

Table 4.9 Effect of acid and base solution on conductivity of CNR sheet. (CNR sheet weight $0.156/\text{cm}^2$).

Concentration [M]	Conductivity(s/cm)	
	H_2SO_4	NaOH
0	7.122E-03	7.122E-03
0.25	2.850E-03	1.440E-03
0.50	2.180E-03	8.510E-04
0.75	1.880E-03	7.320E-04
1.00	1.760E-03	6.110E-04

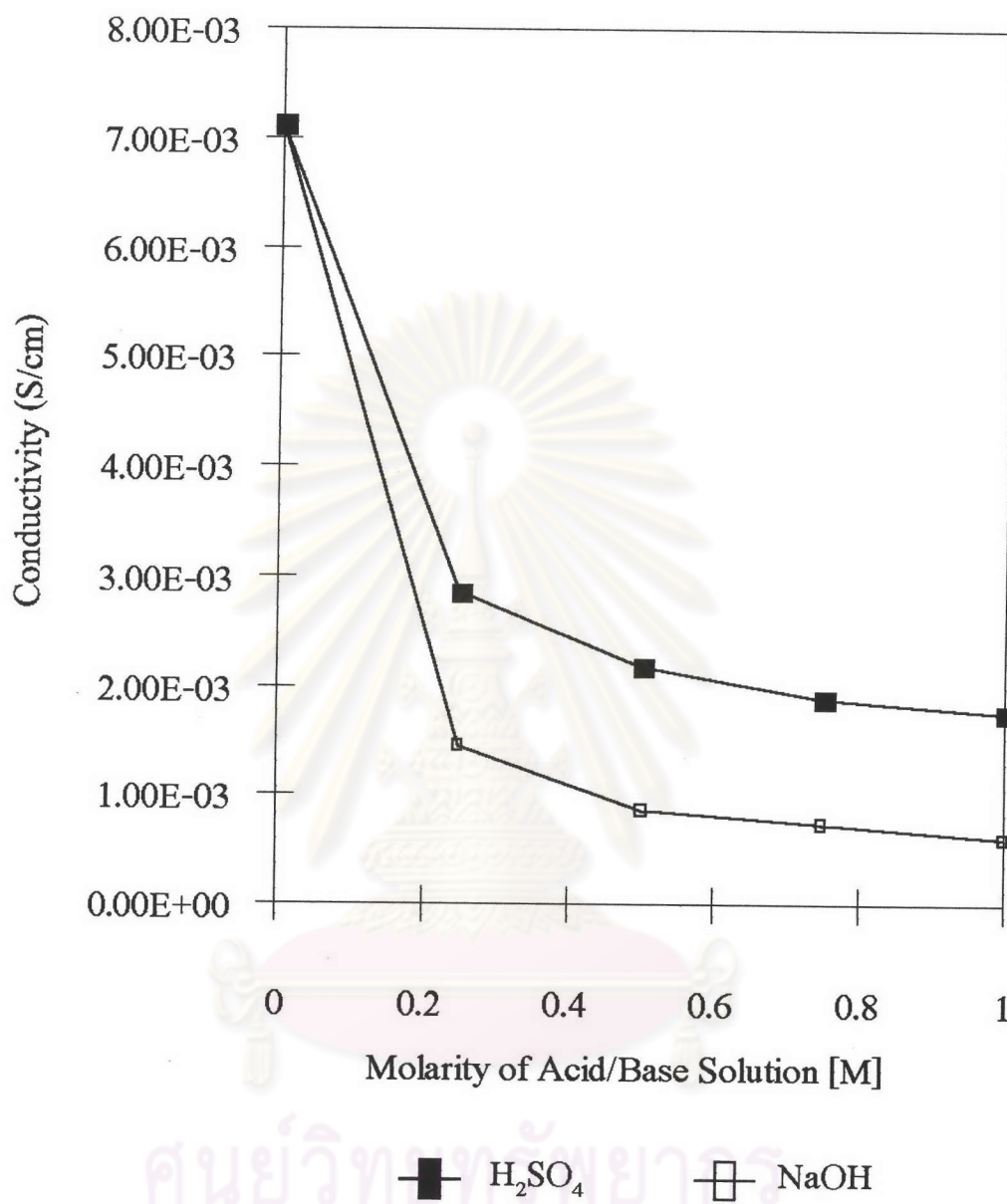


Figure 4.17 Conductivity change of the polypyrrole after acid/base treatment.

