CHAPTER IV

RESULTS AND DISCUSSION

4.1 Synthesis of Silatrane Precursors

The silatrane precursors were synthesised directly from SiO_2 and triethanolamine as mentioned in the previous chapter. The initial reaction mixture was milky. As the reaction proceeded further, the mixture became clear, indicating that the reaction was completed. Subsequently, the mixture was introduced to a high vacuum distillation in order to remove ethylene glycol. White solid product was isolated easily after distillation.

4.2 Characterisation

4.2.1 Characterisation of Silatrane Precursors

4.2.1.1 *Fourier transform infrared spectrophotometre (FTIR)*

Figure 4.1 shows FTIR spectrum and data of the obtained product of the reaction. Major characteristics FTIR peaks found in Figure 4.1 along with the most likely assignment are summarised in Table 4.1.

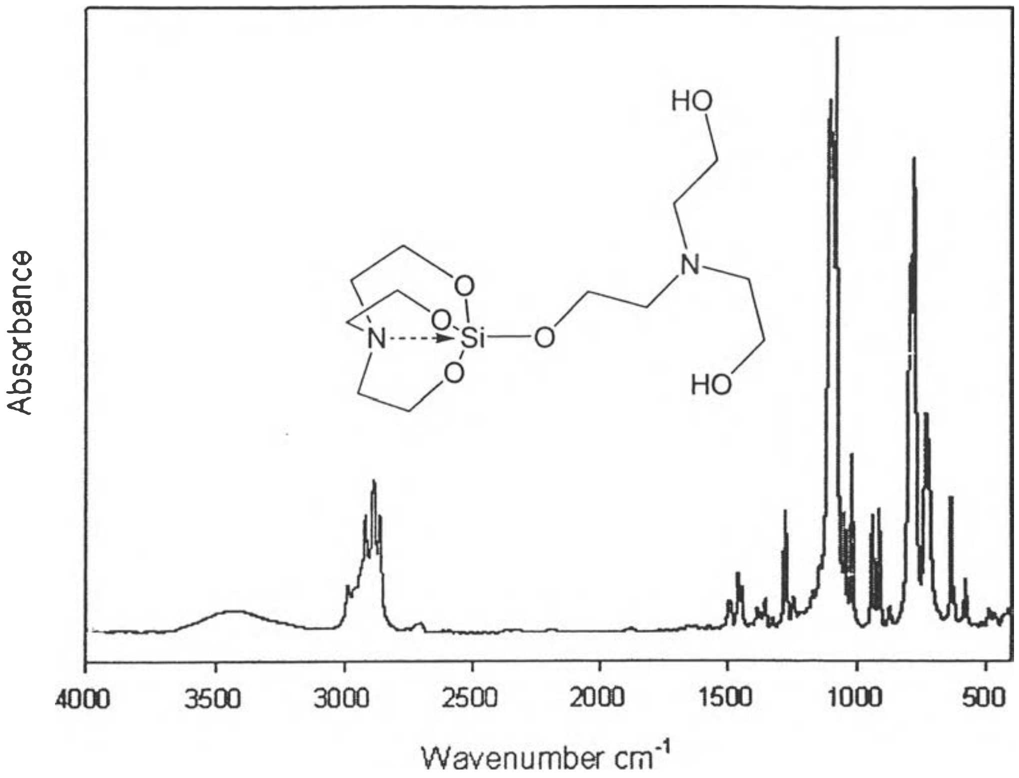


Figure 4.1 FTIR spectrum of obtained silatrane precursors.

Table 4.1 Assignment of infrared spectrum of the products

Characterisation	**Peak Position (cm^{-1})**
N -> Si stretching	560-590, 2697
Si-O-CH	970,883
C-O	1030-1070
Si-O-CH_2	1015-1085
C-N	1351
C-H bending	1380-1460
C-H stretching	2800-2976
O-H stretching	3422

According to Table 4.1, the peaks at 560-590 cm^{-1} and 2697 cm^{-1} are assigned to N -> Si stretching. The peaks at 970 and 883 cm^{-1} correspond to the Si-O-CH groups, whereas the peaks at 1030-1070 result from C-O groups. The peaks at 1015-1085 cm^{-1} are assigned to Si-O-CH_2 groups, and the peaks at 1351 result from C-N groups. The peaks at 1380-1460 correspond to C-H bending, whereas the peaks at 2800-2976 cm^{-1} are assigned to C-H stretching, and the broad band centering at 3422 cm^{-1} corresponds to O-H stretching. The results confirm the formation of silatrane precursors.

4.2.1.2 *Thermogravimetric Analyser (TGA)*

Figure 4.2 shows TGA result and data of the obtained product of the reaction. Major characteristics TGA result found in Figure 4.2.

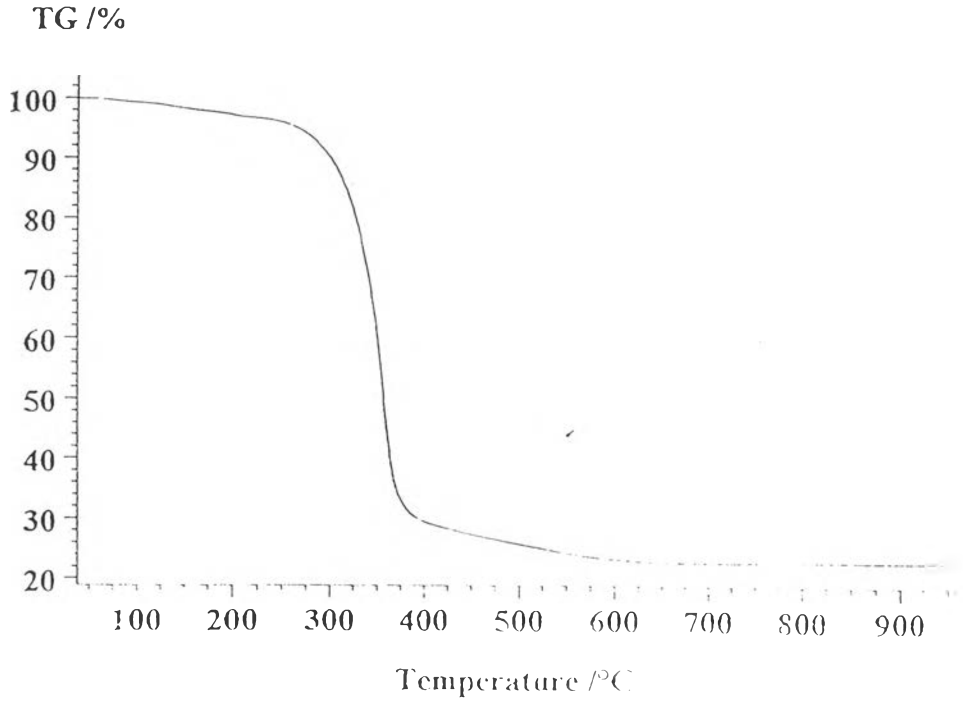


Figure 4.2 TGA result of obtained silatrane precursors.

The first mass loss between 150-250°C corresponds to the loss of trace amount of ethylene glycol. The second mass loss between 290-370°C corresponds to organic ligand decomposition, and the final mass loss from 390 to 620°C corresponds to oxidation of residual carbon char. The final ceramic yield was 21.5%.

4.2.2 Characterisation of Electrospun Silica Fibres

4.2.2.1 *X-ray diffraction (XRD)*

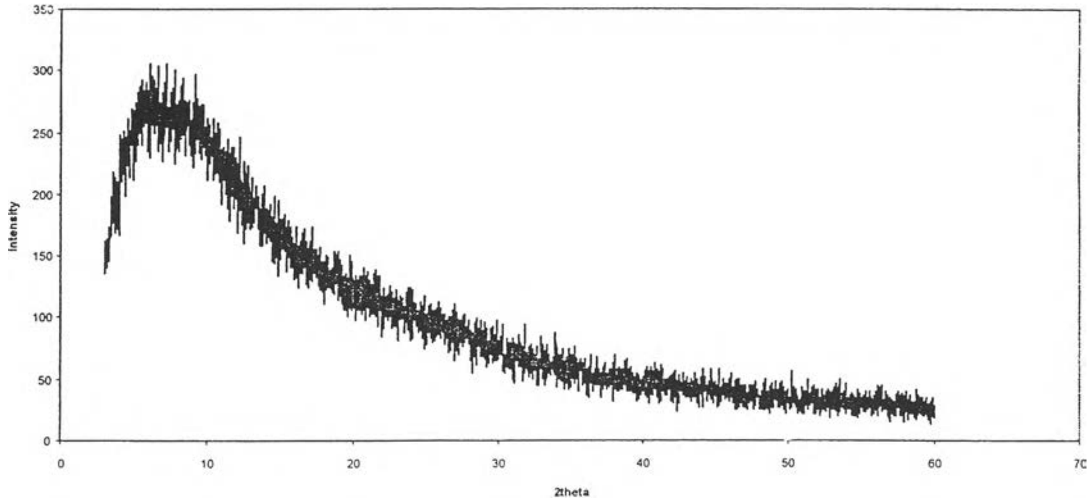


Figure 4.3 XRD pattern of precalcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV.

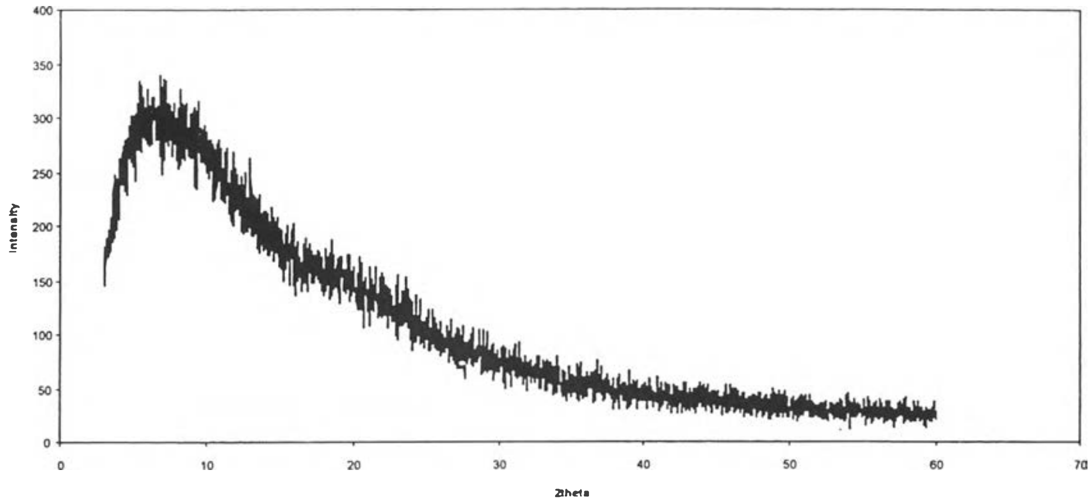


Figure 4.4 XRD pattern of precalcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV.

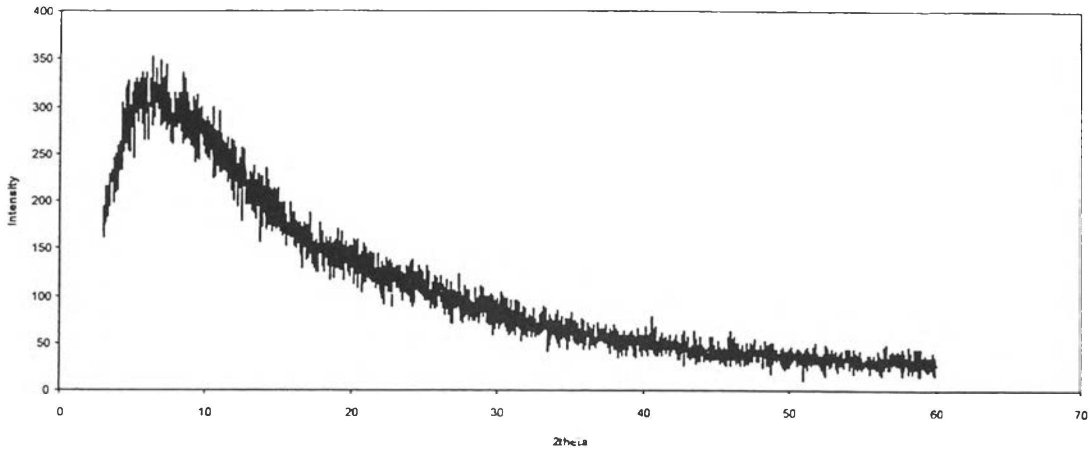


Figure 4.5 XRD pattern of precalcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV.

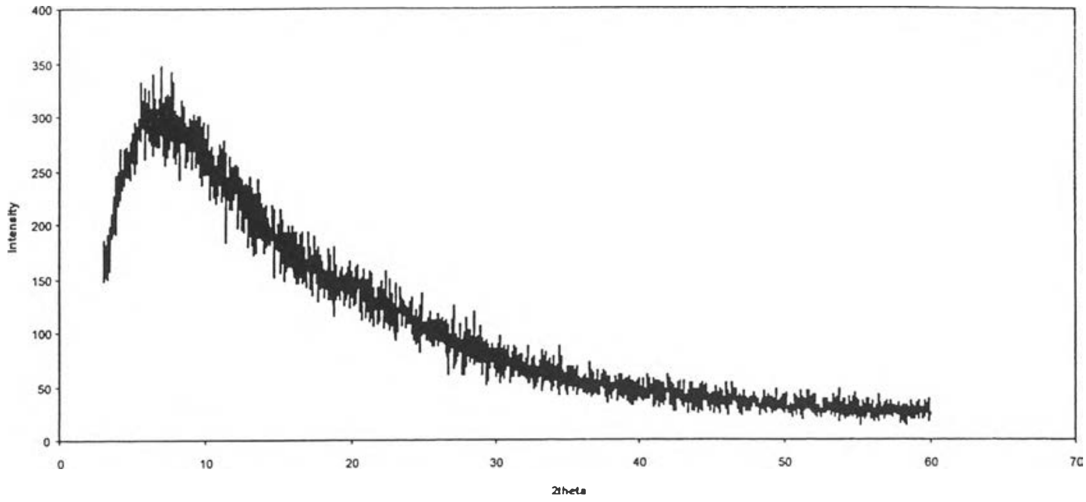


Figure 4.6 XRD pattern of precalcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV.

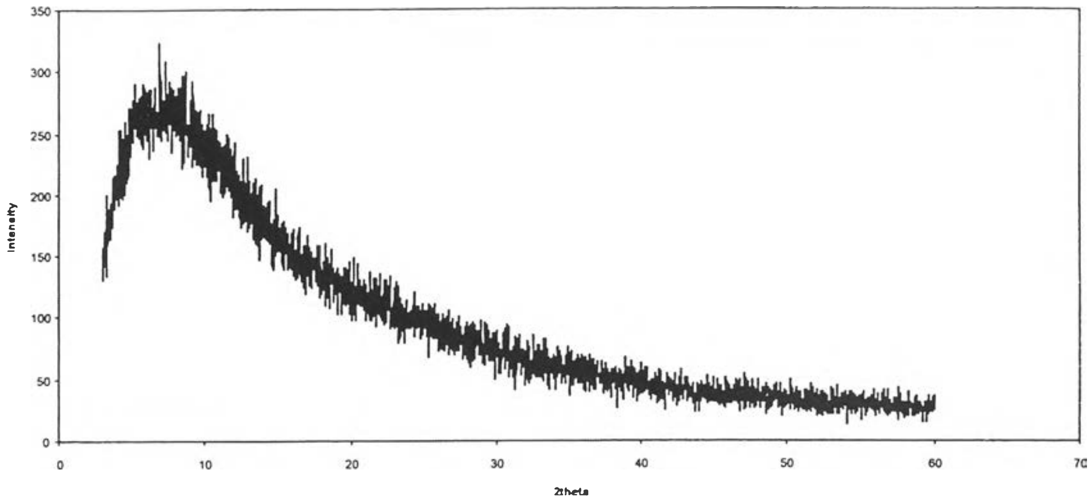


Figure 4.7 XRD pattern of precalcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV.

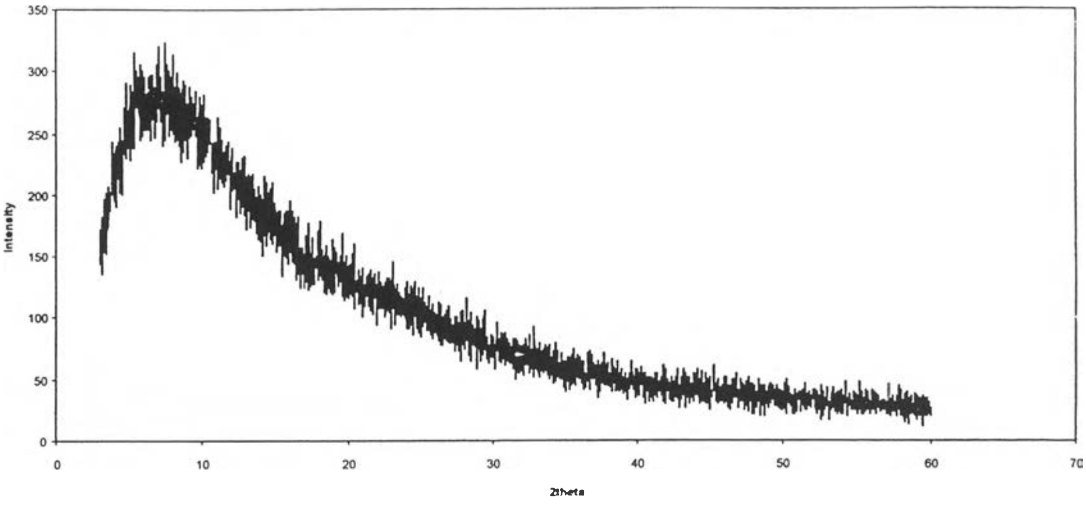


Figure 4.8 XRD pattern of precalcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV.

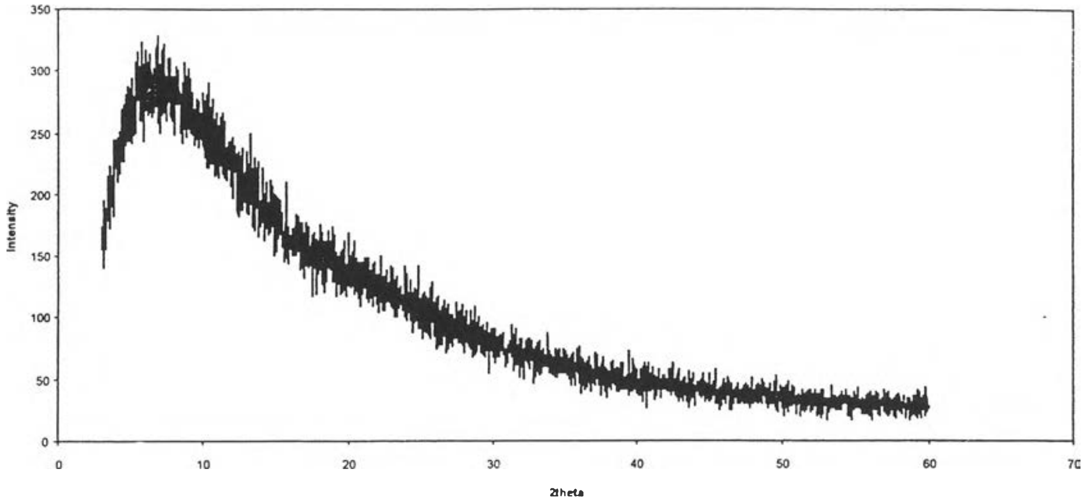


Figure 4.9 XRD pattern of precalcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV.

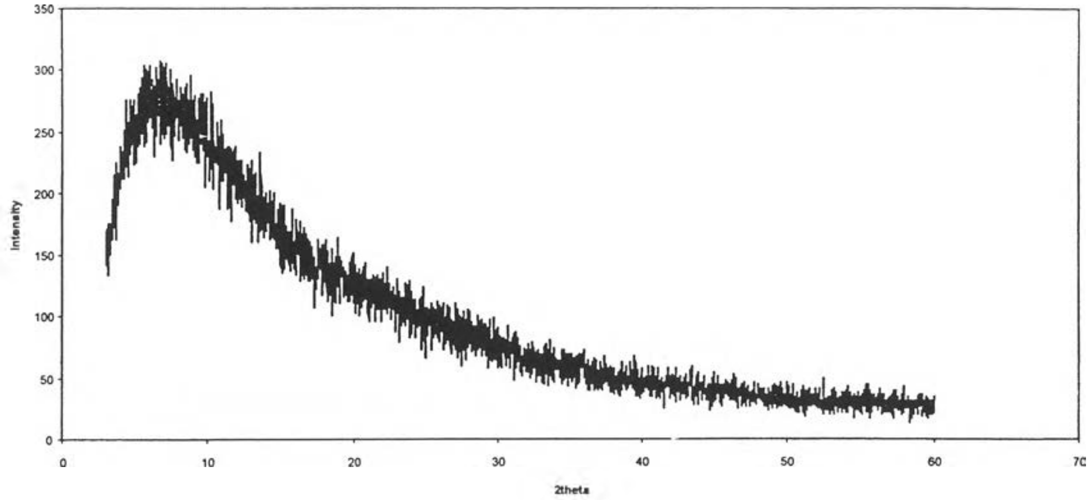


Figure 4.10 XRD pattern of precalcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV.

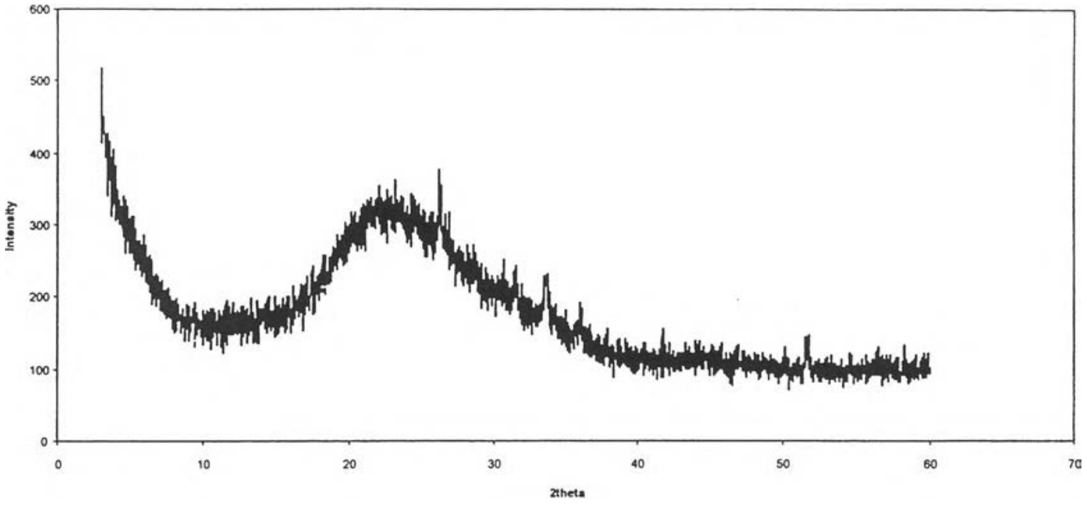


Figure 4.11 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 500°C.

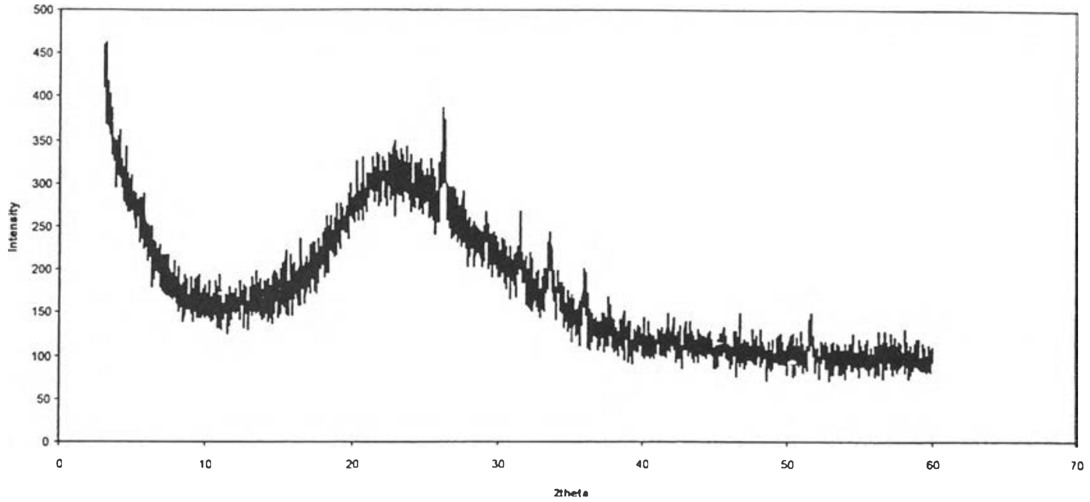


Figure 4.12 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 500°C.

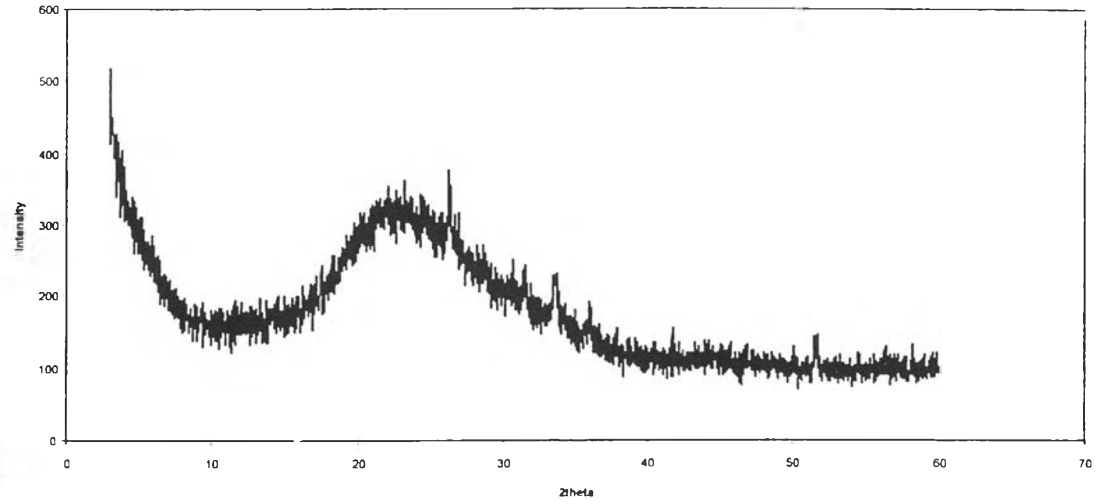


Figure 4.13 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 500°C.

