Use of plasma polymerization to improve the chemical properties of particles and nanoparticles.


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**Interests:** We use Plasma polymerization process to carry out surface modification of particles, nanoparticles, polymers and other materials. **Facilities:** Some pictures of the plasma reactors that we use are represented in Figures 1 to 4. In order to perform the surface modification of the particles and materials we use monomers such as: ethylene, propylene, acrylic acid methyl methacrylate, styrene among others. In our laboratories we have all the necessary equipment to perform morphological and chemical and physicochemical characterization of nanoparticles. We also have the instruments to measure all the mechanical, thermal and conductivity properties of polymer nanocomposites.

*Figure 1. Schematic and Picture of RF Plasma Particle Treatment Reactor.*
Figure 2. Prototype ultrasonic vibrating batch plasma reactor for nanoparticle surface treatment operating at CIQA.

Figure 3. Ultrasonic vibrating continuous plasma reactor for nanoparticle surface treatment.
Figure 4. Plasma Reactor used to carry surface modification of polymers and other materials using plasma polymerization process.

The modification of particles and nanoparticles is carried varying: plasma power, time of treatment and flux of monomer; once nanoparticles are modified by plasma polymerization, they are analyzed by: Electronic microscopy (STEM and TEM), thermogravimetical analyses (TGA), X-ray diffraction, infrared analyses (FTIR), Raman spectroscopy, X ray photoelectron spectroscopy (XPS), among others. We also use different solvent in order to check the dispersion of the nanoparticles in them. In Figure 5 it is presented a micrograph of a pristine copper nanoparticle, and in the same figure is presented a copper nanoparticle coated with a plasma polymer of acrylic acid.

Figure 5. Micrographs obtained by HRTEM of A)Pristine copper nanoparticle and B)Copper nanoparticle coated with a plasma polymer of Acrylic Acid (30W, 30 min).
In other study carried out at CIQA, carbon nanofibers (CNFs) were treated with plasma of acrylic acid, CNFs were analyzed by electron energy loss spectroscopy (EELS) and dispersed in water, and results are presented in Figure 6. The EELS at high energies shows a signal at 534 eV for the treated (CNFs) sample (Figure 6), which is associated with the oxygen K-edge. No such signal is seen on the untreated CNFs spectrum, which confirms the absence of oxygen on the untreated CNFs surface and explains the hydrophobic properties of the untreated nanofibers. It is clear from the evidence provided above that the oxygen present on the treated CNF surface comes from the plasma polymer of acrylic acid deposited. The presence of oxygen on the treated CNF surface, in the form of carboxylic and carbonyl groups, changes its characteristics from hydrophobic to hydrophilic, thereby rendering the treated CNFs water dispersible as evidenced in the inserted photographs in Figure 6. Untreated CNFs remain on the water surface, but treated CNFs are very well dispersed in water as can be appreciated in the inserted picture (right hand side picture).

![Figure 6. EEL spectra of CNFs at the oxygen K-edge.](image)

**Preparation of Polymer Nanocomposites.**

After plasma modification, nanoparticles are mixed with different polymers by melt mixing or by dissolving the polymer and the nanoparticles in a common solvent and once both are mixed, the solvent is evaporated. Nanocomposites prepared with nanoparticles modified by plasma present better properties than nanocomposites prepared with untreated nanoparticles. Plasma polymerization applies a thin coating of polymer on the surface of the particles, and the coating improves the compatibility between polymer and nanoparticles, and as a result we obtain a nanocomposite with enhanced mechanical thermal properties. For example in Figure 7, it is analyzed the thermal behaviour of high density Polyethylene (HDPE), and is compared with nanocomposites of HDPE containing 1% of pristine carbon nanofibers (CNFs) and 1% of carbon nanofibers treated with ethylene plasma. Composites containing treated carbon nanofibers (tCNFs) present higher thermal stability, as can be appreciated in Figure 7, since this composite degrades at higher temperatures.
Figure 7. TGA thermograms of HDPE, HDPE/1%CNFs and HDPE/1%tCNFs.

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