

## NOTES

### *Absorption of CO<sub>2</sub> by Phenolic Resin\**

#### INTRODUCTION

The absorption of CO<sub>2</sub>, He, N<sub>2</sub>, and H<sub>2</sub>O by an ablative material as a function of pressure and temperature has been reported by Wightman and Griest.<sup>1</sup> However, the ablative material studied was a discontinuously filled elastomer composed of colloidal-sized silica fibers, silica spheres, and phenolic resin spheres in a silicone elastomer matrix. The absorption of CO<sub>2</sub>, He, and H<sub>2</sub>O by the components of the ablative material has been studied in subsequent work to determine with which components a given gas was interacting. The absorption of CO<sub>2</sub> by the phenolic resin is reported here since the interaction has been characterized using infrared spectroscopy and may be applicable to other gas-polymer systems.

#### EXPERIMENTAL

##### Absorption of CO<sub>2</sub>

The hollow Microballoon spheres (#BJO-0930), which are made of Bakelite phenolic resin, were supplied by the Union Carbide Company. The phenolic resin spheres have a particle size range of 5 to 127  $\mu$ . Airco CO<sub>2</sub> was used without further purification. Absorption measurements were made in a constant volume (14.8 cc) system. A known quantity of CO<sub>2</sub> was introduced into a previously evacuated 0.34-g sample of phenolic resin and the system pressure was followed as a function of time.

##### Infrared Spectra

Confirmation of absorbed CO<sub>2</sub> in the phenolic resin was sought by an independent technique. Infrared spectroscopy was used to detect the presence of absorbed CO<sub>2</sub>. A 50-mg powdered sample, evacuated for 2 hr and exposed to a CO<sub>2</sub> pressure of 1 atm for 3 hr, was dispersed in Nujol, pressed between NaCl plates, and the infrared spectra were obtained on a Perkin-Elmer 621 spectrophotometer at 10X scale expansion. The spectrum of gaseous CO<sub>2</sub> was obtained using the same spectrophotometer with a Wilks mini-gas cell at 1X scale expansion.

#### RESULTS AND DISCUSSION

A typical pressure-time trace due to CO<sub>2</sub> absorption by the phenolic spheres is shown in Figure 1. The significant decrease in system pressure from 46.2 to 31.6 torr occurred over a 3-hr period. The decrease in pressure on exposure of CO<sub>2</sub> to the phenolic resin is related to the amount of gas absorbed by the resin in a constant volume system. The number of moles of CO<sub>2</sub> absorbed per unit weight of resin (or solubility) was  $9.8 \times 10^{-9}$  moles CO<sub>2</sub>/g-torr calculated from the observed pressure decrease using the ideal gas equation.

The long time required to saturate the sample (see Fig. 1) suggests a diffusion process either into the interior of the hollow spheres or into bulk resin of the spherical annulus.

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Diffusion of  $\text{CO}_2$  is not occurring into the interior of the hollow spheres since absorption measurements using crushed phenolic spheres showed no decrease in the amount of  $\text{CO}_2$  absorbed. The interaction of  $\text{CO}_2$  thus appeared to be with the bulk phenolic resin.

Independent evidence for the presence of absorbed  $\text{CO}_2$  in the bulk resin was sought. A substantial amount of data has been reported<sup>2</sup> on diffusion, permeation, and solubility of gases in polymers. However, there are few reports in the literature of direct experimental confirmation of interacting gases in polymers. It was felt in the present work that infrared spectra might provide such confirmation. The infrared spectrum of crushed phenolic resin which had not been exposed to  $\text{CO}_2$  is shown in Figure 2a in the

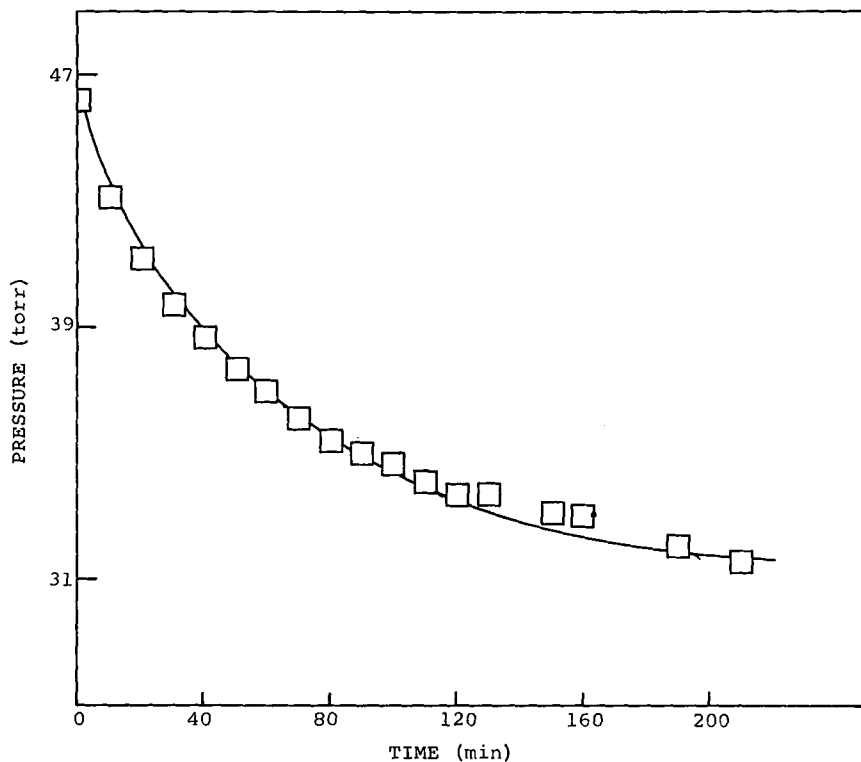


Fig. 1. Pressure-time trace on exposure of phenolic resin spheres to  $\text{CO}_2$  at  $25^\circ\text{C}$ .