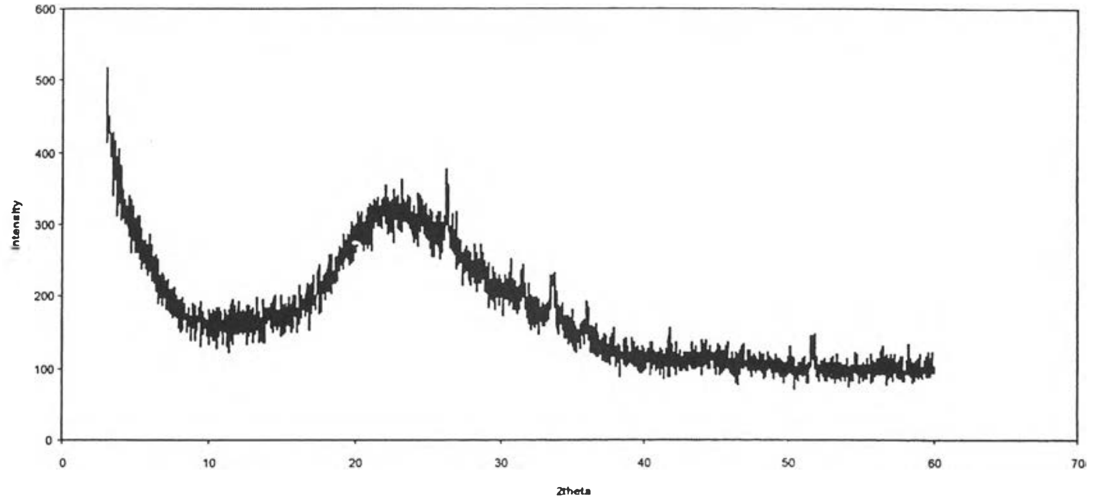


Figure 4.14 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 500°C.

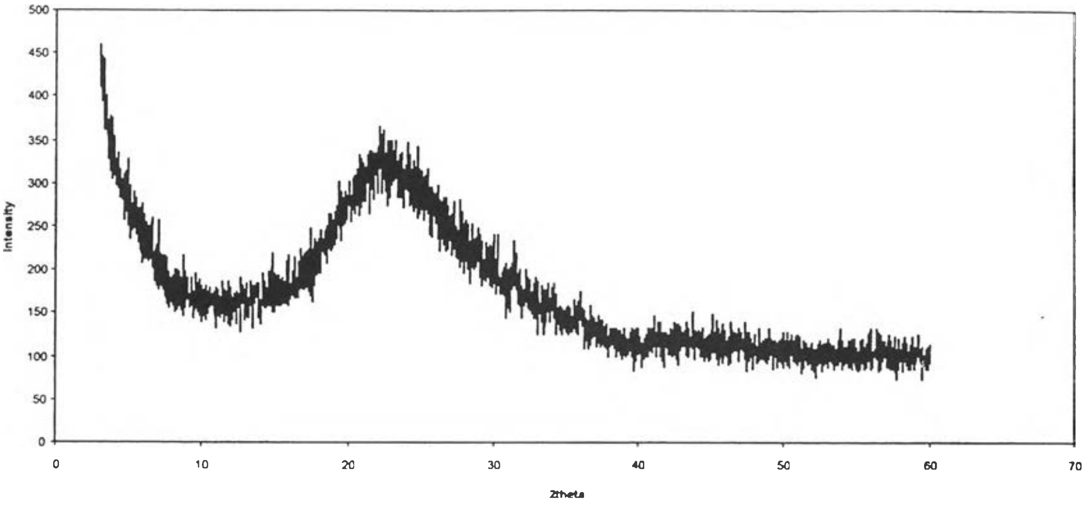


Figure 4.15 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 600°C.

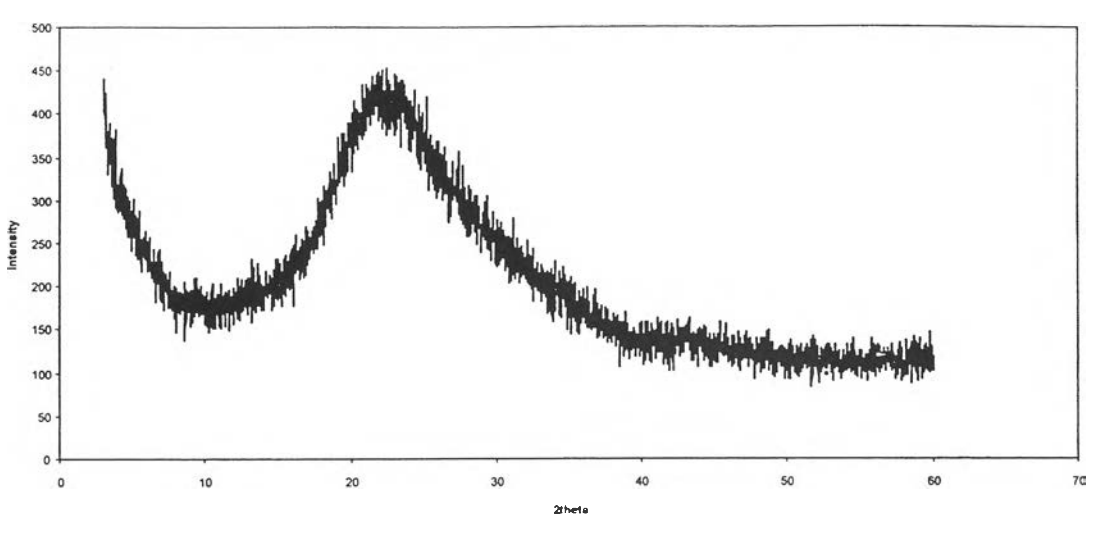


Figure 4.16 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 600°C.

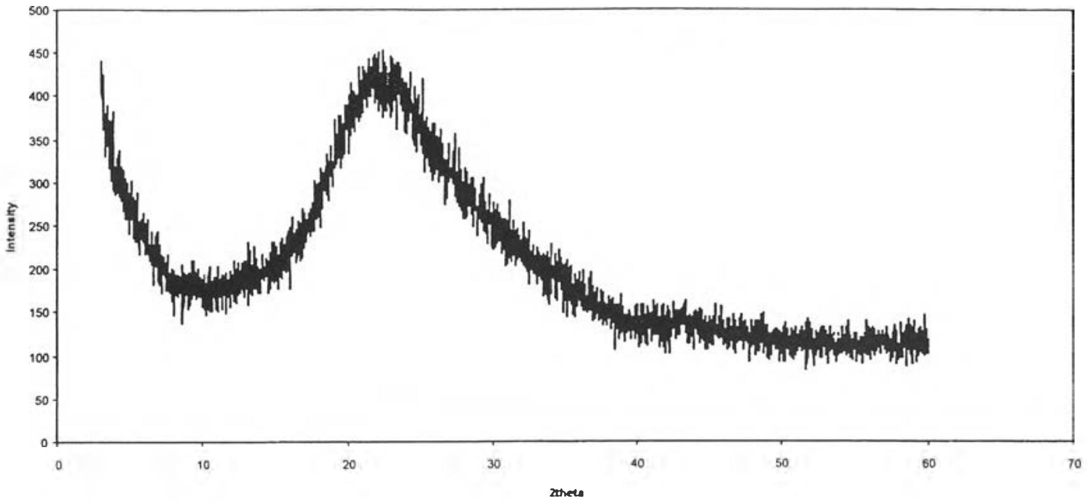


Figure 4.17 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 600°C.

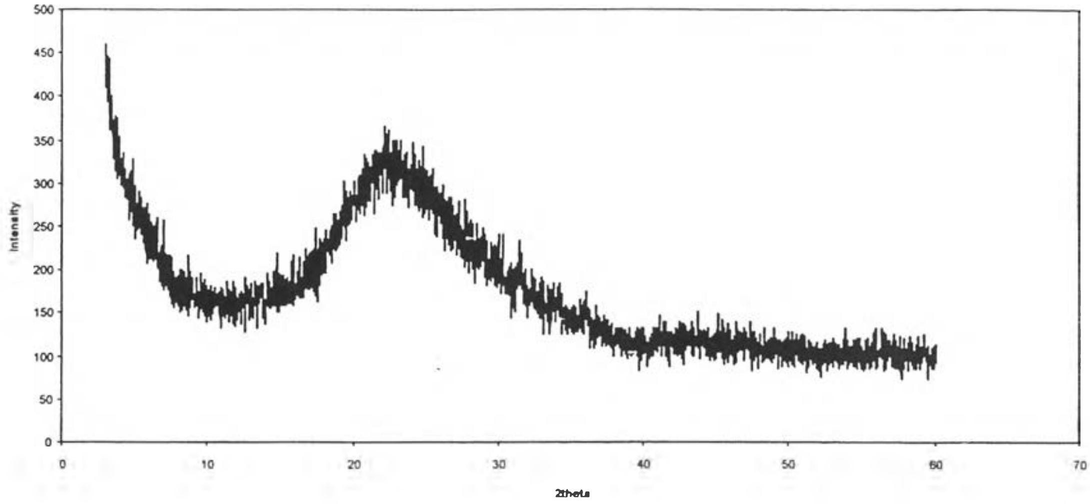


Figure 4.18 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 600°C.

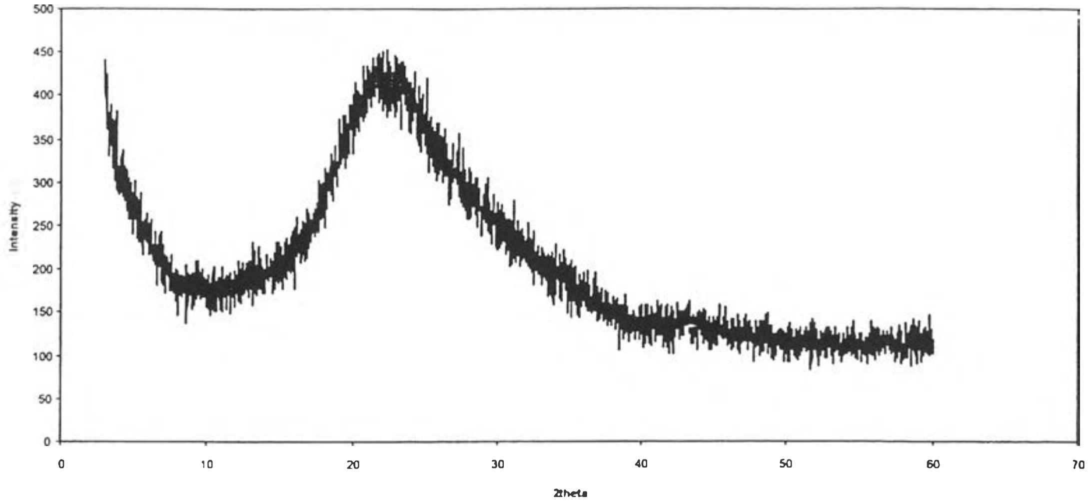


Figure 4.19 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 700°C.

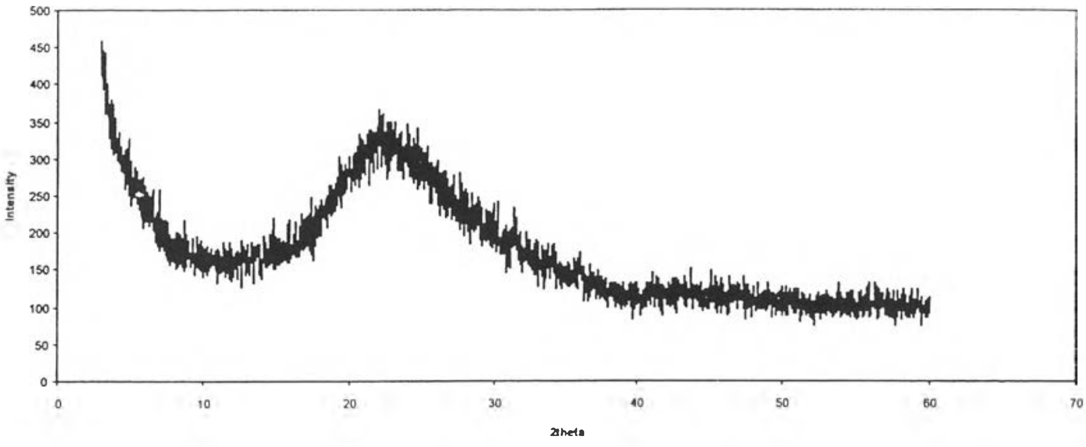


Figure 4.20 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 700°C.

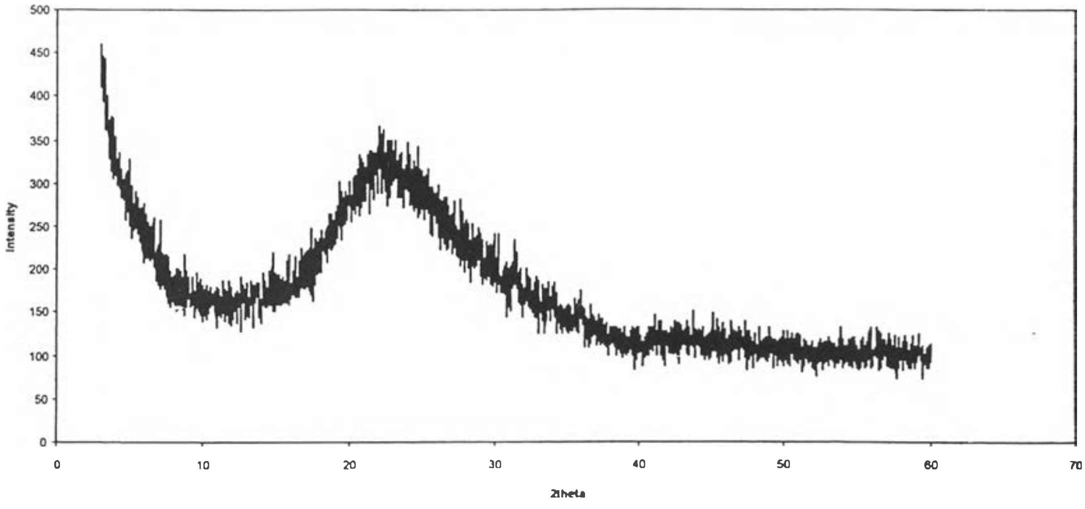


Figure 4.21 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 700°C.

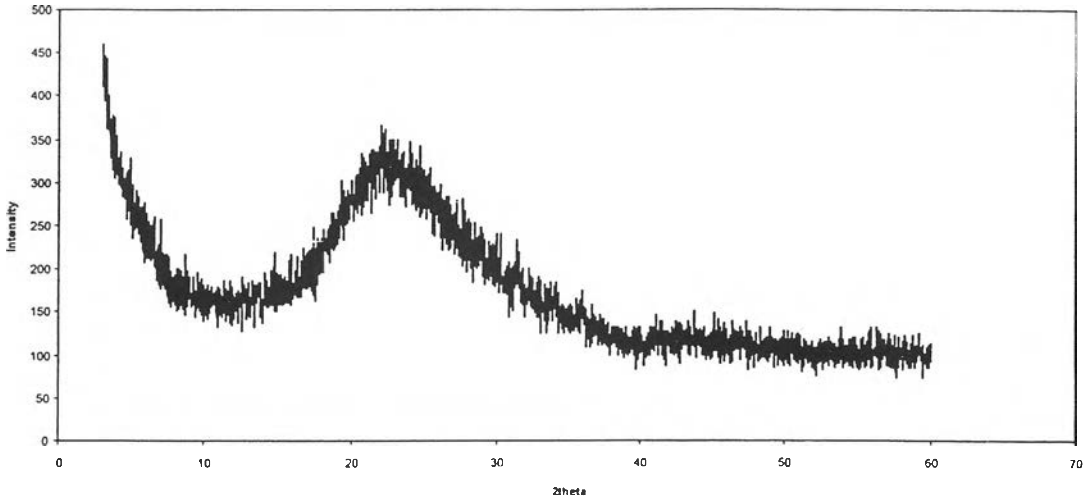


Figure 4.22 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 700°C.

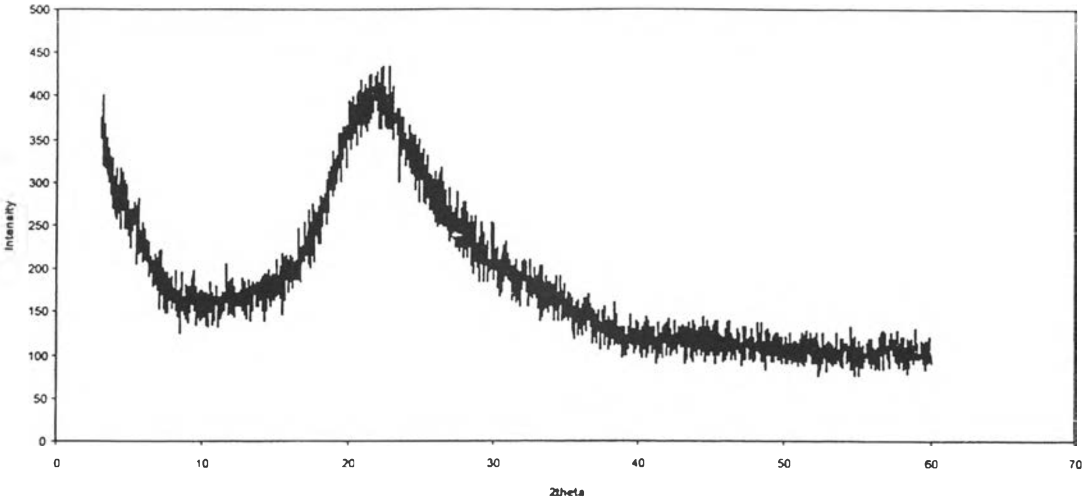


Figure 4.23 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 800°C.

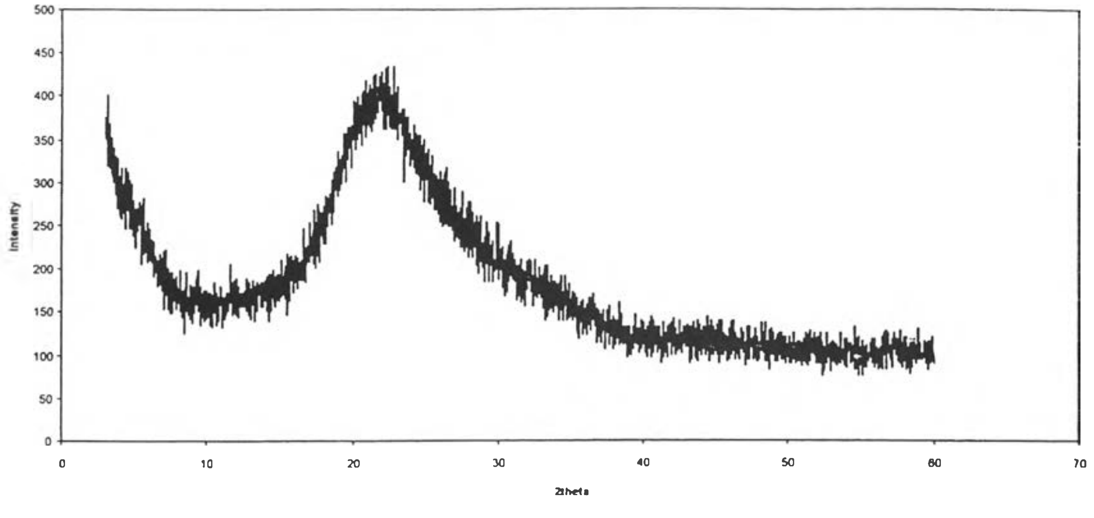


Figure 4.24 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 800°C.

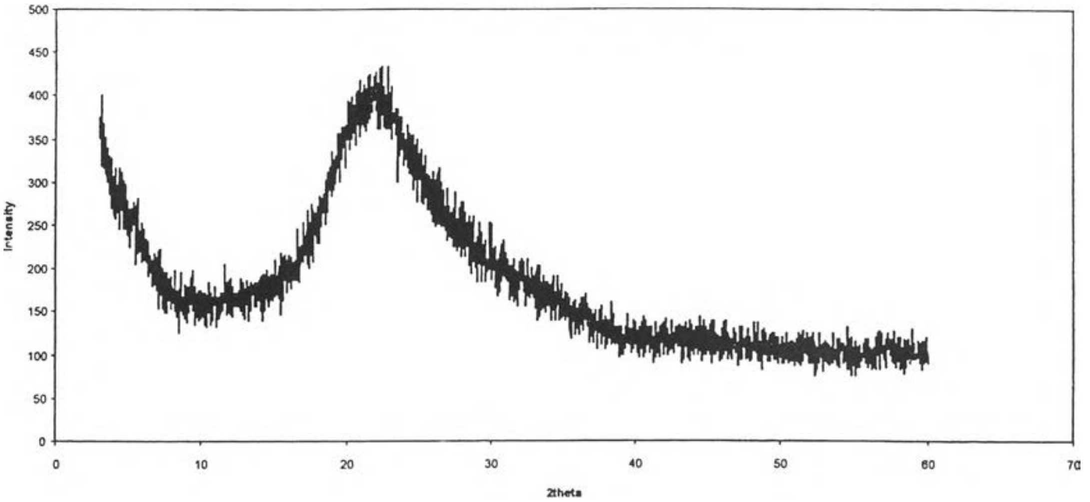


Figure 4.25 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 800°C.

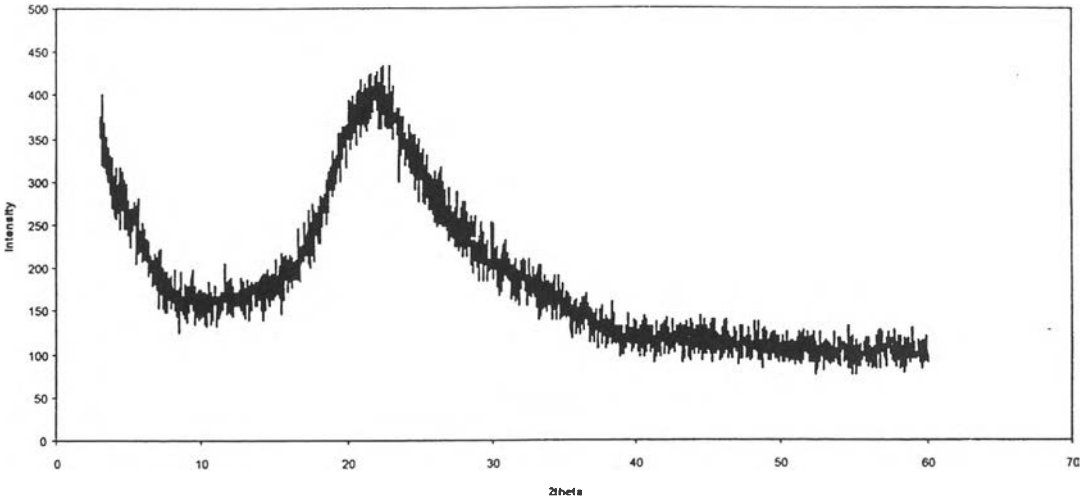


Figure 4.26 XRD pattern of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 800°C.

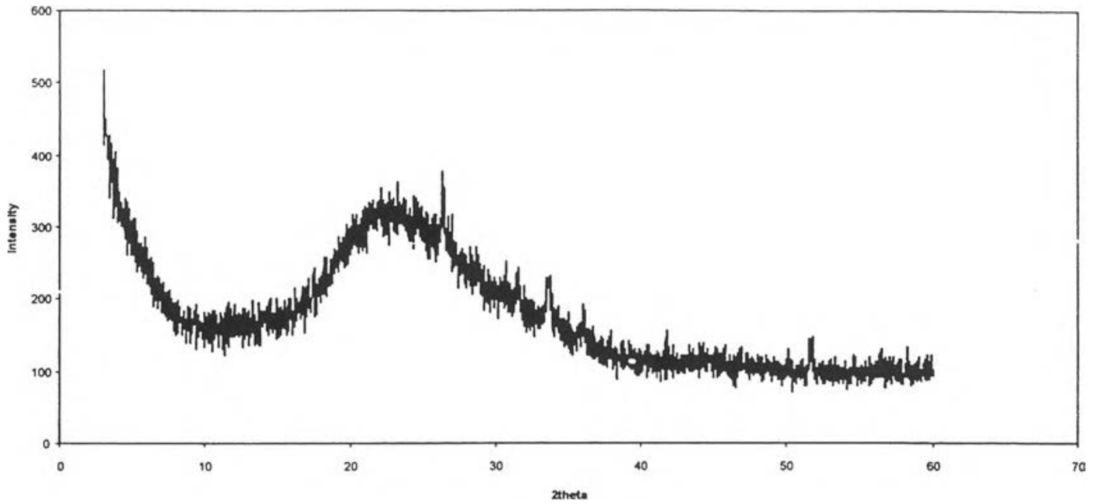


Figure 4.27 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 500°C.

