

## X-RAY PHOTOELECTRON SPECTROSCOPIC MEASUREMENTS OF FULLEROPYRROLIDINE CLAYS

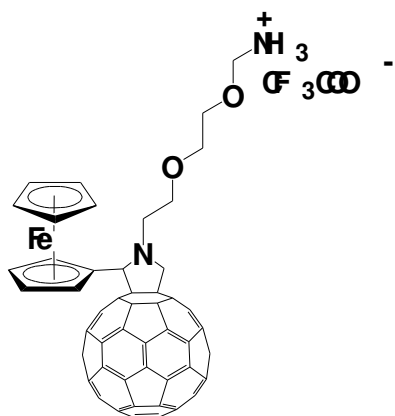
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The design of new properties of nanocomposites makes them useful for a constantly broadening range of applications and consequently opens more and more interesting market opportunities. One of the most relevant materials in this respect are polymer nanocomposites in which polymers are reinforced with low quantities of nanometric-sized clay particles. In general, smectite clays are used as nanoclays since these layered aluminosilicate minerals present a unique combination of swelling, intercalation and ion exchange properties. The main approaches to preparing polymer nanocomposites require the platelets to be rendered organophilic through the exchange of the interlayer cations with a charged organic species. This can be achieved by intercalating fulleropyrrolidine derivatives possessing positive charges (Fig. 1) into the interlayer space of the clays. The outstanding electro- and electrophotocatalytic properties of fulleropyrrolidine derivatives open the way for a variety of unique properties of the final product, i.e. not only better mechanical properties but also the combination of a barrier function with optical properties. This leads to a number of possible applications including the food packaging and automotive industry.



**Fig 1.** Fulleropyrrolidine derivative with an ammonium and with a ferrocene group.

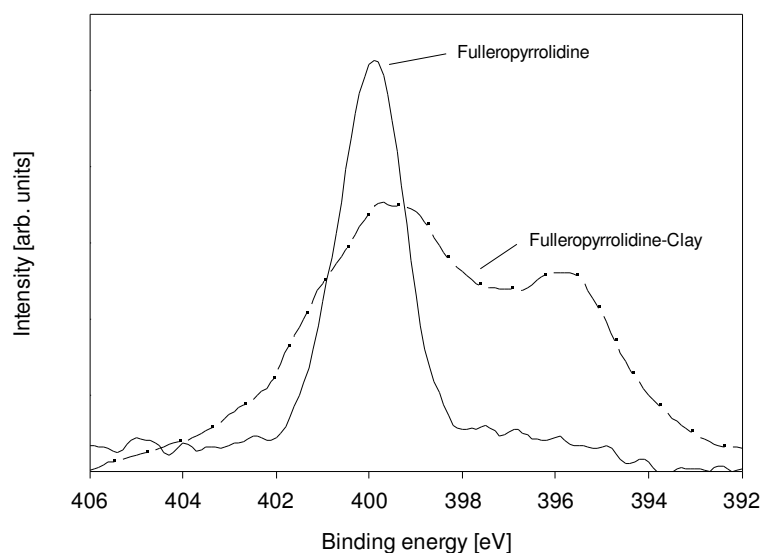
In the framework of this project the smectite clays -sodium ion exchanged Wyoming Na-montmorillonite (Swy-2) [1] and commercial laponite [1]- were manipulated with various fulleropyrrolidine derivatives. For the intercalation procedure and composites characterisation see D. Gournis et al [2]. The as received organo-clays were characterised by means of X-ray photoelectron spectroscopy (XPS) since this method not only allows for the elemental analysis but also provides information about the chemical environment of an element on the surface. The main purpose of this study was to investigate the changes caused by the intercalation process on the different elements of the clays and fulleropyrrolidine derivatives.

All photoemission spectra of the fulleropyrrolidine clays show nitrogen and carbon photoelectron lines which result from the presence of fulleropyrrolidine in the clay galleries (Fig. 2). These N1s and C1s photoemission lines show a pronounced shift to lower binding energy when the fulleropyrrolidine is part of the organoclay, which could indicate that charge transfer takes place between the clay and the fulleropyrrolidine derivative present in the clay galleries. If we compare the spectra of the clays before and after intercalation with fulleropyrrolidine derivatives we can identify an additional component in the O1s photoemission line of the organoclay providing additional evidence for the presence of the fulleropyrrolidine derivative (Fig. 3). Another clear evidence for the intercalation process is the decrease interlayer cation concentration as documented by the lower intensity of the Na 1 s photoelectron line after intercalation.

In conclusion, XPS measurements clearly demonstrate that all the different fulleropyrrolidine compounds are intercalated and that we have succeeded in manipulating smectite clays with fulleropyrrolidine derivatives .

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[2] D. Gournis, V. Georgakilas, M.A. Karakassides, T. Bakas, K. Kordatos, M. Prato, M. Fanti and F. Zerbetto, J. Am. Chem. Soc., in press.



**Fig. 2** Nitrogen 1 s photoemission line for a fulleropyrrolidine and the corresponding fulleropyrrolidine-clay

**Fig. 3** Oxygen 1 s photoemission line for a fulleropyrrolidine, the corresponding fulleropyrrolidine-clay and the fulleropyrrolidine subtracted from the fulleropyrrolidine-clay