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APPENDIX A

Fourier Transform Infrared Spectroscometer (FT-IR) [18]

Recently, infrared spectroscometers based on Fourier transform interferometry have gained acceptance. The infrared instruments employing the Fourier transform , offer even more favorable resolution and signal-to-noise characteristics than dispersive instruments.

The Fourier transformation is a perfectly general mathematical operation. By the use of this operation , one can transform results obtained as a function of time (interferogram) and express them as a function of frequency (absorbance spectra). The infrared spectroscometers utilizing dispersive instruments do have certain limitations. For example : measurements are relatively slow requiring several minutes to obtain spectra with acceptable resolution and signal-to-noise for the common spectral region from 400-4000 cm^{-1} ; the detectability of low level components is not good; and useful spectra cannot be routinely obtained for extremely small samples. Development of Fourier transform infrared spectroscometers greatly alleviated these problems.

The basis of Fourier transform infrared spectroscopy is the Micheson Interferometer shown in figure A1. Collimate energy from an infrared source passes to a beam splitter. The two equal beams are then directed to moving mirror and stationary mirror. If the mirrors are positioned such that optical paths of the beams are equal, the beams are in phase when they return to the beam splitter. Consequently, the beams interfere constructively, giving an energy maximum. Displacing the movable mirror by one-quarter wavelength of the incident light will bring the two beams 180 degrees out of phase when they return to the beam splitter. This results in destructive interference (an energy minimum). Movement of moveable mirror gives an alternating energy maximum and minimum for each quarter wavelength movement of the mirror. This corresponds to a wavelength change of $\lambda/2$ since the beam travels this distance twice.

If monochromatic energy of wavelength λ is passed through the interferometer and moveable mirror is moved with velocity, V, the signal detected will have a frequency, f, given by the following equation :

$$f = \frac{2V}{\lambda}$$

The result of plotting detector response versus mirror travel is a pure cosine function. When polychromatic radiation is used as the source, the detector output signal becomes a complex cosine function. The resulting output is called an interferogram and must undergo a Fourier transformation to yield the familiar absorption spectrum. This transformation requires the use of a digital computer.

Advantages of Fourier transform spectroscometer

The Fourier transform infrared technique offers certain advantages over dispersive infrared technique. These include :

1. Fellgett's Advantage

The Fourier transform spectroscometer measures all wavelengths of the infrared spectrum simultaneously. Dispersive spectroscometer measures only one wavelength at a time. Therefore,a Fourier transform system is capable of measuring a complete spectrum in the same time it takes a dispersive spectroscometer to measure one resolution element. The Fourier transform system is N times faster than dispersive spectroscometer ,where N is the number of resolution elements in the spectrum. Alternately, for the same measurement time, the Fourier transform system has better signal-to-noise characteristics than a dispersive spectroscometer.

2. Jacquinot's Advantage

This advantage concerns comparison of the light through-put of the interferometer versus a dispersive instrument at a given resolution. The interferometer has a circular aperture of, for example, 50 nm diameter, and has no slits. A dispersive instrument operating at 1 cm^{-1} resolution has a slit area of approximately 1 mm^2 . Comparison of these figures suggests a through-put advantage of nearly 2000 for the interferometer. However, the solid angle of view for a dispersive instrument is about 50 times that of an interferometer, hence the actual through-put advantage for the interferometer is more nearly a factor of 40.

3. Connes's Advantage

Fourier transform infrared spectroscometers use a laser to calibrate the wavelength of each scan. This insures wavelength precision.

4. Stray Light Advantage

In Fourier transform systems, each infrared frequency is chopped by the interferometer (according to above equation) at a different frequency, resulting in essentially zero stray light reaching the detector.

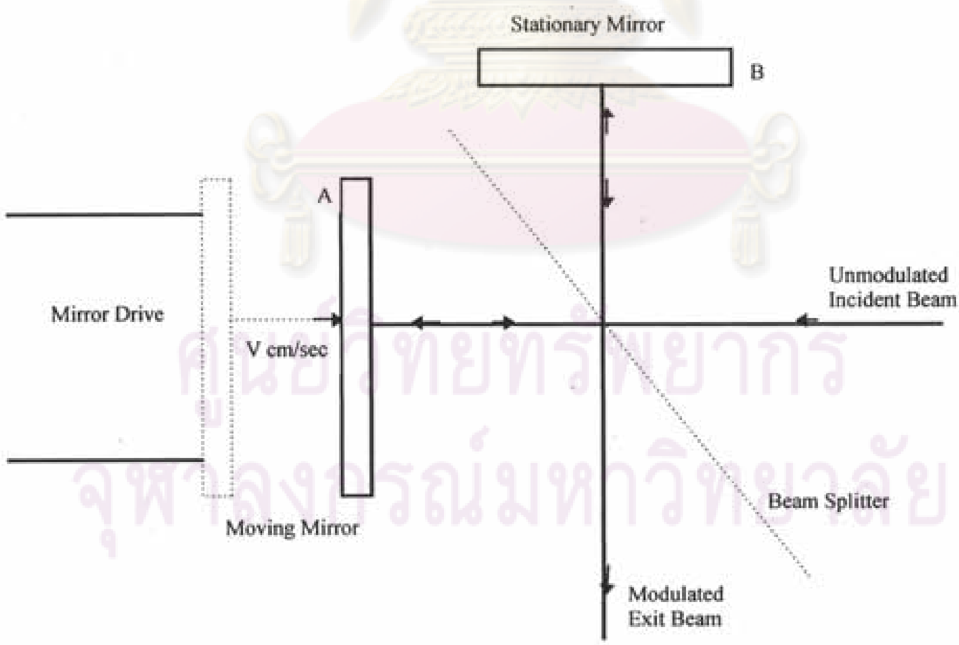

Figure A.1 Diagram of a Michaelson Interferometer [18]

APPENDIX B

IR spectra of coked catalysts at 300°C, 400°C, and cooled down to room temperature in N_2 atmosphere