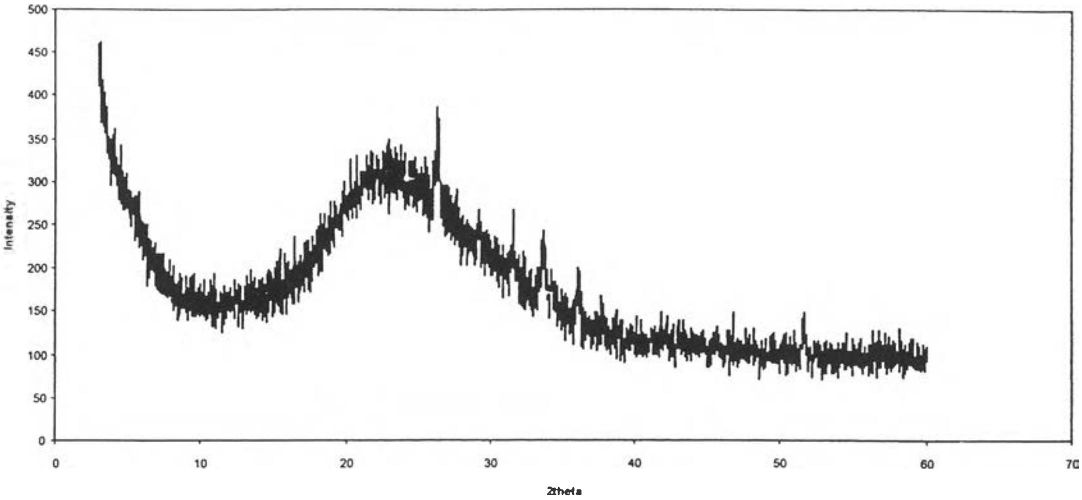


Figure 4.28 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 500°C.

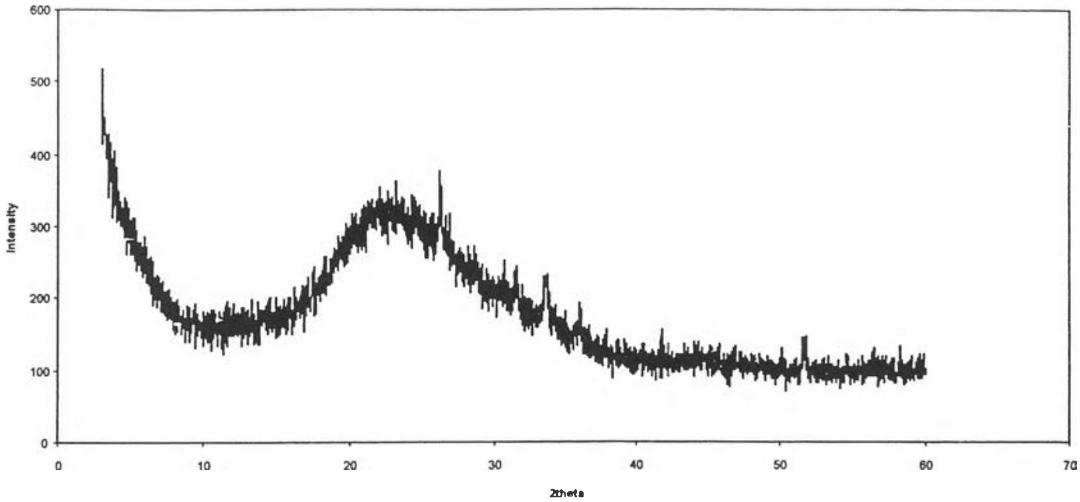


Figure 4.29 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 500°C.

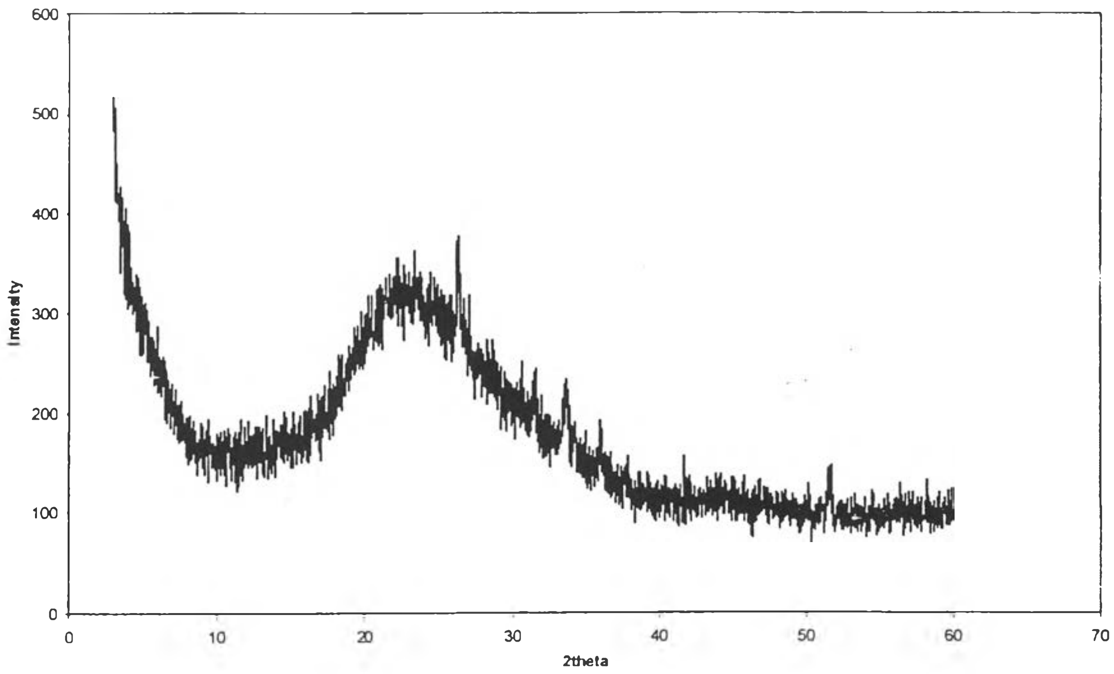


Figure 4.30 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 500°C.

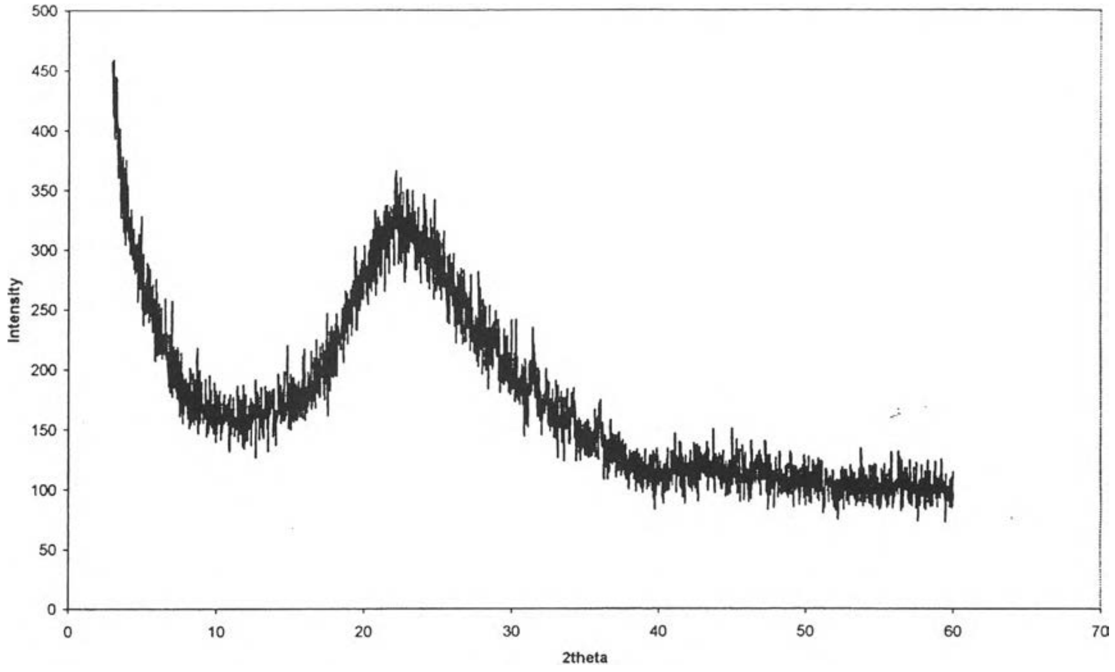


Figure 4.31 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 600°C.

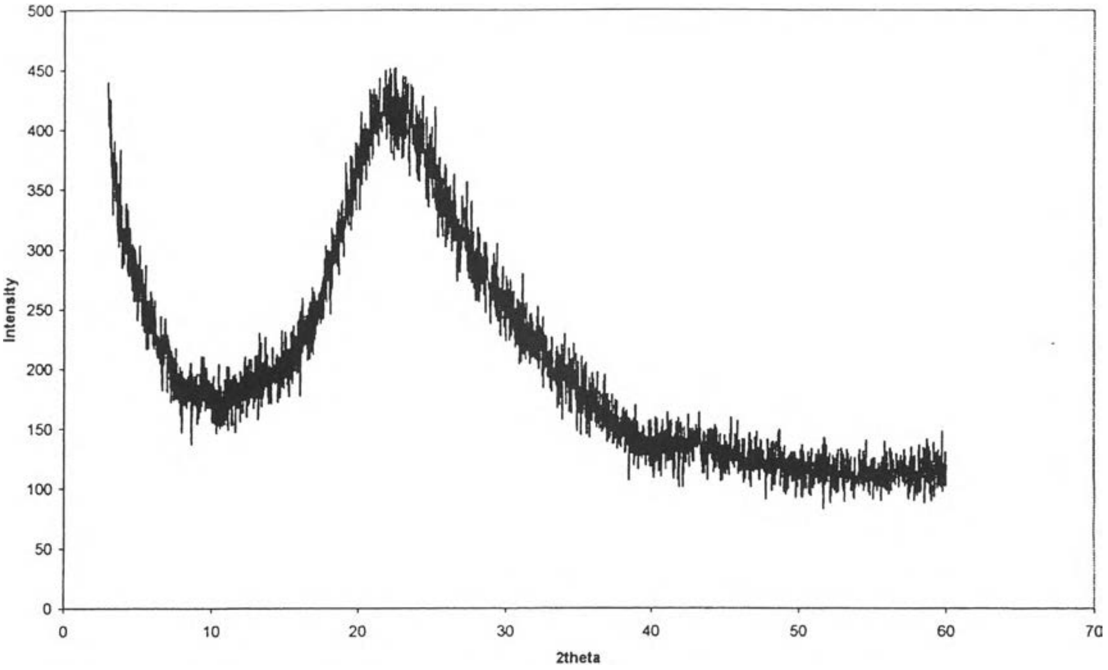


Figure 4.32 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 600°C.

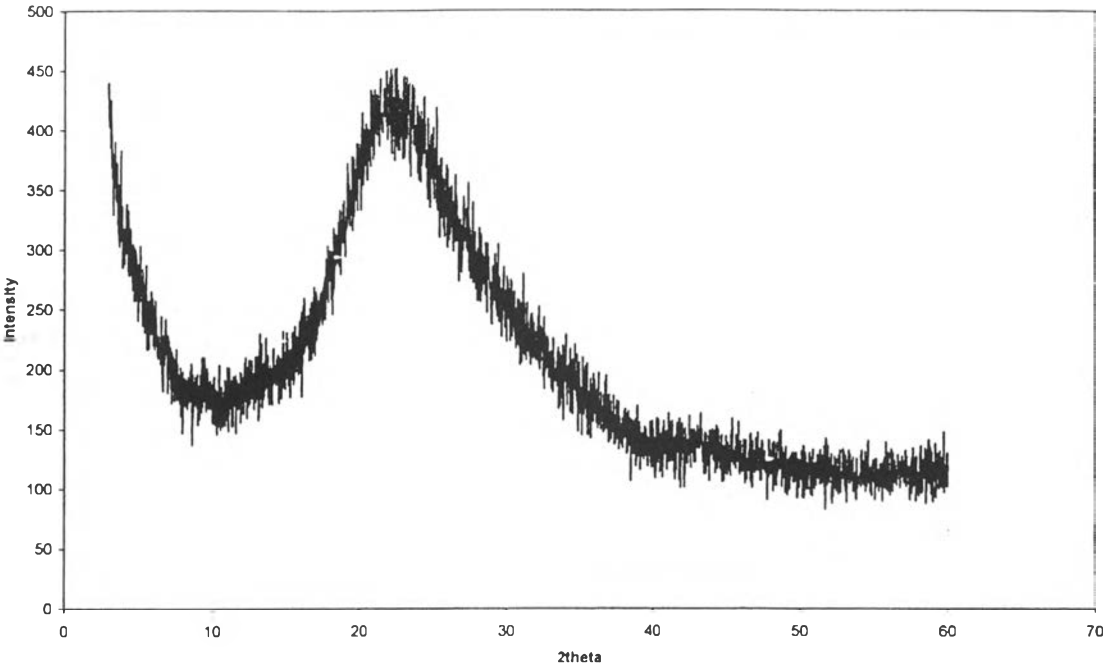


Figure 4.33 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 600°C.

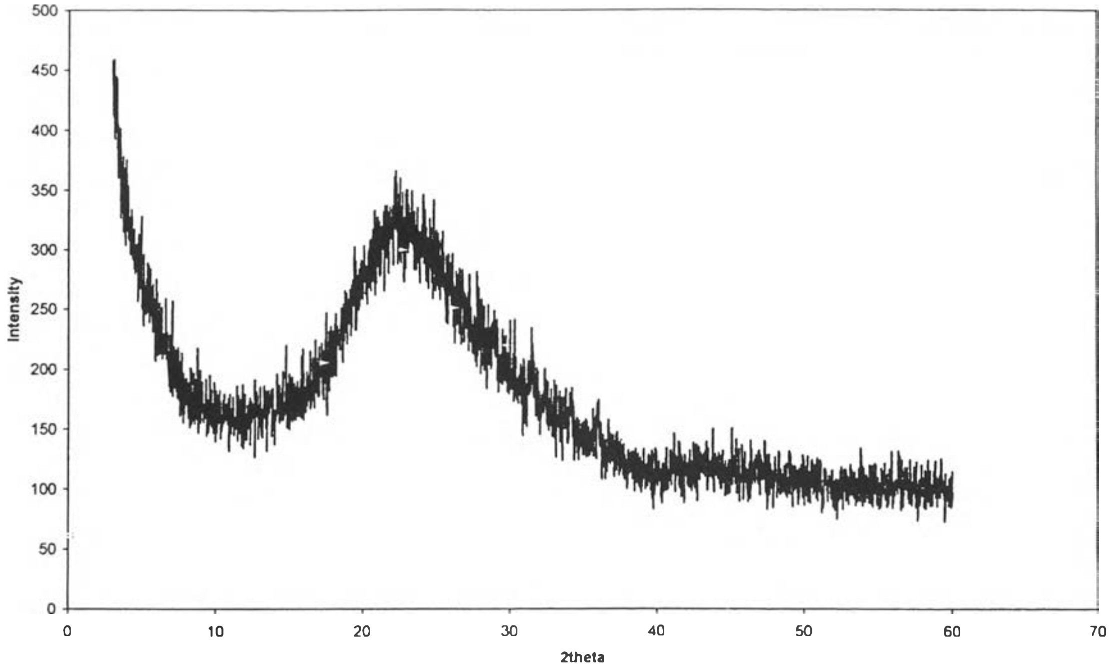


Figure 4.34 XRD pattern of calcined fibres obtained from 11.76 wt% of silatranc in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 600°C.

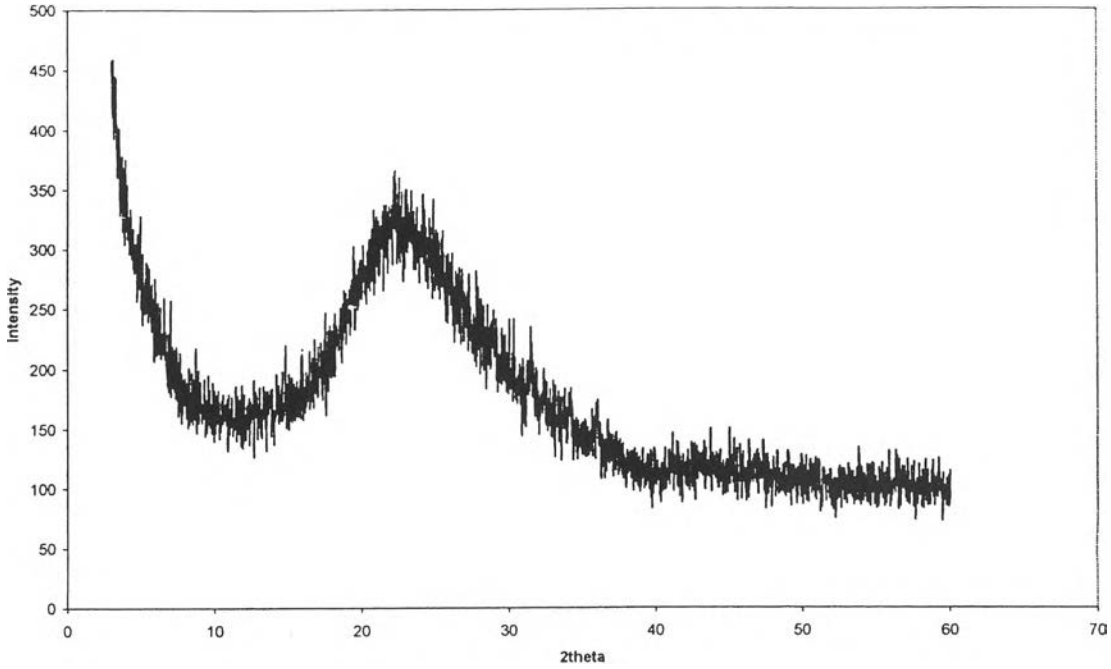


Figure 4.35 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 700°C.

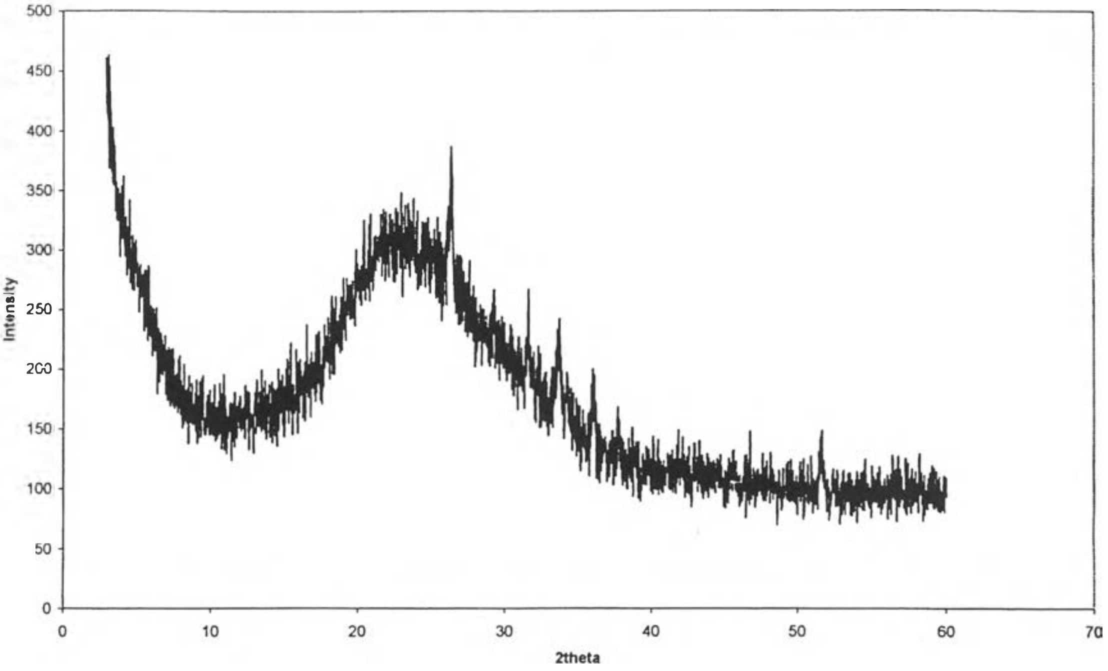


Figure 4.36 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 700°C.

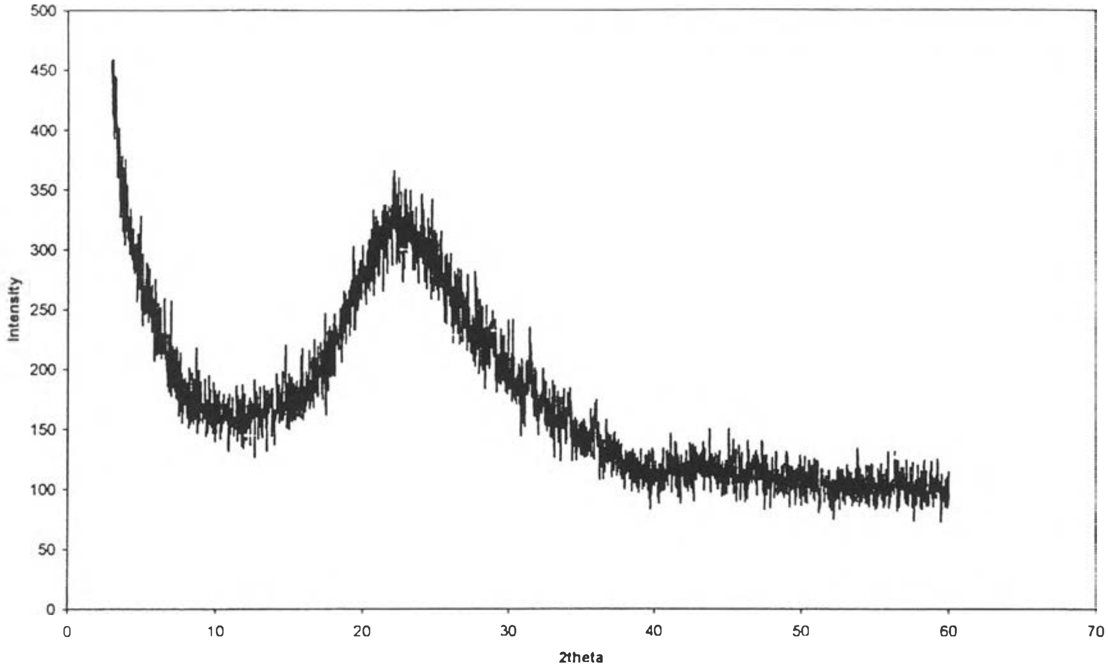


Figure 4.37 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 700°C.

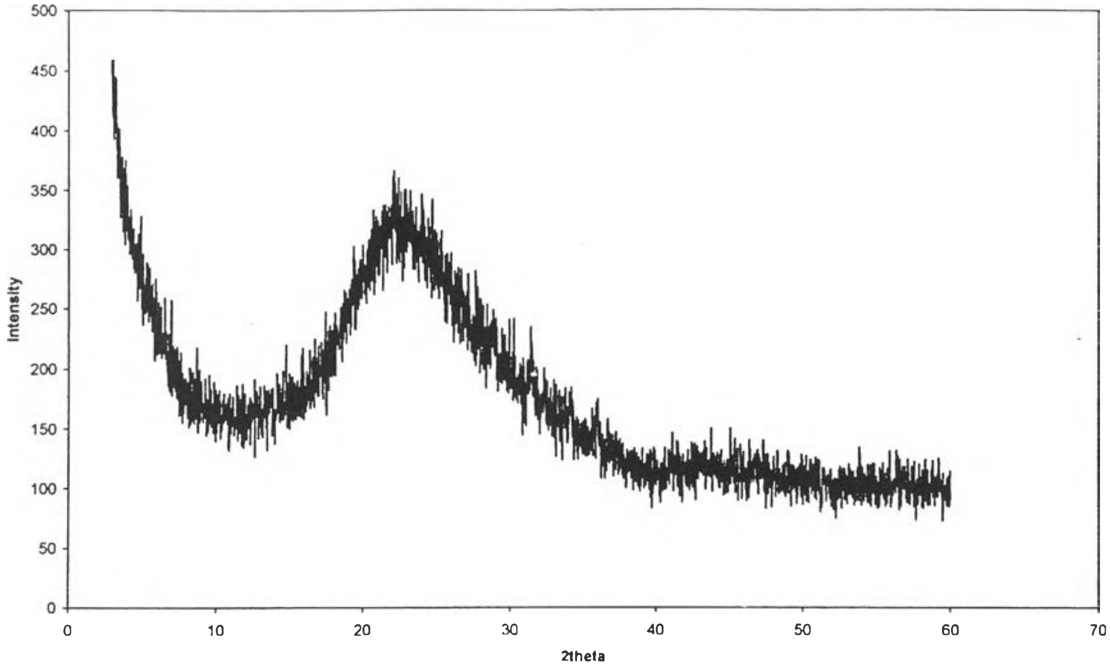


Figure 4.38 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 700°C.

