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Title:
DEVELOPMENT AND STUDY OF POLYMERIC MATERIALS FOR THEIR USE IN CHEMICAL SENSORS

Summary

The aim of this PhD thesis was the study of polymeric materials for their use in gas sensor applications. Inherently conducting polymers and conducting polymer composites comprise the two classes that could be used as sensitive elements in conductivity type gas sensors based on polymers. The sensing mechanism of the above sensors is based in the fact that the resistance of the active material of the sensor is changed upon exposure to analyte gases.

The sensitivity of the above materials is based in the alternation of their electrical properties. For that reason, the factors that influence the conductivity are systematically studied. The conductivity of polymer nanocomposites comes from the incorporation of conductive filler, at an efficient concentration, that can form a conducting network throughout the insulating polymer matrix. The conducting phase could be either an inherently conductive polymer or conducting nanoparticles. The critical concentration of the conducting filler, which is responsible for the transition from the insulating to the conducting phase, is dependent on the type of the inclusion and the connectivity of the conducting network. For the above reason, the study of percolation aspects is of great importance in this work.

The preparation of the studied materials was done using facilities available in Dielectric Spectroscopy Laboratory of Physics Department and in other associate institutes abroad. The same stands also for the techniques and equipment for the characterization of the prepared materials. The study of morphology was done using Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Atomic Force Microscopy (AFM). Thermomechanical characterization was performed using Differential Scanning Calorimetry (DSC) and Dynamic Mechanical Analysis (DMA). Electrical/dielectric properties were studied using Dielectric Relaxation Spectroscopy (DRS) in a wide temperature and frequency range, as well as by dc conductivity measurements. A special set-up for testing the prepared materials as gas sensors, was developed. The above set-up comprises of an apparatus responsible for preparing gas mixtures of the desired concentration of the analyte and a measurement cell for making electrical measurements. The sorption and diffusion of water in hydrophilic polymers were studied using the gravimetric method with a moisture sorption analyzer.

The sorption/desorption of water in hybrid materials based on hydrogels, was also studied. The inorganic phase consisted of silica nanoparticles prepared by sol-gel process. The above nanoparicles enhanced the mechanical properties of the hybrids. Furthermore, morphological, thermomechanical characterization and immersion experiments into water evidenced the existence of a continuous silica network for concentrations above 15 % w.t silica.

The formation of the above network influences the water sorption capacity of the hybrid, as well as the diffusion of water through them.

Percolation aspects were studied in composites based on polypyrrole. Particularly, the influence of the filler content in an insulating polypropylene matrix, the preparation procedure and the incorporation of montmorillonite clay on the morphology and electrical and dielectric properties, was studied in detail. The results showed that the incorporation of the clay promotes conductivity and lowers the percolation threshold.

For the study of inherently conducting polymers as gas sensors, thin films of polypyrrole and polyaniline were prepared by chemical polymerization. Their response to different concentrations of ammonia, was studied. The response showed good sensitivity and reversibility indicating that these polymers are good candidates for ammonia sensors. Furthermore, the influence of moisture in their response to ammonia, was also studied.

Nanocomposites consisted of carbon nanotubes dispersed in polymethylmethacrylate matrix were prepared for the investigation of their sensing properties. The results from morphology study and electrical/dielectric characterization showed good dispersion of the filler with low percolation threshold. Furthermore, the response to water and ethanol vapour, at different concentration, was also studied. A variety of different polymers were also used as matrices for the preparation of composites by solution mixing. Their response to water and ethanol showed better responses for the more hydrophilic polymers and those with glass transition temperature below room temperature.