# Application Note

050-AT-0223

## Monitoring of reaction process using the fiber probe

### Introduction

The reaction mechanism and dynamic parameter of the chemical compounds can be analyzed by monitoring the reaction process using FTIR. Monitoring of reaction process has been widely used for the research in organic synthesis, enzymatic reaction and electrochemistry as well as the on-site measurement such as reactor, and there is a method of putting a fiber probe in the reaction system for such measurement. VIR-100/200/300 series spectrometer mounted with the Fiber connection unit has a capability to control maximum 6 lines of fiber through PC, enabling to monitor multiple reactors by one system. Moreover, since VIR-200/300 allows the rapid scan measurement of up to 25 msec interval optionally, it is also capable to monitor in real time basis the relatively rapid chemical reaction such as enzymatic reaction. The type of fiber probe can be selected from the one for transmission, reflection and ATR according to the purpose. In this report, the reaction process between oil applied on the surface of ATR prism and surfactant was measured by the rapid scan measurement using ATR probe.

### Experimental

Figure 1 shows the configuration of the system used in this measurement. Since ATR method can analyze the interface between ATR prism and solution, the measurement can be easily implemented by simply putting tip of the ATR prism on the solution without any adjustment of optical pathlength which is usually needed for the transmission method. ATR fiber whose material is chalcogenide (made by Remspec) with ZnSe prism was used. After applying cooking oil to the surface of ATR prism, ATR prism was put on the stirred surfactant in the vial and the reaction process was monitored by rapid scan.

**<Measurement conditions>**

| | |
|---|---|
| Instrument: | VIR-200 |
| Accessory: | Fiber connection unit |
| Fiber probe: | Chalcogenide (Made by Remspec) |
| Measurement method: | ATR (ZnSe prism) |
| Light source: | High-intensity ceramic source |
| Beam splitter: | KBr/Ge |
| Detector: | Mid-band MCT |
| Resolution: | 4 $cm^{-1}$ |
| Interferometer drive system: | Rapid scan |
| Measurement interval: | 80 msec |
| Max. measurement time: | 80 sec |

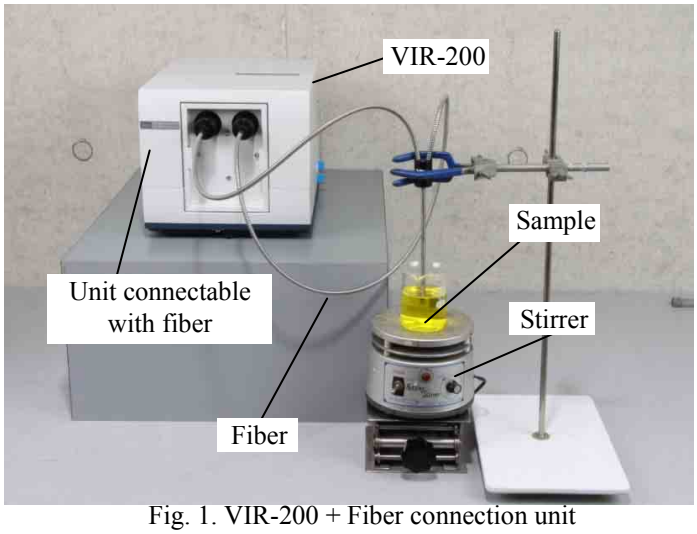


Fig. 1. VIR-200 + Fiber connection unit



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## Results and Discussions

Figure 2 shows the change of spectra with time as 3D spectra. In this measurement, ATR prism was put on surfactant solution after 25 seconds from the start of measurement. As a result, a decrease in intensity of -CH peak (at 2925 $cm^{-1}$) attributed to oil and an increase in intensity of -OH peak (at 1639 $cm^{-1}$) attributed to surfactant were observed as time goes by. Also, Figure 3 shows a spectrum (for surfactant and oil) after the reaction between oil and surfactant on the surface of ATR prism and a spectrum (for surfactant only) measured on ATR prism without oil. As shown in the results (pink area) in Figure 3, it was found that the peak shape of spectrum of surfactant only was broader as compared with the spectrum of surfactant + oil. This is considered to be due to the ordered structure of surfactant forming micell by the interaction of hydrophobic groups between surfactant and oil, and the disordered structure of surfactant in case that oil is not included.