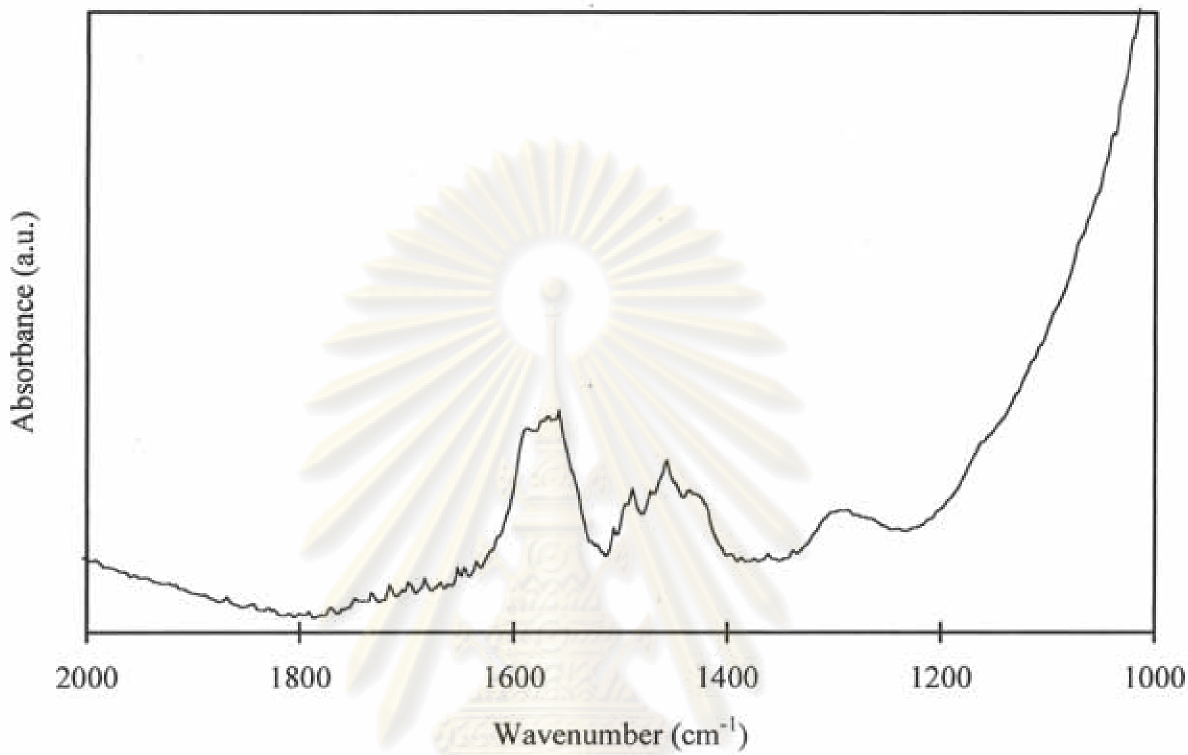

Figure B.1 IR spectrum of 5 min.coked catalyst at 300°C in N_2 atmosphere

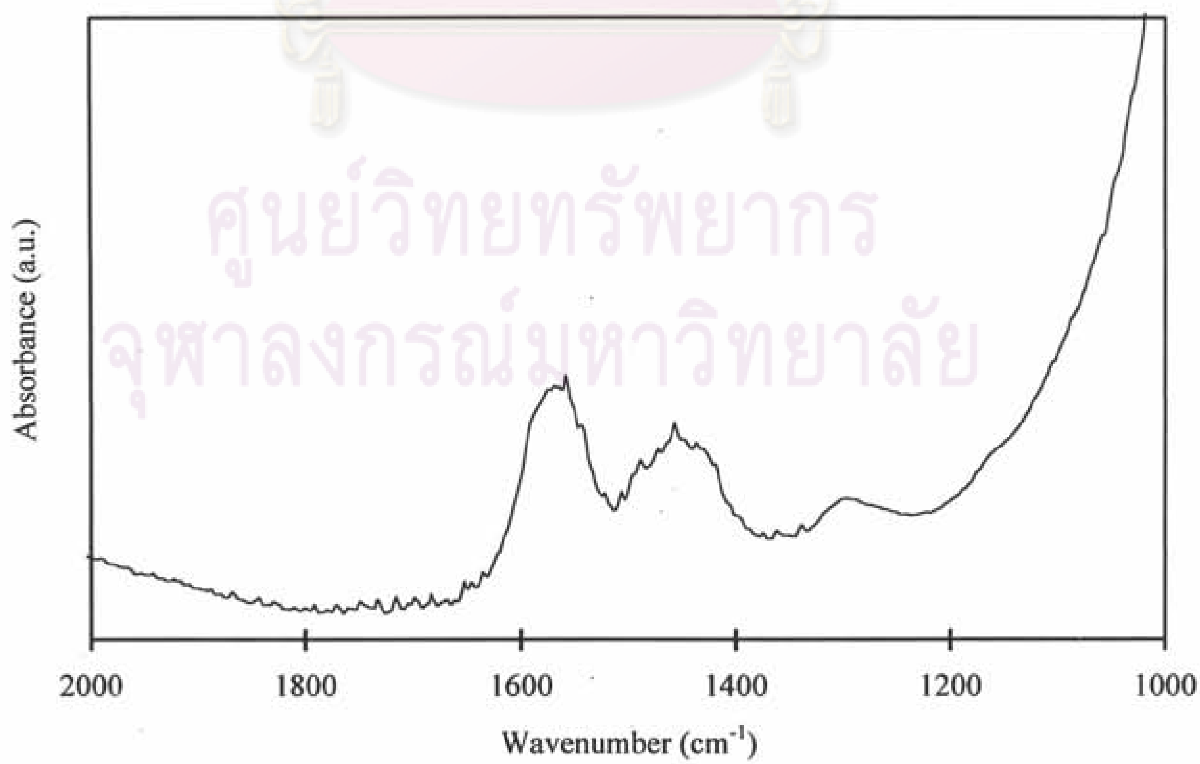

Figure B.2 IR spectrum of 5 min.coked catalyst at 400°C in N_2 atmosphere

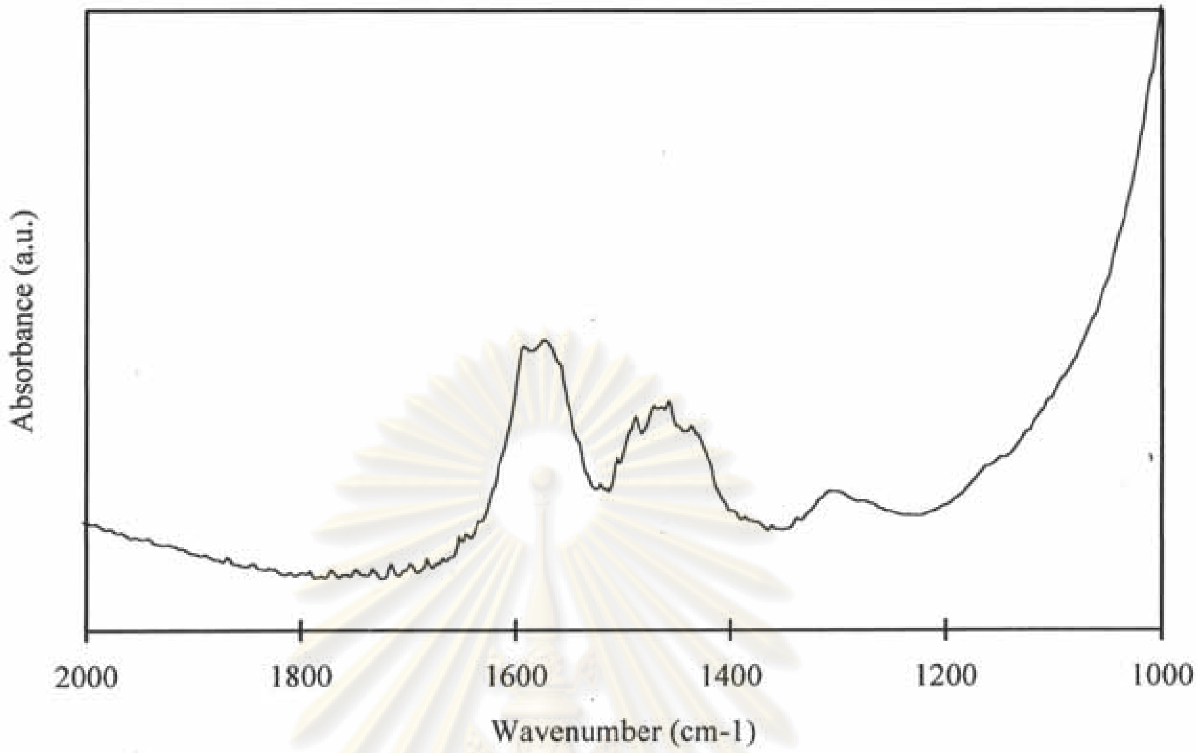

Figure B.3 IR spectrum of 5 min.coked catalyst cooled down to room temperature in N_2 atmosphere