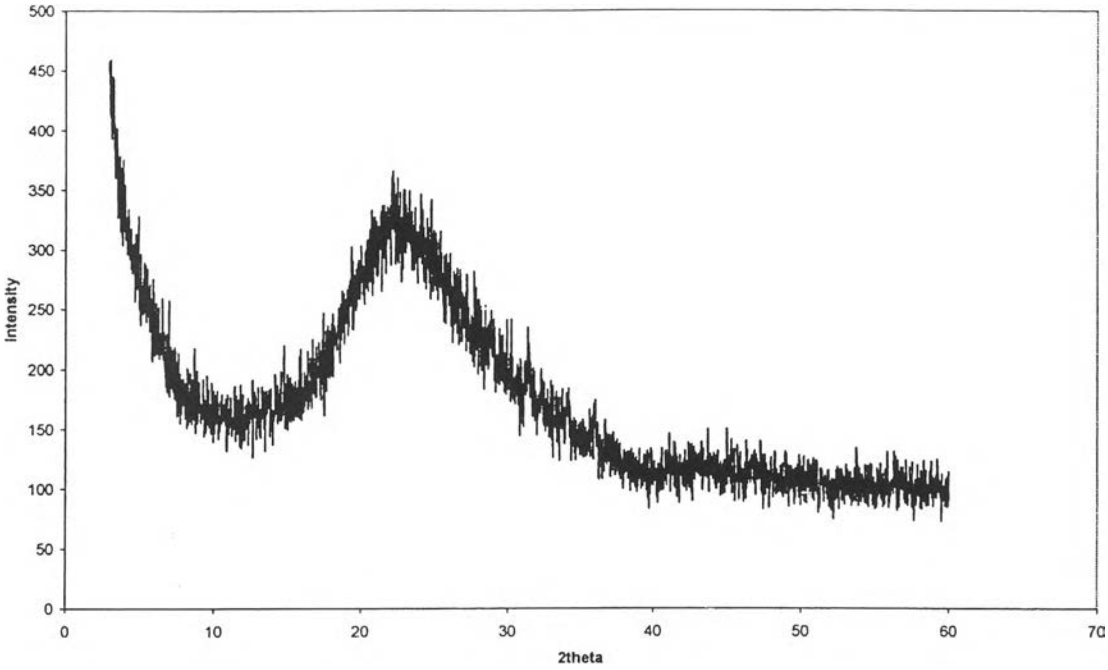


Figure 4.39 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 800°C.

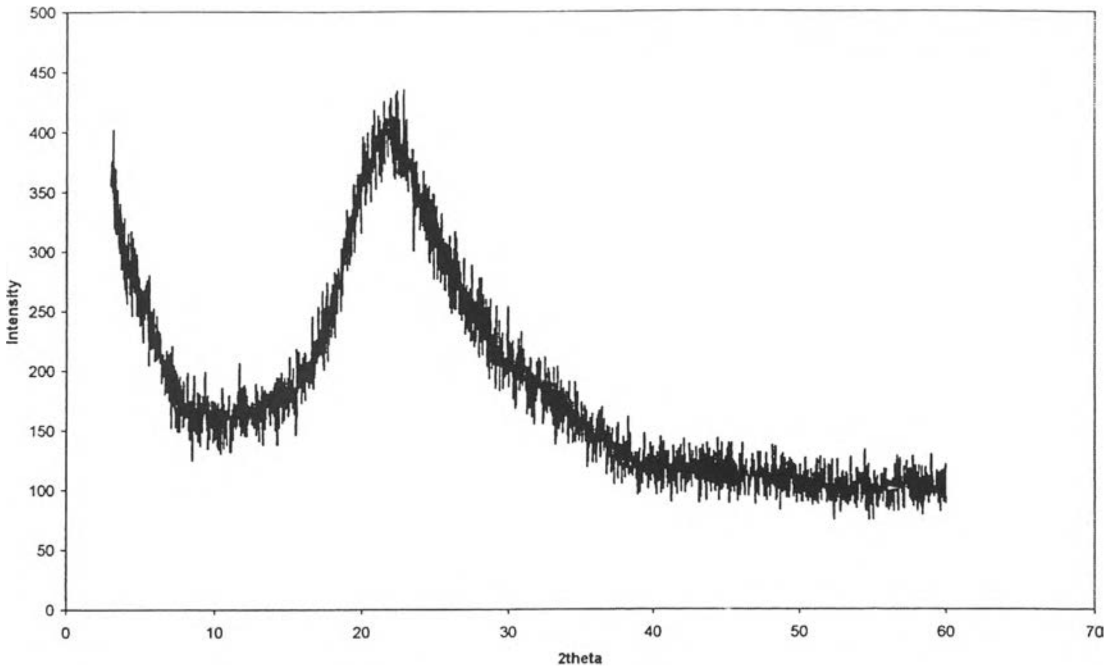


Figure 4.40 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 800°C.

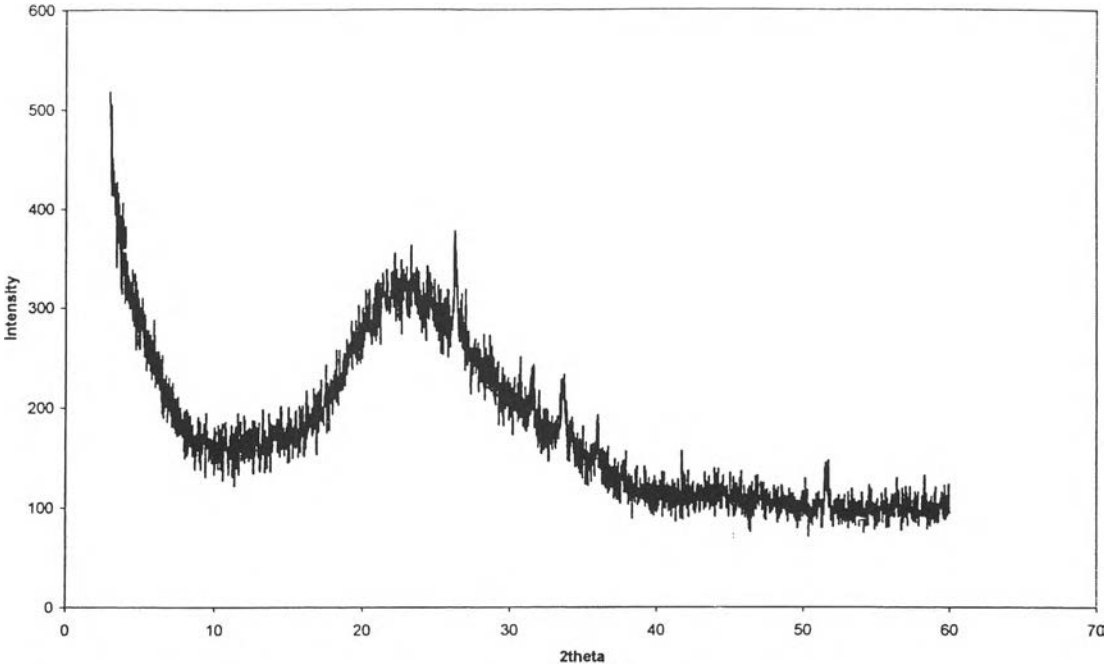


Figure 4.41 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 800°C.

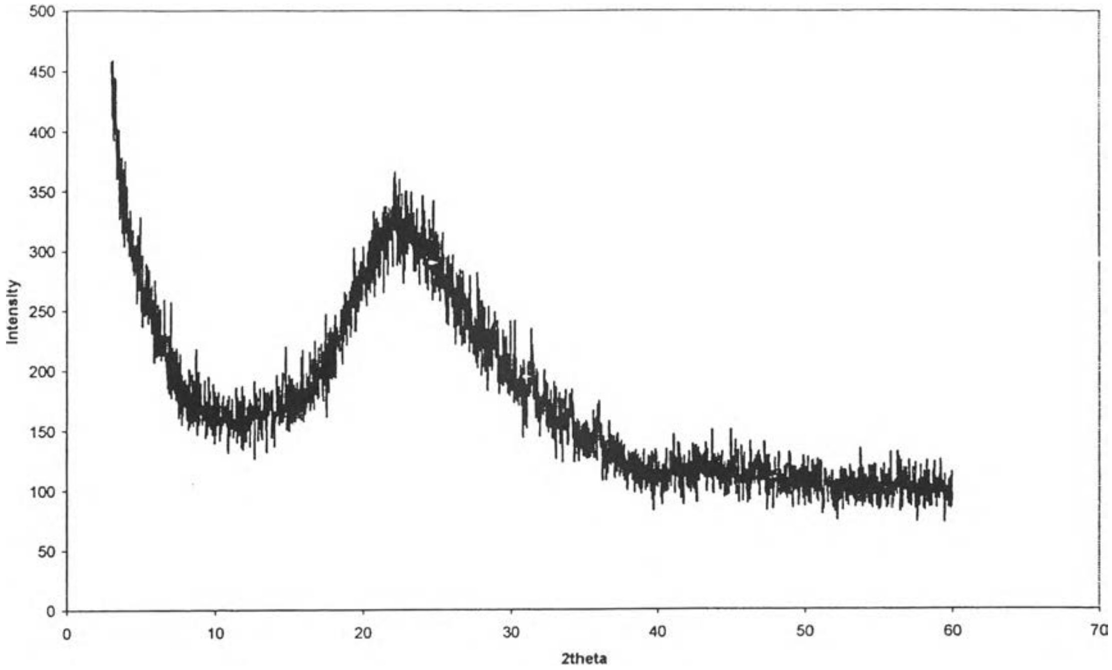


Figure 4.42 XRD pattern of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 800°C.

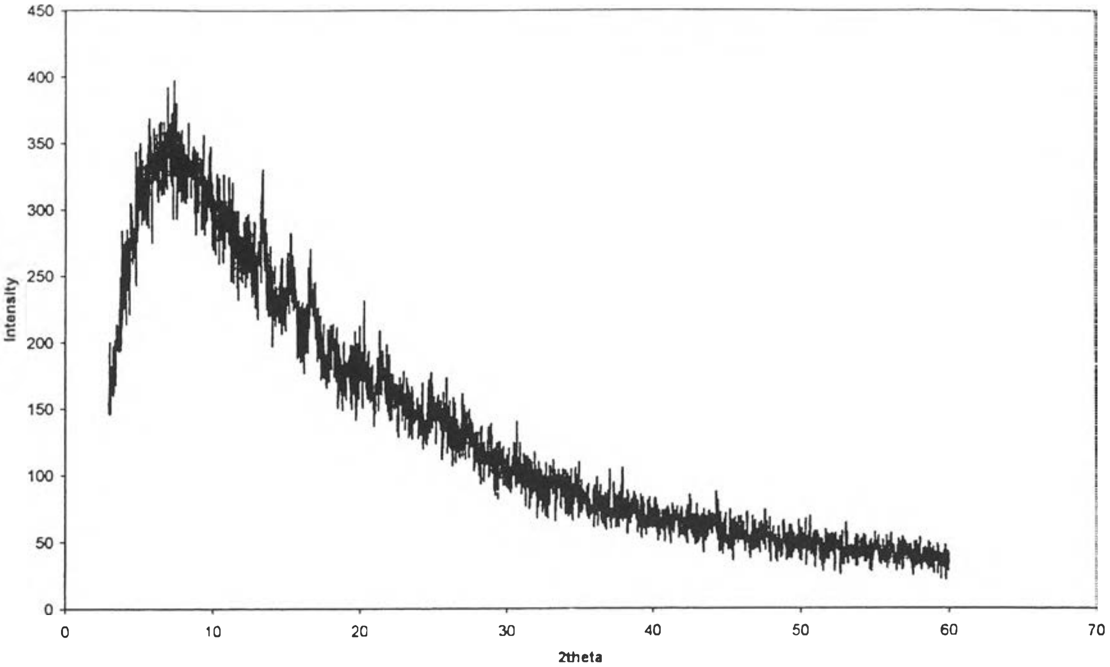


Figure 4.43 XRD pattern of silatrane complexes.

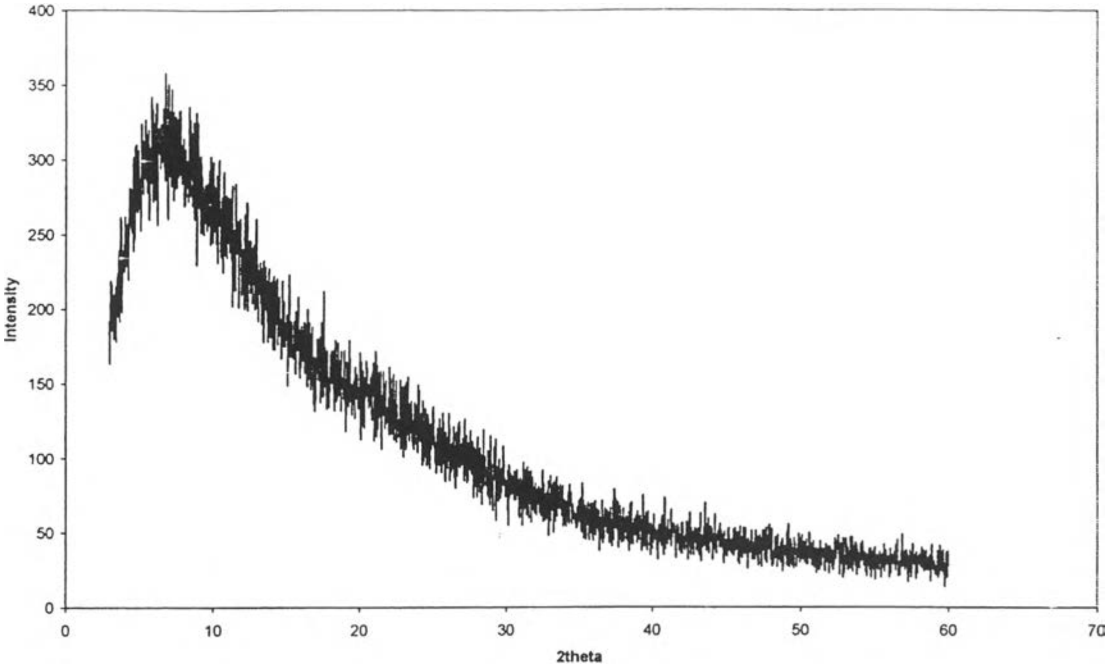


Figure 4.44 XRD pattern of pure silica.

Crystalline structure of the silica fibres is investigated by means of X-ray diffraction (XRD). The XRD pattern of the obtained silica fibres generated from every experimental condition is shown in figures 4.11–4.42. This XRD pattern exhibits the characteristics of amorphous silica agreeing with the results from the previous work (Choi *et al*, 2003). However, it should be noted that the width of XRD peak of calcined fibres are narrower than that of precalcined fibres and pure silica, and the position of XRD peak of calcined fibres shift to higher 2theta compared to that of pure silica. They can be implied that although the phase of the calcined silica fibres is amorphous, it gives silica which holds more order when compared to reactant silica partuicles. Moreover, the XRD pattern of the precalcined fibres derived from every experimental condition shown in figure 4.3–figure 4.10 exhibits the characteristics of amorphous materials, and the pattern of the precalcined one and the calcined one is different significantly. It indicates the phase changing to more order state.

4.2.2.2 *Fourier transform infrared spectrophotometre (FTIR)*

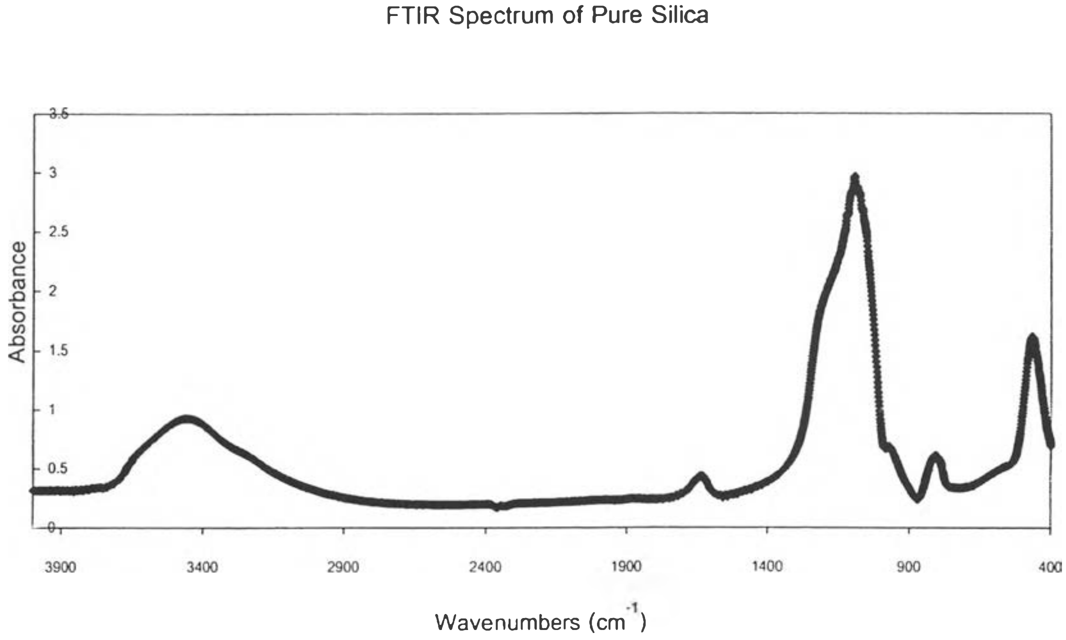


Figure 4.45 FTIR spectrum of pure silica.

Figure 4.4 shows the FTIR spectrum of pure silica. The peaks at 466, 806, and 1100 cm^{-1} are assigned to Si-O stretching and the broad band centering at 3458 cm^{-1} is assigned to O-H stretching. The broad band peak centering at 3458 cm^{1} results from intra- and intermolecular hydrogen bonds of the silanol groups. This indicate that a substantial amount of silanol groups exists on the surface of silica particles.

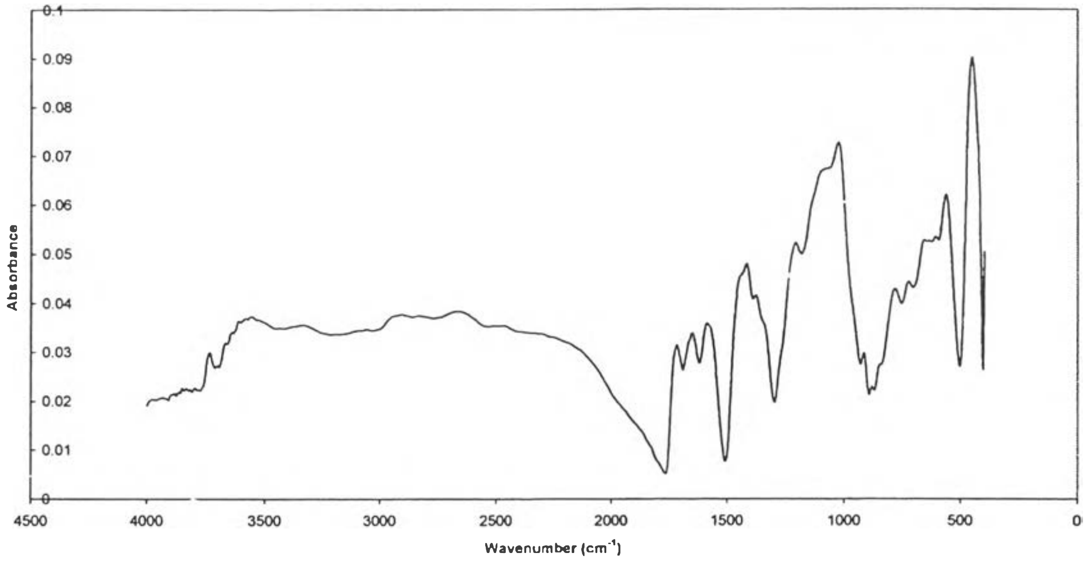


Figure 4.46 FTIR spectrum of precalcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV.

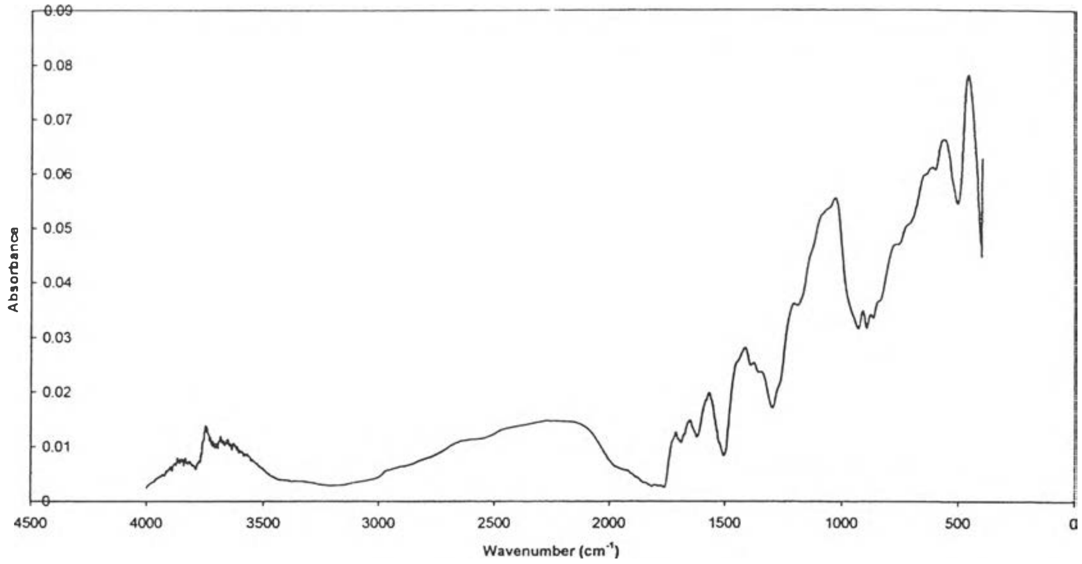


Figure 4.47 FTIR spectrum of precalcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV.

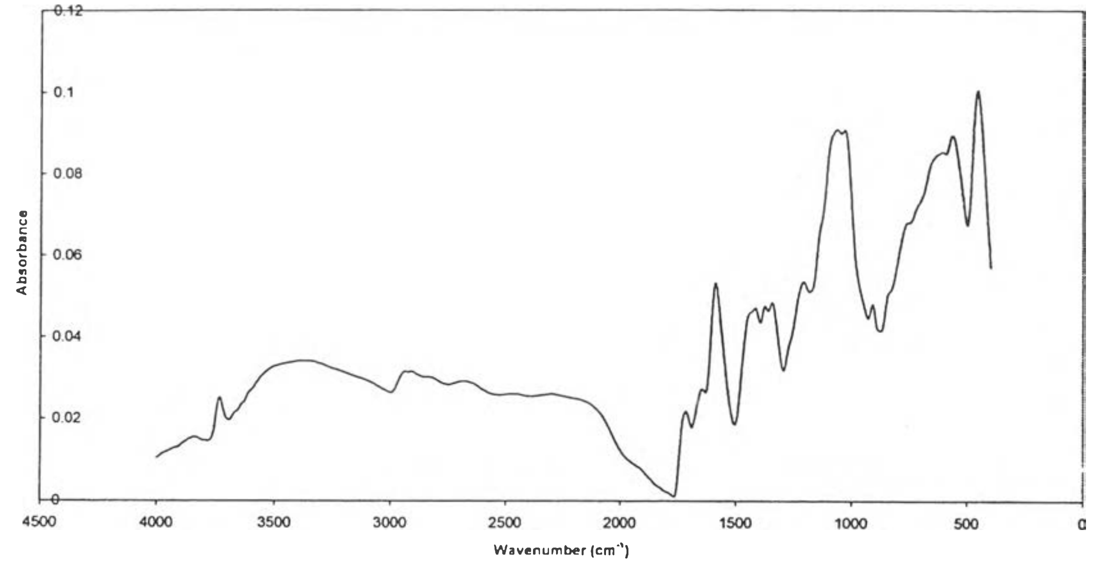


Figure 4.48 FTIR spectrum of precalcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV.

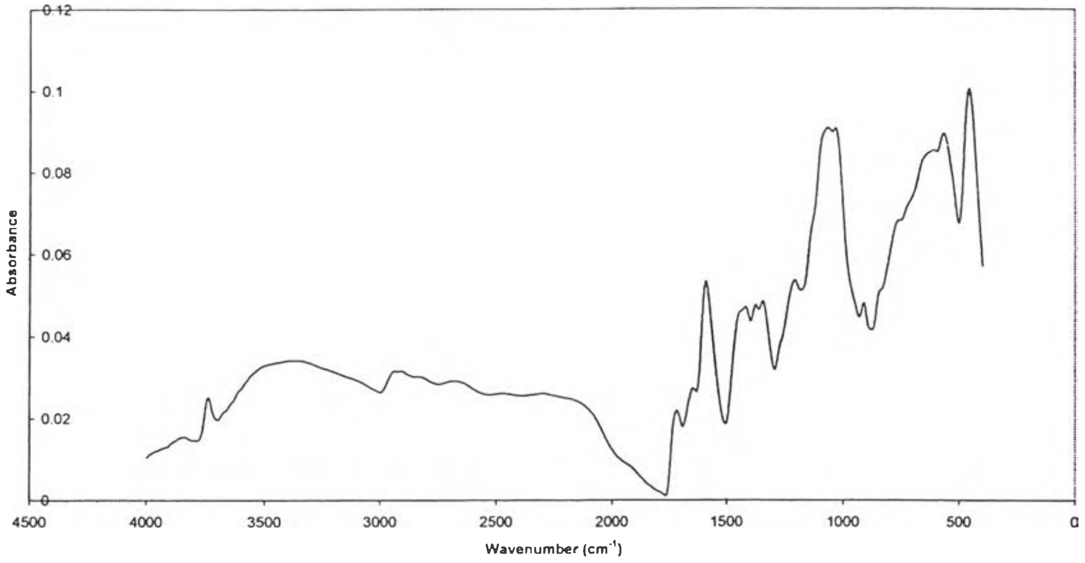


Figure 4.49 FTIR spectrum of precalcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV.

