Determination of pesticide by near infrared spectroscopy with a new DESIR technique

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Introduction

Determination of pesticides is time consuming and very expensive. Although, near infrared (NIR) spectroscopy has been applied to a large number of analytes in food and agriculture, application of NIR to the determination of pesticides has not been reported previously.

To offer trace level analysis, dry extract spectroscopy by infrared reflectance (*DESIR*) has been used.[1-4] However, in *DESIR*, liquid sample distribution on a glass fibre paper is not even because pipetting is used to apply a small aliquot of the sample solution on to the paper, which may cause errors in NIR measurement. To compensate the mentioned problem, we have developed a new *DESIR* technique with immersion of the filter paper in the analyte solution in place of pipetting.

In this work, the possibility of a new *DESIR* technique for the rapid and inexpensive estimation of a pesticide using acephate in ppm level was investigated.

Materials and methods

Samples

Acephate solutions in the range of 2 to 50 ppm were prepared by dissolve acephate powder (Wako Chemicals, Japan) in water.

The Whatman Glass microfibre filter papers (40 mm diameter) were used as a substrate material.

Preparation of the new *DESIR* technique

DESIR samples were prepared by immersing the respective filter paper in the standard solutions in petri dishes for 1 minute. Initially the filter paper was immersed in the solution for 30 seconds and it was turned up side down and kept immersed for another 30 seconds. Then the excess solution on the filter paper was allowed to drain for 15 seconds with a forcep. Each concentration was prepared in quadruple. The samples were then fixed on to a stainless steel drying frame by double paper clips and were dried with a forced air drying oven "Yamato DN-42" (Yamato Co., Japan) at 60°C for 1 hour.

Spectral acquisition

NIR spectra of the *DESIR* samples were measured with the NIRSystems 6500 spectrophotometer (Foss NIRSystems, USA). A commercially available rotating drawer arrangement was used as a sample holder. A specially-designed sample cell was used to hold the *DESIR* filter paper. A ceramic plate as a reference material was placed in the sample cell behind the filter paper position. The NIR spectra were measured in the long wavelength region of 1100 nm to 2500 nm with 2 nm intervals in reflectance mode. Each spectrum was the average of 50 scans.

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NIR spectrum of acephate powder was measured with the InfraAlyzer 500 spectrophotometer (Bran+Luebbe GmBH, Germany). A commercially available aluminum sample cup (2 mm sample thickness, 20-mm diameter) was used as a sample cell.

Results and discussions

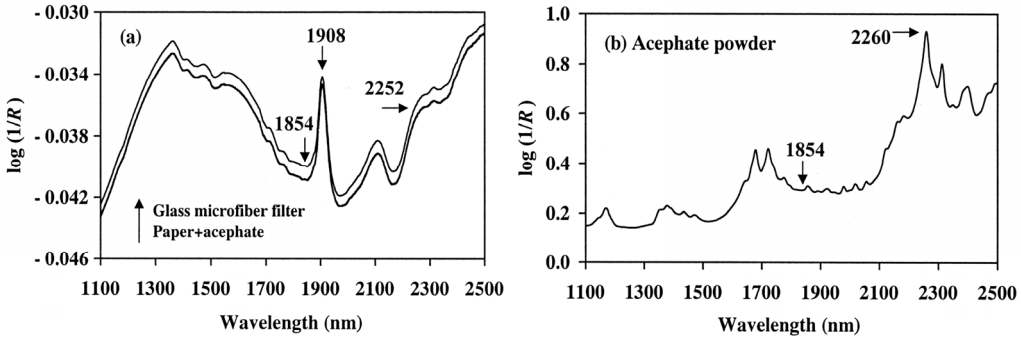

Figure 1. (a) Original [log(1/*R*)] spectra of a glass microfiber paper and the new *DESIR* filter paper with acephate measured with the NIRSystems 6500. (b) Original [log(1/*R*)] spectrum of acephate powder measured with the InfraAlyzer 500.

Original [log(1/*R*)] spectrum of a typical *DESIR* sample with acephate is shown in Figure 1(a). The shape of the spectrum was mainly affected by absorption of filter paper [Figure 1(a)] in place of acephate [Figure 1(b)].

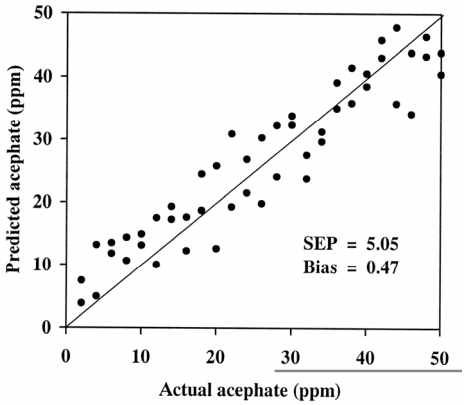

Figure 2. Scatter plots for validation set of actual acephate concentrations vs. NIR-predicted acephate concentrations.

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By PLS regression, a sufficiently accurate calibration equation for acephate could be obtained. The calibration results are given in Table 2.

Table 2. PLS calibration results of acephate determination.

Wavelength region (nm)	*F*	*R*	*SEC*	*SEP*	*Bias*
1100-2500	3	0.94	4.94	5.05	0.47

Scatter plots for validation set of actual acephate concentrations vs. NIR-predicted acephate concentrations is shown in Figure 2. From the regression coefficient plot of the calibration equation developed, it was found that the wavelengths of 2252 nm and 1854 nm had important role in the equation. The absorption peak at the wavelength of 2260 nm near the 2252 nm could be noticed in the spectrum of acephate powder [Figure 1(b)] while there was no peak in that area for the filter paper one [Figure 1(a)].

Conclusion

It was concluded that the new *DESIR* technique developed was capable to determine minute amount of the pesticide "acephate". Since the method is rapid, accurate enough and does not involve complex instrumentation, it has a significant potential for the rapid determination of pesticides in many kinds of liquid samples.

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