

CHAPTER III

RESULTS AND DISCUSSION

3.1 Characterization of Benzoxazine Monomer

3.1.1 *Size Exclusion Chromatography (SEC)*

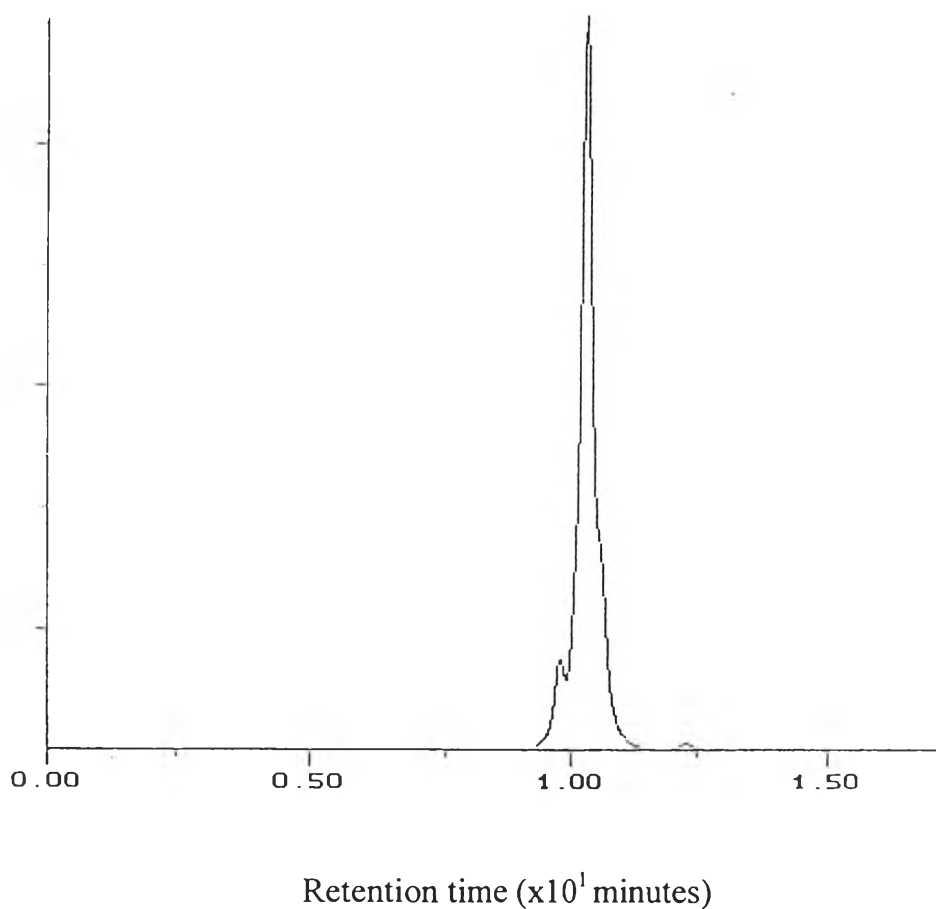


Figure 3.1 SEC chromatogram of purified B-a.

Figure 3.1 represents the SEC chromatogram of purified B-a. The weak peak with shorter retention time (9.5 min) is assigned to the oligomeric benzoxazine. The stronger peak at 10.5 min retention time is assigned to the difunctional benzoxazine monomer. The percentage of B-a monomer from

the area under the peak is 98.6 % so the purified product has almost 100 % monomer.