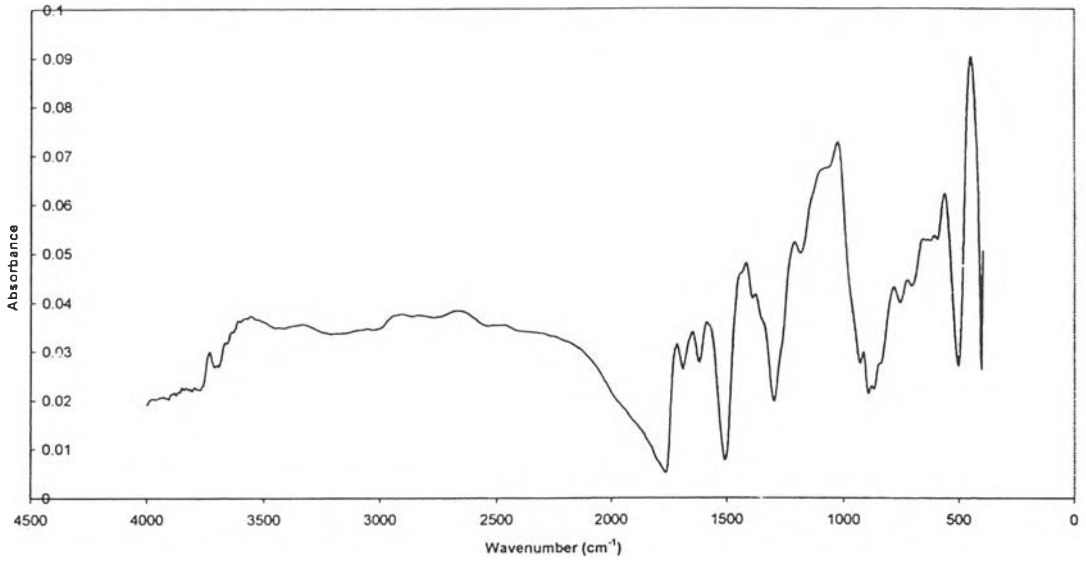


Figure 4.50 FTIR spectrum of precalcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV.

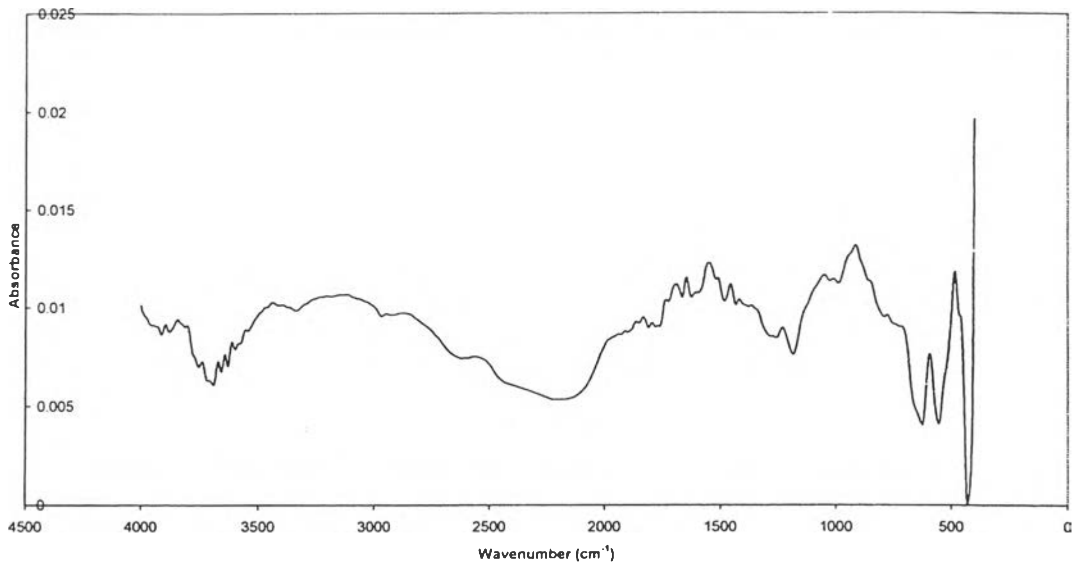


Figure 4.51 FTIR spectrum of precalcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV.

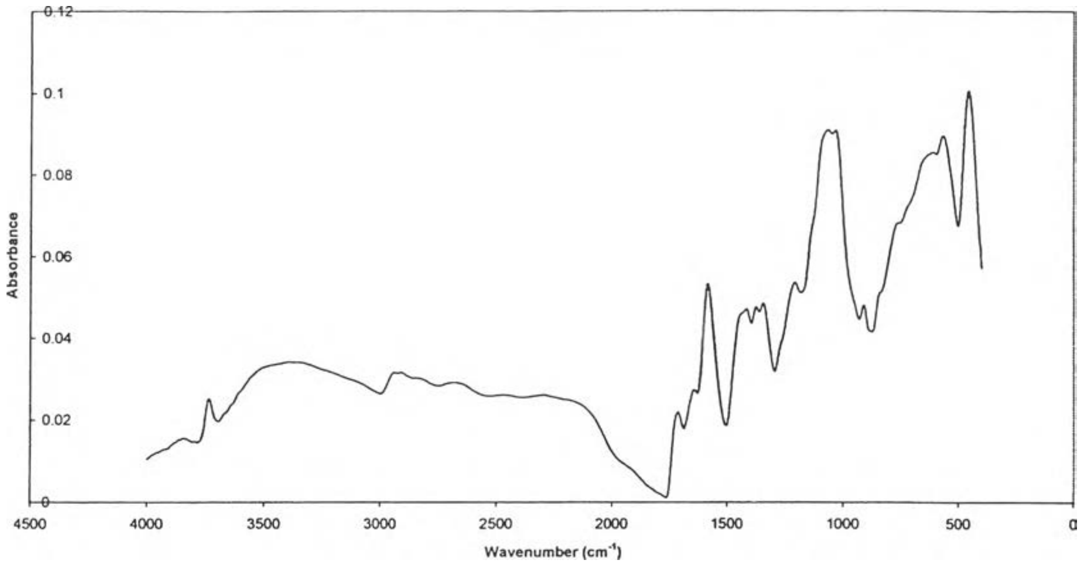


Figure 4.52 FTIR spectrum of precalcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV.

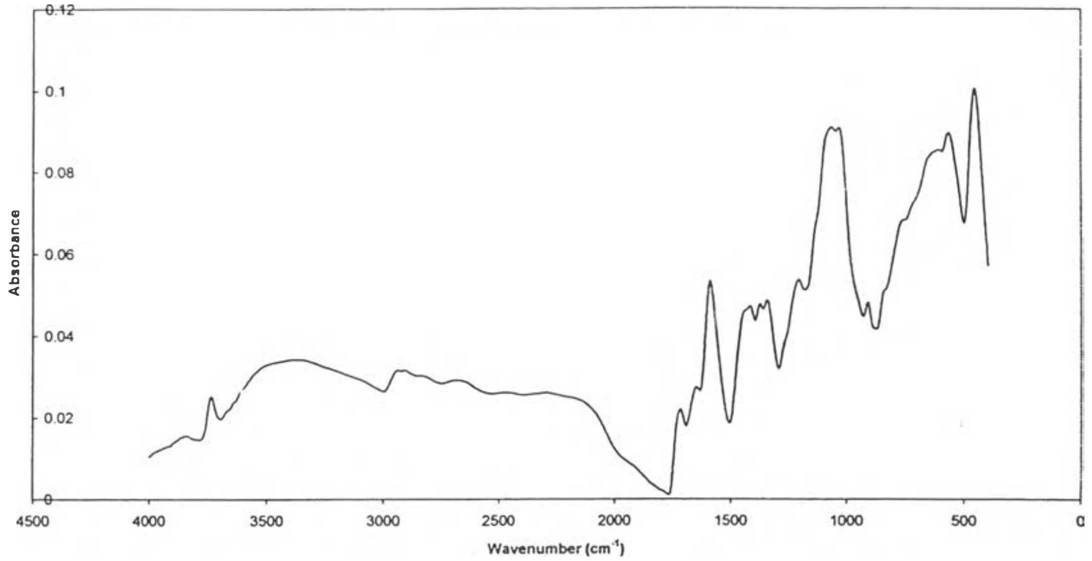


Figure 4.53 FTIR spectrum of precalcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV.

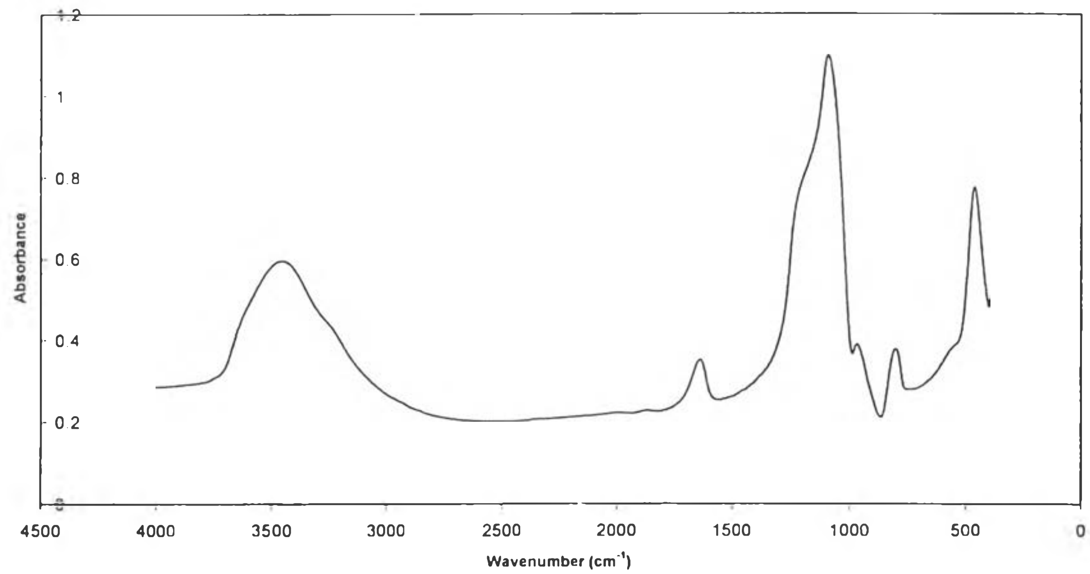


Figure 4.54 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 500°C.

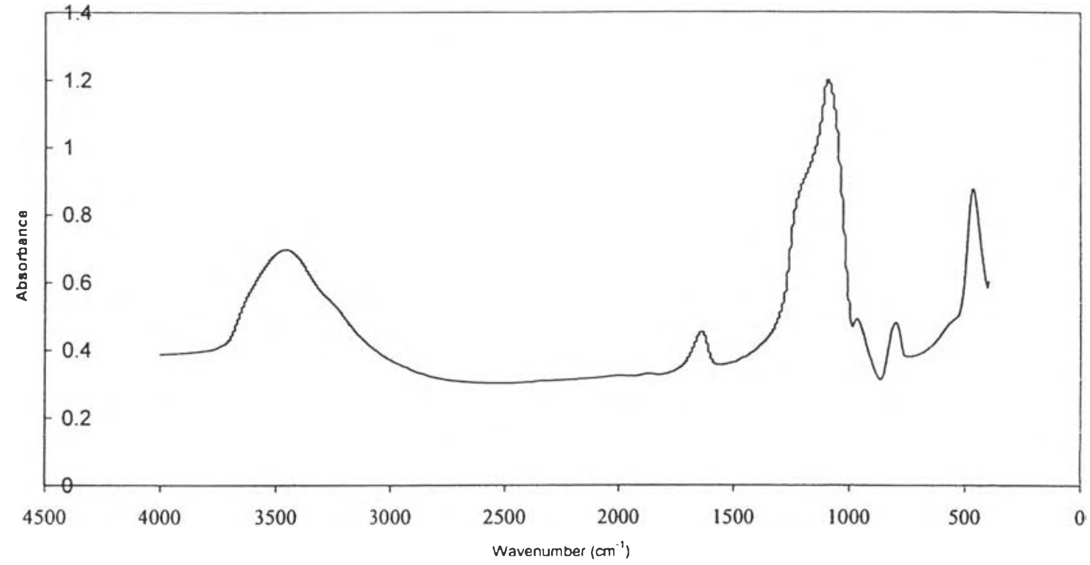


Figure 4.55 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 500°C.

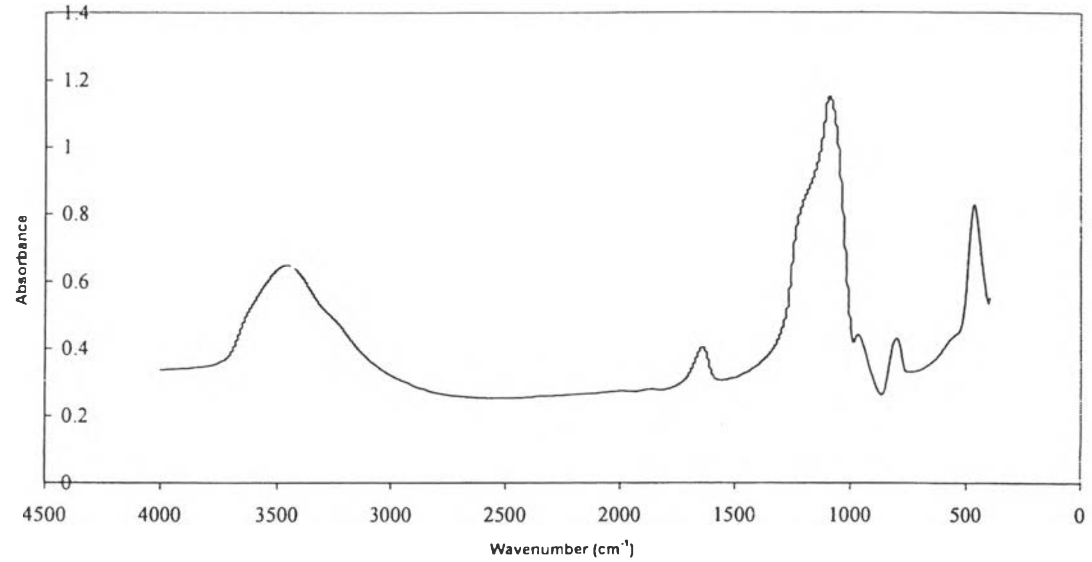


Figure 4.56 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 500°C.

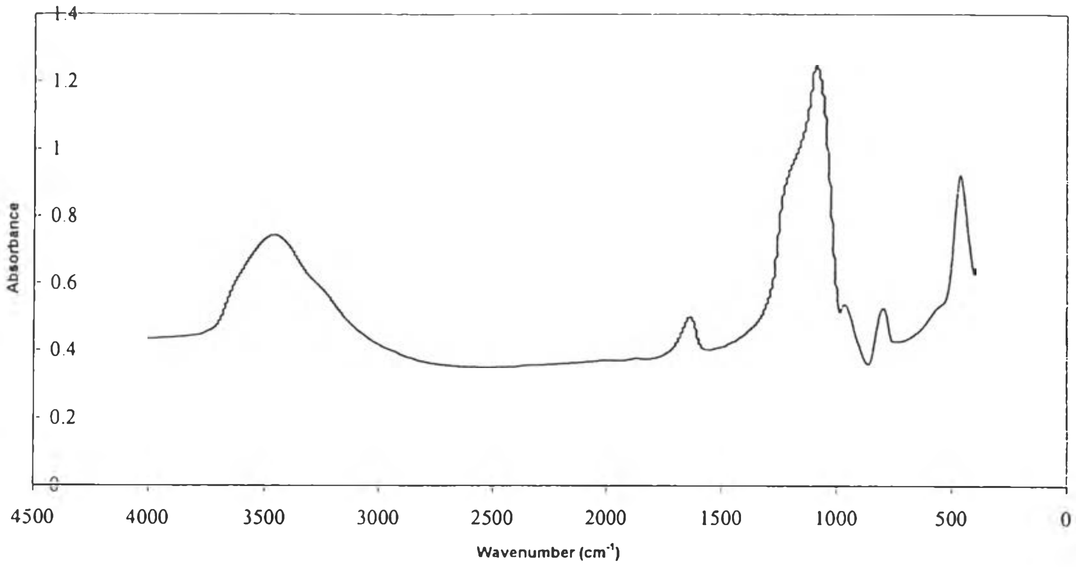


Figure 4.57 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 500°C.

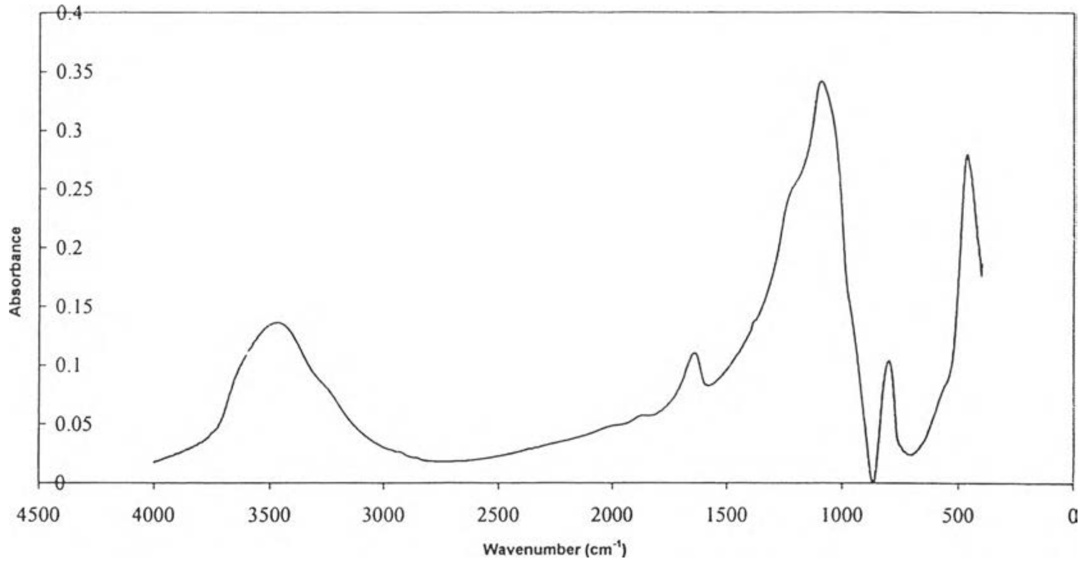


Figure 4.58 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 600°C.

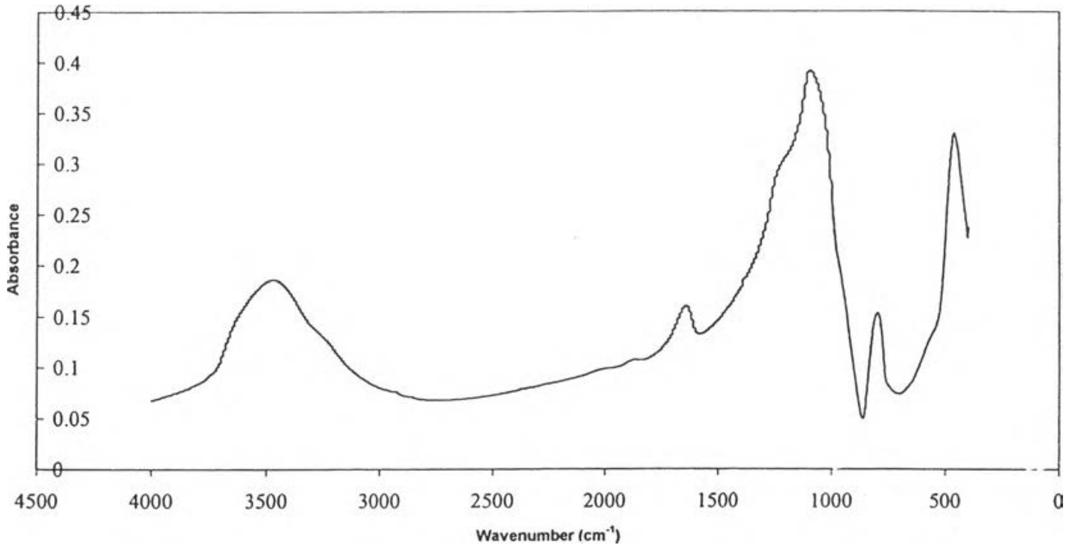


Figure 4.59 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 600°C.

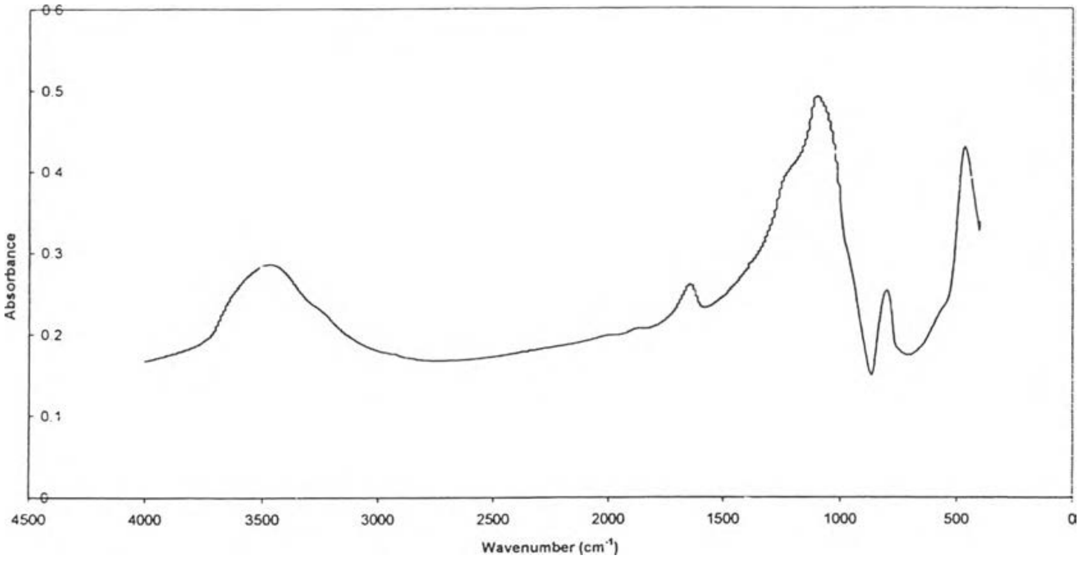


Figure 4.60 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 600°C.

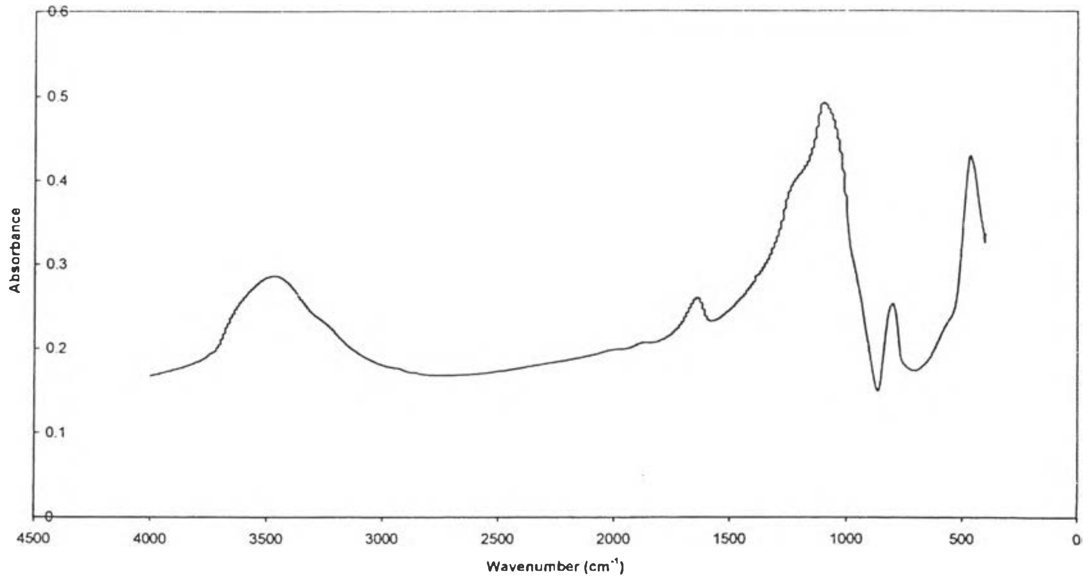


Figure 4.61 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 600°C.

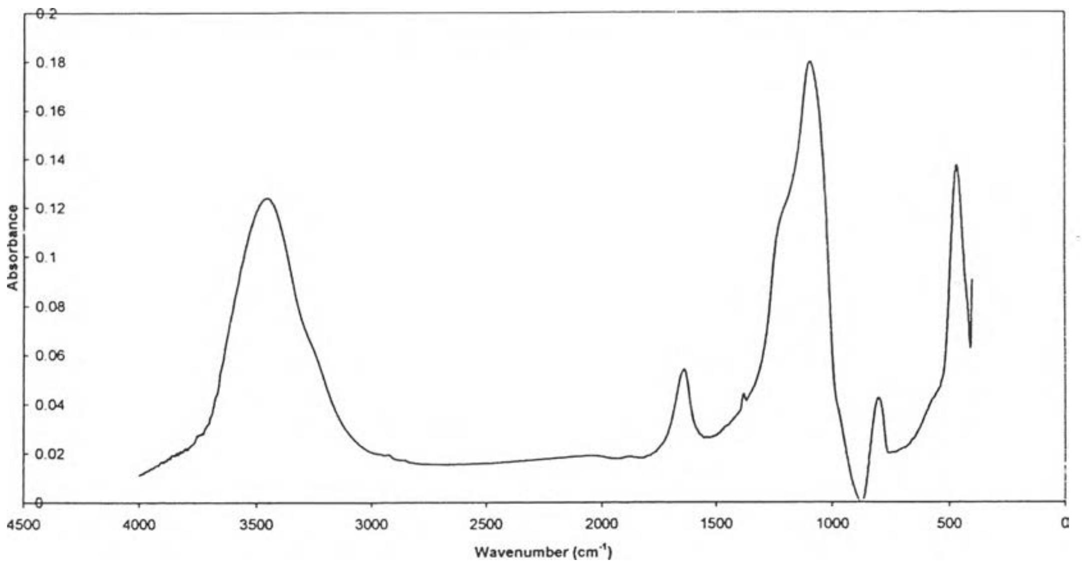


Figure 4.62 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 700°C.

