MASS SPECTROMETRIC PROBING OF C60 WITH MoS, COMPOSITE PRODUCED IN AQUEOUS MEDIUM



V.S. Shelkovsky¹, M.V. Kosevich¹, O.A. Boryak¹, P.O. Kuzema², A.V. Dolbin¹, N.A. Vinnikov¹, S.V. Cherednichenko¹, V.A. Karachevtsev¹

¹ B. Verkin Institute for Low Temperature Physics and Engineering of the National Academy of Sciences of Ukraine, 47, Nauky Ave., 61103, Kharkiv, Ukraine, e-mail: shelkovsky@ilt.kharkov.ua ² Chuiko Institute of Surface Chemistry of the National Academy of Sciences of Ukraine, 17, General Naumov Str., 03164, Kyiv, Ukraine, e-mail: coralchance@gmail.com

Preparation of aqueous dispersions of nanomaterials is required for tests of their biological activity and further biomedical applications.

It is remarkable that fullerenes were first synthesized and detected in mass spectrometric experiments on laser ablation of carbon. Since then, various versions of mass spectrometric techniques are intensively applied in studies of fullerenes in the gas, liquid, and solid states.

The discovery of fullerenes in 1985 gave a significant impetus to the development of nanotechnology of carbon-based nanomaterials. To test biological activity of fullerenes aqueous dispersions were required, which was a real challenge for hydrophobic C60. A problem of transfer of C60 to water was solved at the ILTPE as early as 1995 [1] by applying ultrasonic treatment to the toluene-water system. Recently, an advanced method of vacuum-sublimation cryogenic deposition for the preparation of hydrated fullerenes was proposed [2] at the ILTPE, which allowed for the avoidance of traces of organic solvents in an aqueous medium. Presence of pure C60 in thus prepared samples was confirmed by mass spectrometry [3]. Exfoliated MoS₂ is a 2D nanomaterial promising for various nanotechnological, including biomedical applications. Nanocomposites or nanohybrides of several compounds are fabricated to obtain new materials with new properties.

Laser Desorption/Ionization Mass Spectrometry of (C60 + MoS₂) nanocomposites

Dried solid nanocomposites (C60+MoS₂) were studied by LDI technique using an Autoflex II mass spectrometer (Bruker Daltonics, Germany) equipped with a nitrogen laser (337 nm).

Comparison of LDI mass spectra of pure C60 film dried on steel and (C60+MoS₂) nanocomposite prepared in aqueous medium.

['n: e	I D 35	700 0	~ +-	35-Pos-(60-40)-M5-C3+H2O_glass-sum10-LD1/0_M5\1\1Lin Raw
si 2000 -	LF JJ	720 0	60	hydrated fullerene dried on steel
- 1				

The AIM of the present work was to fabricate nanocomposite of C60 fullerene with MoS₂ (molybdenum disulfide) 2D nanoflakes in aqueous medium and to probe it by laser desorption/ionization (LDI) mass spectrometry.

Experimental. Water solutions of C60 prepared by vacuum-sublimation cryogenic deposition were mixed with water dispersions of MoS₂ for synthesis of (MoS₂ + C60) nanocomposite by ultrasound (1700 kHz) treatment. Stable grey water dispersions were obtained.



What do we know about the mass spectrometry of fullerenes?

- Fullerene produces radical ions both in positive (C60+•) and negative (C60⁻•) ion modes under different ionization techniques.

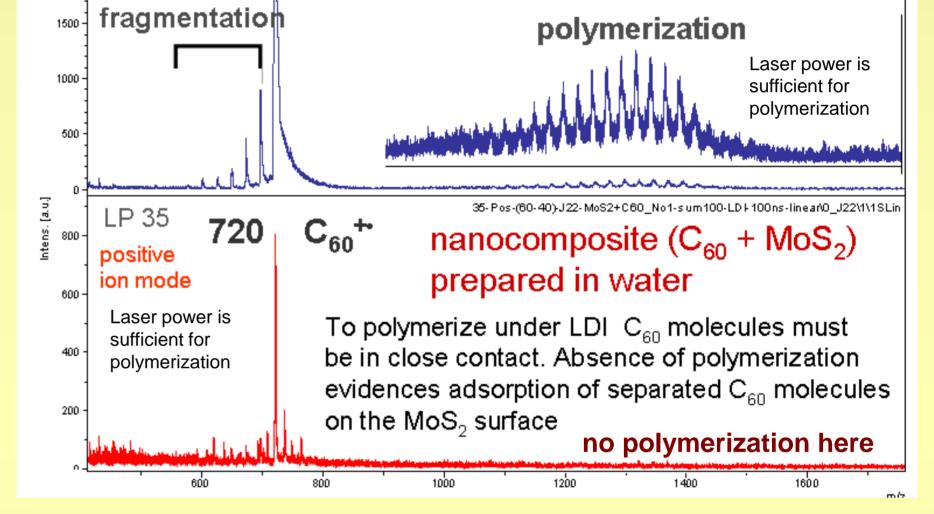
- C60⁺• can undergo fragmentation via subsequent loss of 2C units $[C60 - 2C_n]^+$.