3.1.2 Fourier transform infrared spectroscopy (FTIR)

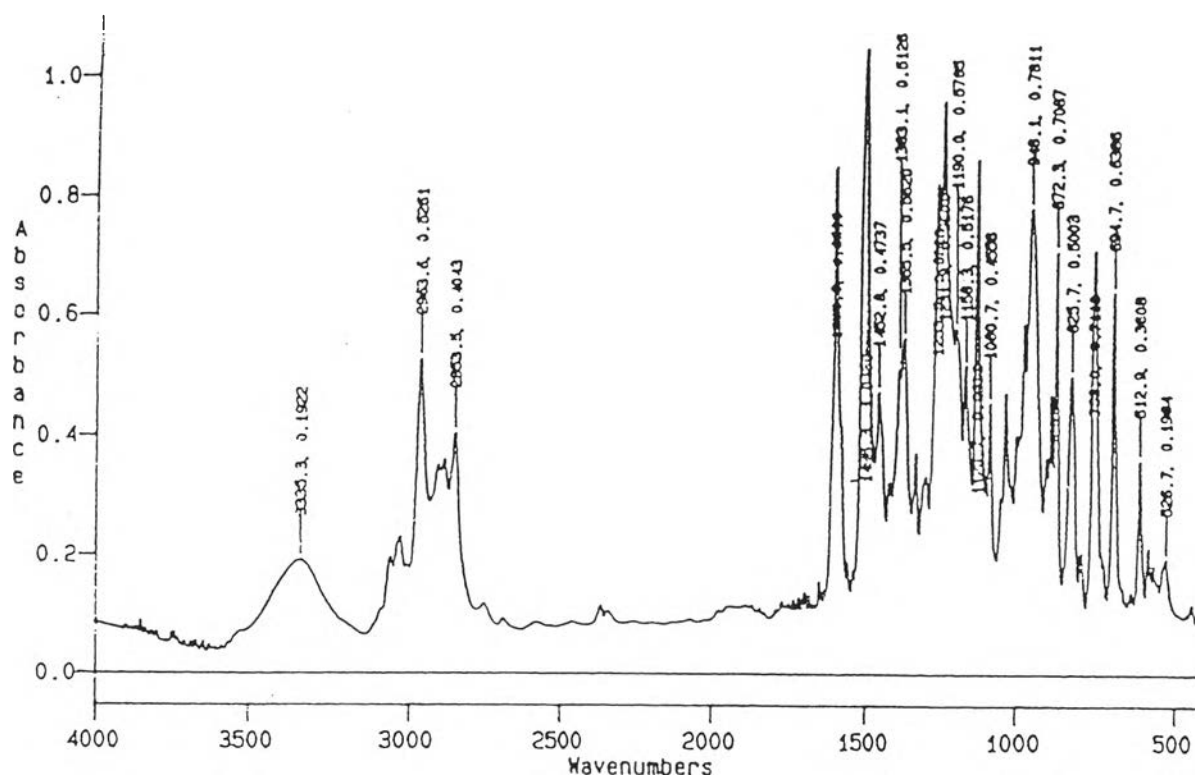


Figure 3.2 FTIR Spectrum of as synthesized B-a.

FTIR spectrum of as-synthesized B-a is shown in Figure 3.2. The broad band around 3500 cm^{-1} is the OH stretching of free phenolic structure in dimers and oligomers.

