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THE CONCEPT OF FRACTALS IN THE STRUCTURAL ANALYSIS OF NANOSYSTEMS: A RETROSPECTIVE LOOK AND PROSPECTS

The concept of fractals is widely used in various fields of science. By an example of the results obtained by L.A. Bulavin's scientific school, the tendency toward a more intense application of the fractal analysis to structural studies of nanosystems has been demonstrated. It is shown that the peculiarities in the distribution of nanosystems over their fractal dimensions are related to the mechanisms of growth and aggregation of the dispersed phase. An important aspect of the considered issue is the kinetics of the process under the influence of various factors. The leading role of small-angle scattering methods (analysis in the reciprocal Fourier space) together with microscopy ones (analysis in the direct space) used to study advanced nanostructured materials in various states of matter is emphasized.

K e y w o r d s: fractals, fractal dimension, nanosystems, aggregation.

1. Introduction

Forty-five years have passed since B. Mandelbrot introduced the term fractal [1]. Since then, the fractal concept has deeply penetrated into various areas of our life, such as mathematics and physics, chemistry, biology, geology, economics, painting, and design [2]. This approach enables a quantitative description of the systems characterized by scale invariance to be performed. This type of symmetry is naturally associated with the power distributions of elements of the system. The exponents of such distributions include its dimension. In the case of fractals, the latter differs from the dimension of the space that contains the system and even can take fractional values. Examples of such objects have been known to mathematicians for a long time. In particular, these are the Weierstrass function and the von Koch and Peano curves [2]. However, the development of unified approaches to describe both abstract and real natural objects belongs exactly to B. Mandelbrot.

The concept of fractal was especially successful in the physics of nanosystems, where its application has made clearer the understanding of the structural features in disordered, at first glance, systems. Fractal relationships found their reflection in a number of *in situ* methods, which allowed a precise determination of the substance structure parameters on the nanoscale to be performed. The scattering of radiation of various types (neutrons, X-rays, light), various kinds of microscopy (scanning and transmission electron, atomic force, and optical polarization ones), adsorption and rheological techniques, and so forth should be classified to this group of methods. They are actively applied to a diversity of nanoobjects that are included into the substance in various aggregate states, which is evidenced by a steady growth in the number of scientific publications devoted to the fractal analysis of structural data.

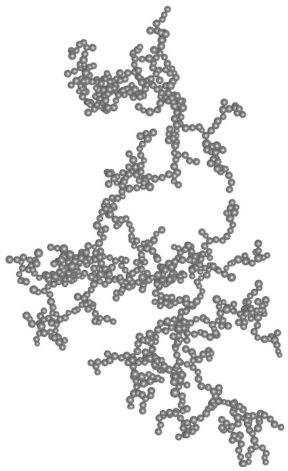

Fig. 1. Example of a volume fractal: a model cluster of colloidal particles with a fractal dimension of 1.5

It seems impossible to trace this trend in detail on the basis of the whole body of available literature sources, especially in the framework of a single review article. However, focusing the attention on a certain set of works that have a common basis, it is still possible to reveal certain regularities in the application and combination of different methods, the statistical distribution of fractal dimensions, and the chronology of the interest growth to this topic. For instance, publications of scientists belonging to the same scientific school can be sampled for the analysis. A remarkable example of such a school is the community of disciples of Academician of the National Academy of Sciences of Ukraine, Professor Leonid Bulavin. The scientists who started their careers at the Department of Molecular Physics of the Faculty of Physics at the Taras Shevchenko National University of Kyiv are now working at many institutions in Ukraine and abroad. It is their publications that were taken as a basis for this review. Thus, the aim of this work was to analyze the works of the scientific school of Prof. L.A. Bulavin in which the concept of fractal was applied to the structural diagnostics of nanosystems, as well as to establish the regularities in the distribution of researched objects over the types of fractal structures.

2. Fractal Nanosystems and Methods of Their Research

Natural fractals are objects (systems) that are identically ordered in a certain scale range. Therefore, in a statistical sense, any fractal system is self-similar [2] (Fig. 1). A mathematical quantity that makes a description of disordered systems possible is the parameter of fractal dimension. It can acquire any value within an interval from 0 to 3. For connected sets, such as nanoparticle clusters, this is an interval from 1 to 3. Very prolate or very oblate objects can also be formally classified to fractals because their dimensions (1 and 2, respectively) also differ from the dimension of the Euclidean space, where they are located.

In the general case, the volume fractal dimension D_V is introduced using the mass-to-radius ratio: the number of structural units N confined in a sphere of the radius r is determined as

$$N \propto r^{D_V}. \tag{1}$$

Against the background of the global problem of physical chemistry concerning the determination of the specific surface area of colloidal formations, we note that the fractal clusters are characterized by the following relation between the area and the volume:

$$S \propto V^{D_V/3}, \tag{2}$$

which means a larger specific surface area for systems with a higher fractal dimension. From whence, one can see that the surface occupies a substantial volume in considerably branched fractals ($D_V \to 3$).