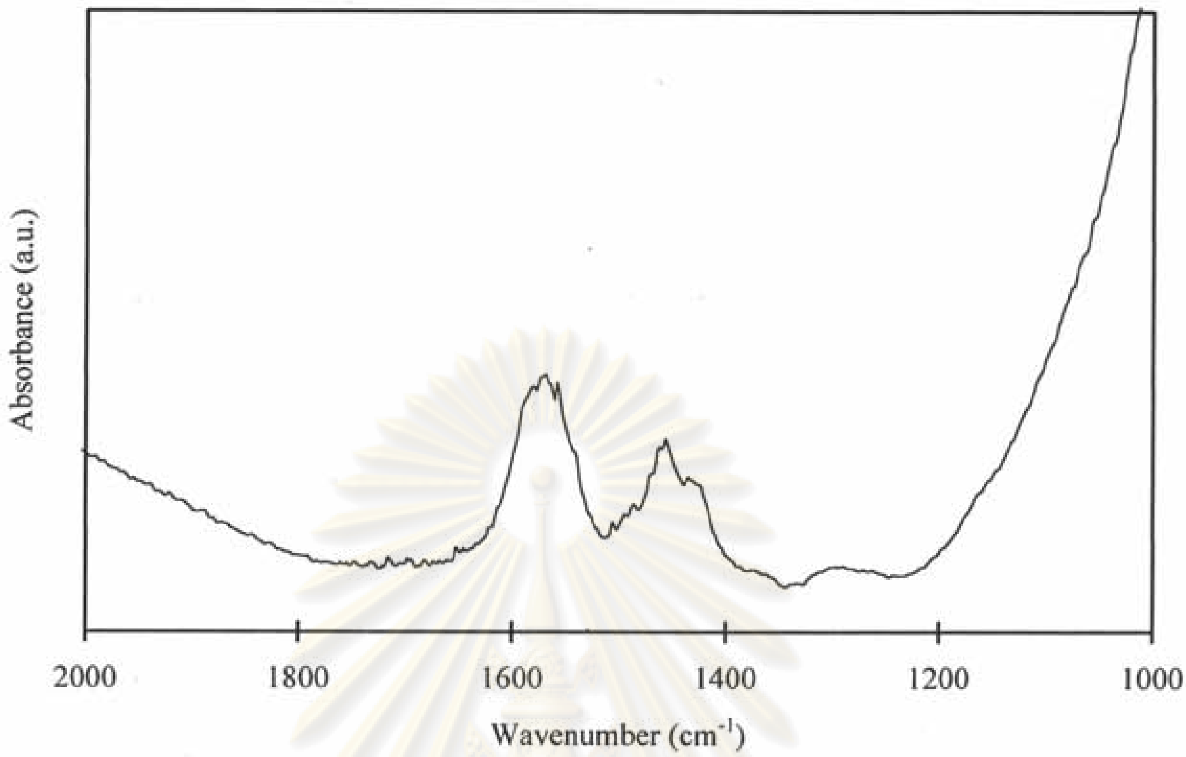

Figure B.4 IR spectrum of 10 min.coked catalyst at 300°C in N_2 atmosphere

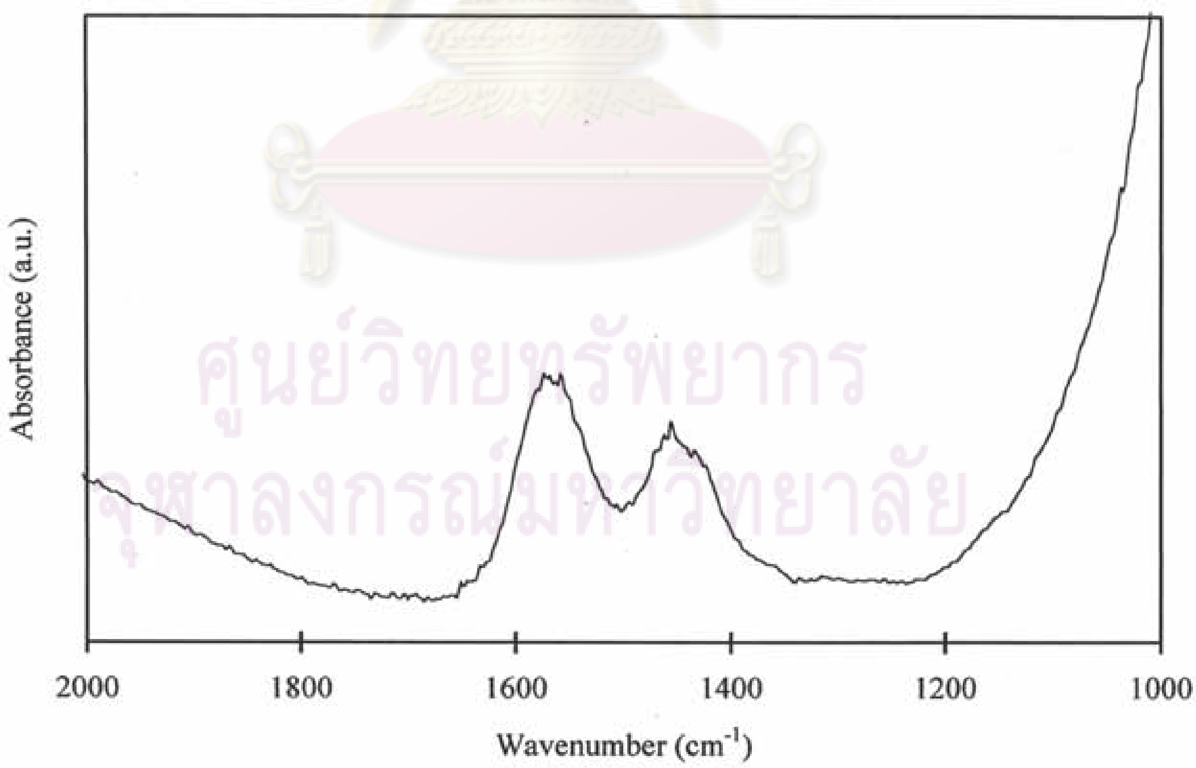

Figure B.5 IR spectrum of 10 min.coked catalyst at 400°C in N_2 atmosphere

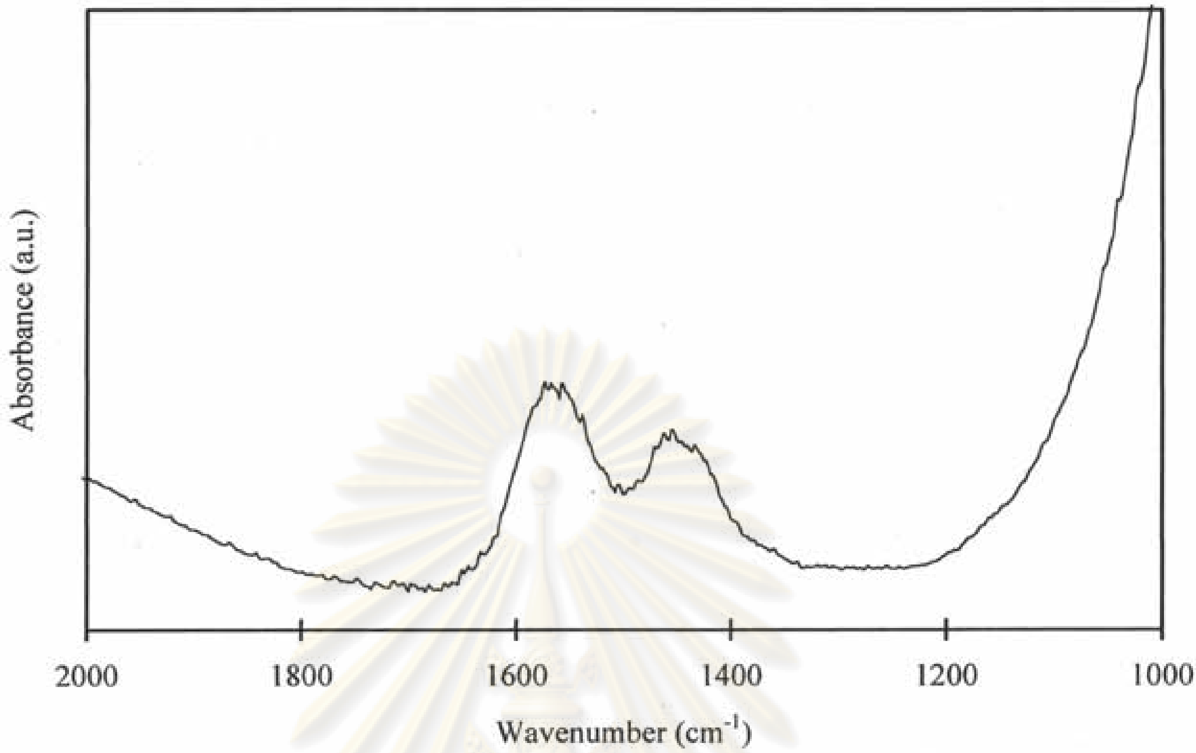

Figure B.6 IR spectrum of 10 min.coked catalyst cooled down to room temperature in N_2 atmosphere