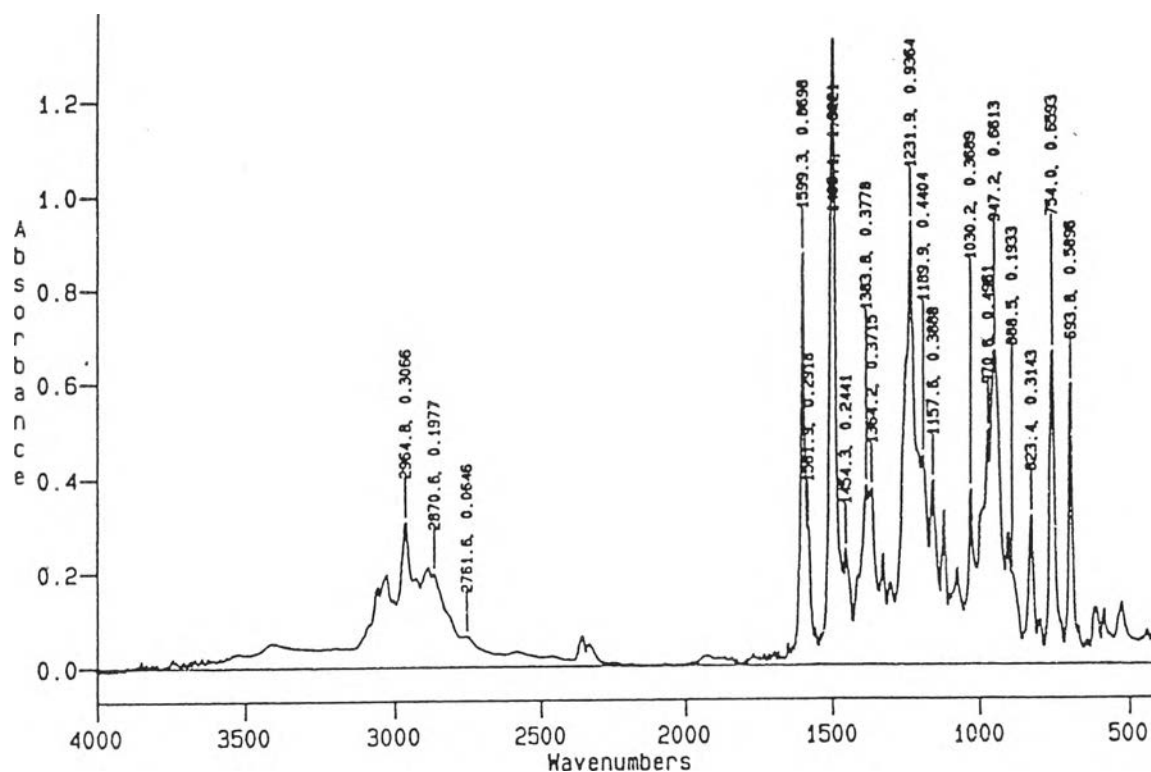


Figure 3.3 FTIR Spectrum of purified B-a.

Figure 3.3 represents the FTIR spectrum of purified B-a. The band around 3500 cm^{-1} has almost disappeared. The C-H stretching region can be found from $3100\text{-}2700\text{ cm}^{-1}$. The tri-substituted benzene ring of B-a monomer is observed at 1496 cm^{-1} . The antisymmetric and symmetric C-N-C stretching modes are found in the region from $1240\text{-}1020\text{ cm}^{-1}$ and $830\text{-}740\text{ cm}^{-1}$ respectively. The region around $1230\text{-}1070\text{ cm}^{-1}$ is assigned to

antisymmetric C-N-C stretching modes. The region around 750-700 cm^{-1} is assigned to C-H out of plane mode.

3.2 Mechanical Testing

The results of shear strength testing of benzoxazine adhesives are shown in Table 1, Table 2 and Figure 3.4, 3.5 and 3.6.

Table 1 Average shear stress, shear strain and shear modulus of B-a adhesive and B-a mixed with ATBN tested at room temperature

% ATBN	Avg.shear stress (MPa)	Avg.shear strain (cm/cm)	Avg.shear modulus (MPa)
0	5.78	0.027	214.07
2	7.40	0.064	115.63
5	8.21	0.065	126.31
10	5.58	0.041	136.10
15	5.51	0.056	98.39
20	4.37	0.028	156.07