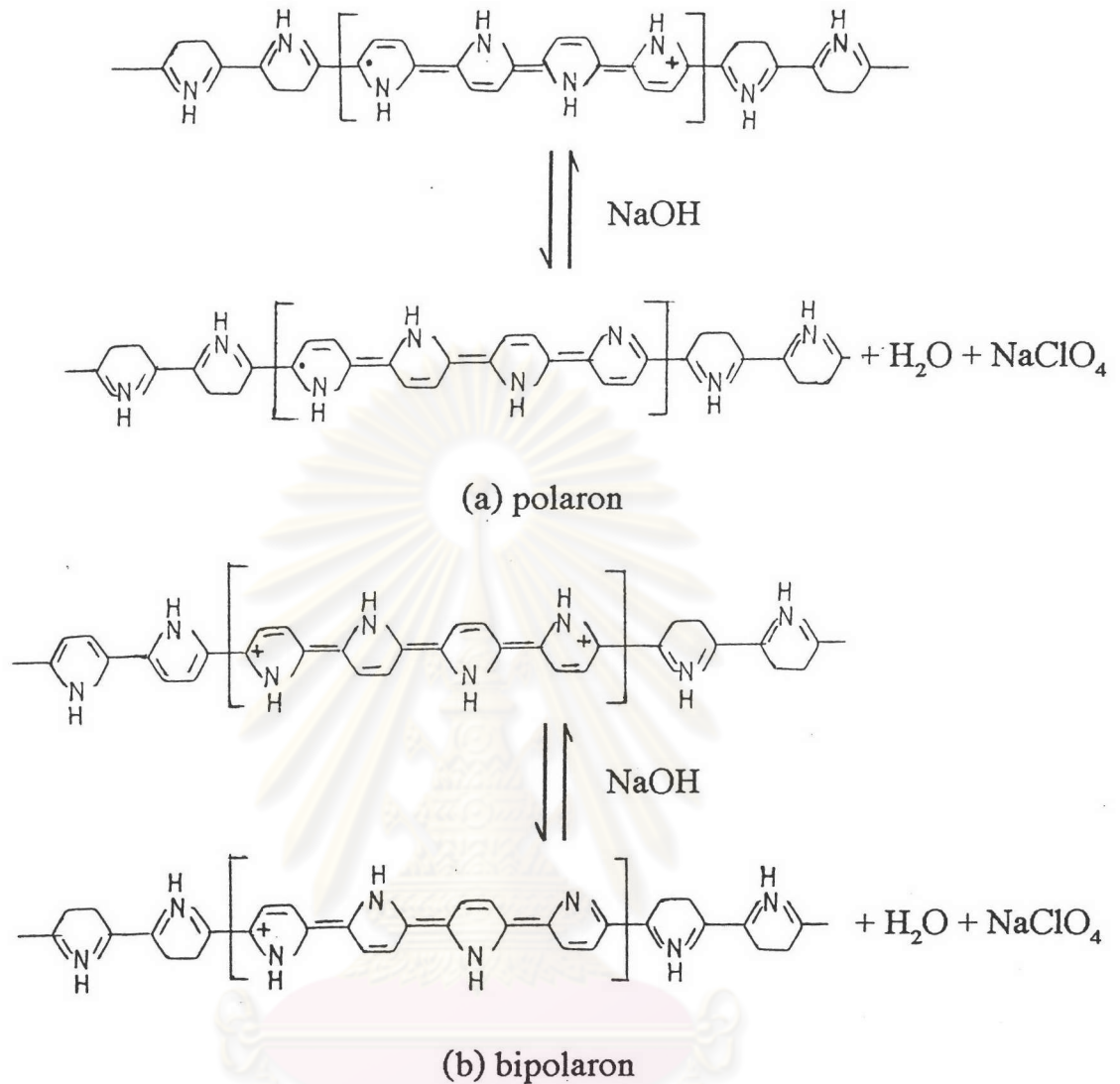
As can be seen in Figure 4.17 NaOH treatment resulted in less conductivity of polypyrrole in porous of NR than H₂SO₄ treatment. This decrease, by any mean, was not a function of the concentration in the range

studied. From the result, it was found that the treatment of CNR with acid and base solution led to a decrease of conductivity.

When it was treated with NaOH, it was expected that there was also reversible deprotonation at the nitrogen atoms in PPy (in the porous medium (NR matrix)). This will affect the electronic structure of the polymer, by leaving a single electron on the polymer backbone during deprotonation. The polymer deprotonation may also lead to a decrease of polaron or bipolaron of the conjugated chain length of the polymer as proposed below. Thought the conductivity of CNR was decreased too.



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Deprotonation at nitrogen atom in the polymer [28].