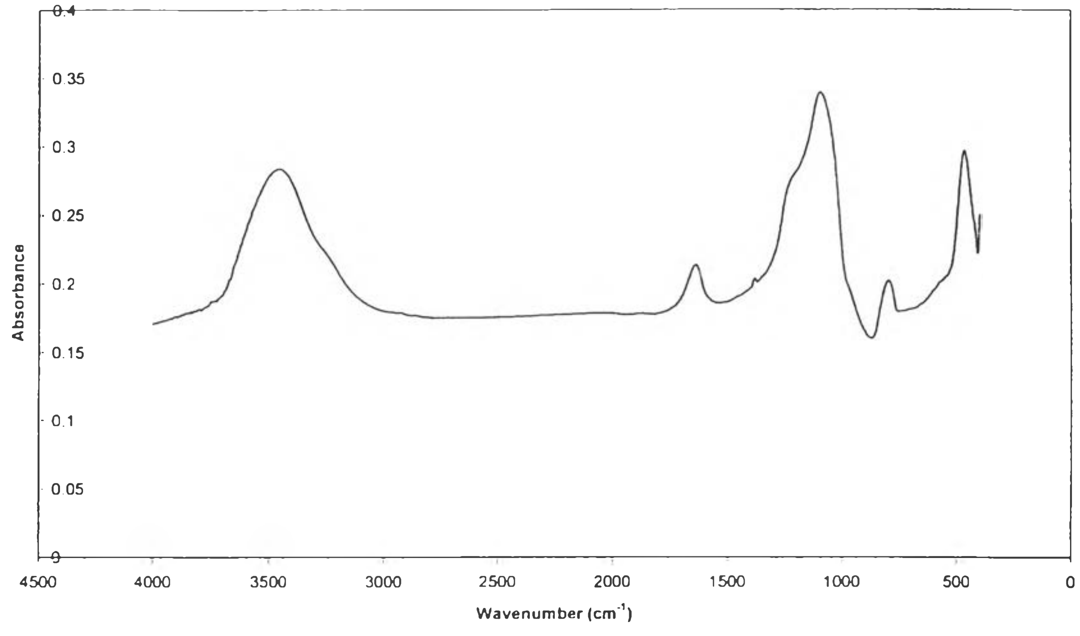


Figure 4.63 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 700°C.

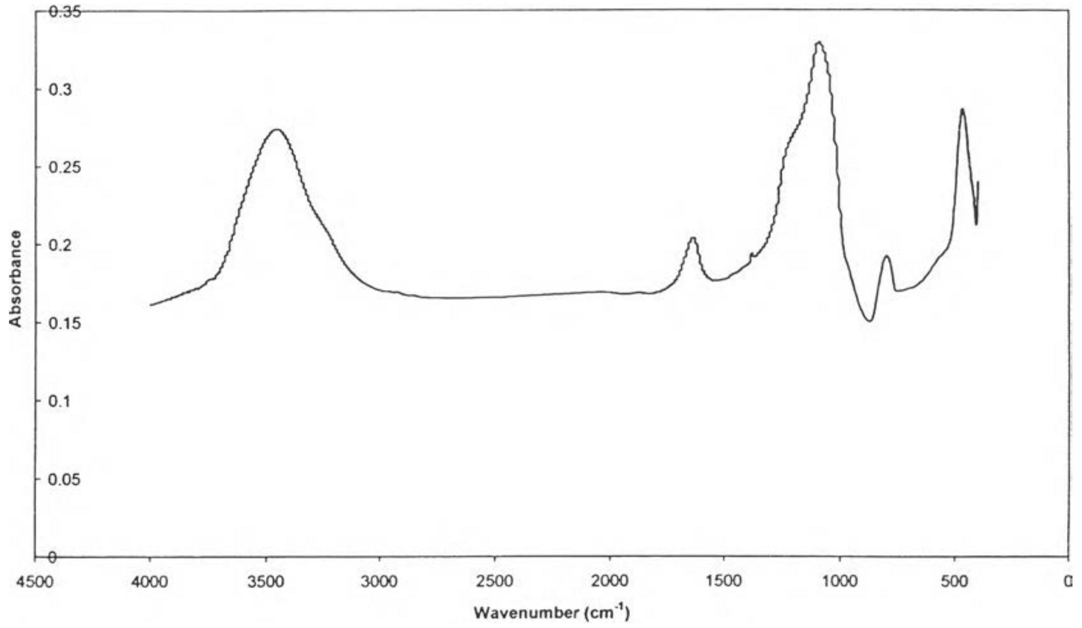


Figure 4.64 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 700°C.

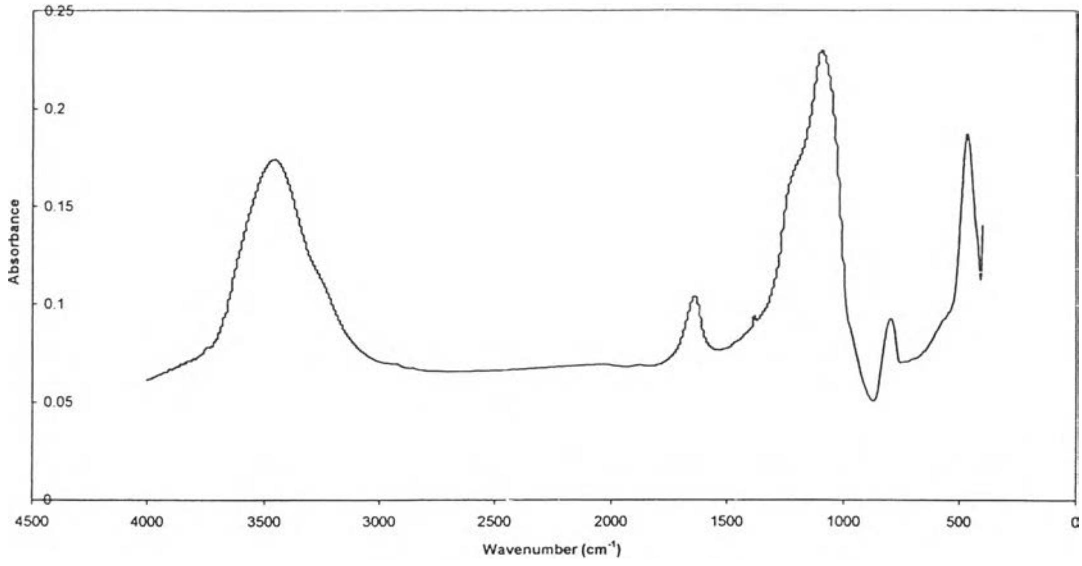


Figure 4.65 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 700°C.

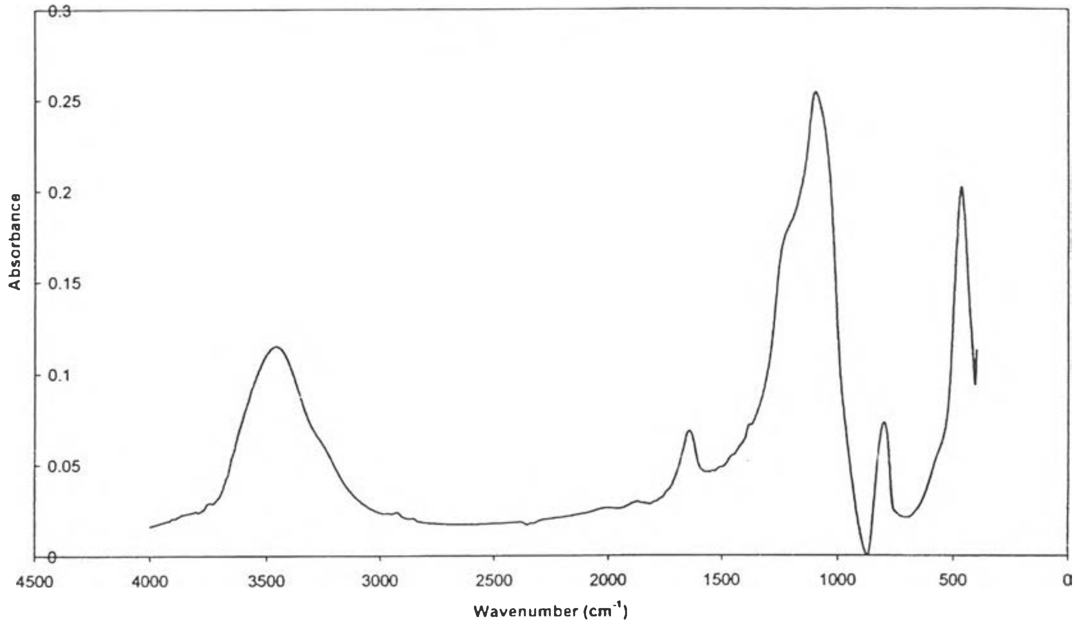


Figure 4.66 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 800°C.

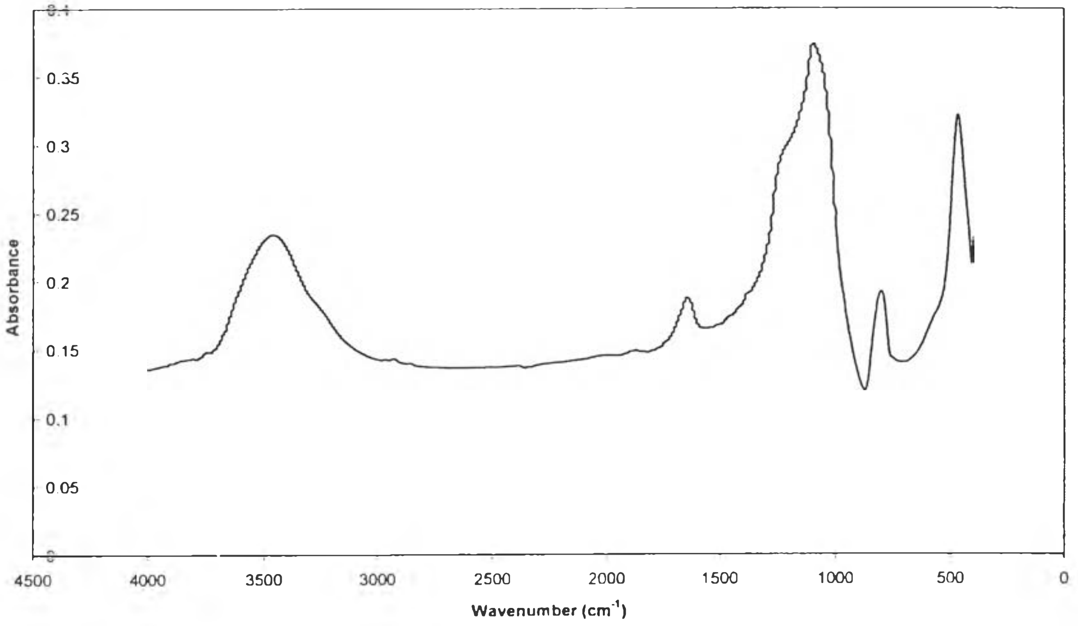


Figure 4.67 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 800°C.

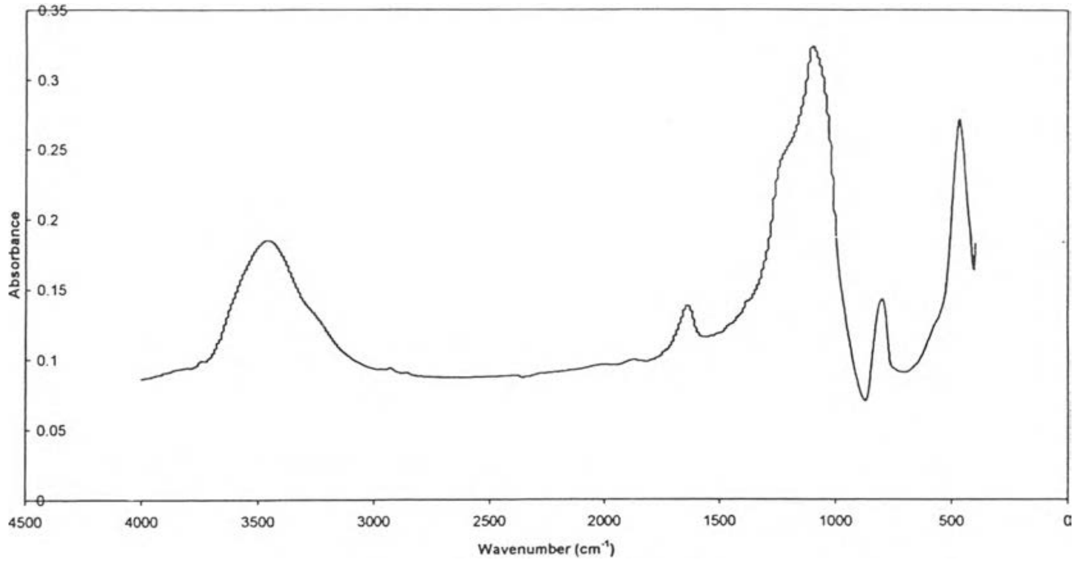


Figure 4.68 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 800°C.

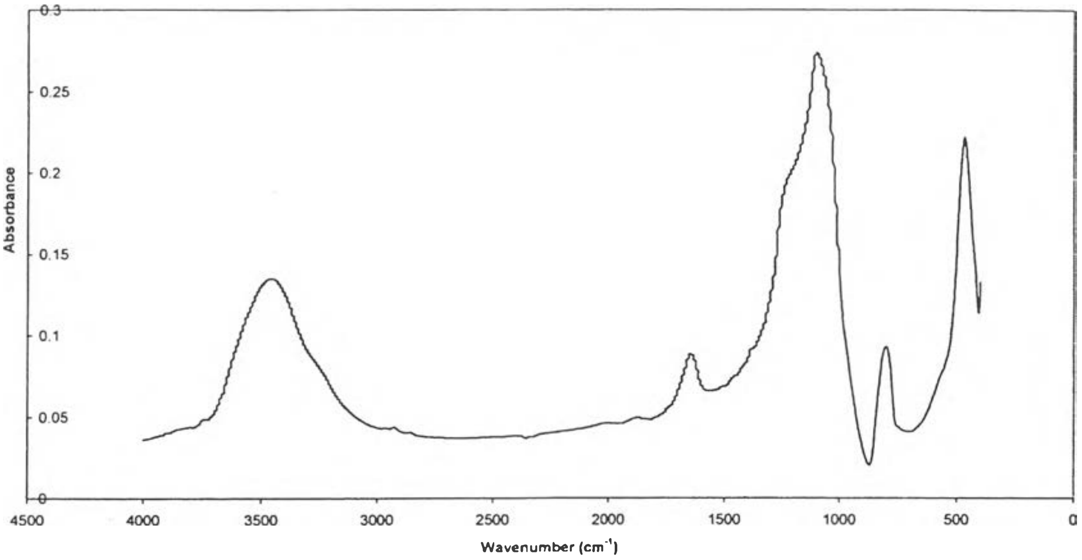


Figure 4.69 FTIR spectrum of calcined fibres obtained from 10 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 800°C.

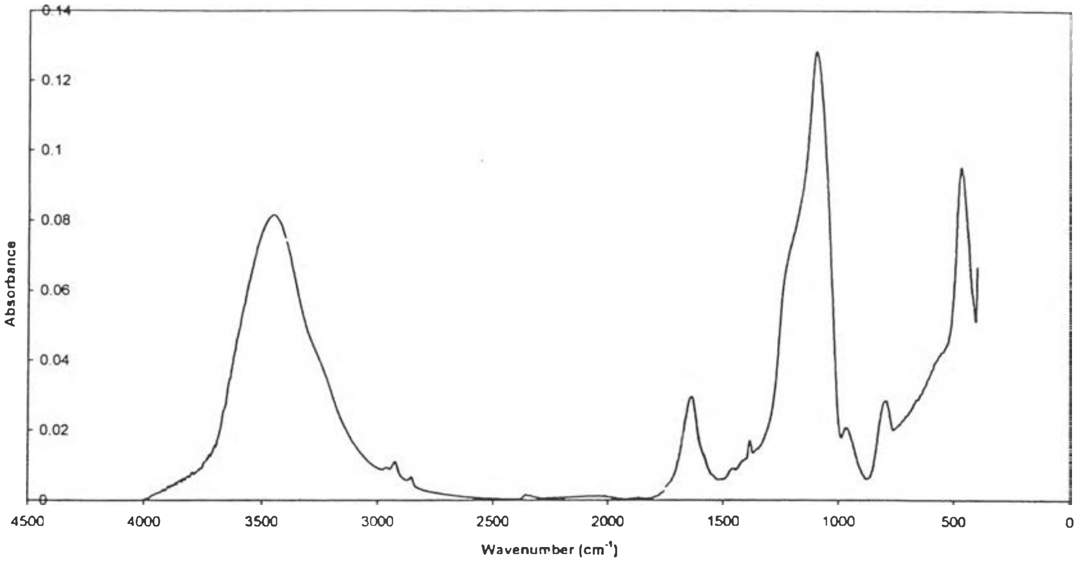


Figure 4.70 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 500°C.

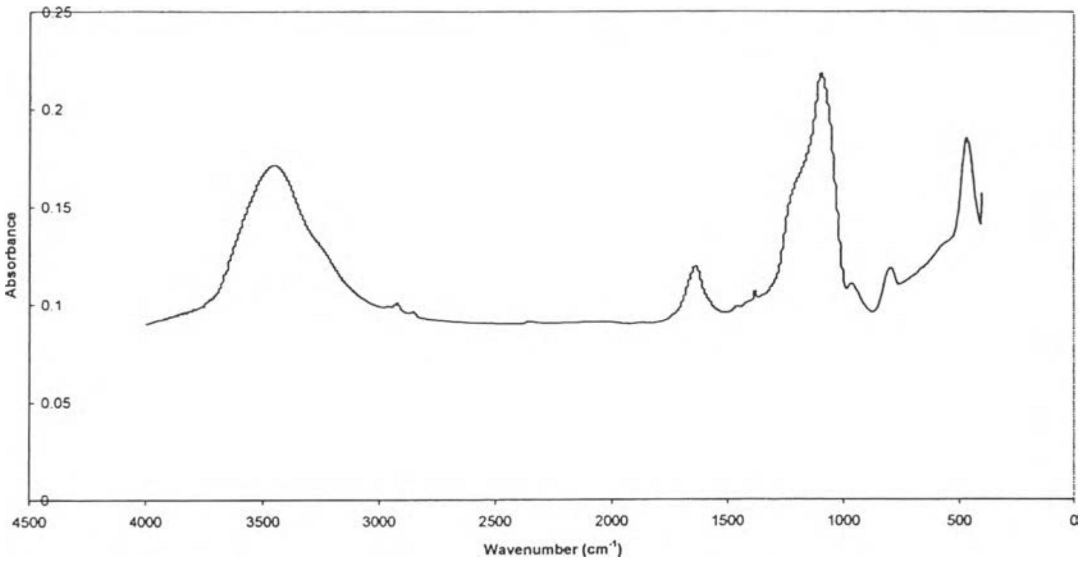


Figure 4.71 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 500°C.

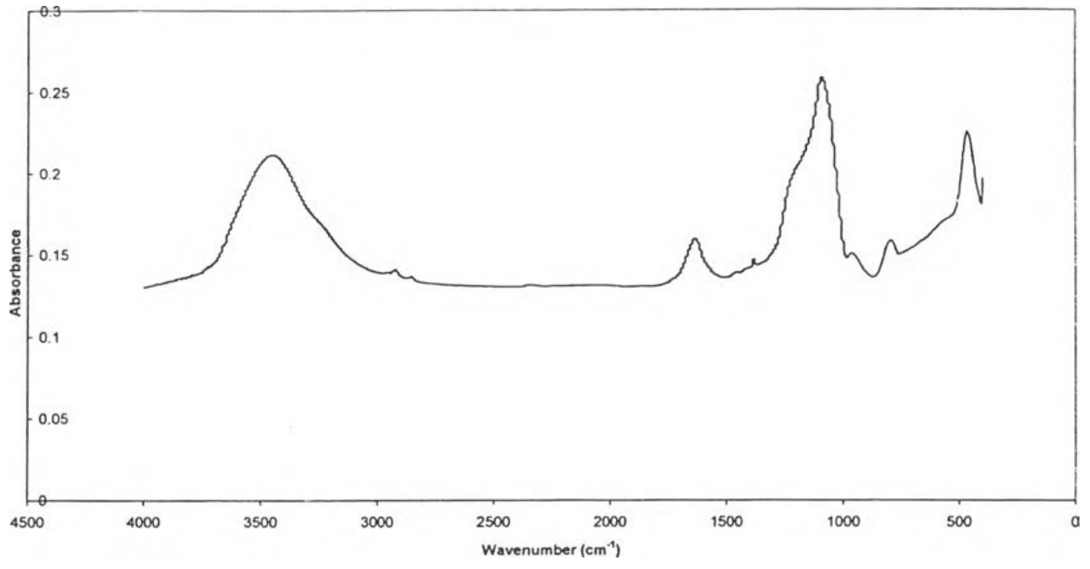


Figure 4.72 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 500°C.

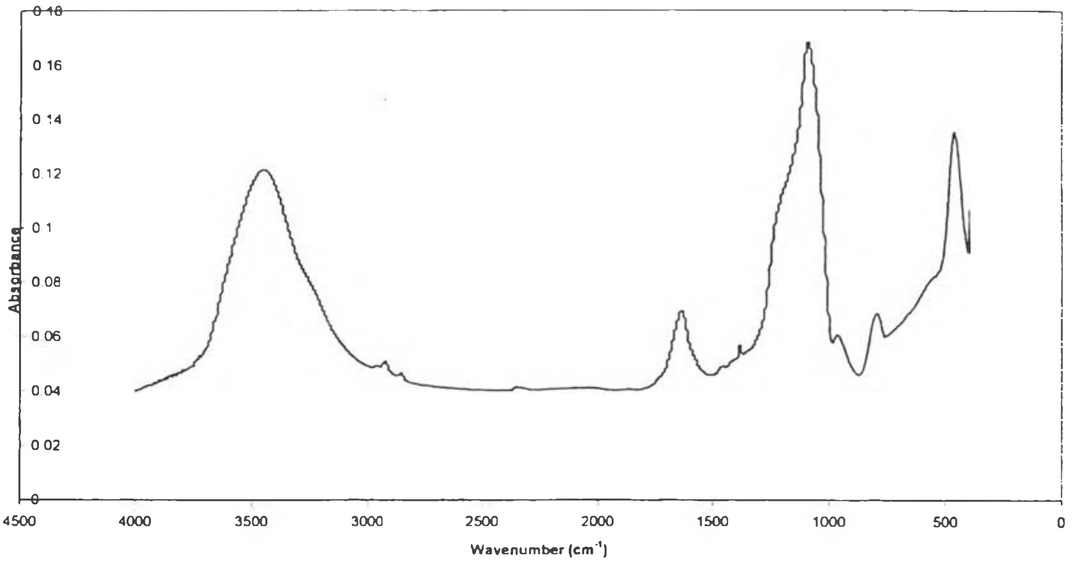


Figure 4.73 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 500°C.

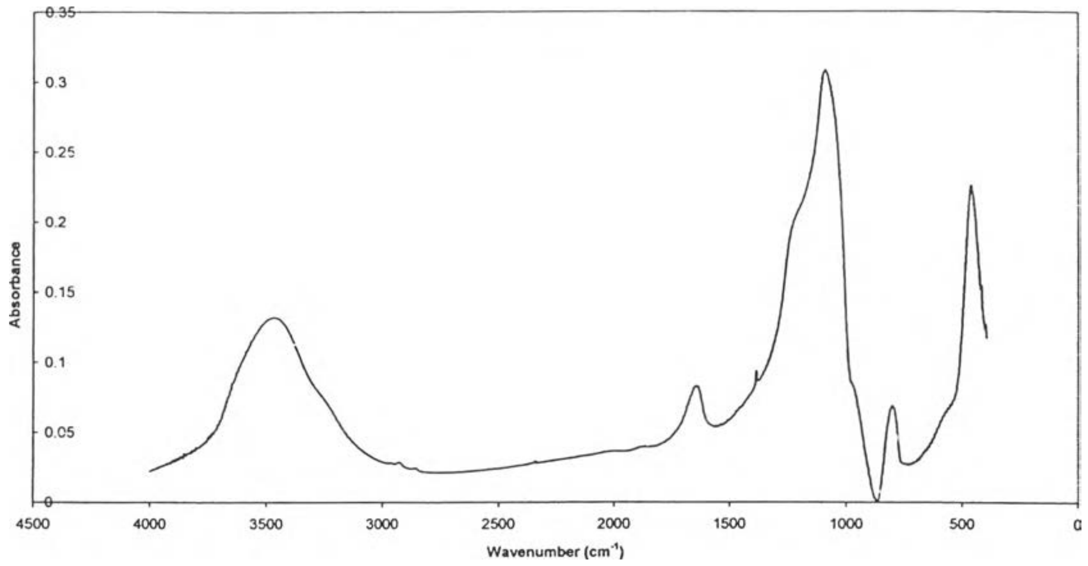


Figure 4.74 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 600°C.

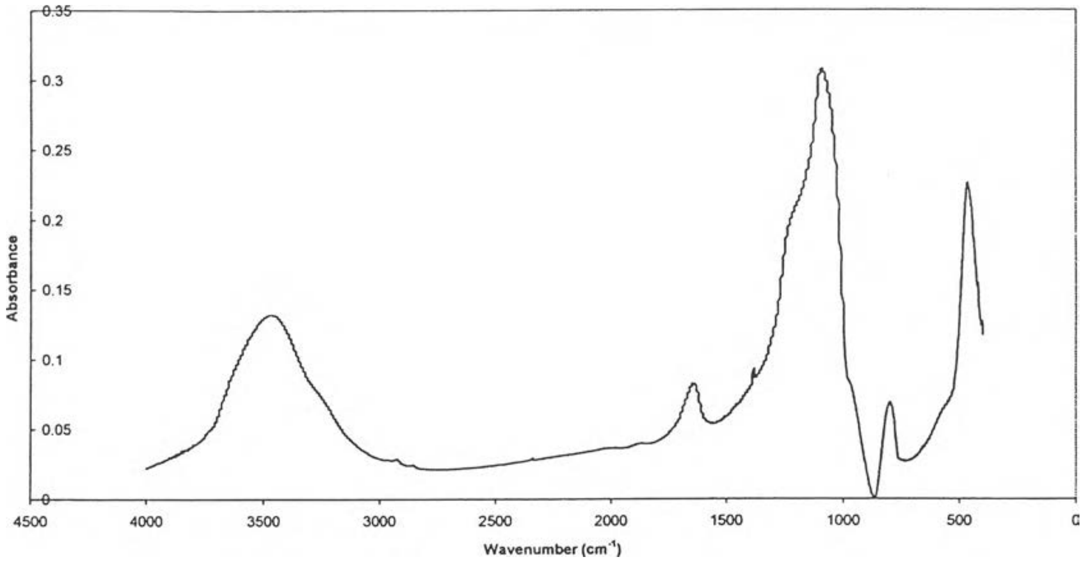


Figure 4.75 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 600°C.

