Cheap method for synthesis of highly water soluble fullerene derivatives – fullerenols-d

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The applications of the fullerenes are often retained by their almost total incompatibility with water solutions. So, an interest in the production of water-soluble derivatives of the fullerene is steadily high.

A polyhydroxylated water soluble fullerenol $C_{60}(OH)_n$ has a simple structure, small toxicity and high ability to capturing radicals. However the cost of pure fullerenol is too high for its industrial applications. Therefore a technology of the fullerenols allowing different functional groups, not necessarily hydroxyl ones, is important. We choose a direct synthesis of the fullerenols as the most simple and steady. The saturated C_{60} solution in benzene (600 mg of C_{60}/800 ml of benzene) was added with a solution of NaOH (20g/20 ml) and with tetrabutyl-ammonium hydroxide solution $[(n\text{-}C_4H_9)_4N]OH$ in intensive mixing mode.

Then benzene was driven away and the reactionary mix was added with water. In an optimized course of subsequent procedures, principal of which was the fullerene salting out from the water solution by means of methanol, small-dispersed red-brown crystals of the fullerenol-d synthesized were deposited. The quantity of the product was ~70% from the theoretically predicted yield.

The electron spectrum of the water solution of the fullerenol-d does not have notable absorption bands in the both visible and near UV regions. In particular, the absorption peaks at ~ 472nm (fullerene C_{70}), ≈ 335nm (both C_{60} and C_{70}), 320–330 nm (for the bromine fullerenes $C_{60}Br_n$ (n = 6,8,24), typical of the light fullerenes and their derivatives, are absent. Though, UV-spectra of the fullerenols are able to be used for finding their concentration, following the Lambert-Ber law at non-characteristic wavelengths, e.g. at $\lambda \approx 300 \div 350$ nm. More informative are the IR absorption spectra, registered at SHIMADZU FTIR-8400S device for solid state samples. Notable are the absorption region for $\tilde{\nu}_1 \approx 3420$ cm^{-1} (hydro-xyl OH absorption), $\upsilon_{2(1)}$'=1590 cm^{-1} and $\upsilon_{2(2)}$'=1450 cm^{-1} (duplet), υ_3'=1040 cm^{-1}. So, IR- spectra of the fullerenol-d are also useful for its identification.

High performance liquid chromatography (HPLC) of fullerenol-d in water as well as the mass-spectrometry characterize the content of the fullerenol-d composition as a mix of polyalcohols $C_{60}(OH)_n$, oxy - polyalcohols $C_{60}(OH)_{n1}O_{n2}$ and their salts. Owing to the features of our synthesis the formation of sodium salts with a common formula of $C_{60}(OH)_{n1}O_{n2}(ONa)_{n3}$ is also possible.

Two examples of promising fullerenol-d application are presented in the materials of the Workshop.