When treated CNR with H_2SO_4 , the conductivity of this sample was slightly reduced, compared to that of NaOH treatment. It was probably resulted in the exchange of sulfate ions for perchlorate ions, see Table 2.5. Polypyrrole films containing perchlorate and sulfate anions have the conductivities which are in the range 60-200 S/cm and 0.01-50 S/cm respectively. This result may lead to the conductivity of CNR sheet decreased.

Thanawadee (1993) studied the FTIR spectra for acid and base treated polypyrrole powder. The FTIR spectra in all three cases ; PPy treatment with H₂O, PPy treatment with 1.00 M H₂SO₄, PPy treatment with 1.00 M NaOH showed a long absorption from 4000 to about 2000 cm⁻¹ [26]. These long absorption tails have been assigned to be the tail of OH covalent bond formation with polypyrrole. The FTIR spectra of H₂SO₄ or NaOH treated still showed bands characteristic of pyrrole rings in the polymer. The FTIR spectra of polypyrrole when treated shows absorption bands at 1100-610 cm⁻¹ [26] which is a characteristics of the sulfate anion.

FTIR spectrum of the CNR sheet treated with H₂SO₄ and NaOH (Figure 4.18) indicated absorption band of olefin (cis-1,4 polyisoprene) at 820 cm⁻¹, CH in-plane bending and CH out of plane bending 1310-690 cm⁻¹ [see Appendix B]. There was an absorption band at 1100-600 cm⁻¹ which presumably the sulfate anion of polypyrrole.

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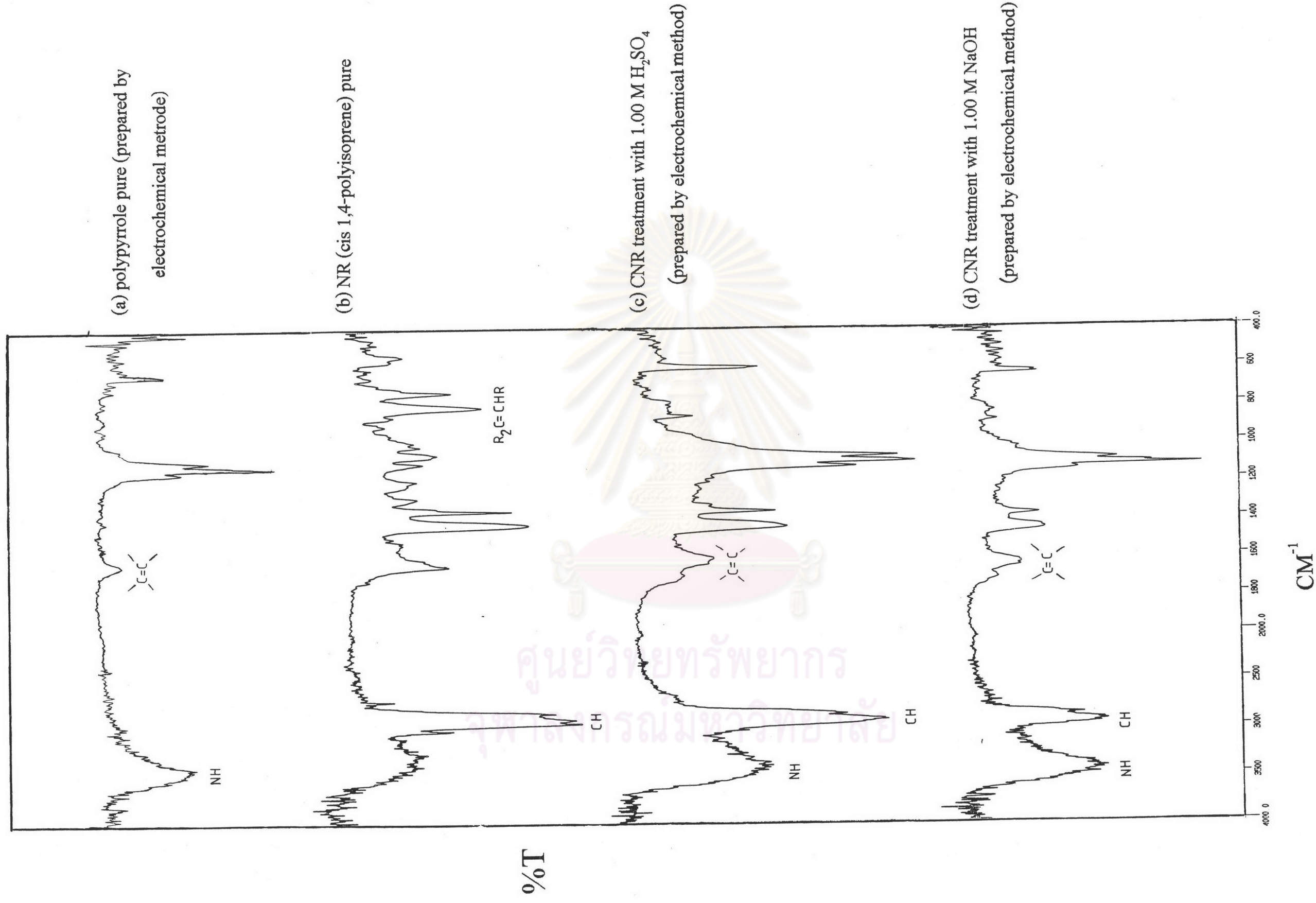