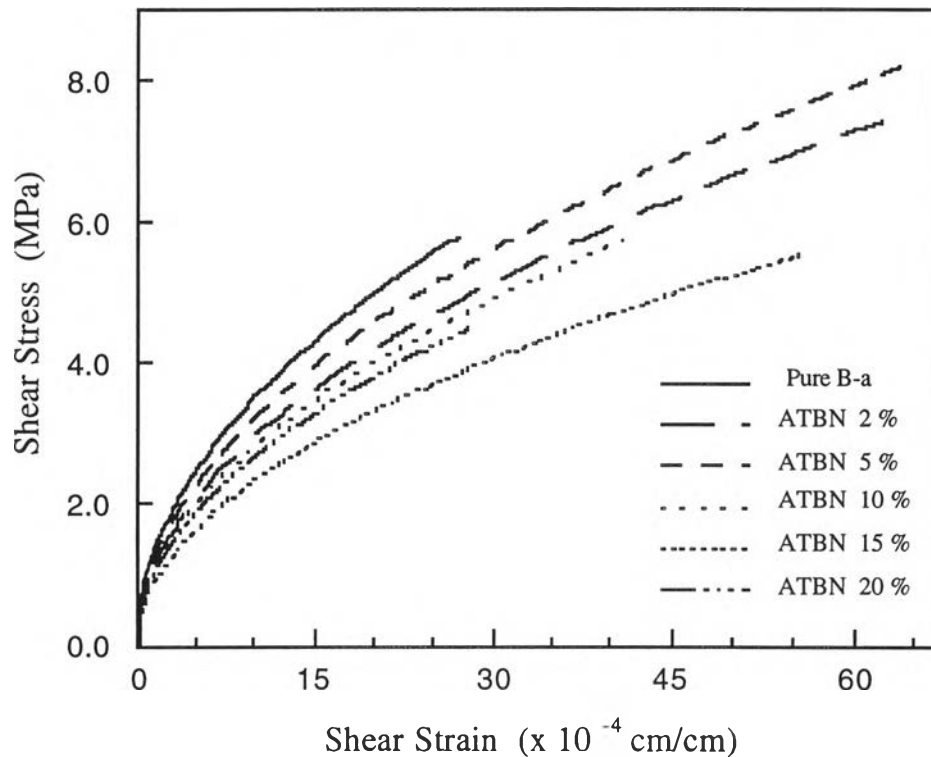


Figure 3.4 Shear Stress - Strain curves of pure B-a and B-a mixed with ATBN.

The shear strength of unmodified epoxy adhesive for aluminium ranges from 4.82-13.8 MPa, while that of B-a is 5.87 MPa [Shields, J.(1987)]. The shear strength of B-a can be improved by about 30-40 % by incorporating small amount of ATBN (2-5 %) as shown in Figure 3.5. The 5 % ATBN gives the maximum strength at 8.21 MPa. The shear strength of B-a adhesive is in the range of epoxy adhesive so it can be a candidate to epoxy for bonding aluminum. However greater amount of ATBN decreases the adhesive strength. This trend is also observed for B-m adhesive.

