

CHAPTER 6

FILM SYNTHESIS AND PRELIMINARY RESULT

6.1 Film Synthesis

Polyacetylene was first synthesized by addition polymerization. This method requires initiator and catalyst to form the polymer chain. Nowadays polymer can be prepared from monomer vapor by the glow discharge techniques. This technique differs from addition polymerization in that it doesn't use initiator and catalyst. This technique is more convenience than first technique. According to many advantages, glow-discharge techniques have been chosen in this thesis.

6.1.1 Synthesis procedures

The glass substrate is placed in the parallel position at the center close to the mirror plate where plasma is formed. The reactor chamber is evacuated to a low pressure of 5.0×10^{-3} torr. Acetylene gas to be used in this thesis for synthesis polyacetylene thin-film by using glow discharge process of the RF-PECVD system. Acetylene gas is introduced into the reactor chamber via gas flow control.

Acetylene gas is introduced into the reactor for 3 minutes. After that, the reactor chamber is evacuated again to pressure of 0.01 torr and repeated the same procedure for three times to minimize the contaminants in the chamber. Introduce acetylene gas and adjust the pressure to desire value of 0.5 torr and let the pressure to achieve equilibrium.

Finally, switch on the RF generator. The vacuum capacitor of matching network was tuned frequently to obtain minimum reflected power. Increasing RF power to the RF-PECVD system until the E-mode of plasma generated. The RF generator is adjusted to a RF power of 100 watt for 5 minutes. The treated substrates were then

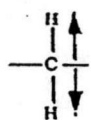
analyzed by NMR spectroscopy and IR spectroscopy. Photograph of polyacetylene thin-film is shown in figure 6.1.



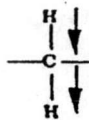
Figure 6.1: Photograph of polyacetylene thin-film on glass substrate

6.2 Film Analysis

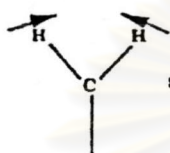
NMR spectroscopy and IR spectroscopy techniques are used in the study of polymer thin-film in this thesis. IR spectroscopy can give information about the chemical structure of a polymer. Furthermore, strength of IR absorption measurements can give quantitative information about the composition of any mixture [15, 16]. By allowing infrared radiation to pass through the sample, an infrared absorption spectrum of a material is obtained. The frequency at which any peak in the absorption spectrum appear is equal to the frequency of one of the normal modes of vibration of the molecules of the sample. The vibration of molecular bond can be divided into three modes as shown in Figure 6.2.

Stretching:

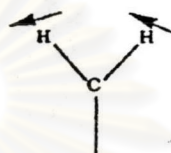
symmetric



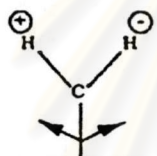
asymmetric

In-plane bending

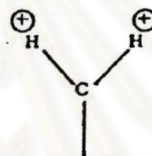
scissor



rocking

Out-of-plane bending

twisting



wagging

Figure 6.2: Vibration modes

Figure 6.3 shows a summary of the absorption bands (group frequencies) from important groups. This scheme is very useful in chemical analysis. The presence of absorption bands in certain frequency regions suggests the presence of certain groups in the sample. For example, IR spectra of methanol (CH_3OH) show absorption band of the C-O bond stretching between $1000\text{-}1320\text{ cm}^{-1}$ and the O-H bond stretching show peak between $3300\text{-}3700\text{ cm}^{-1}$.

are given in parts per million (ppm) downfield from tetra methylsilane as internal standard.

6.2.2 Results and discussion

From the structure of polyacetylene ($\text{---CH=CH-CH=CH-CH---}$). IR spectra should show absorption band of ---C=C--- bond stretching. From the IR spectra in Figure 6.4 shows an absorption band at 1630 cm^{-1} due to ---C=C--- bond stretching [17,18,19,20]. The =C-H bond stretching shows an absorption peak at 2921 cm^{-1} . The broad band at 3446 cm^{-1} due to C-OH bond stretching. It due to the structure of thin-film has hydroxyl group (OH) in molecule. That may be occur from the result of conjugate double bonds (---C=C-C=C---) in polyacetylene are easy to oxidized by oxygen and moisture in air, that convert ---C=C--- to $\begin{array}{c} \text{---C---C---} \\ | \\ \text{OH} \end{array}$