Figure 4.18 FTIR spectrum of CNR after acid and base treatment.

4.3 Preparation of Conductive Natural Rubber by Chemical Dissolution Method.

PPy can be prepared electrochemically as well as chemically. The physical appearance and conductivity of PPy strongly depend upon the preparation route employed. The chemical oxidation route led to PPy powder, while the electrochemical route provides free-standing PPy films.

It is therefore appropriate to compare the prepared conducting polymer composites (CNR sheet) by chemical oxidation method and the electrochemical method.

A NR sheet was prepared by electradecantation method at 4 V for 4 min of coagulation time (see section 3.2), then it was blended with powder PPy prepared by chemical oxidation method (see section 3.4) in toluene solvent with various the amount of PPy at a concentration of 10, 20, 30, 50, 70 and 90 %w. Blending was carried out as described in section 3.4, by stirring at 50°C for 24 h.

Finally, the CNR sheet was obtained, and its electrical conductivity measured (see section 3.5). The result of this experiment is summarized in Table 4.10.

Table 4.10 Conductivity of CNR sheet at various PPy content.

Experiment No.	Part by Weight Percent of PPy (%)	Conductivity (S/cm)
1	0	0
2	10	0
3	30	2.819E-04
4	50	7.604E-03
5	70	2.770E-02
6	90	2.082

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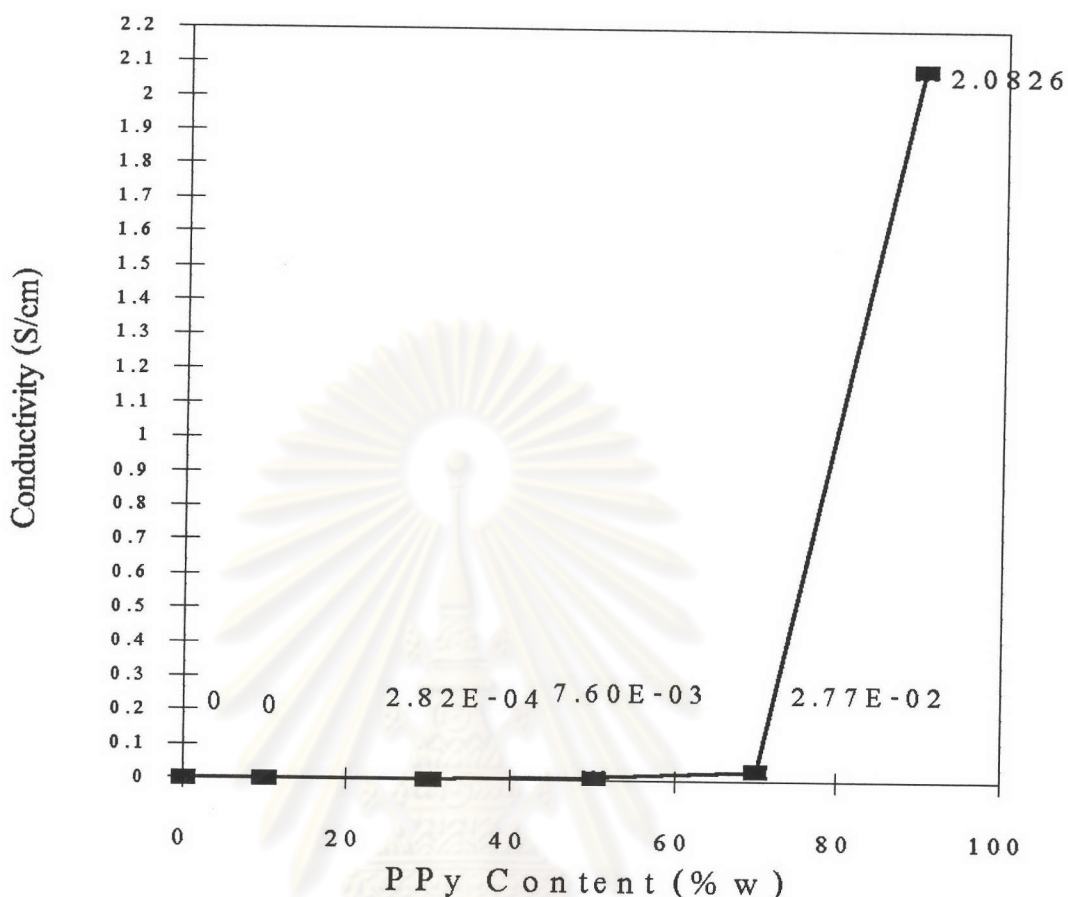