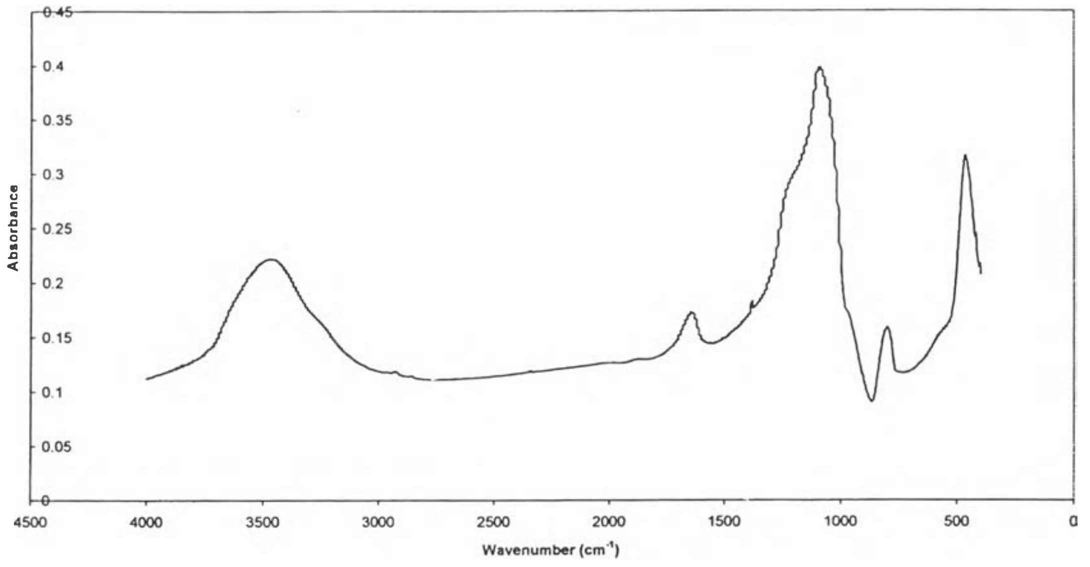


Figure 4.76 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 600°C.

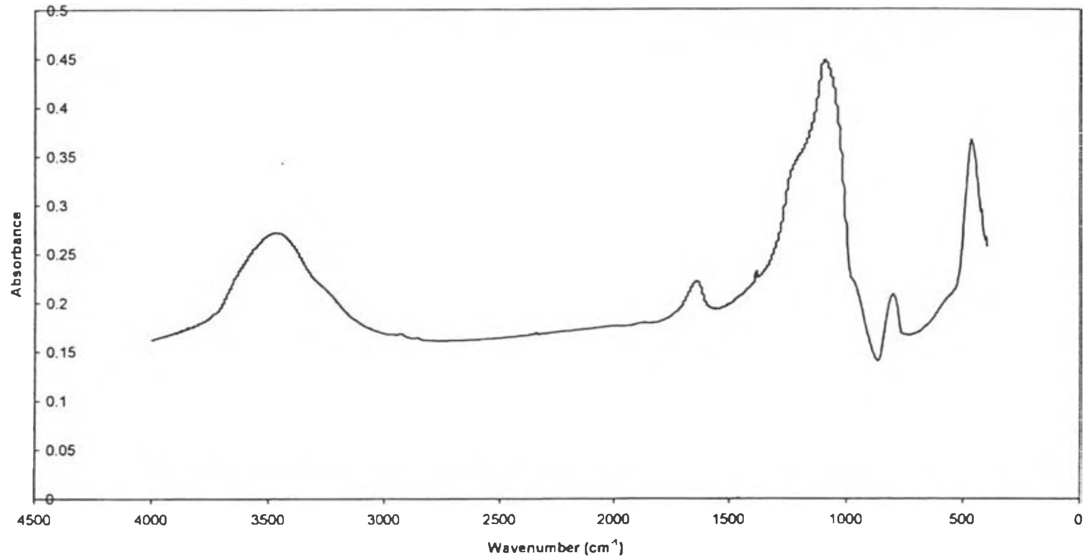


Figure 4.77 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 600°C.

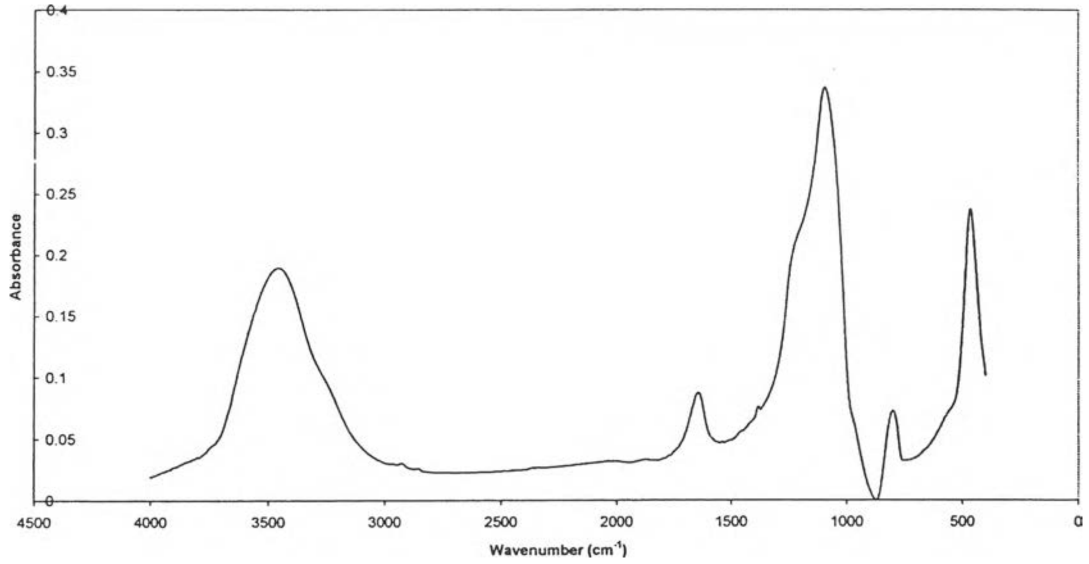


Figure 4.78 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 700°C.

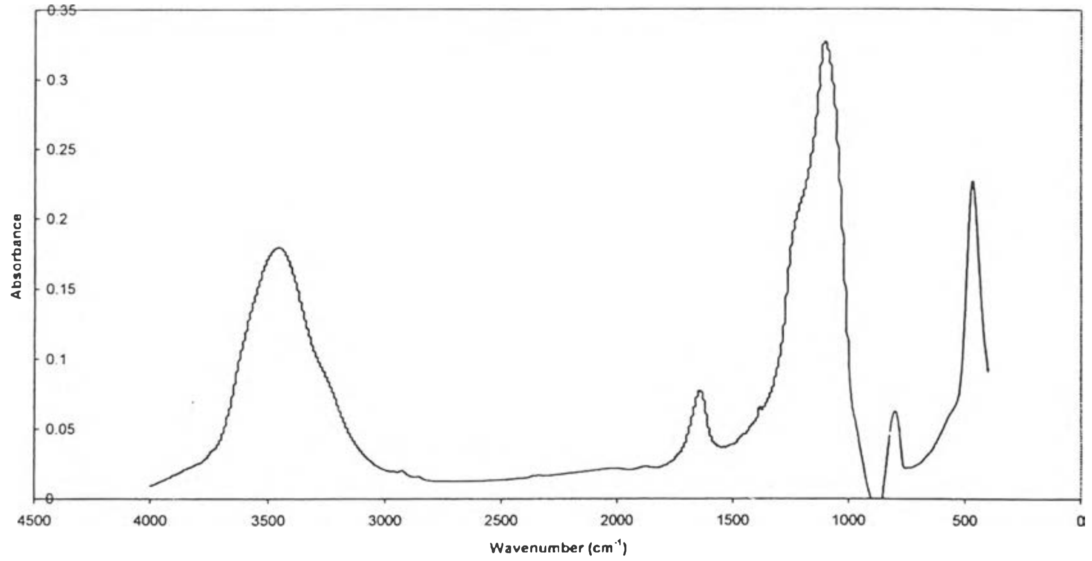


Figure 4.79 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 700°C.

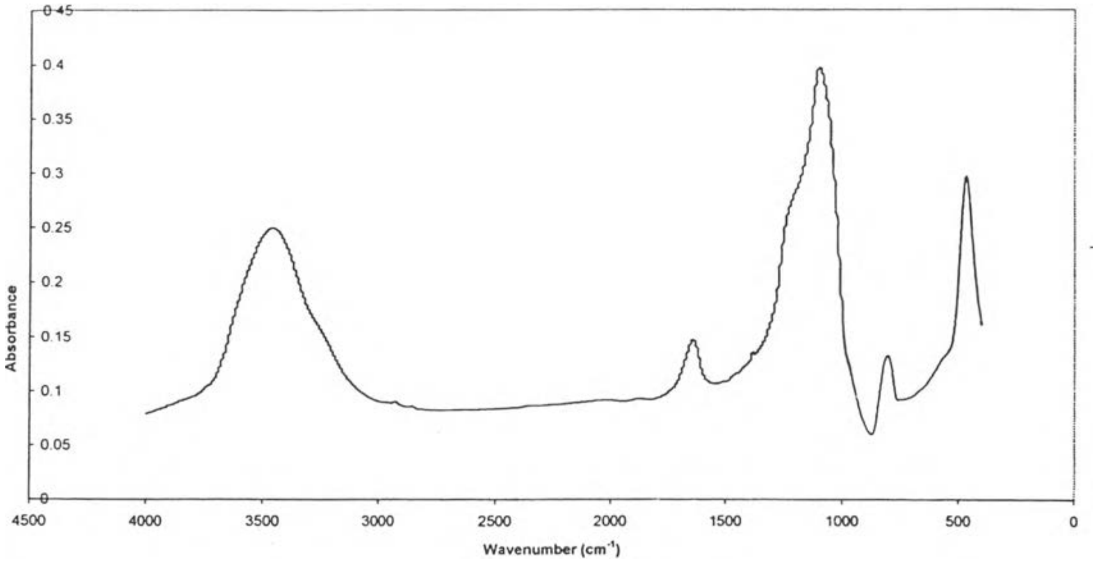


Figure 4.80 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 700°C.

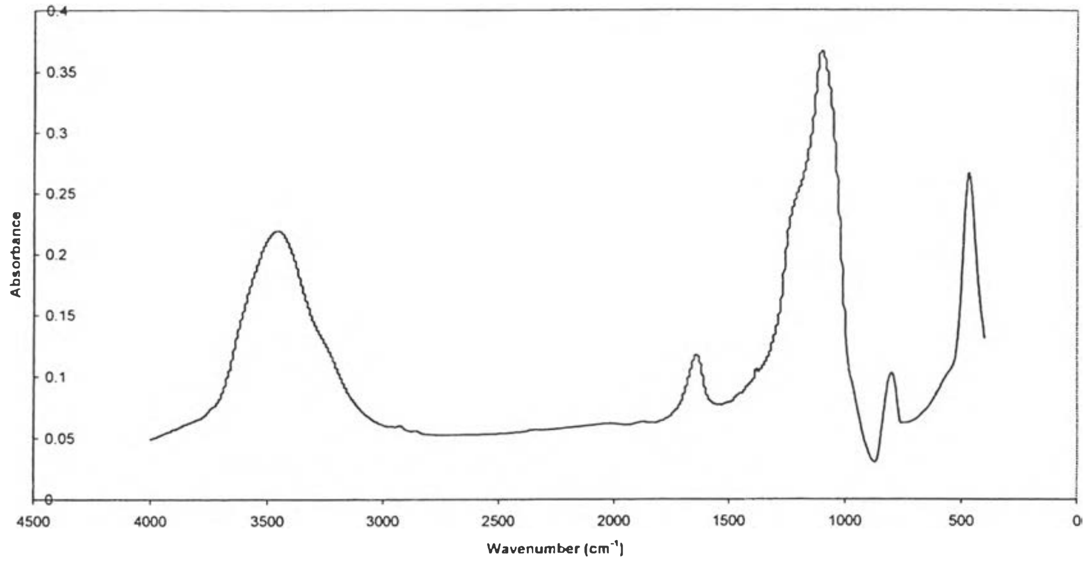


Figure 4.81 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 700°C.

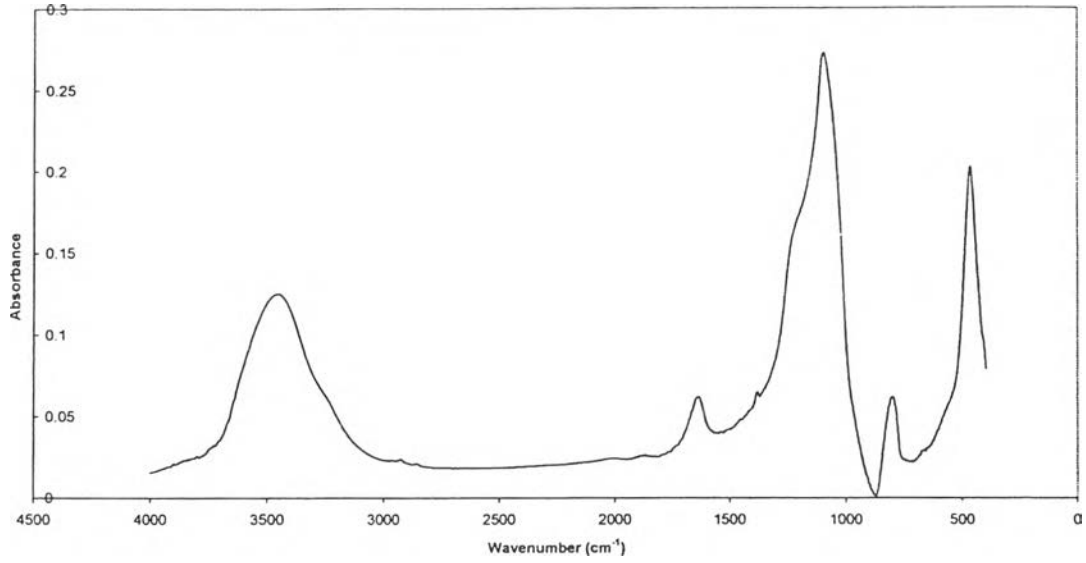


Figure 4.82 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 11.25 kV, and a calcination temperature of 800°C.

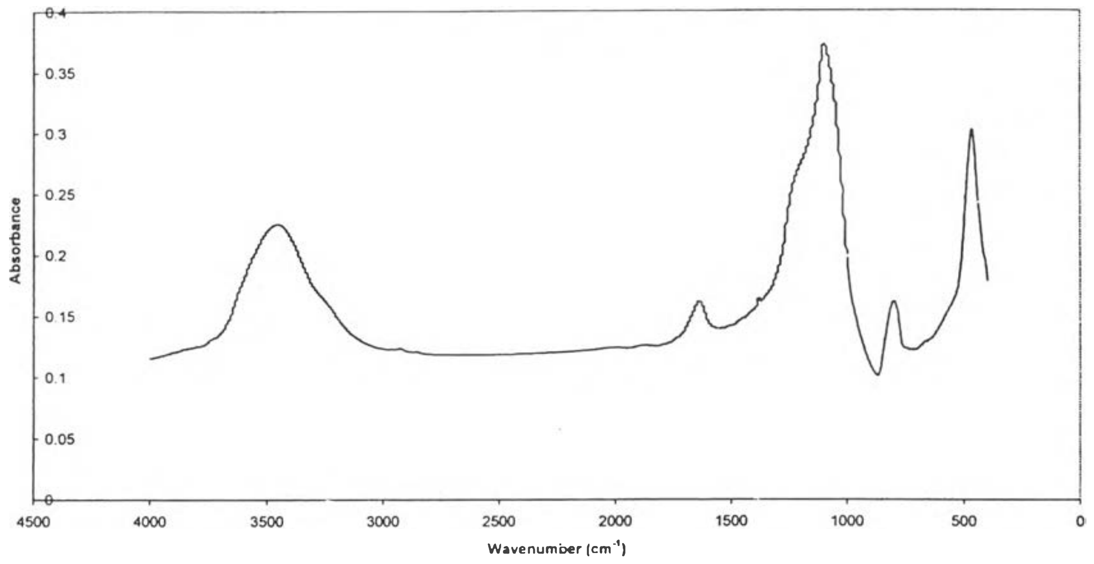


Figure 4.83 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 15 kV, and a calcination temperature of 800°C.

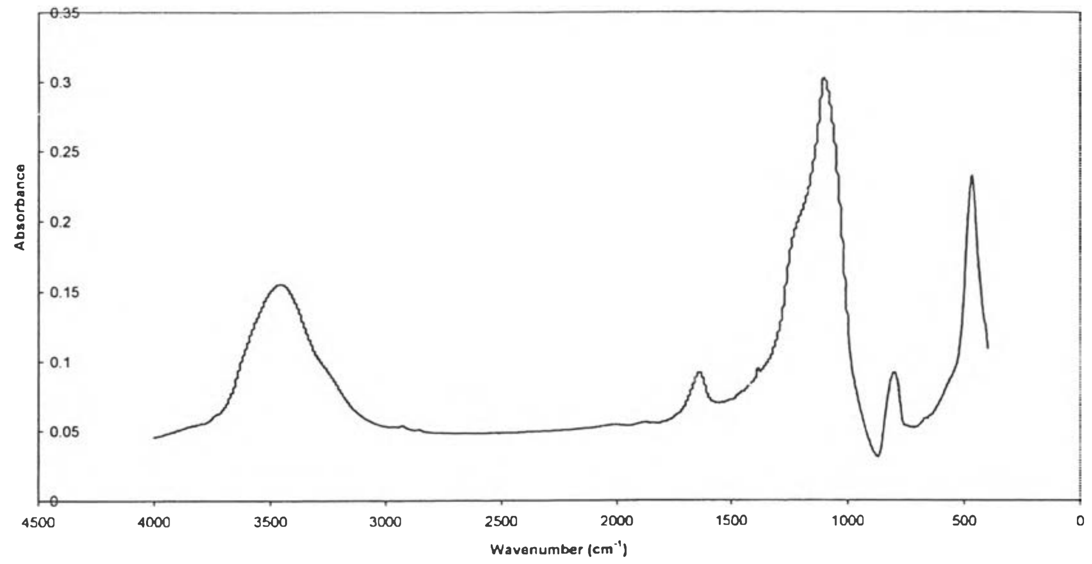


Figure 4.84 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 18.75 kV, and a calcination temperature of 800°C.

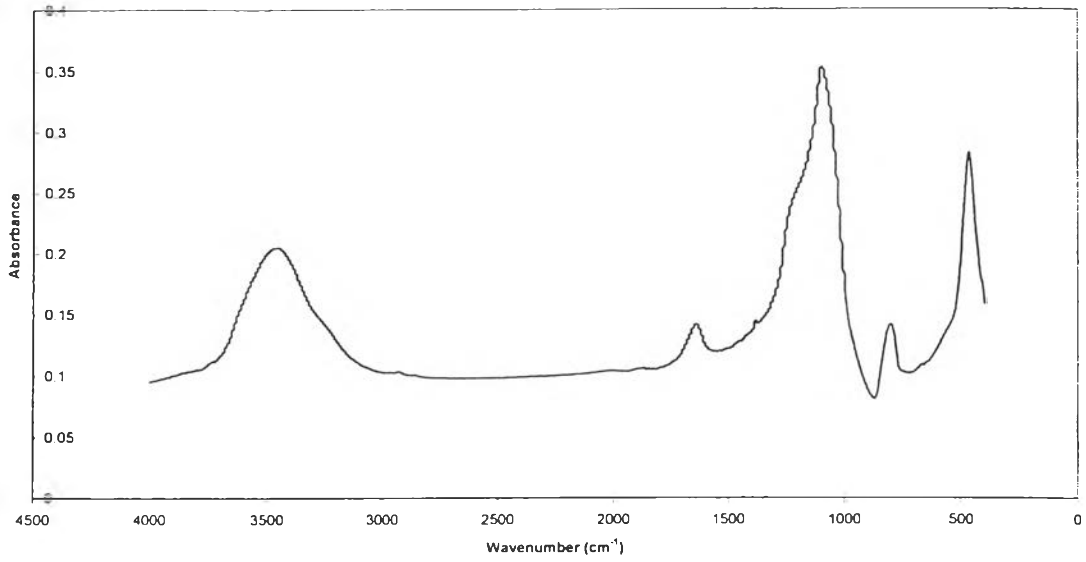


Figure 4.85 FTIR spectrum of calcined fibres obtained from 11.76 wt% of silatrane in 6 wt% PVA solution using an applied potential of 22.50 kV, and a calcination temperature of 800°C.

Figure 4.86 FTIR spectrum of PVA.

Figures 4.46–4.53 show the FTIR spectra of precalcined fibres. The O-H stretching band in the IR spectrum is by far the most characteristic feature of alcohols, and appears at 3575 cm^{-1} in pure PVA. The hydroxyl band is displaced from the initial position in precalcined fibres. This gives a clear indication of hydrogen bonding in the composite fibres between PVA and silatrane complexes. The vibrational peak at 1452 cm^{-1} is assigned to a CH_2 scissor mode of pure PVA. The peak at 1726 cm^{-1} is assigned to C=O stretching in the acetate group of PVA. The characteristic vibrational band at 1100 cm^{-1} is assigned to C-O stretching of secondary alcohols. The peaks at 560-590 cm^{-1} are assigned to N -> Si stretching and the peaks at 970 and 883 cm^{-1} are assigned to Si-O-CH group. The bands at 1015-1085 cm^{-1} are assigned to Si-O-CH_2 group. The peaks at 1351 and the band at 1380-1460 are corresponded to C-N and C-H bending respectively. However, some of the peaks and the bands are broadened and displaced from the initial position in precalcined fibres because there are secondary interaction, particularly hydrogen bonding in the composite fibres between PVA and silatrane complexes. All of them can confirm that prepared precalcined fibres are composed of both PVA and silatrane complexes.

Figures 4.54-4.85 show the FTIR spectra of electrospun silica fibres. The peaks at 466, 806, and 1100 cm^{-1} are assigned to Si-O stretching and the broad band centering at 3458 cm^{-1} is assigned to O-H stretching. The broad band peak centering at 3458 cm^{1} results from intra- and intermolecular hydrogen bonds of the silanol groups. It indicate that a substantial amount of silanol groups exists on the surface of calcined silica fibres like that of silica particles.