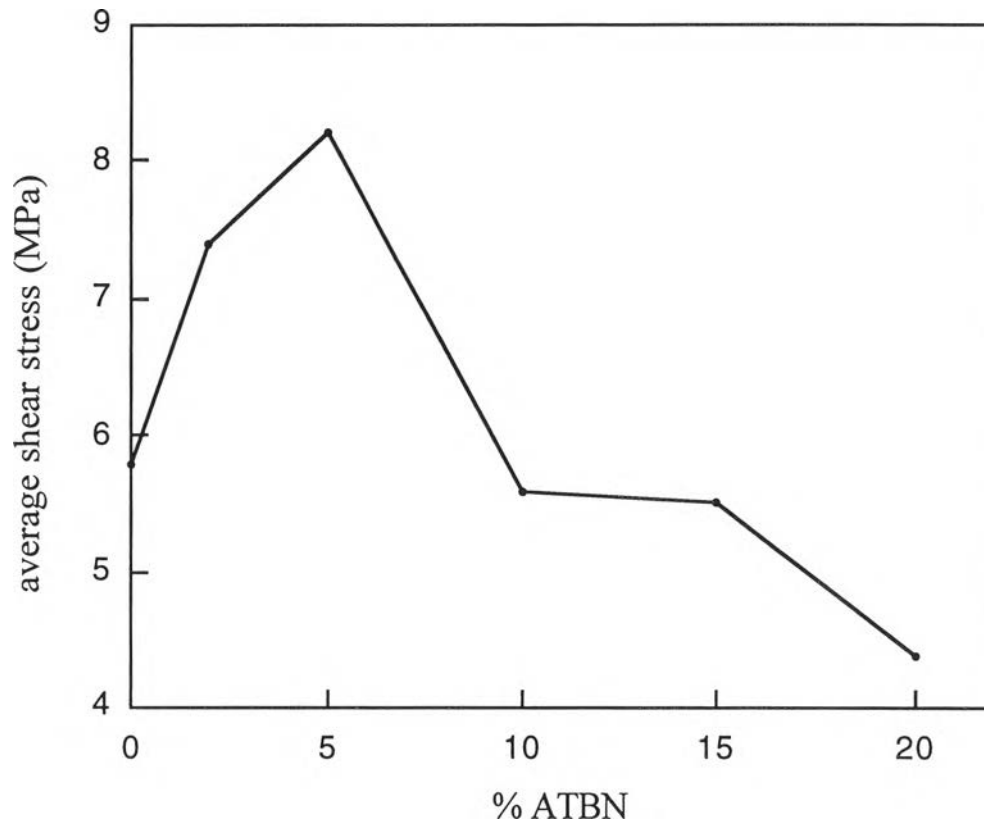


Figure 3.5 % ATBN vs average shear stress of B-a.

The ATBN acts as a flexibilizer in adhesive. The presence of flexibilizer produces a loose network which allows a greater degree of segment mobility. The enhanced mobility increases the ability of the system to relax the internal stress that may have developed upon curing [Wu, S (1982) and Edward, W.T. and Raymond W.S. (1985)]. The large amount of ATBN leads to weaker bond strength in adhesive joints because the rubber component causes the slippery chain and lowers the strength.

Table 2 Average shear stress, shear strain and shear modulus of B-m adhesive tested at 150 °C and B-m mixed with ATBN tested at room temperature

%ATBN	Avg.shear stress (MPa)	Avg.shear strain (cm/cm)	Avg.shear modulus (MPa)
1) 150°C			
0	9.51	0.006	1585.00
2) Room temp.			
0	8.18	0.007	1168.57
2	10.23	0.028	365.38
5	12.58	0.030	419.33
10	6.05	0.023	263.04
15	5.36	0.015	357.33
20	5.12	0.014	365.71