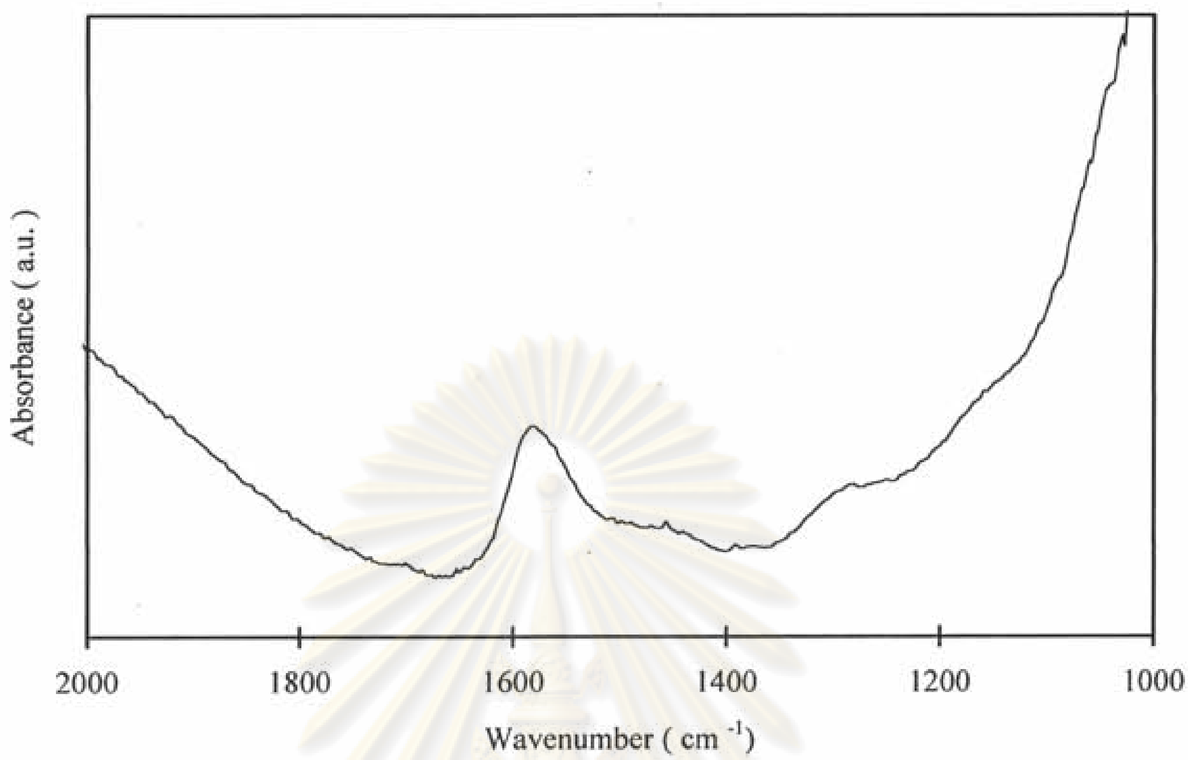

Figure B.7 IR spectrum of 30 min.coked catalyst at 300°C in N_2 atmosphere

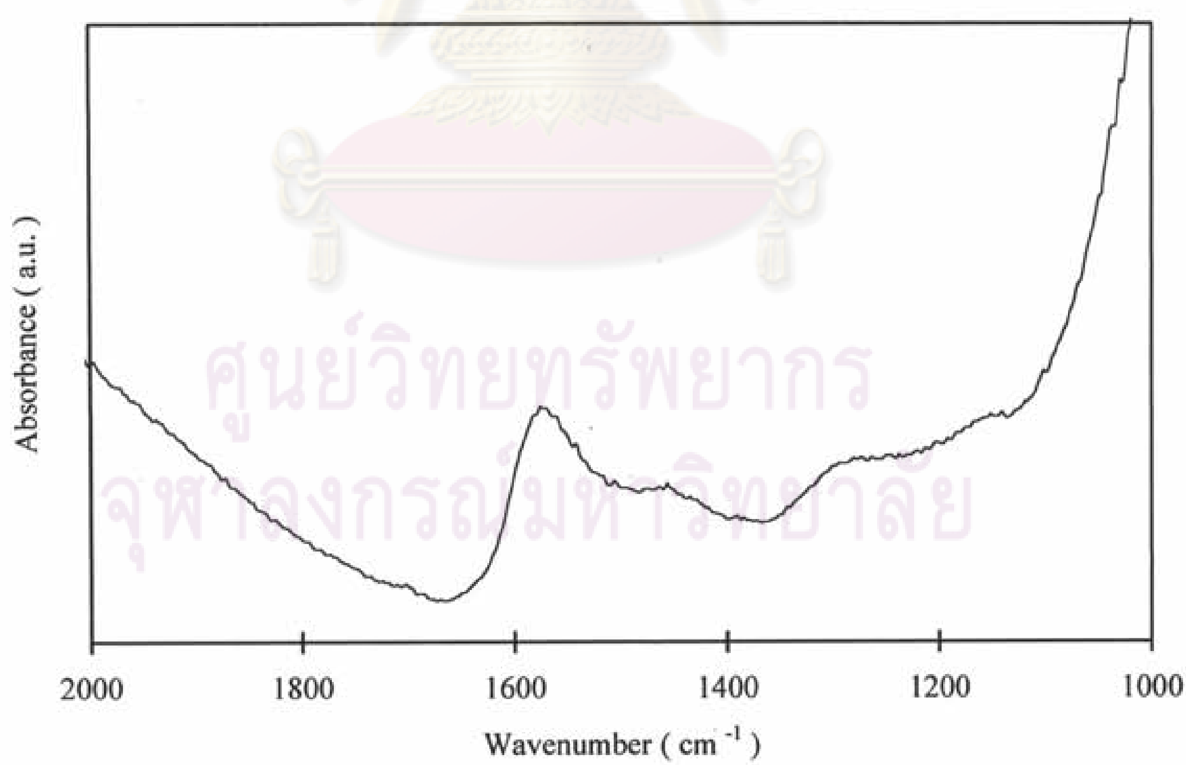

Figure B.8 IR spectrum of 30 