CHAPTER V

CONCLUSIONS

In this research, the homemade FI-colorimetric and HSI-spectrophotometric methods were designed, constructed and developed to determine ethanol.

The FI-colorimetric system was designed and developed for determination of ethanol in distilled liquor. The reactions used for this system are based on two simple redox reactions. First, ethanol reduces the potassium permanganate in sulfuric acid solution and decrease in color intensity of permanganate was monitored using green-LED light source and then the positive-FI peaks were appeared under the light emitting principle of LED colorimetric detection. Second, ethanol reduces the potassium dichromate in sulfuric acid solution and increase in color intensity of chromium-(III) was monitored using red-LED light source and then the negative-FI peaks were also showed under the same principle. For an acidic potassium permanganate reagent solution, it was found that 0.2 mmol/L of $KMnO_4$, 0.25 mol/L of H_2SO_4, 1.0 mL/min of flow rates of both reagent and carrier streams, 140 cm of reaction coil length and 90 μL of sample volume were selected. Because of the instability of this reagent by using the batch spectrophotometric procedure, it could be used and operated this permanganate reagent within three hour (180 min). Under optimum conditions, a linear calibration graph in the range of 10-40 %v/v ethanol ($y = 0.0492x-0.1474$, $r^2 = 0.9994$, %RSD = 1.7-3.7) was obtained. The detection limit was 1.2 %v/v ethanol. The sample throughput was 72 injections per hour. For an acidic potassium dichromate reagent solution, it was found that 0.15 mol/L of $K_2Cr_2O_7$, 4.0 mol/L of H_2SO_4, 1.0 mL/min of flow rates of both reagent and carrier streams, 200 cm of reaction coil length and 90 μL of sample volume were selected. Under optimum conditions, a linear calibration graph in the range of 4-8 %v/v ethanol ($y = 0.737x-0.9481$, $r^2 = 0.9978$ and % RSD = 1.4-2.5) was obtained. The detection limit was 0.3 %v/v ethanol. The sample throughput was 80 injections per hour. The proposed system, with two reagent solutions and two redox reactions used, was successfully applied for analysis of ethanol in Thai white distilled liquor samples and results

obtained agree well with those obtained by other methods of FI-spectrophotometric, AOAC redox titration and micro-scale potentiometric redox titration (t-test, 95% confidence). This proposed system was simple, convenient, automation, low cost of instruments, low chemical consumption, low sample consumption, precise and accurate.

The HSI-spectrophotometric system with on-line gas diffusion unit was designed and developed for determination of ethanol in alcoholic beverages (i.e. beer, wine and distilled liqour) by using a simple redox reaction of ethanol and potassium dichromate in sulfuric acid solution. Reduction reaction of potassium dichromate to chromium-(III) with ethanol is spectrophotometrically monitored at 600 nm. By incorporating a gas diffusion unit prior to the HSI-spectrophotometric system, most of the interferences could be eliminated. Under optimum conditions, a linear calibration graph in the range of 2–10 %v/v ethanol ($y = 0.3041x-0.1674$, $r^2 = 0.999$, %RSD = 0.7-2.3) was obtained. The detection limit was 0.4 %v/v ethanol. The sample throughput was 15 injections per hour. The proposed system was successfully applied for determination of ethanol in some commercial alcoholic beverage samples and results obtained agree well with those obtained by other methods of FI-spectrophotometric, AOAC redox titration and micro-scale potentiometric redox titration (t-test, 95% confidence). Advantages of this system were simple, semi-automation, low chemical consumption, low sample consumption, precise and accurate.

To enhance the analysis efficiency, these two systems of FI-colorimetric and HIS-spectrophotometric will be applied for determination of other analytes and other samples.