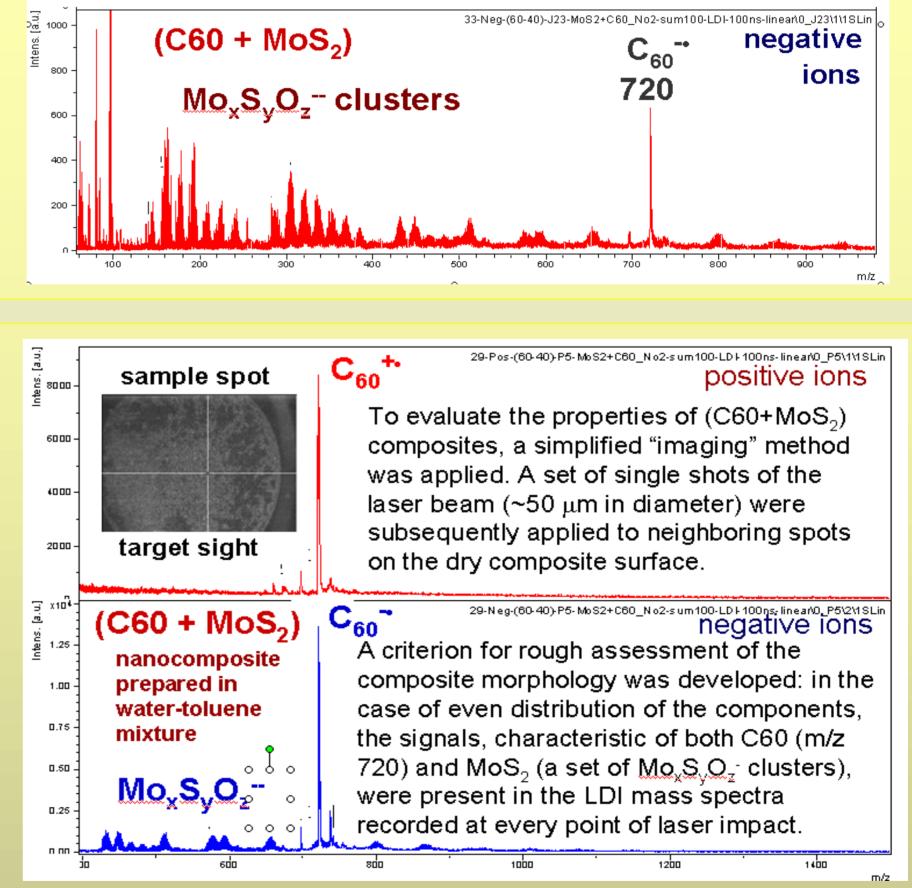
- C60 can be polymerized by addition of 2C units [C60+2C_n]⁺.

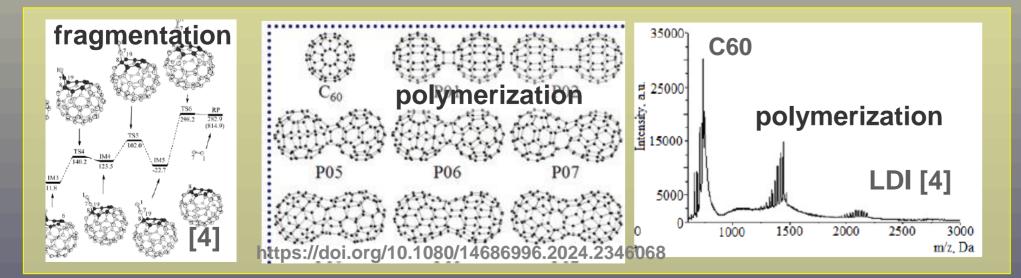
- Under LDI, molecular ions are obtained in desorption mode (under moderate laser power); polymerization occurs in ablation mode (high laser power); its degree depends on the solvent and substrate.

These processes are studied, in particular, at the Institute for Surface Chemistry [4].

What do we know about the mass spectrometry of MoS₂? LDI mass spectra of MoS₂ are recorded in the negative ion mode in the form of sets of clusters $Mo_xS_vO_z^-$. Each cluster is represented by a peak envelope emerging due to polyisotopic nature of molybdenum.







[1] G.V. Andrievsky, M.V. Kosevich, O.M. Vovk, V.S. Shelkovsky, L.A. Vashchenko, J. Chem. Soc. Chem. Commun. 12, 1281 (1995). https://doi.org/10.1039/C39950001281

[2] N.A. Vinnikov, S.V. Cherednichenko, A.V. Dolbin, V.B. Eselson, V.G. Gavrilko, R.M. Basnukaeva, A.M. Plokhotnichenko, Low Temp. Phys. 48, 336 (2022). https://doi.org/10.1063/10.0009739

[3] Cherednichenko S.V., Andrievsky G.V., Vinnikov N.A., Dolbin A.V., Kosevich M.V., Shelkovsky V.S., Basnukaeva R.M., Gnatyuk O.P., Bezkrovnyi O., Ptak M., Chaika M., Kuzema P.O., Dovbeshko G.I., Low Temp. Phys. 50, 248 (2024). https://doi.org/10.1063/10.0024965

[4] V.O. Pokrovskiy, A.G. Grebenyuk, E.M. Demianenko, V.S. Kuts, O.B. Karpenko, S.V. Snegir, N.T. Kartel, Laser desorption/ionization of fullerenes: experimental and theoretical study Him. Fiz. Tehnol. Poverhni, 4, 78 (2013).

CONCLUSIONS. Applying aqueous colloidal solutions of C60 [2] permits obtaining high-quality (C60 + MoS₂) nanocomposites. Even and rare distribution of C60 over the MoS₂ surface is confirmed by the absence of C60 polymerization under LDI conditions.

Acknowledgements.

This work was supported by the NAS of Ukraine grant N 0123U100628

V International Conference "Condensed Matter & Low-Temperature Physics", 2-6 June, 2025, Kharkiv, Ukraine