min.coked catalyst at 400°C in N_2 atmosphere

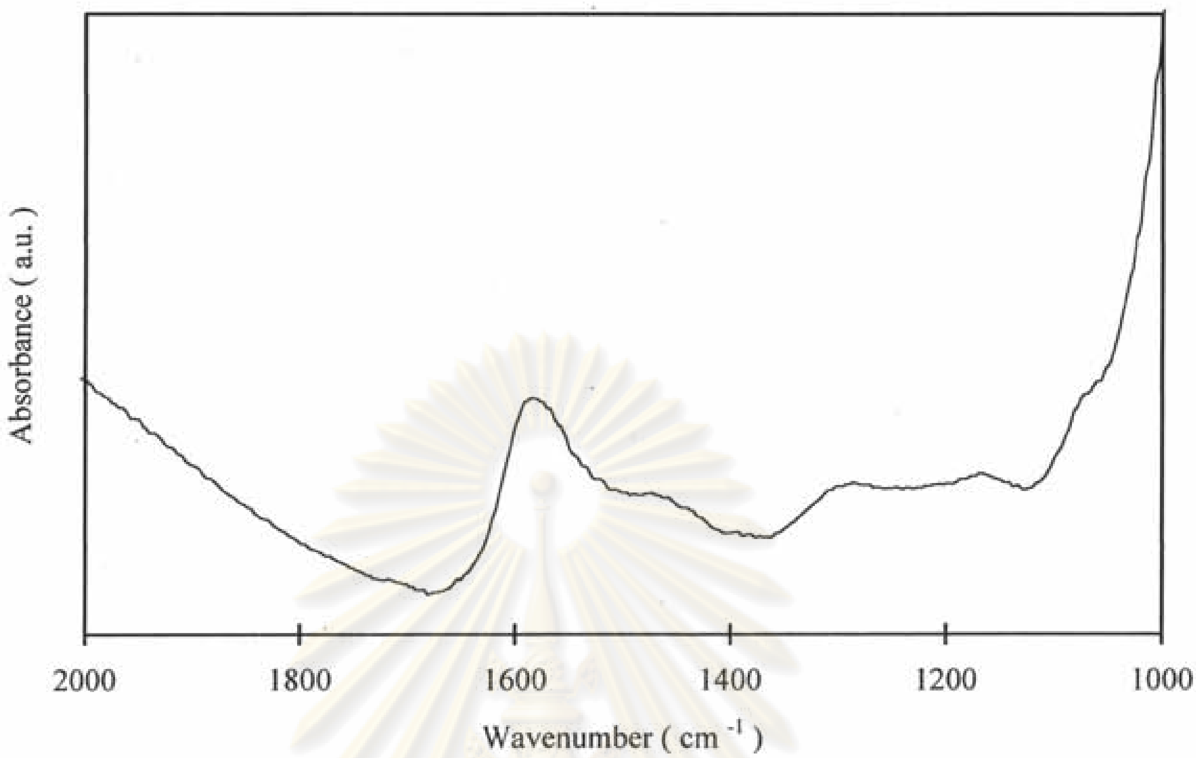

Figure B.9 IR spectrum of 30 min.coked catalyst cooled down to room temperature in N_2 atmosphere