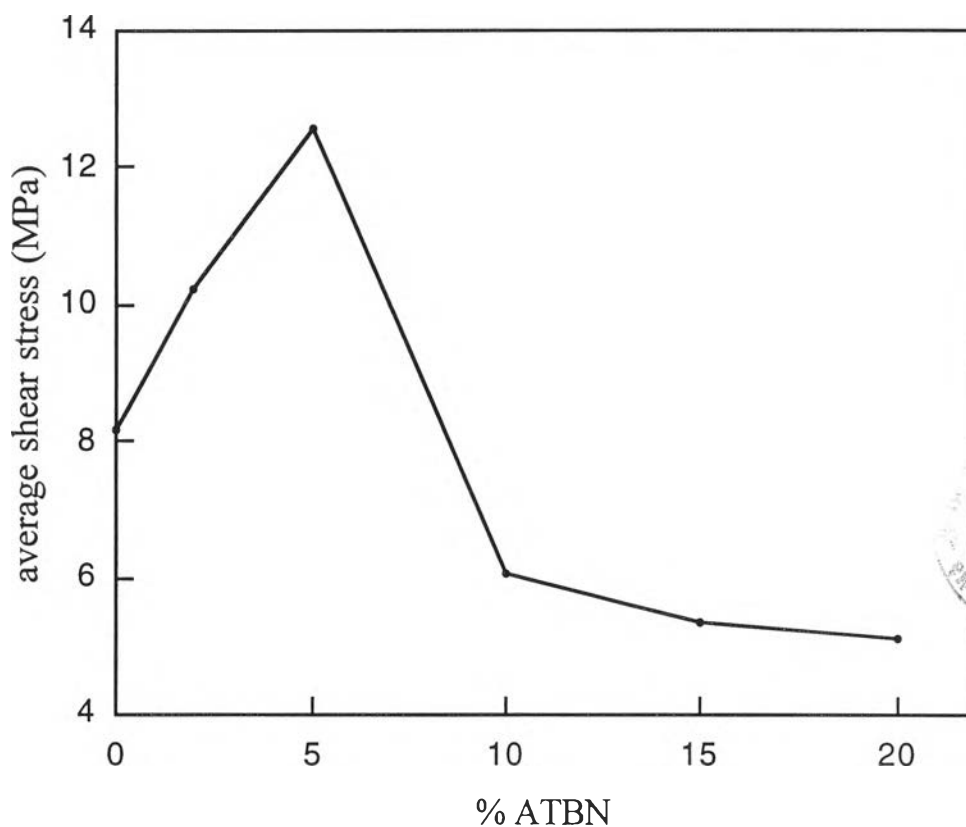


Figure 3.6 % ATBN vs average shear stress of B-m.

The shear strength of B-m at 150 °C is slightly higher than that of B-m at room temperature. The incorporation of small amount of ATBN (2-5 %) improves the shear strength of B-m adhesive by 25-40 %. However greater amount of ATBN also decreases the adhesive strength.

The shear strength of B-m adhesive is higher than that of B-a adhesive because B-a has more benzene rings in its structure than B-m. The benzene rings impart brittleness to the adhesive.

The high strength of B-m at 150 °C shows that B-m can be used as an adhesive at high temperature.

3.3 Corrosion Investigation

Humidity test was made for B-a coated Cu plates at 80 °C for 24, 48, 72 and 96 hrs. The results were compared with bare Cu plate. The corrosion product of bare Cu in humidity is Cu₂O. The assignment of Cu₂O in FTIR spectrum is 650 cm⁻¹ [Ishida, H and Johnson, R (1986)]. Therefore, the corrosion of Cu can be monitored by observing the band at 650 cm⁻¹. The FTIR-RAS spectra of bare Cu after exposure to humidity at 80 °C for 24, 48, 72 and 96 hrs are shown in Figure 3.7.