Figure 4.19 Conductivity of CNR (S/cm) prepared by blending.

From Figure 4.19, it was found that the conductivity of CNR disc increases as the content of powder PPy increases. But it is not higher than the conductivity of CNR sheet prepared by electrochemical method. It is clear from Figure 4.20 that at 10% by weight of PPy in porous medium, the conductivity of CNR prepared by electrochemical method is 4.2×10^{-3} S/cm where that prepared by the other method is zero S/cm. It is proposed that the prepared CNR disc by chemical method underwent many steps and took longer time for its preparation, therefore the conductivity was reduced because the charge decay was accelerated.

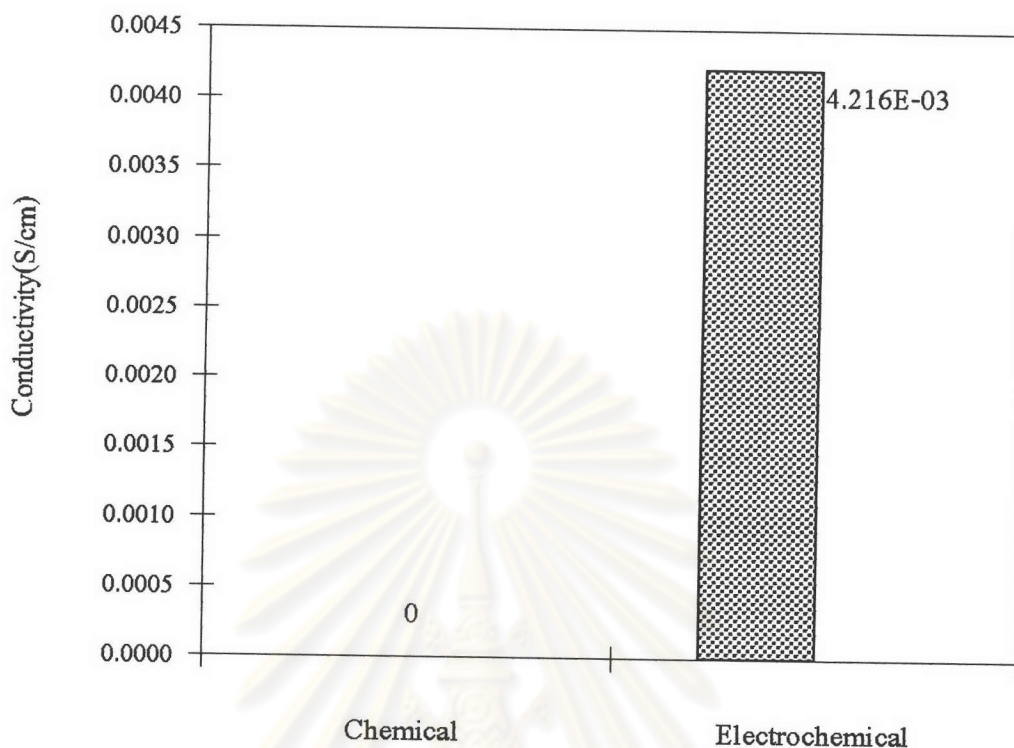


Figure 4.20 Conductivity of CNR prepared by two method (10%w of PPy).

Another possibility is that, the CNR disc prepared by chemical solution dissolved in toluene solvent and became a suspended colloid similar to that at the start of reaction before preparation of CNR by chemical mean. But the CNR sheet prepared by electrochemical method did not dissolve in toluene solvent. The FTIR spectrum of CNR composition prepared by electrochemical method and chemical method, were shown in Appendix B.

It may be concluded that to prepared a composite that has pores sufficient for high conductivity, good mechanical strength, and allow ease of processibility (i.e., are readily able to be shaped). The electrochemical method must be used.