Figure 4 shows time-dependent change of the -CH peak and -OH peak. It was found that the intensity of -CH peak slowly decreased gradually after increased rapidly when ATR prism touched the surfactant. On the other hand, the intensity of -OH peak rapidly increased after about 25 seconds from the start of measurement and then slowly increased. This phenomenon indicates that the solubilization (emulsification) and dispersion was occurring in two steps on the surface of ATR prism and it can be said that the high speed reaction process was traced successfully by the rapid scan. It is also expected that the molecule behavior on the solid-liquid interface as well as the monitoring of ordinary chemical reaction can be analyzed by using this system. Accordingly, there is a possibility that this system can be applied to the research of liposome which attracts attention due to DDS as well as the basic study of surfactant and emulsion.

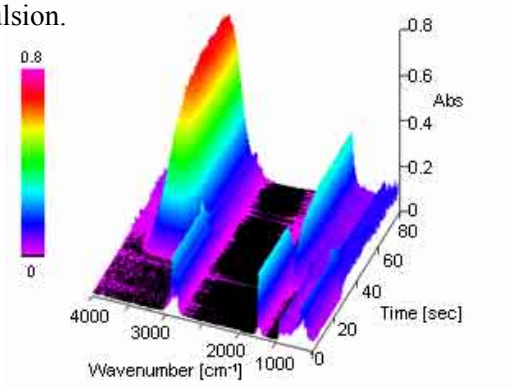


Fig. 2 3D spectrum of reaction process

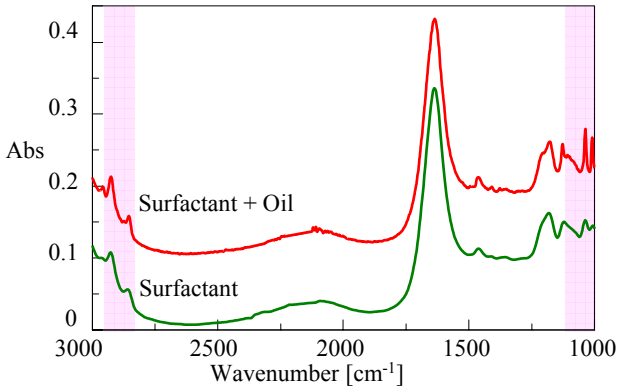


Fig. 3 Spectrum of surfactant after measurement and spectrum of surfactant only

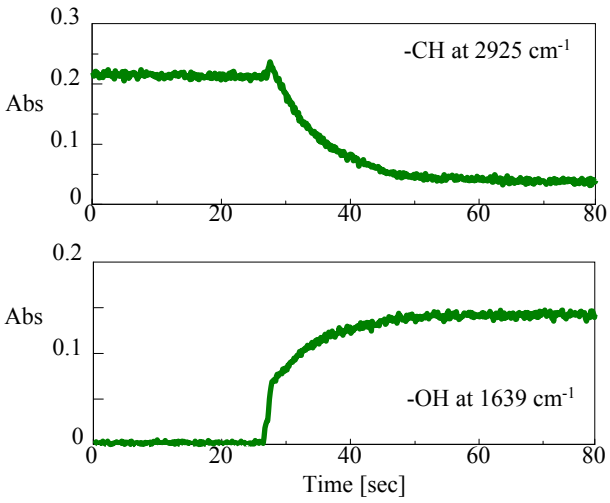


Fig. 4 Time-dependent change of Oil(-CH) & $H_2O$(-OH) group)

## Conclusion

In this report, the two-steps reaction was monitored by using the fiber probe and rapid scan. In addition, it is expected that VIR-100 series can be utilized in a wide range of field such as the evaluation of lithium-ion battery materials that can be measured by the instrument installed in the glove box, the remote measurement in reactor by multi-probes and the measurement in manufacturing line, making full use of the features such as compact size, robustness and free usage of sample compartment.



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