

CHAPTER V

CONCLUSION

5.1 Conclusion

Conducting electrospun fibers with diameters ranged from 400-700 nm have been successfully produced using the electrospinning process by blending of the conducting polymer, polyaniline, and polyvinyl alcohol. Polyaniline base (PANi-EB) as substrate was synthesized by chemical oxidation polymerization in the acid condition. Citric acid was used to dope the PANi-EB and to cross link the PVA. Synthesized PANi-EB and PANi doped citric acid (PANi-citric acid) were characterized by Fourier-Transform Infrared Spectrometer (FT-IR). Results indicated that PANi-citric acid was conductive form and was in the form of high doped emeraldine salt.

The morphology of electrospun fibers which were via electrospinning processing, were investigated by Scanning Electron Microscope (SEM). The morphology and diameter of the obtaining fibers were strongly affected by the flow rate, the diameter of needle, the electric potential and the distance between the needle and the collection screen. The electrospun citric acid doped PANi-PVA fibers with regular, straight, uniform diameter fibers, low diameter distributions and without bead defect were prepared through optimizing electrospinning parameters: the flow rate at 15 $\mu\text{l}/\text{min}$, diameter of needle at 0.55 mm, the electric potential at 15 kV and distance between the needle and the collection screen at 25 cm. The average diameter of electrospun fibers was 589 nm.

Electrospun fiber had large surface area and high porosity to interact with gas analyte. The resistance of electrospun citric acid PANi-PVA mats was immediately decreased when exposed to ammonia gas which is unusual for ammonia sensing by conventional polyaniline. Although, the exact mechanism of sensing was not very

clear at this stage, the decreasing of resistance on exposure to ammonia could be probably the interaction of NH_3 with the dopant citric acid. The proposed mechanism of this phenomenon was the competition of two available sites for ammonia attraction into electrospun citric acid PANi-PVA mats, the proton at PANi chain and proton at carboxylic group of citric acid. The first site led to a resistance increase and the latter site led to a resistance decrease. The predominant of the latter resulted in the overall resistance of electrospun citric acid PANi-PVA mats on the ammonia gas exposure. The mechanism of such an effect lies in the reversible protonation of PANi molecules under ammonia removal. The optimized concentration of PANi was 2% wt/v because the increased percent of PANi did not change the viscosity of polymer solution in electrospinning process, hence, the morphology of electrospun fibers observed at various percent of PANi were similar. For ammonia sensing, the resistance of electrospun mats at varied percent of PANi did not significantly change. The optimized citric acid doped was 1.2 g because initial and final resistance was clearly different, which resulted in good sensitivity and efficient sensing of ammonia gas. The variation of solvents such as chloroform and acetone was studied to compare with NMP. Fibrous structures obtained from chloroform were uniform in diameters, straight and also their change of resistance was higher than those from NMP and acetone. However, the initial resistance of PANi in NMP was higher than that from using chloroform because mostly undissolved PANi in chloroform. Therefore, concentrations of PANi after filtration were not real and will have influence on measured electrical resistance. Therefore, the NMP solvent was selected for ammonia sensing. This electrospun mat showed stable, good repeatability and reversibility of ammonia gas sensing. The response time and recovery time were 5 and 10 minutes, respectively. The responses of repeated exposure and removal of ammonia gas were reproducible up to 12 cycles. Besides, the change of resistance of various samples resulted in a good precision. In the presence of 1-100 ppm of ammonia gas, the changes of resistance were well related to the concentration of ammonia gas. The resistance of electrospun mat was compared with a cast film of the same polymer condition. It was found that the initial resistance (R_0) of the electrospun PANi-PVA mat was lower than that of PANi-PVA cast film at the same polyaniline concentration. The result showed that the conductance of electrospun mat was better

than that of cast film. The change of resistance of electrospun mat was higher and also the response time was faster than those of cast film. The electrospun mat sensor was applied to gaseous sample and the result was compared with the OLDHAM Mx2100 sensor. The ammonia gas concentration of sample using electrospun mat sensor of site 1 and site 2 were 2 ppm and 4 ppm, respectively. The ammonia concentration obtained from electrospun mat sensor was similar to the OLDHAM Mx2100. Therefore, PANi-PVA electrospun mat showed a good performance as a sensing material for ammonia gas.

We have successfully developed an ammonia gas sensor based on a sensitive layer made of conducting polymer by electrospinning which is sensitive, stable, reversible, low cost, fast in response, and easy to regenerate. Moreover, this new sensor is suitable for use in chemical industries.

5.2 Suggestion of future work

As the initial resistance of material was too high, it was difficult to measure and observe the change of resistance. The advance multimeter should be performed for the future experimental. Many factors can influence the morphology of fibers such as molecular weight of polymer, surface tension, collector geometry, etc. Hence, these important parameters will be studied during electrospinning process development. Moreover, it is well known that interaction with water vapor also decrease the resistance of PANi and other conducting polymers. Thus, humidity surrounding a sensor will be influent the ammonia detection capability of electrospun PANi-PVA mats. Consequently, humidity is a parameter that must be dealt within the process of sensor developing.