

CHAPTER 4

CONCLUSION

For the determination of the total iron content in milled and brown rice samples, the rice samples were prepared by wet digestion with concentrated HNO_3 using microwave digestion and followed by analysis of iron concentration using GFAAS. The main propose of this research are study on the iron extraction from rice samples using suitable extracting solvent and separation Fe^{2+} and Fe^{3+} in rice sample by solvent extraction technique. The parameters affecting for the extraction Fe were optimized which included extraction solvent, the ratio of rice weight per solvent volume and extraction time. From the results, the suitable solvent for extraction of Fe can be ordered into milli-Q water, 0.005M HCl, 0.01M HCl, 0.1M HCl, 1% CH_3COOH , MIBK and CHCl_3 , respectively. So, the milli-Q water was chosen for the Fe extraction method because the form of extractable Fe was not changed.

A method for speciation of Fe^{2+} and Fe^{3+} ions was developed using solvent extraction technique and determination by GFAAS. The complexing reagent for Fe^{2+} were 1-(2-pyridylazo)-2-naphthol (PAN). The Fe^{2+} -PAN complex was extracted into chloroform phase while Fe^{3+} remained in water phase. After the complex of Fe^{2+} -PAN was extracted into chloroform and decomposed by heating of organic solvent followed adding 4.0 ml of concentrated HNO_3 . Then, 2.0 ml of milli-Q water was added and Fe was determined by GFAAS.

The parameters affecting the separation of Fe^{2+} were optimized which included the pH, the extraction time, the PAN amount and the chloroform volume.

The optimized separation conditions were defined as the conditions which yielded the maximum recovery for Fe^{2+} in chloroform phase and the minimum recovery for Fe^{3+} in aqueous phase were obtained. The optimum conditions were pH at 1, extraction time of 15 min, PAN amount of 0.1% in ethanol and chloroform volume of 10 ml.

The accuracy expressed in term of percentage recovery of this method for Fe^{2+} was found in the range of 76.30-114.91%. Limit of detection was 0.29 ppb. The precision expressed in term relative standard deviation was found to be 1.65%. Linearity of Fe was obtained in the range of 10-120 ppb. And the calibration curve of Fe was obtained in the range of 10-100 ppb and given the correlation coefficient (R^2) in the range of 0.9925 -0.9998.

The developed methods were applied for the determination of Fe^{2+} and Fe^{3+} in rice bran samples that harvested from Tung Kula Ronghai, Thailand. From the results, Fe^{2+} contents are determined in the range of 31.60-83.44 ppb and Fe^{3+} was found in the range of 9.68-27.73 ppb in rice bran samples. The extracted Fe was obtained in the range of 41.28-111.17 ppb. The amount of Fe^{3+} was obtained from the calculation because the precipitation was occurred in the sample after adding the reducing agent in the extracted solution for reducing Fe^{3+} to Fe^{2+} form. In ICP-MS method, the results shown that the amount of the extracted Fe was obtained in the range of 50.76-90.53 ppb. So, the results from GFAAS technique were comparable with the results from ICP-MS technique, which can be concluded that both techniques were similarly reported in the amount of the extracted Fe.