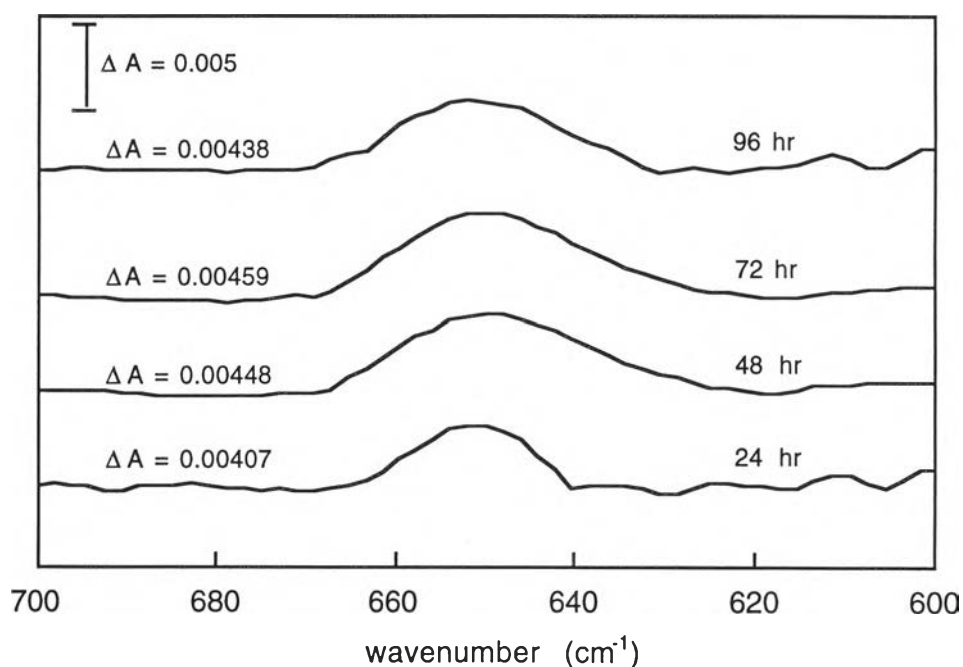


Figure 3.7 FTIR-RAS spectra of bare Cu after exposure to humidity at 80 °C.

Bare copper is corroded in the presence of water after exposure for 24 hr because the strong peak of Cu₂O is observed at 650 cm⁻¹. The more exposure time, the more bare Cu is corroded. The evidence is that the

intensity of Cu_2O at 650 cm^{-1} increases with the exposure time. Figure 3.8 shows the FTIR-RAS spectra for B-a coated Cu plates.

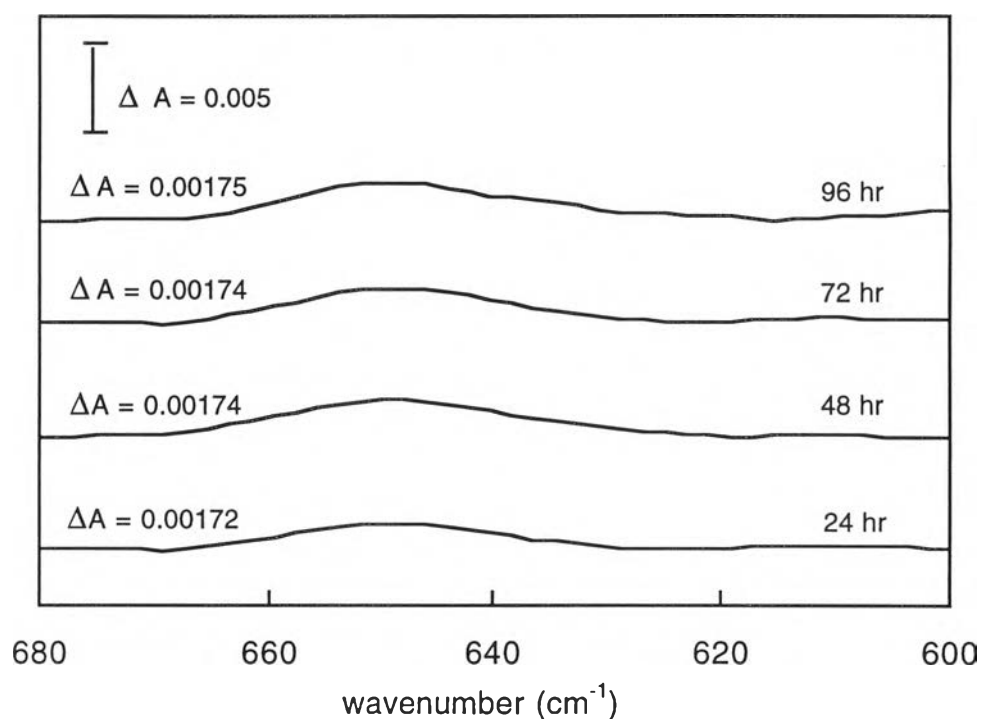


Figure 3.8 FTIR-RAS spectra of B-a coated Cu after exposure to humidity at 80 °C.

After 24 hours, the peak at 650 cm^{-1} appears very weak compared to the peak of cuprous oxide on bare copper at the same time. Furthermore, the intensity of cuprous oxide changes very slightly after 24 hours, so copper is protected from corrosion in humid environment when it is coated with B-a.