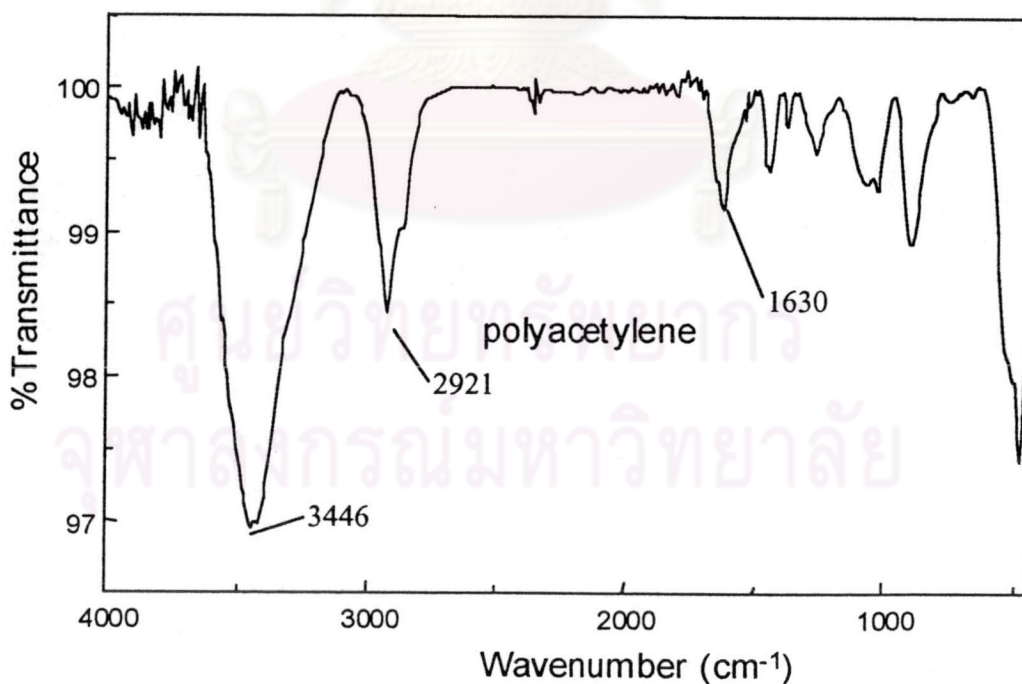


Figure 6.4: The IR spectra of thin-film

The ^1H NMR spectra of the thin film are shown in Figure 6.5. The spectra of the thin-film shows peaks around 5.69-6.51 ppm for the vinylic protons (H-C=) of polyacetylene due thin-film molecules have C=C . The hydroxylic protons (OH) show peak at 2.0 ppm [17,18,19,20]. The peaks around 2.10-2.30 ppm and 3.4-4.2 ppm due to protons near the hydroxyl protons (Figure 6.6). These peaks show thin-film has hydroxy groups that form by oxidation of conjugate double bonds. The protons $-\text{CH}-\text{CH}$ show peak between 1.3-1.5 ppm.

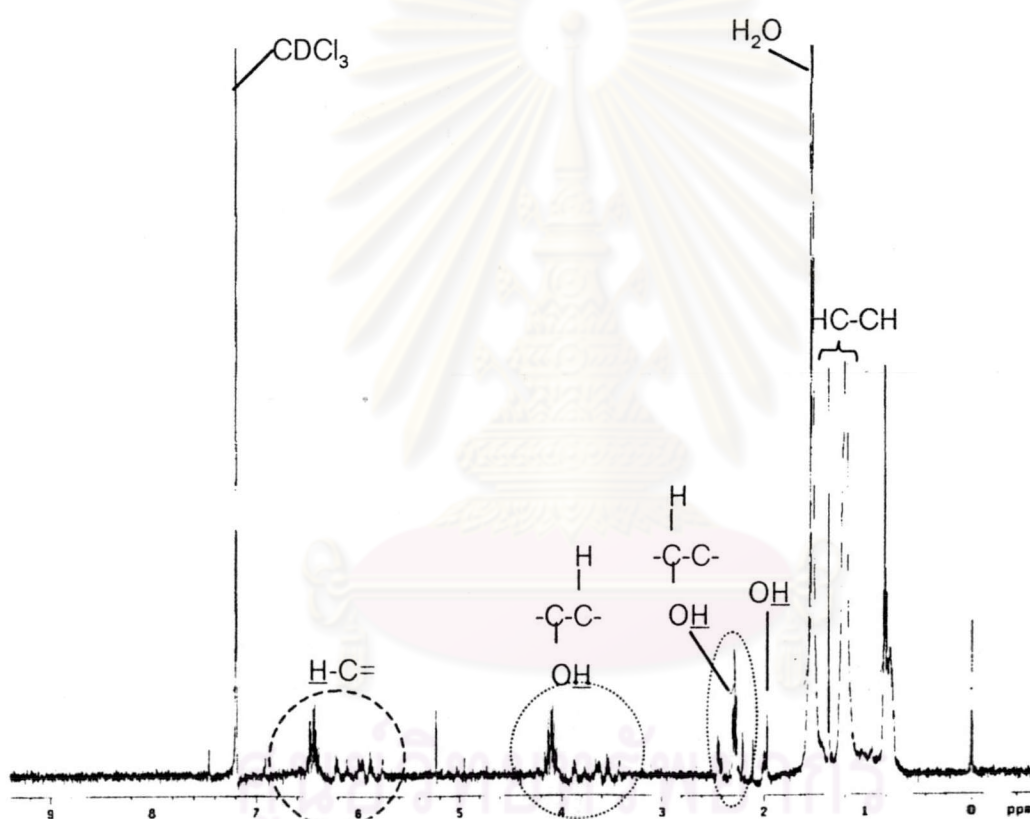
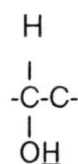
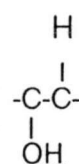


Figure 6.5: The NMR spectra of thin-film



Show peak at 2.10-2.30 ppm



Show peak at 3.4-4.2 ppm

Figure 6.6: Proton environment structures

The data from IR and NMR spectroscopy can concluded that the thin-film has -C=C- bonds, =C-H bonds and -C-OH bonds. The -C=C- bonds and =C-H bonds due to the functional group of polyacetylene and the -C-OH bonds due to oxidation of polyacetylene by oxygen and moisture in air. Therefore, this thin-film should be polyacetylene.



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