

Application News

Spectrophotometric Analysis

No. A422

Rubber Analysis by FTIR -Method for Removing Silicate from Rubber-

A silicate is formed from silicon and oxygen, and talc and kaolinite are some typical silicate compounds. Silicon dioxide (silica), which does not contain a metallic oxide, is also a type of silicate. Silicate, like carbonate, is used as an alkaline builder in laundry soap, and is used widely as an additive in rubbers and plastics.

When measuring the infrared spectrum of rubber that contains silicate, an extremely large silicate

peak appears in the vicinity of 1000 cm^{-1} , which conceals nearby rubber peaks and makes it difficult to perform qualitative analysis. Here we introduce a pretreatment procedure for removing talc, one type of silicate, from rubber which contains talc. For a description of the procedure for removing calcium carbonate, another additive used in rubber, refer to Application News No. A416.

■ Measurement Method

Infrared spectral measurement was conducted using a single reflection total reflectance ATR accessory (DurasamplIR) with a diamond prism. The measurement conditions are shown in Table 1.

Table 1 Analytical Conditions

Resolution	: 4 cm^{-1}
Accumulation	: 45
Detector	: DLATGS

■ Spectrum of Talc

The chemical formula of talc is $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$, and the fine powder is used as a lubricant, in pharmaceutical products, as a cosmetic (talcum powder), as a filler in paper making, and as a source of magnesium in ceramics¹⁾, to name a few.

Fig. 1 shows the infrared spectrum of talc measured using the single reflection ATR method.

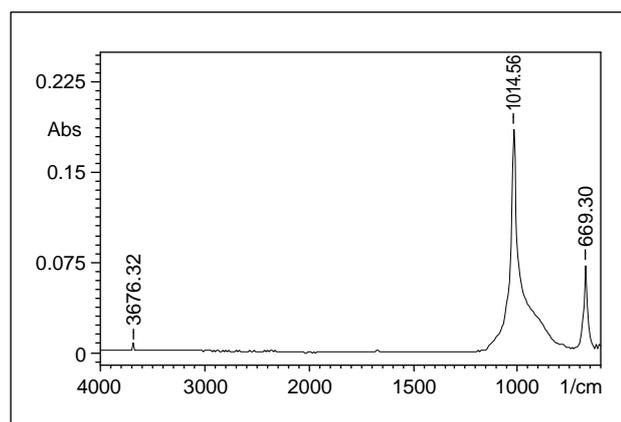


Fig. 1 Spectrum of Talc

The infrared spectrum of talc characteristically reveals an extremely strong absorption peak at 1014 cm^{-1} due to an Si-O stretching vibration, in addition to a sharp absorption peak at 3676 cm^{-1} attributed to the stretching vibration of O-H without a hydrogen bond.

■ Polychloroprene Rubber Containing Talc

The sample used for this measurement is chloroprene rubber (CR) that contains talc. Fig. 2 shows the results of the measurement, conducted by single reflection ATR.

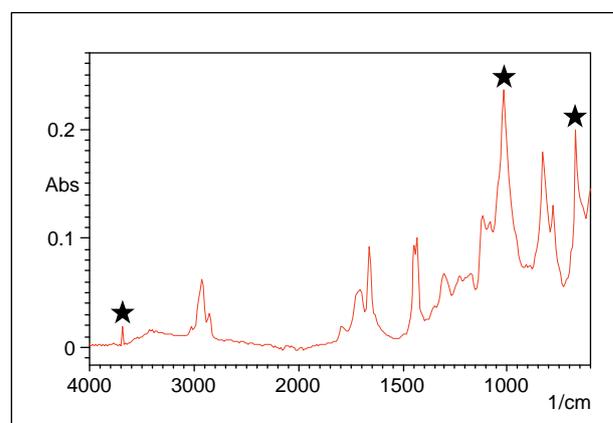


Fig. 2 Spectrum of CR Containing Talc

The star (★) annotations in Fig. 2 indicate peaks originating from the talc. To remove these peaks, the sample was immersed in hydrofluoric acid for 30 minutes, and after thoroughly rinsing it with water, the infrared spectrum was measured. Fig. 3 shows the infrared spectra before and after the hydrofluoric acid treatment.

