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The resistant agglomerate formation of bismuth ammonium potassium citrate in water solutions and their investigation by SAXS and ablation with using powerful terahertz radiation

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Abstract

The bismuth ammonium potassium citrate water solutions was investigated by small-angle x-ray scattering of synchrotron radiation X-ray diffraction and by ablation with using powerful terahertz radiation. It was found the existence of a resistant agglomerate of the bismuth ammonium potassium citrate in water solution. The ablation experiment confirms the existence of resistant agglomerate. The structure and the size of which depends on the concentration of solutions. WAXD experiment gives information about agglomerate - water molecules interaction. SAXS experiments was made at accelerators complex VEPP-3/VEPP-4.

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Keywords: bismuth potassium citrate ammonium, synchrotron small-angle X-ray scattering, THz laser ablation, colloidal particles, resistant agglomerate

1. Introduction

Developing since the 70s of the 20th century investigating methods of the structure of matter on the basis of the Siberian Synchrotron and Terahertz Radiation Center INP SB RAS allow studies of various levels of the organization the crystal lattice parameters and the internal structure of solids with the use of synchrotron radiation to

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study the structure of supramolecular aggregates and biological objects using complex Novosibirsk free electron laser (Kozlov, 2011).

As an object of research we selected anti-ulcer drug substance based on bismuth ammonium potassium citrate (BAPC) developed in ISSCM SB RAS (Yukhin, 2001). This drug helps the disintegration of Helicobacter pylori, increases the effect of some antibiotics and inhibits gastric secretion (Lambert, 1997). Also forming "polymer" protective film in the recesses ulcers crystalline bismuth subcitrate was discovered in animal models of prepared ulcers (Koo, 1982). The drug is practically not absorbed from the gastrointestinal tract.

To determine the in-situ study of transformations of potassium bismuth ammonium citrate used the best practices developed and implemented in the Siberian center of synchrotron and terahertz radiation. High-intensity synchrotron radiation (SR), several orders of magnitude excess power of the X-ray tubes, linear polarization and a small angular divergence allows to explore ultra-small concentrations of drugs in a reasonable exposure time does not exceed a few seconds.

Typically, the crystalline sample is required for X-ray diffraction studies. However, this is not typical for living systems. A widely used non-destructive analytical method to obtain information about the shape and structure of disordered materials such as complex systems, protein molecules of various dispersed systems, a small-angle x-ray scattering (SAXS). The intensity of the SAXS curves is proportional to the square of the difference between the electron densities of the medium and the scattering center, and for multiphase samples - additively. When bismuth complexes studying it gives an opportunity to get a signal at low concentrations due to their large atomic number. Another way to study the state of matter in solution without substantial degradation it is the method of ablation from solution by submillimeter radiation.

The aim of this work was to investigate water solutions of bismuth potassium citrate ammonium by SAXS and by submillimeter radiation from Novosibirsk free electron laser.

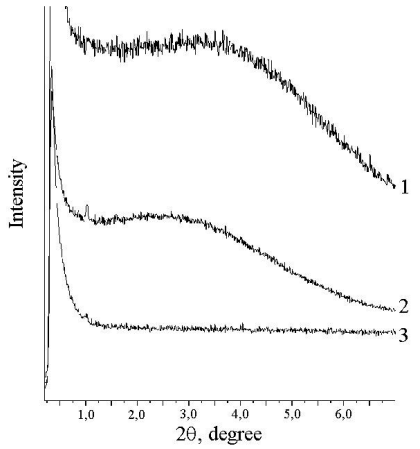

Fig. 1. Small-angle scattering from BAPC substance solutions: 1 - 0.5 M, 2 - 0.125 M, 3 - water. The radiation energy E = 8.333 keV.

2. Materials and methods

SR-SAXS measurements were performed at the station of channel 5b of VEPP-3 storage ring (Siberian Center for Synchrotron and Terahertz Radiation, INP SB RAS, Novosibirsk, Russia). Monochromatic radiation with the wavelength of 1.516 Å and a one-coordinate detector OD-3 (Aulchenko, 2009) were used for diffraction studies.

To study samples by means of the submillimeter laser ablation (Kozlov, 2010), the colloidal sample was placed into a horizontal cell. Filtered gaseous nitrogen was subsequently added to this cell in excess, which was necessary to prevent the arrival of aerosol from the surrounding environment. Before starting the work, the absence of aerosol in the gas line was tested. Ablation was performed by irradiation with 130±5 μm wavelength radiation, having average power of 15 W. The surface of the sample was located in the focal point of the sector mirror; exposure times varied from 3 to 10 seconds. The resulting aerosol was carried with nitrogen into a buffer reservoir (25 L) to stabilize the number of particles and prolonged analysis. The flow was subsequently brought to the aerosol classifier (SMPS+C, Grimm Aerosol Technik GmbH & Co. KG, Germany) to analyze the particle size distribution. Each measurement took 4 minutes to complete. Each sample was measured five times, and the size distributions were averaged across this series. Irradiation of pure solvents did not lead to the formation of particles.

Atomic force microscopy (AFM) studies were carried out in a tapping mode using an “INTEGRA” scanning probe nanolaboratory (NT-MDT, Russia). Nanoparticles were deposited on a freshly cleaved mica surface (3 x 3