$2500\text{--}2200\text{ cm}^{-1}$  region at scale expansions of 1X and 10X. There is no significant absorption by the unexposed sample. The infrared spectrum of the phenolic resin saturated with  $\text{CO}_2$  is shown in Figure 2b. The intense and sharp absorption at  $2335\text{ cm}^{-1}$  is due to the asymmetric stretching mode of  $\text{CO}_2$ . The spectrum of gaseous  $\text{CO}_2$  is shown in Figure 2c. The unresolved vibration-rotation band is seen with the frequency of the band center located at  $2352\text{ cm}^{-1}$ . The loss of rotation of  $\text{CO}_2$  in interaction with the phenolic resin is seen by comparing Figures 2b and 2c. Further, the fundamental frequency of  $\text{CO}_2$  is shifted from  $2352$  to  $2335\text{ cm}^{-1}$  due to the interaction of  $\text{CO}_2$  with the phenolic resin.

We have shown that infrared spectroscopy could be used advantageously to demonstrate the interaction of  $\text{CO}_2$  with phenolic resin and may have application in a variety of other gas-polymer systems.

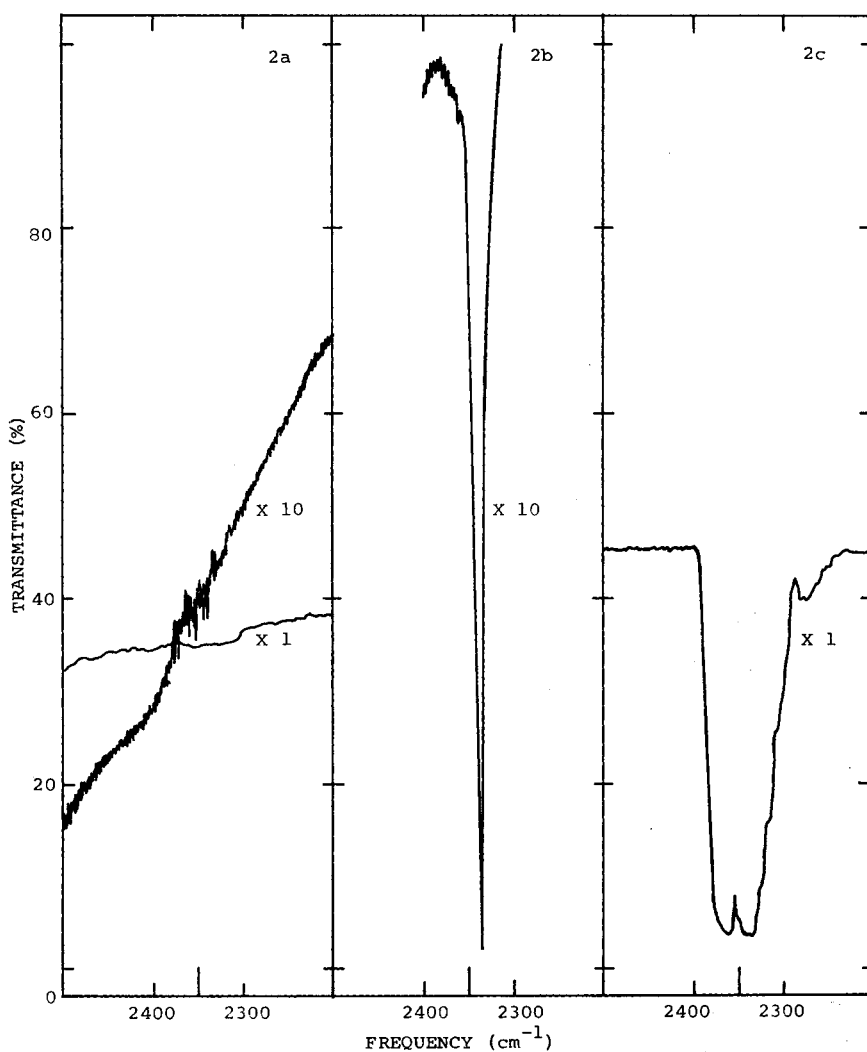


Fig. 2. Infrared spectra crushed phenolic resin: (a) phenolic resin not exposed to  $\text{CO}_2$ , at scale expansions of  $1\times$  and  $10\times$ ; (b) phenolic resin exposed to  $\text{CO}_2$ ; (c) gaseous  $\text{CO}_2$ .

#### References

1. J. P. Wightman and W. H. Griest, *Va. J. Sci.*, **19**, 200 (1968).
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