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OPTICAL PROPERTIES FROM COMPOSITES OF C₆₀ AND FAU ZEOLITE

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We have investigated the photophysical properties of C₆₀ (guest)-FAU zeolite (host) composite, focusing mainly on the properties of a single crystallite using microscopic techniques (UV-Vis-IR absorption and photoluminescence). The composite C₆₀-FAU is of great interest because it exhibits new optical properties different from that of either C₆₀ powder or FAU zeolite.

Keywords: C₆₀, FAU zeolite, UV-Vis-IR absorption, luminescence

INTRODUCTION

On the one hand, fullerenes have been under intense scrutiny, because they possess a quasi-spherical carbon shell structure and an extended π -electron system. We have already demonstrated that the photophysical properties of fullerenes are very sensitive to any alteration of the electronic structure and that intermolecular charge transfer excited states play important roles in C₆₀ film^[1,2]. We have also already reported that C₆₀/Si composite films show strong white light emission, never observed for neat C₆₀ or Si films. The origin of the latter phenomenon has been already discussed^[3,4]. On the other

hand, we know that the most striking aspect of semiconductor nano-crystals and molecular clusters is the dramatic change in their optical properties as a result of the confinement effect in zeolite cages ^[5]. Faujasite-type zeolite (FAU) consists of a regular framework of corner-sharing SiO_4 and AlO_4 tetrahedrons, which generate well-defined system of cages. FAU zeolite can be described as a network of dot-type "0-D" cages with cavity size in the range of 8 Å. A remarkable aspect is that FAU zeolite forms single crystal of 10 to 5 μm size.

In this context, we have investigated the photophysical properties of the composite C_{60} (as guest) and FAU zeolite (as host), focusing mainly on the properties of a single crystal using microscopic techniques (analysis on the scale of 10 to 5 μm). This methodology is very different and more powerful than those already used by other groups (mainly on zeolite powders and using macroscopic techniques) ^[6,7]. Here we report our preliminary results.

PREPARATION

C_{60} -FAU composites were prepared in gas phase. Typically 20 mg of C_{60} powder and 500 mg of FAU zeolite were mixed and loaded into a glass tube. Under dynamic vacuum the system was pre-heated at 200 °C for 6 hours, in order to remove adsorbed gases and to activate the zeolite. Subsequently the tube was sealed, always under vacuum, and heated at 550 °C for 2 days. Under the above conditions a high partial pressure of C_{60} gas is generated and this gas can readily diffuse into the zeolite cages and/or adsorb on the surface of the zeolite. Finally, the composite was washed several times with toluene to remove the excess of C_{60} powder and was dried under vacuum (to remove toluene), before experimental investigations.

RESULTS AND DISCUSSIONS

Under the above experimental conditions we can discard the possibility of chemical and physical degradation of each constituent, namely C_{60} and FAU zeolite. Indeed, the UV-visible absorption spectrum of the solution used to wash the C_{60} -FAU composite shows absorption bands corresponding to those of C_{60} molecules. In other words there is no chemical degradation of C_{60} during sublimation process under vacuum in the presence of FAU

zeolite. Moreover, the characterization by X-ray diffraction shows that the C₆₀-FAU composite retains the crystallographic structure of the zeolite in terms of 2θ and width of the peaks. In other words the crystal structure of the zeolite FAU is stable under the present experimental conditions with no significant amount of amorphous phase. In the light of recent reports by HR-TEM [8], it has been observed that C₆₀ molecules are not only adsorbed on the surface of the zeolite but also a small fraction of C₆₀ molecules can be entrapped into the zeolite cages.

The photophysical properties of the C₆₀-FAU zeolite composite were investigated using microscopic techniques such as micro-UV-visible-IR absorption (Zeiss Microscope Spectrophotometer USMP 80), micro- and macro-photoluminescence spectroscopies.