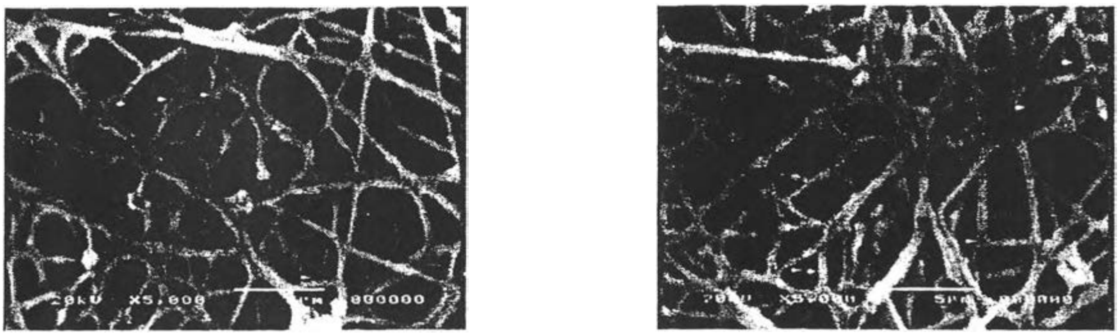
Table 4.2 Assignment of infrared spectra of the PVA/silatrane composite fibres

Characterisation	**Peak Position (cm^{-1})**
N -> Si stretching	560-590
Si-O-CH	970,883
C-O stretching	1100
Si-O-CH_2	1015-1085
C-N	1351
C-H bending	1380-1460
C=O stretching	1726
O-H stretching	3575

Table 4.3 Assignment of infrared spectra of the calcined fibres and pure silica

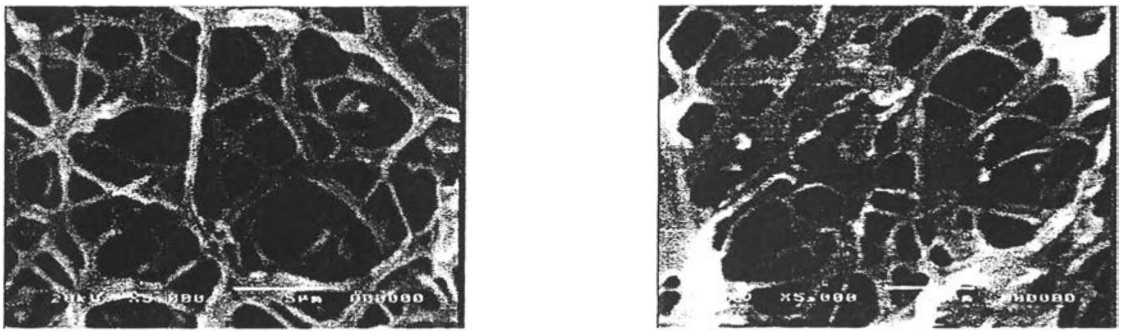
Characterisation	**Peak Position (cm^{-1})**
Si-O stretching	466, 806, 1100
O-H stretching	3458

4.2.2.3 *Scanning electron microscope (SEM)*



(a) 10.00 wt %, 11.25 kV (x5,000)

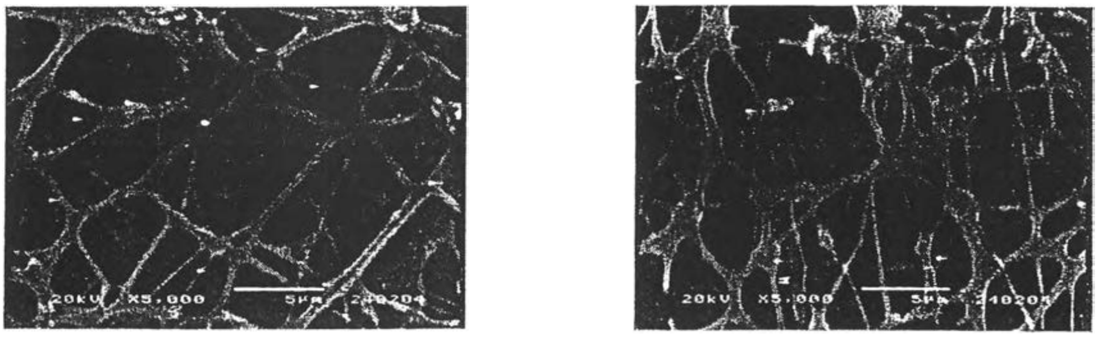
(b) 10.00 wt %, 15.00 kV (x5,000)



(c) 10.00 wt %, 18.75 kV (x5,000)

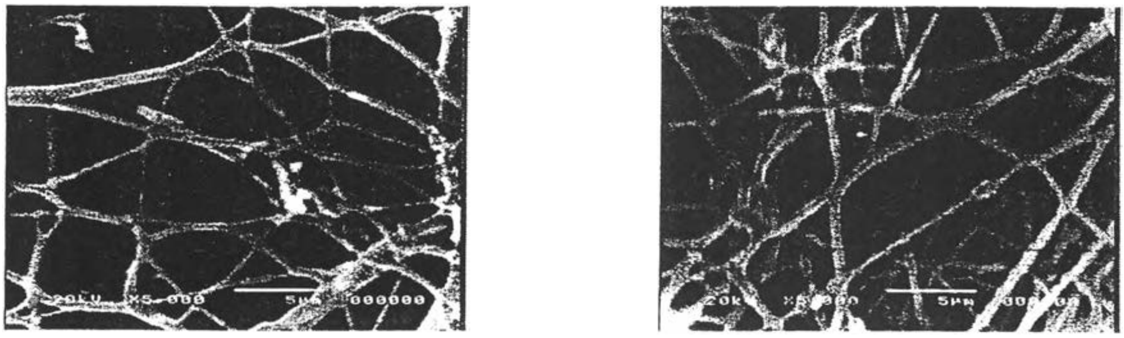
(d) 10.00 wt %, 22.50 kV (x5,000)

Figure 4.87 SEM micrographs of obtained PVA/silatrane composite fibres derived from 10 wt % silatrane spinning solution at various applied voltages.



(a) 11.76 wt %, 11.25 kV (x5,000)

(b) 11.76 wt %, 15.00 kV (x5,000)



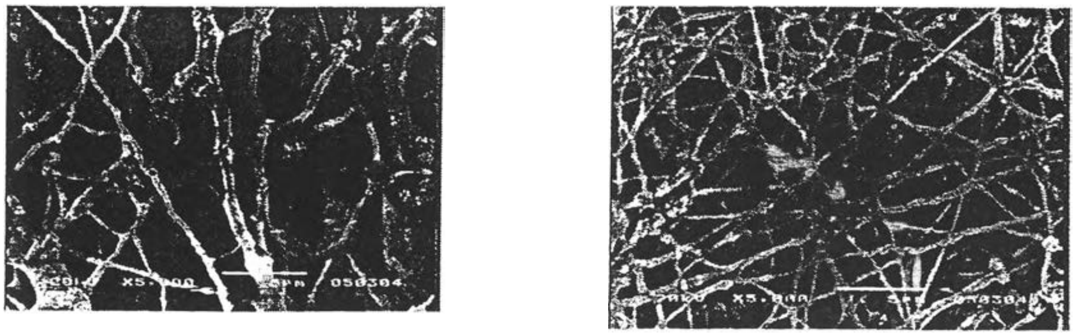
(c) 11.76 wt %, 18.75 kV (x5,000)

(d) 11.76 wt %, 22.50 kV (x5,000)

Figure 4.88 SEM micrographs of obtained PVA/silatrane composite fibres derived from 11.76 wt % silatrane spinning solution at various applied voltages.

(a) 10.00 wt %, 11.25 kV, 500°C (x5,000)

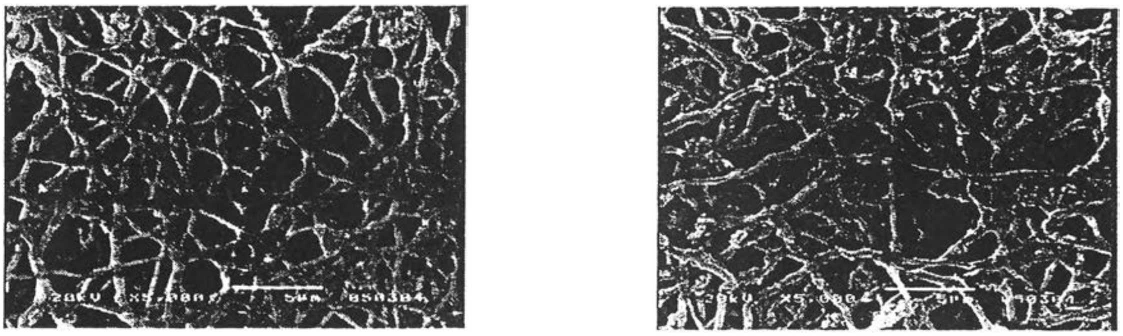
(b) 10.00 wt %, 11.25 kV, 600°C (x5,000)



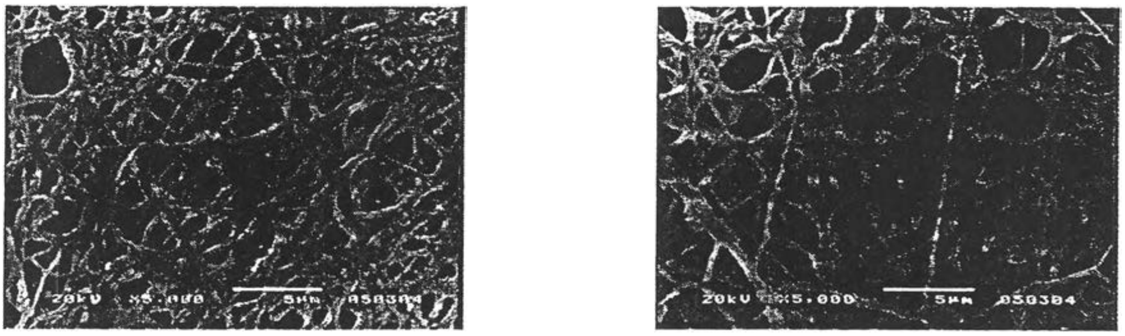
(c) 10.00 wt %, 11.25 kV, 700°C (x5,000)

(d) 10.00 wt %, 11.25 kV, 800°C (x5,000)

Figure 4.89 SEM micrographs of calcined silica fibres derived from 10 wt % silatrane spinning solution at 11.25 kV.

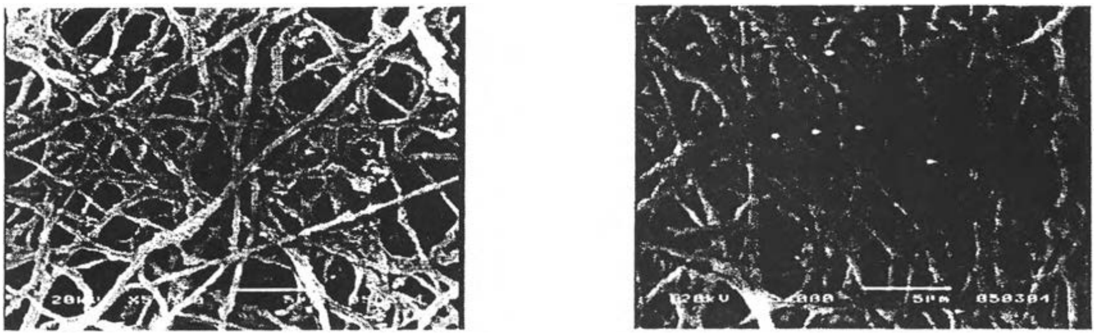


(a) 10.00 wt %, 15.00 kV, 500°C (x5,000) (b) 10.00 wt %, 15.00 kV, 600°C (x5,000)

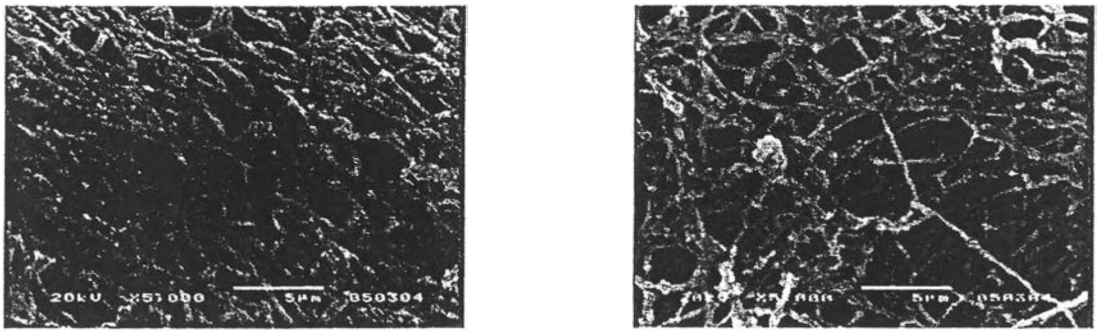


(c) 10.00 wt %, 15.00 kV, 700°C (x5,000) (d) 10.00 wt %, 15.00 kV, 800°C (x5,000)

Figure 4.90 SEM micrographs of calcined silica fibres derived from 10 wt % silatrane spinning solution at 15.00 kV.



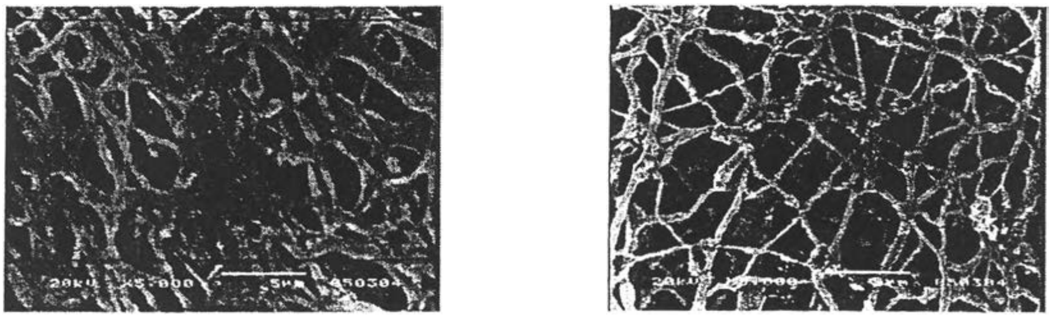
(a) 10.00 wt %, 18.75 kV, 500°C (x5,000)   (b) 10.00 wt %, 18.75 kV,600°C (x5,000)



(c) 10.00 wt %, 18.75 kV, 700°C (x5,000)   (d) 10.00 wt %, 18.75 kV, 800°C (x5,000)

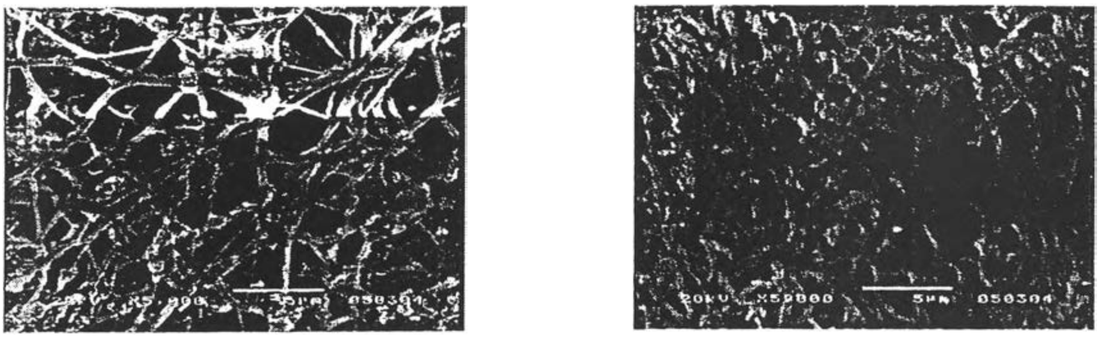
Figure 4.91 SEM micrographs of calcined silica fibres derived from 10 wt % silatrane spinning solution at 18.75 kV.

(a) 10.00 wt %, 22.50 kV, 500°C (x5,000) (b) 10.00 wt %, 22.50 kV, 600°C (x5,000)



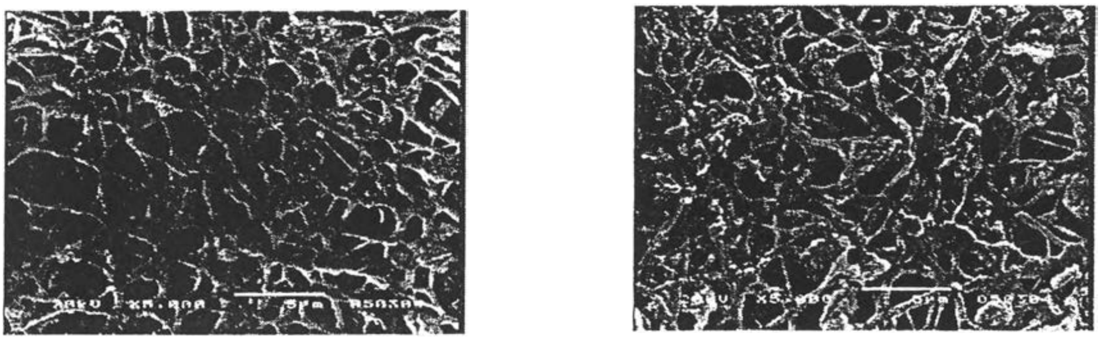
(c) 10.00 wt %, 22.50 kV, 700°C (x5,000) (d) 10.00 wt %, 22.50 kV, 800°C (x5,000)

Figure 4.92 SEM micrographs of calcined silica fibres derived from 10 wt % silatrane spinning solution at 22.50 kV.



(a) 11.76 wt %, 11.25 kV, 500°C (x5,000)

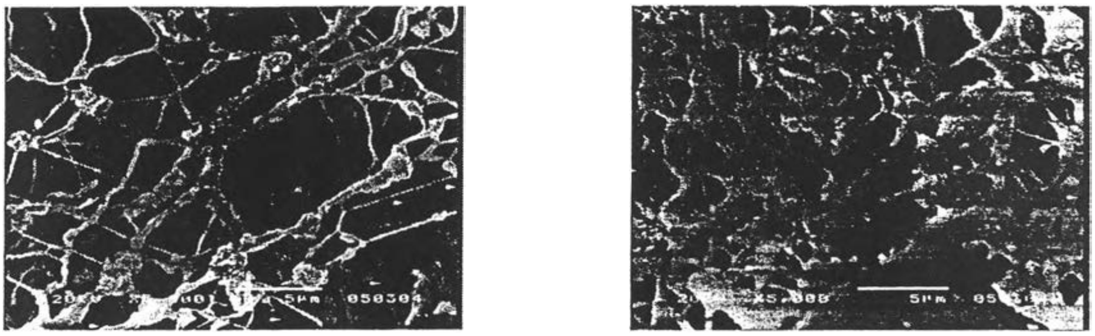
(b) 11.76 wt %, 11.25 kV, 600°C (x5,000)



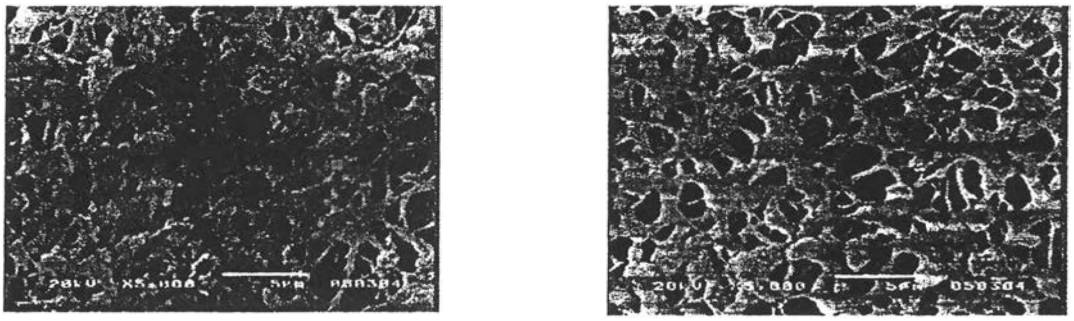
(c) 11.76 wt %, 11.25 kV, 700°C (x5,000)

(d) 11.76 wt %, 11.25 kV, 800°C (x5,000)

Figure 4.93 SEM micrographs of calcined silica fibres derived from 11.76 wt % silatrane spinning solution at 11.25 kV.

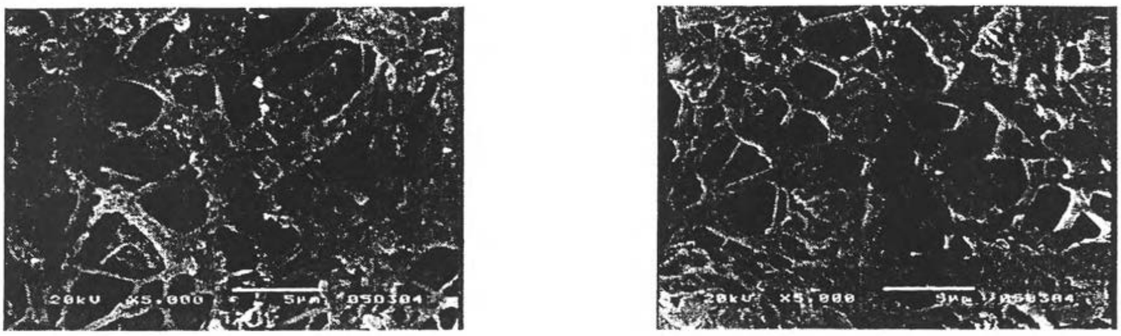


(a) 11.76 wt %, 15.00 kV, 500°C (x5,000) (b) 11.76 wt %, 15.00 kV, 600°C (x5,000)

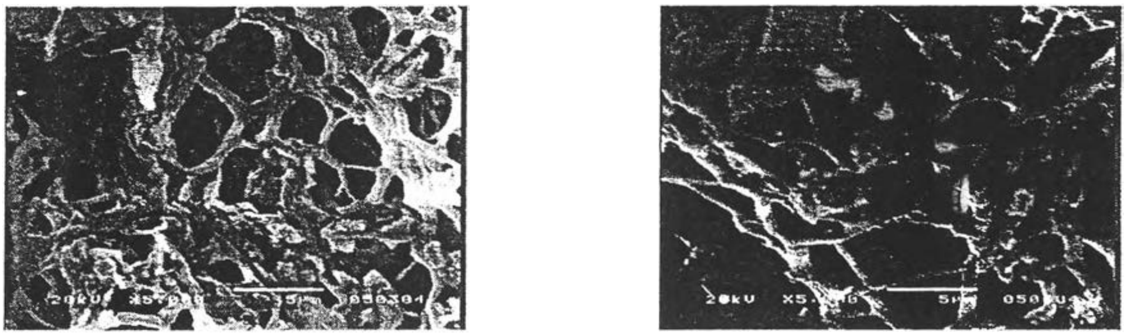


(c) 11.76 wt %, 15.00 kV, 700°C (x5,000) (d) 11.76 wt %, 15.00 kV, 800°C (x5,000)

Figure 4.94 SEM micrographs of calcined silica fibres derived from 11.76 wt % silatrane spinning solution at 15.00 kV.

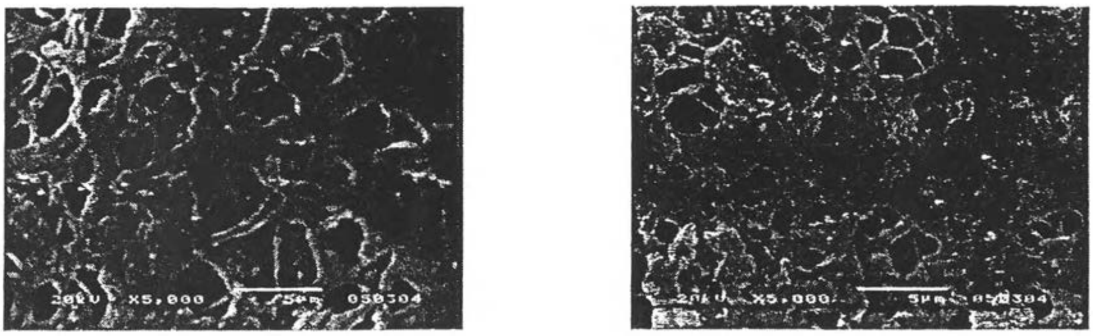


(a) 11.76 wt %, 18.75 kV, 500°C (x5,000) (b) 11.76 wt %, 18.75 kV, 600°C (x5,000)

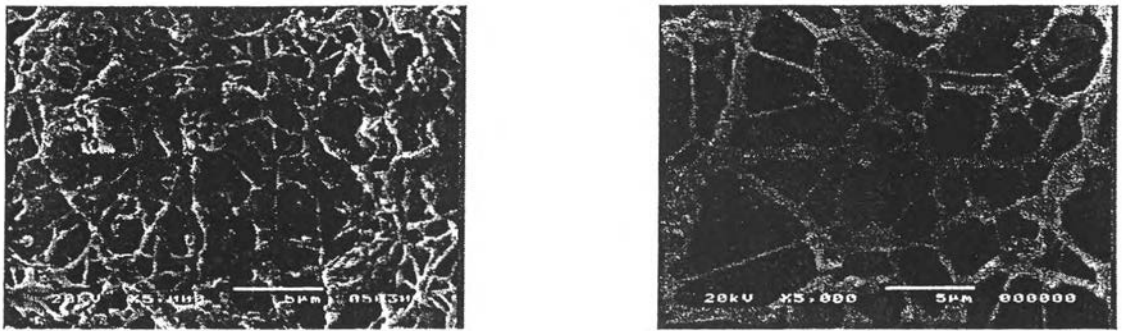


(c) 11.76 wt %, 18.75 kV, 700°C (x5,000) (d) 11.76 wt %, 18.75 kV, 800°C (x5,000)

Figure 4.95 SEM micrographs of calcined silica fibres derived from 11.76 wt % silatrane spinning solution at 18.75 kV.



(a) 11.76 wt %, 22.50 kV, 500°C (x5,000) (b) 11.76 wt %, 22.50 kV,600°C (x5,000)



(c) 11.76 wt %, 22.50 kV, 700°C (x5,000) (d) 11.76 wt %, 22.50 kV, 800°C (x5,000)

Figure 4.96 SEM micrographs of calcined silica fibres derived from 11.76 wt % silatrane spinning solution at 22.50 kV.

Figures 4.87–4.96 show the SEM micrographs of precalcined and calcined fibres. In this part, the SEM micrographs will be used to observe the effect of experimental parameters on the morphology of fibres. Hence, the relationships between them will be divided and discussed as the following sections.

a) Effect of applied potential

It was found that nanofibre diameter increased with increasing applied voltage. It resulted from the charge transport. The higher the charge transport, the greater the electrospinning current. Consequently, mass flow rate of polymer jet increased and fibre diameter increased as well, when the other parameters (i.e., concentration of silatrane of spinning solution, collection distance) are constant.

b) Effect of silatrane concentration

The nanofibres obtained from the concentrations of 10 wt % and 11.76 wt % are shown in Figures 4.87-4.96. It sould be noted that fibres cannot be produced at concentration lower than 10 wt % because the microgel formation occurred. This phenomenon can be explained in terms of chemistry of sol-gel process. Because polyvinylalcohol (PVA) molecules act as nucleophile in the hydrolysis step, the rate of hydrolysis and polycondensation increases with the number of PVA molecules proportionally. Thus, if the PVA concentration of spinning solution is greater than 90%, the microgel resulting in the inhomogeneity of spinning solution will occur. However, it was impossible to prepare spinning solution with silatrane concentration greater than 11.76% because this concentration is the highest concentration to prepare silatrane solution in 6 wt % PVA.

When the SEM micrographs were taken into account, it was found that the number of sticking fibres increased with the increment of silatrane concentration. The effect of silatrane concentration on the number of sticking fibres can be explained in terms of the solidification of nanofibres. The rate of nanofibre solidification depended largely on the distance of fibre travel. The greater the applied voltage, the less the instability. As a result, the relation between the applied voltage and the path of nanofibres is reverse. Hence, nanofibres produced from the higher applied voltage are wetter than that from the lower applied voltage. Thus, the wetter the nanofibres, the more the trend of sticking nanofibre formation. For calcined nanofibres, when the PVA left from the composite nanofibres, the nanofibres tended to shrink and close together by cohesive forces between silica molecules. Consequently, the calcined fibres tended to stick together with the increment of silatrane concentration.

c) Effect of calcination temperature

It was found that the calcination temperature hardly influenced morphology of the obtained silica nanofibres because the polymer content in composite fibres degraded almost completely before 500°C. It should be noted that the phase of obtained silica fibres was consistent with varying the operating temperature indicating that there is no phase transformation of silica fibres below 800°C.