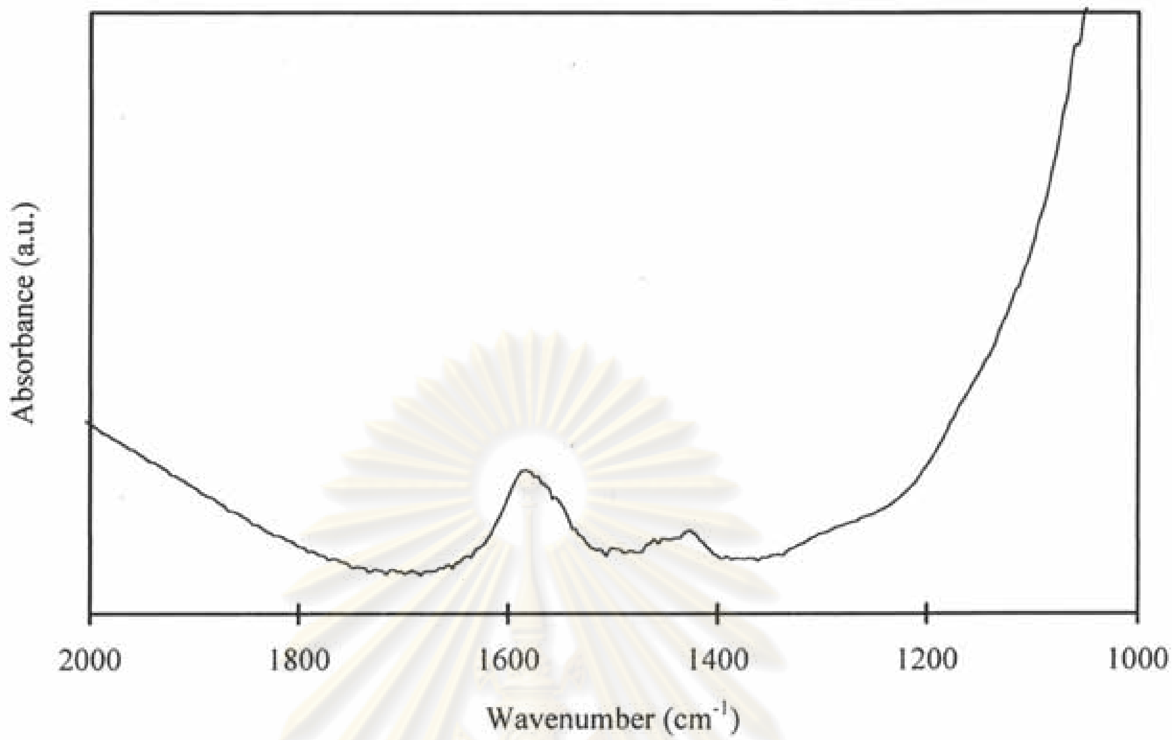

Figure B.10 IR spectrum of 1 hr.coked catalyst at 300°C in N_2 atmosphere

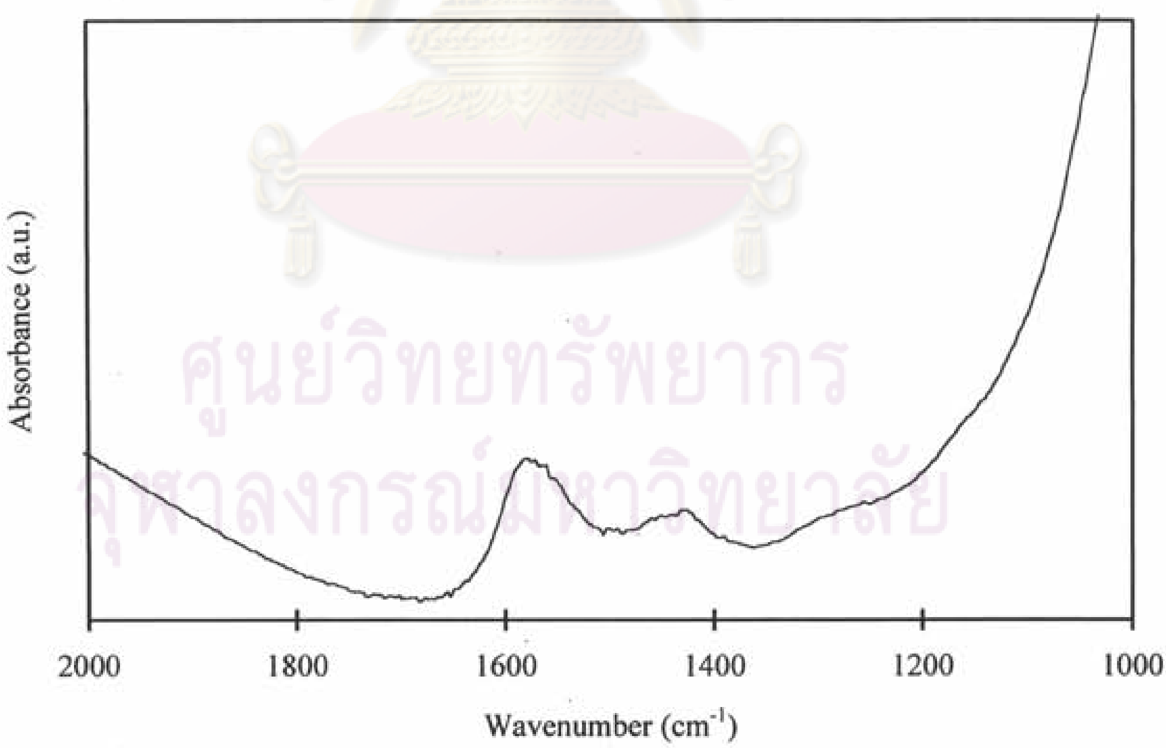

Figure B.11 IR spectrum of 1 hr.coked catalyst at 400°C in N_2 atmosphere

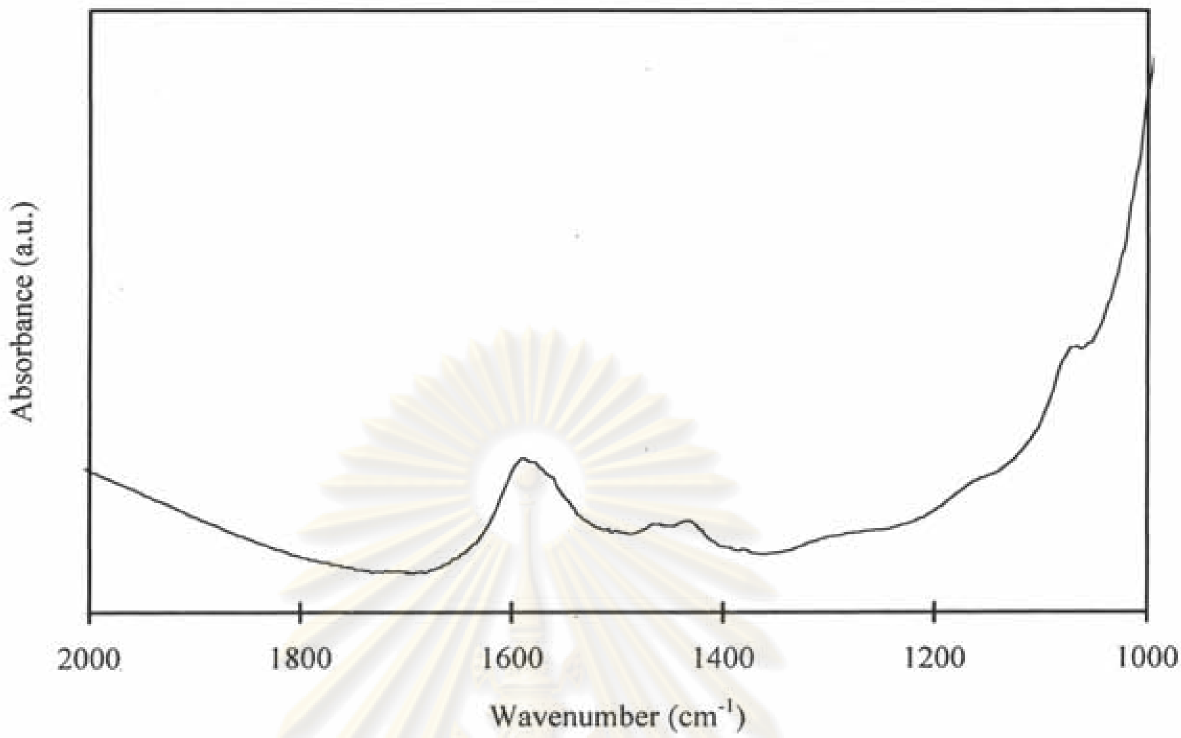

Figure B.12 IR spectrum of 1 hr.coked catalyst cooled down to room temperature in N_2 atmosphere

