CHAPTER V

CONCLUSIONS

This research work, studied the application of thermoreactor and UV digestion for NR latex preparation before the determination of the total phosphorus by UV-Vis spectrophotometry and digital image-based colorimetry coupled with artificial neural networks (DIC-ANNs). The conclusions achieved in this study are discussed in the following sections.

Oxidizing agents were studied in order to find the optimum oxidizing agent for the decomposition of biological matrices in NR latex by using a standard thermoreactor. Ammonium peroxodisulphate and potassium peroxodisulphate were used as oxidizing agents and investigated for NR latex digestion. The digestion efficiency of both oxidizing agents shows no statistical difference. However, solubility of ammonium peroxodisulphate is higher and it is cheaper than that of potassium peroxodisulphate. Therefore, ammonium peroxodisulphate was selected to use as oxidizing agent in the next study.

Then, the absorption spectrum of phosphorus with molybdenum blue solution was studied by scanning the wavelength in the range of 400 - 900 nm with UV-Vis spectrophotometer in order to observe the effect of ammonium peroxodisulphate. After digestion by both techniques, the absorption spectra of phosphorus standard solution and phosphorus in NR latex obtained were similar. In addition, the maximum wavelength of all results was at 710 and 880 nm. Because of the higher sensitivity at the wavelength of 880 nm, it was selected in this study.

The concentration of the ammonium peroxodisulphate that was appropriate to react with the matrices in NR latex sample was investigated. The oxidizing agent concentration of 30 g L^{-1} shows high efficiency for NR latex digestion by both methods. Therefore, this concentration was selected for the next study.

The digestion temperature was experimented. The temperature of the thermoreactor was studied in the range of 60 - 150 °C. The results found that the normalized absorbance was observed to increase with increasing the digestion

temperature up to 100 °C after which the normalized absorbance become constant. The good result was obtained at 100 °C. Thus, the temperature at 100 °C by using thermoreactor was chosen for NR latex digestion. For UV digestion unit, the digestion temperature could not be controlled because the temperature increasing followed the heat of the UV lamp. When starting the UV lamp, the temperature is increased from 60 ± 5 °C to 170 ± 5 °C in 50 minutes and was used for NR latex sample digestion. As a consequence, the digestion temperature of UV digestion unit was studied.

The effect of decomposition reaction time providing the completeness of the NR latex digestion was studied. For the thermoreactor, the digestion time was experimented in the range of 10 - 120 minutes. The highest of the normalized absorbance was obtained by using the digestion time at 70 minutes and was chosen as the optimized value. For the UV digestion unit, the digestion time was experimented in the range of 10 - 60 minutes. The best result was obtained at 50 minutes. Thus, it was chosen for the next experiment.

The reaction time in the range of 5 - 60 minutes for color development was studied by testing molybdenum blue reagent with phosphorus standard solution and NR latex sample (without and with added phosphorus standard). It was found that, all colorimetric reactions were stable after 10 minutes. Therefore, the solution left for 10 minutes before phosphorus determination was chosen in this study.

Under the optimum conditions, using thermoreactor and UV digestion unit for NR latex preparation presented good recoveries for total phosphorus determination at 82.28 - 101.4% and 84.67 - 103.3%, respectively. The effect of interfering ions such as arsenate (AsO_4^{3-}), sulfide (S^{2-}), nitrite (NO_2^-), hexavalent chromium (Cr^{6+}) and silicate (SiO_3^{2-}) on the recoveries of total phosphorus were studied. The cations and anions used can interfere with the total phosphorus determination. The tolerance level was found at the concentrations of 0.001, 1.0, 10.0, 1.0 and 1.0 mg kg^{-1}, respectively. The amount of total phosphorus in NR latex sample by using thermoreactor and UV digestion unit and determining with UV-Vis spectrophotometer and digital image-based colorimeter-artificial neural networks (DIC-ANNs) showed no statistically significant difference at 95% confidence level by applying paired t-test.

The comparison of features and performances between thermoreactor and UV digestion unit are shown in Table 12. Both require the same type and the same

concentration of oxidizing agent. UV digestion unit offer more application and lower cost (~10 fold) than thermoreactor, but thermoreactor is more secure and the life time is longer than UV digestion unit. However, UV digestion unit in this work is still more interesting than thermoreactor because using UV radiation assisted with thermal energy provides higher radical generating from oxidizing agent for decomposition reaction than using only thermal energy. Therefore, UV digestion unit is suitable for laboratory and small industrial factory in NR latex digestion before total phosphorus determination.

Table 12 Features and performances comparison between digestion with thermoreactor and UV digestion unit

Parameters	Thermoreactor	UV digestion unit
Oxidizing agent concentration (g L^{-1})	30	30
Digestion temperature (°C)	100	gradient (60-170)
Digestion time (minutes)	70	50
Reaction time (minutes)	10	10
Number of sample (per batch)	14	10
Capital price (Bath)	44,000	4,500

Suggestions for future works

As for further works, the two digestion method for NR latex pretreatment could be applied to other chemical residues determination e.g. copper, manganese or magnesium.

UV digestion unit could be improved on increasing the number of sample per batch (more than 10 samples).

The lifetime of UV lamp is relatively short. In addition, UV digestion unit could not be used continuously for the digestion in order to prevent the overheat in the UV digestion unit.

It could be emphasized that the developed procedure is simple, rapid, safety, accurate, precise and low cost which are the main advantages of NR latex digestion techniques for determination of total phosphorus in this work.