The absorption spectrum of C₆₀-FAU zeolite single crystal revealed broad bands in the range 300-850 nm, different from neat C₆₀ films and C₆₀ dispersed in PMMA films (Fig. 1). This suggests that the electronic structure of C₆₀ molecules in the FAU matrix has been considerably altered.

It is important to stress that the C₆₀-FAU composite exhibits a broad and intense photoluminescence emission with excitation wavelength 514 nm (Fig. 2, curve a) in the range 600-900 nm, dramatically different from that of either C₆₀ (powder (Fig. 2, curve b), solution, film), FAU zeolite (untreated powder, (Fig. 2, curve c)) or of unheated mixture C₆₀-FAU zeolite (Fig. 2, curve d). Using a confocal microscope (Leica TCS system) either in transmission or reflection mode we have been able to map the photoluminescence in 3-D (x40 lens, and 0.1 μm resolution in the thickness direction) and to measure the photoluminescence spectrum of an individual crystal ($\lambda_{\text{ex}}=488$ nm). We have observed that the photoluminescence originates not only from the surface of the crystal but also from the bulk. Its intensity is homogenous all over the crystal, within the experimental resolution.

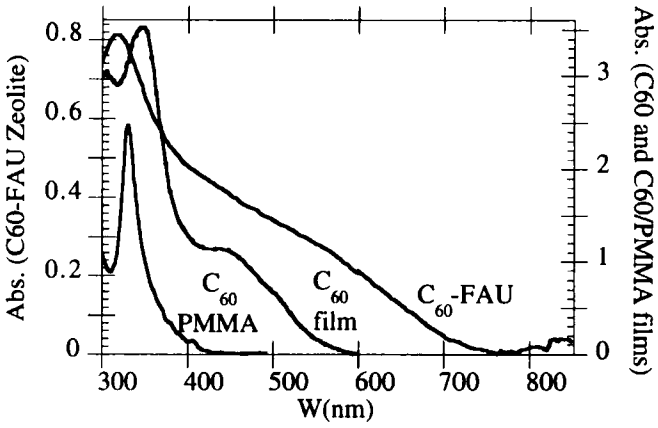


Fig. 1: Absorption spectra of C₆₀-FAU Zeolite compare to C₆₀ and C₆₀/PMMA films

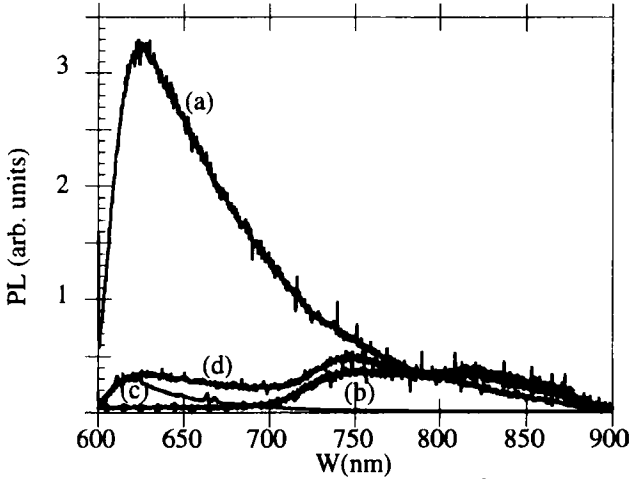


Fig. 2: Luminescence emission spectra $\lambda_{ex}=514\text{nm}$ (see text for legend)

The above results suggest a dramatic alteration of the electronic structure and vibrational modes of C₆₀ molecule as a result of strong interactions between C₆₀ and FAU zeolite. Therefore, the observed changes in the photophysical properties of C₆₀-FAU composite should be related either to the adsorption of C₆₀ on the zeolite and/or the confinement effect of C₆₀ in the zeolite cages.

Further investigations are necessary to confirm the above results and we must elucidate the origin of the broad photoluminescence in the composite C₆₀-FAU zeolite. For that purpose we are going to use several types of zeolite with different dimensionality of cages (dot-type "0-D", tube-type "1-D", layer-type "2-D") and size.

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