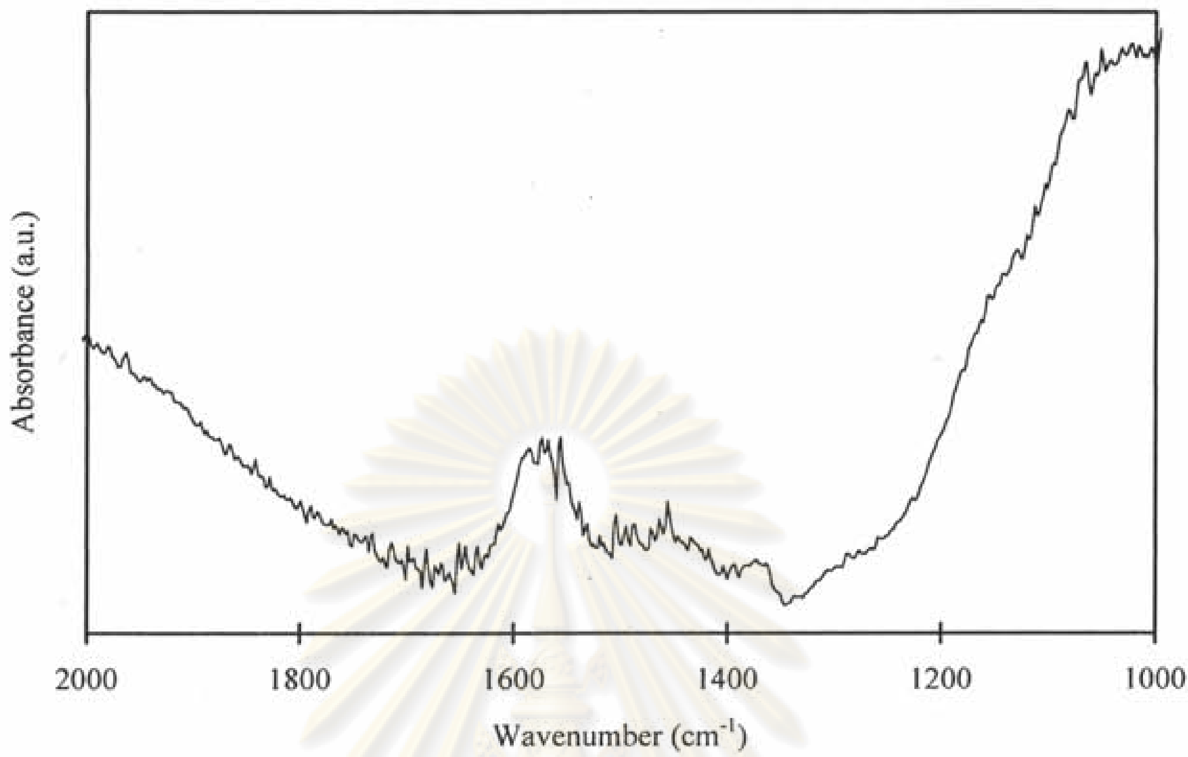

Figure B.13 IR spectrum of 2 hr.coked catalyst at 300°C in N_2 atmosphere

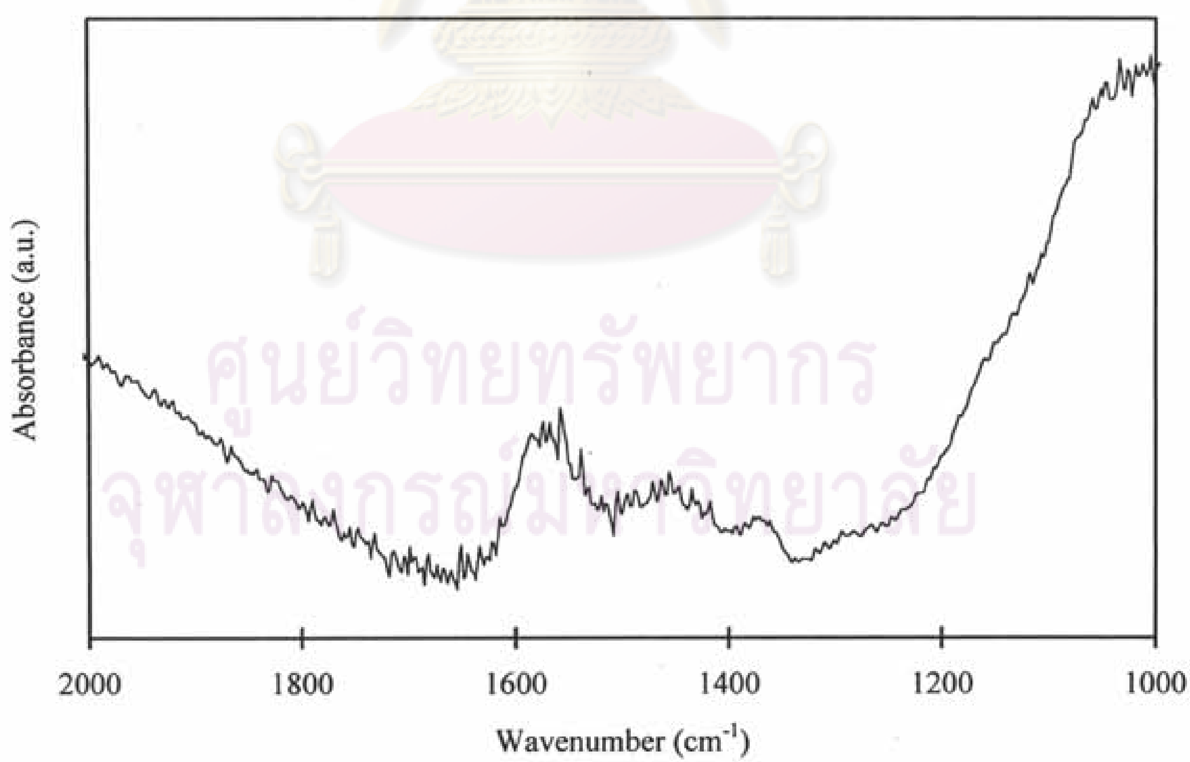

Figure B.14 IR spectrum of 2 hr.coked catalyst at 400°C in N_2 atmosphere

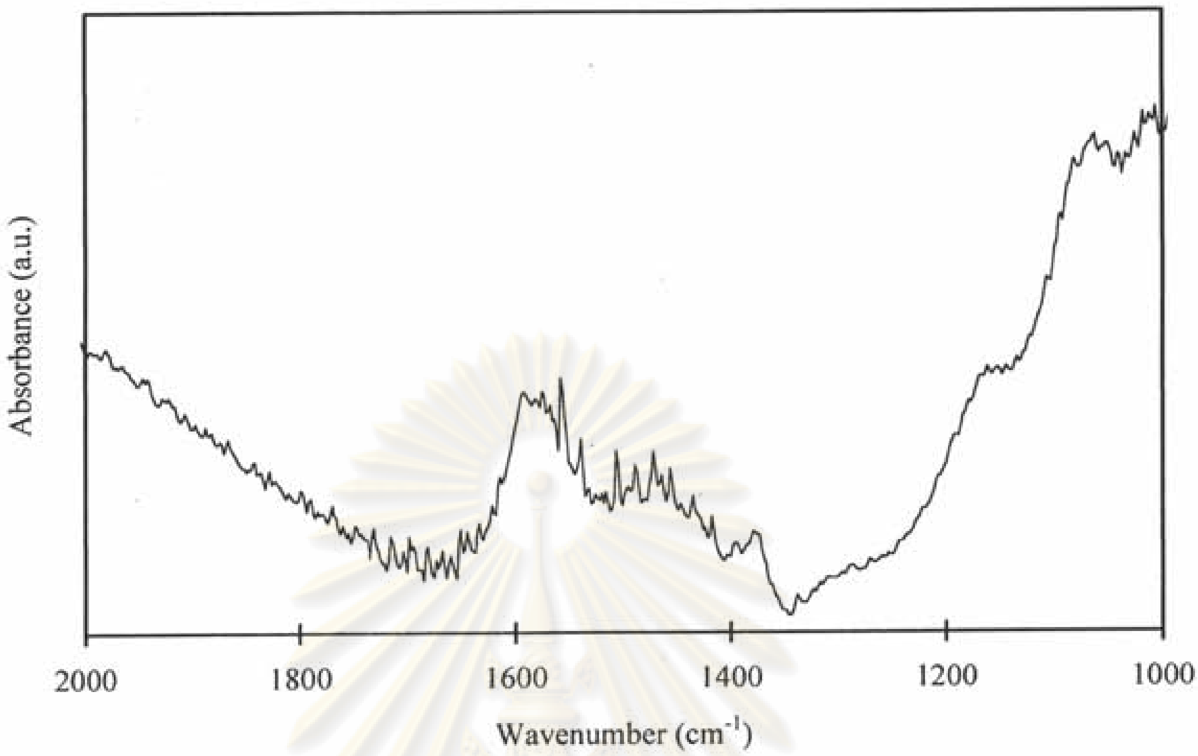

Figure B.15 IR spectrum of 2 hr.coked catalyst cooled down to room temperature in N_2 atmosphere

