TiO2 nanoparticles were coated uniformly, with a controllable thickness of conductive polymer polythiophene, in a rotating capacitively coupled radio-frequency (RF) plasma reactor. Plasma parameters such as power, pressure, and time affected the properties of the TiO2/polythiophene nanocomposites, and plasma characteristics were examined using an optical monochromator, as well as in situ residual gas analysis (RGA), Fourier transform infrared (FTIR), X-ray, transmission electron microscopy (TEM), thermogravimetric analysis (TGA), and four-probe techniques. The plasma optical emission results showed that the peaks from TiO2 decreased by time as the polythiophene coating thickness increases. TEM results confirmed the polythiphene coating onto TiO2 in order of nanometers. It was obtained data from main peaks (molecular masses of 2 (H-2), 18 (H2O), 28 (C2H6), and C2H4) of mass spectra recorded by RGA. The shift and the changes in the intensity of the FTIR peaks are credited to the substantive internalization between PT and TiO2 materials. The characterization of the structure has indicated that thiophene molecules are adsorbed onto, and then polymerized on, the surface of the TiO2 particles.