In the literature, there are many kinetic models of aggregation processes giving rise to the formation of stochastic fractal structures. The fractal construction algorithms can be divided into two types: the particle-cluster and cluster-cluster aggregations. Some aggregation algorithms developed on the basis of stochastic processes are introduced in accordance with their natural analogs (e.g., the diffusion-limited aggregation, the reaction-limited aggregation, and so on) [2]. In addition, there are a number of models for the fractal growth of clusters. Some of them came from the physics of polymers (the statistical coil model, the Florey model), the others from the physics of phase transitions and critical phenomena (the percolation cluster). Thus, the formalism of fractal dimension allows peculiar classes of universality to be introduced in accordance with the models of fractal aggregation. As was marked above, the fractal dimension characterizes the degree of disorder of the system. From this point of view, the pair

correlation function has a monotonically decreasing asymptotics at $r \to \infty$,

$$g(r) \propto r^{D_V - 3}. \tag{3}$$

The physical processes that are used to measure a certain system are often convenient to be considered in a reciprocal space. The intensity of small-angle scattering is known to be proportional to the Fourier transform of the correlation function [3, 4]. From whence, it follows that the scattering intensity (the differential scattering cross-section per unit volume) for bulk fractal systems with the dimension D_V looks like

$$I(q) \propto q^{-D_V}. \tag{4}$$

In the works devoted to the analysis of fractal structures, only the asymptotic expression (4) is used, as a rule, to estimate the fractal dimension, although a more detailed analysis is available in the framework of the form-factor method.

Besides volume (mass) fractals, nanosystems can also contain surface fractals (Fig. 2). The latter can be defined as interfaces that are also characterized by self-similarity. Their dimension D_S differs from a value of 2 (this value is a characteristic of Euclidean surfaces) and varies within an interval from 2 to 3. Thus, we have a certain branched interface on a uniform substrate. In this case, the pair correlation function has the following asymptotics at $r \to 0$:

$$g(r) \propto 1 - Cr^{3-D_S}. \tag{5}$$

For surface fractals, the intensity of small-angle scattering looks like

$$I(q) \propto q^{D_S - 6}. \tag{6}$$

From whence, it follows that, for a fractal interface, the variation interval for the power exponent differs from that inherent to mass fractals, so that the method can distinguish between objects of two types. A limiting case for a fractal surface is a smooth Euclidean surface, for which $D_S = 2$. In this limit, the isotropic scattering by a two-phase system with a smooth interface is called the Porod scattering.

Another interval of the power exponent (from 4 to 6) characterizing the scattering intensity recession corresponds to the so-called diffusive surface around a homogeneous core [5]. Here, we deal with a weak inhomogeneity, but without scale-invariant properties.

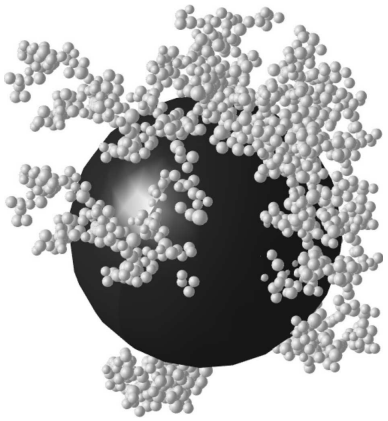

Fig. 2. Example of a surface fractal: a specific aggregation of nanoparticles on the surface of a submicron particle. The surface fractal dimension equals 2.5

Hence, the method of small-angle scattering allows the structures of various types to be identified by analyzing the behavior of the power exponent in their dependences of the scattering intensity decay as a function of the transferred wave vector magnitude q. In other words, an experimenter can immediately determine whether the examined objects are prolate or oblate, whether they are scale-invariant or not, whether the surfaces of nanoparticle possess diffusive or fractal properties, or whether they are smooth.

While analyzing the structure of both the surface and volume fractals in the direct space, it is important to determine the so-called cell dimension. The corresponding method is based on replacing the complicated shape of an object with a grid of d-dimensional cubes, each with the side ε. Let $N(\varepsilon)$ be the number of cubes containing at least one point of the object. Then, for the volume V, we may approximately write

$$V \approx N(\varepsilon)\varepsilon^d. \tag{7}$$

Hence, the number of test bodies required to cover the given set of points depends on the cube side length according to the relation

$$N(\varepsilon) \propto \varepsilon^{-D}. \tag{8}$$

The quantity $N(\varepsilon)$ can be interpreted as the number of pixels required to represent a system with the resolution ε.