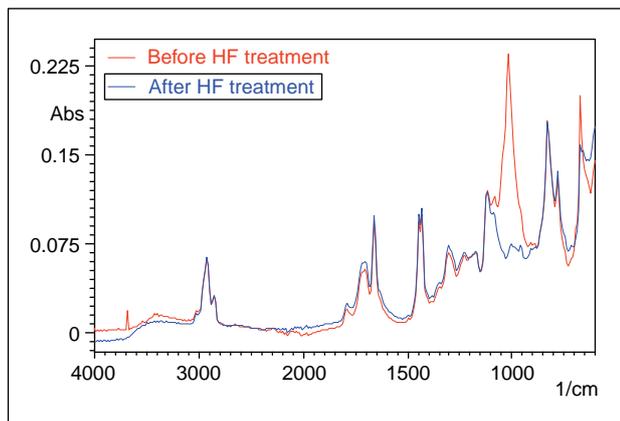


Fig. 3 Spectra Before and After Hydrofluoric Acid Treatment

From Fig. 3, it is clear that the peaks originating from the talc have disappeared from the spectrum. This is because the talc was removed from the surface of the rubber as a result of the hydrofluoric acid treatment.

Fig. 4 shows an overlay of 2 infrared spectra, the red one, obtained following ATR correction of the hydrofluoric acid-treated rubber sample (blue-colored spectrum of Fig. 3), and the blue spectrum, which is the result of a search of the Bio-Rad Laboratories Sadtler database (Monomers & Polymers Vol. II)

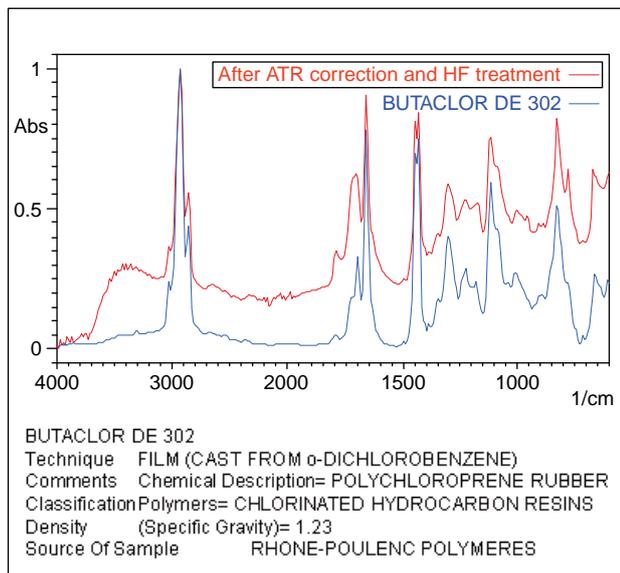


Fig. 4. Spectrum Search Result after Hydrofluoric Acid Treatment, and Detailed Information

From Fig. 4, it is clear that the infrared spectrum obtained following hydrofluoric acid treatment corresponds well with that of a typical chloroprene rubber over the entire wavenumber region.

■ EPDM Rubber Containing Talc

We conducted the same hydrofluoric acid treatment on ethylene propylene diene rubber (EPDM) which also contains additives such as calcium carbonate and talc, as previously described in Application News No. A416 "Analysis of Rubbers by FTIR -Method to Remove Calcium Carbonate in Rubbers-". Fig. 5 shows the overlaid infrared spectra before and after hydrofluoric acid treatment.

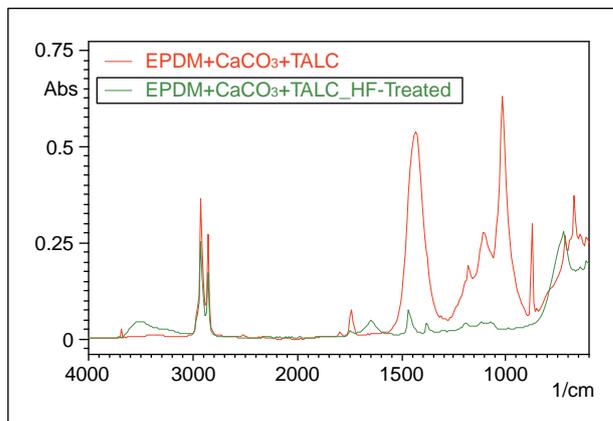


Fig. 5 Spectra Before and After Hydrofluoric Acid Treatment

From Fig. 5, it is clear that the talc peaks can be removed just as in the case of chloroprene rubber as described above. In addition, the absorption peaks associated with calcium carbonate (near 1390 cm^{-1} , 870 cm^{-1} , and 710 cm^{-1} , respectively) were also removed at the same time due to reaction with hydrofluoric acid. Now, the relatively broad absorption peak in the vicinity of 720 cm^{-1} is attributed to calcium fluoride, formed by the reaction of calcium carbonate with hydrofluoric acid. In addition, the absorption peaks at 3500 cm^{-1} and 1650 cm^{-1} are peaks due to water, presumed to have formed as a result of the reactions.

Thus, peaks originating from silicate and calcium carbonate can be removed relatively easily by hydrofluoric acid treatment. However, please note that hydrofluoric acid is designated as a poisonous substance, and as such should be handled with due care, including the use of gloves and protective goggles, and adequate ventilation.

[References]

- 1) Physical and Chemical Dictionary, Iwanami Shoten, Publishers