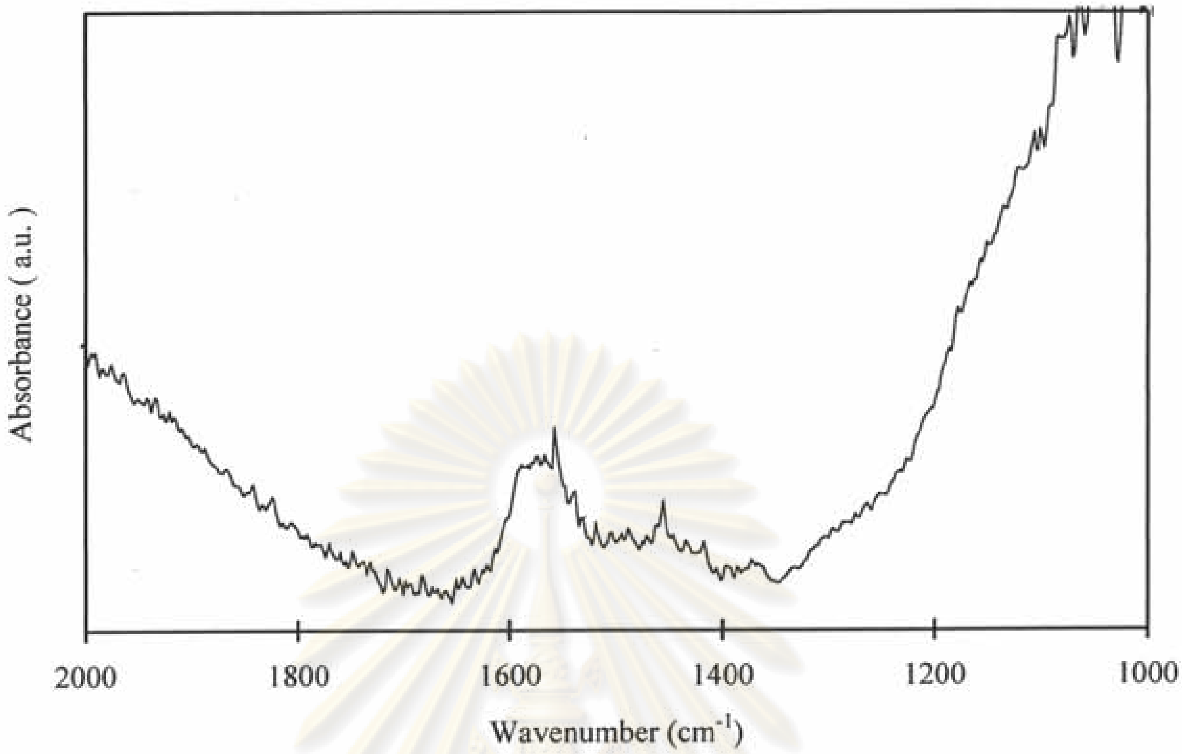

Figure B.16 IR spectrum of 4 hr.coked catalyst at 300°C in N_2 atmosphere

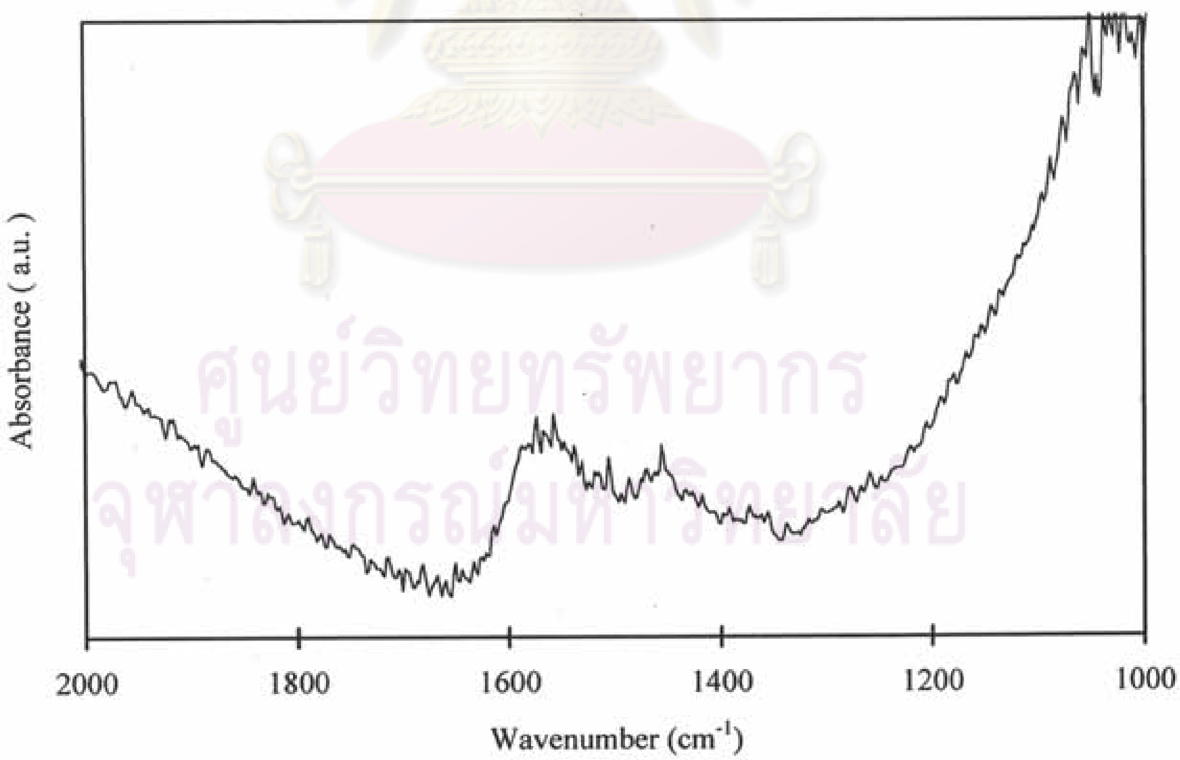

Figure B.17 IR spectrum of 4 hr.coked catalyst at 400°C in N_2 atmosphere

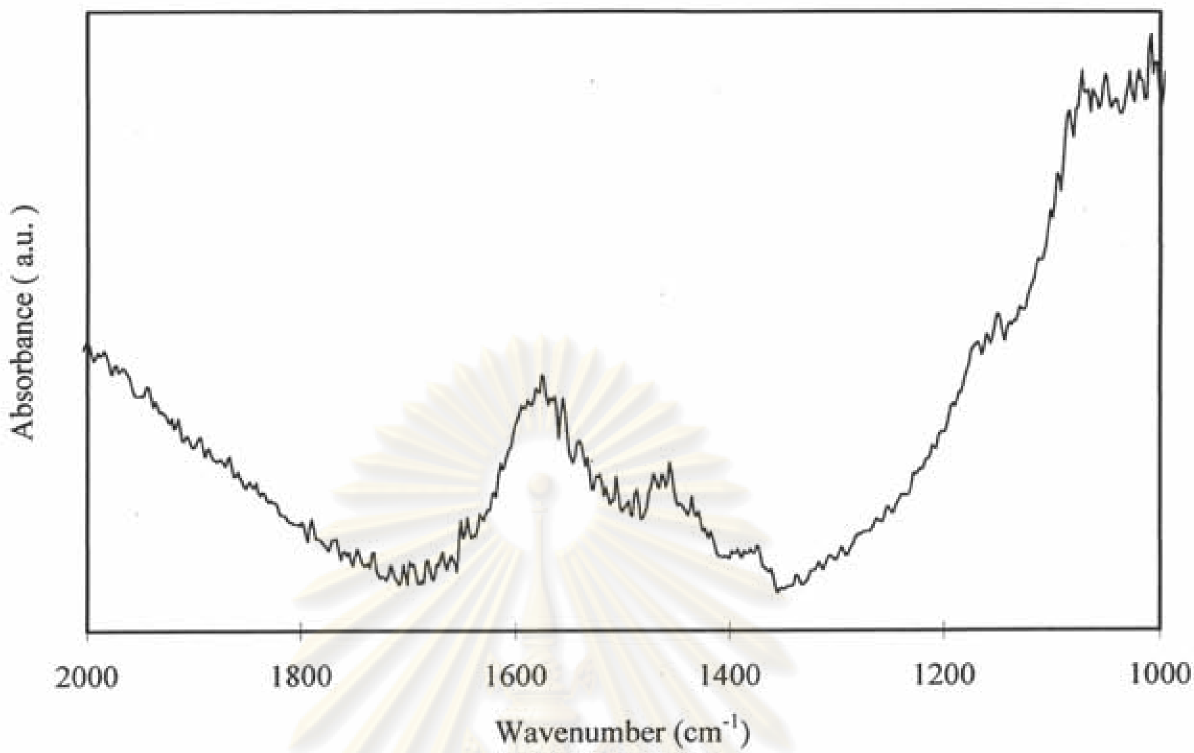

Figure B.18 IR spectrum of 4 hr.coked catalyst cooled down to room temperature in N_2 atmosphere

VITA

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