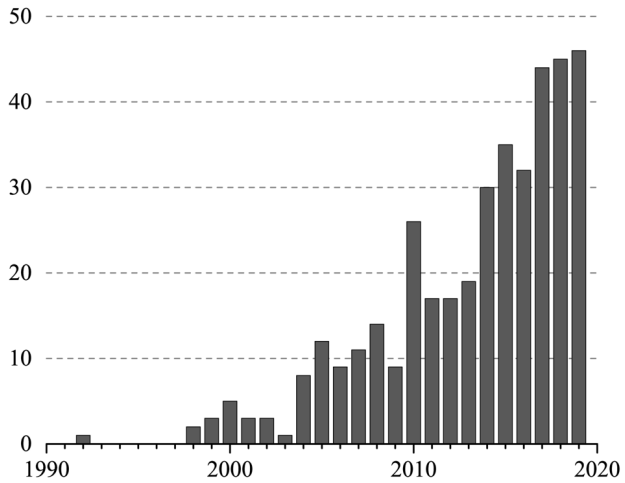

Fig. 3. Growth of the number of publications of L.A. Bulavin's scientific school on fractal topics during the last thirty years [3–394]

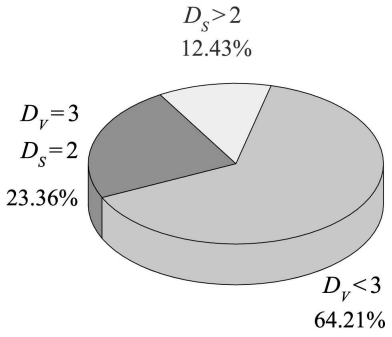

Fig. 4. Comparison of researched systems with respect to their fractal dimensions: volume and surface fractals, as well as non-fractal systems

Surfaces that can be described by a single-valued function $z(x, y)$ are studied with the help of the cross-section method. When the cross-sections of such a surface are oriented perpendicularly to the z-axis, the obtained level lines compose a set of closed curves. Those curves have a topological dimension equal to 1, so that they are easier to study than the surface itself. The fractal dimension D' of the level lines is connected with the surface dimension D_S by the formula

$$D' = D_S - 1. \tag{9}$$

Because of the symmetry of the x- and y-axes, the obtained set is self-similar. To determine the fractal dimension, the perimeter versus area relation, which is similar to relation (2), can be used:

$$P \propto S^{D_S/2}, \tag{10}$$

where P is the length of the cross-section boundary, and S the cross-section area. If the experimental determination of the area becomes a difficult task, the contours are analyzed using the cell dimension method applied to the two-dimensional case [Eqs. (7) and (8)]. This approach is widely used while analyzing the data obtained for the surface of nanoobjects, e.g., using atomic force microscopy.

In every particular case, specific experiments may be required in order to indirectly determine the fractal dimension – alternatively, with the help of Eq. (1). For example, the fractal morphology of aerosol particles can be described on the basis of the results of the sedimentation analysis or by estimating their behavior in the electrical or gravitational field. The results of applications of those methods are discussed in the next section in the framework of the fractal approach.

3. Experimental Studies of Nanosystems with Fractal Properties

During the last thirty years, the representatives of L.A. Bulavin's scientific school have studied, along with other objects, various nanosystems [3–394], including polymers, composites, and liquid systems. The plot in Fig. 3 testifies that the number of works devoted to this topic has been constantly growing since the 1990s. The analyzed sample of works includes the papers, where the studied systems either revealed fractal properties or, on the contrary, presented evidence of their dense packing or homogeneity ($D_V = 3$). In general, this behavior corresponds to the global trend in the science of nanostructured materials. The fractal description is applied more and more frequently because it can reveal new structural details that are often associated with macroscopic physico-chemical properties of the substance.

An analysis of the quantitative distribution of nanosystems over their dimensions demonstrates that less than a quarter of the researched specimens did not reveal fractal properties and, in general, could be described as Euclidean geometric bodies (Fig. 4). It was taken into account that several different systems could be studied in the same article. More than 64% of nanosystems were identified as volume frac-

tals with that or another dimension. About 12.4% of the studied specimens contained nanoformations with a fractal surface, i.e. the dimension of surface inhomogeneities exceeded a value of 2. This unexpected result testifies that a uniform mass distribution or an aggregation with dense packing at the nanolevel is rather an exception than a rule for practically important nanosystems. Physico-chemical conditions occurring at the natural or industrial synthesis often favor the emergence of a substantial inhomogeneity and the branch character of the system. It is the scale-invariant symmetry within a certain interval of sizes not exceeding a few micrometers that helps one to reveal an obscured order in such objects.

Furthermore, the distribution of nanosystems over their fractal dimensions turned out very non-uniform (Fig. 5). The majority of objects had a dimension of 2. This circumstance is a result of several important contributions. First, such objects as thin films [262, 263, 349, 359, 369], lipid bilayers [155, 191, 255], nano-sized laponite disks [162, 193] and some others are quasi-two-dimensional ones. Second, these are polymer systems in the statistical coil state under θ-conditions [315]. Third, these are branched fractal aggregates of nanoparticles, the formation of which is governed by mechanisms that are close to the model of ballistic cluster aggregation or reaction-limited aggregation [71, 171, 288].

The second place, according to the statistics, is occupied by objects with a fractal dimension of 1. These are substantially anisotropic prolate bodies. Their rod-like shape can be close to those of ellipsoids of rotation, cylinders, or prisms, but the main thing is that their size along one of the axes is much larger than the sizes in the other two directions. Such systems include amyloid solutions of proteins [367], nanoparticle chains [285, 286], nanotubes [394], and others.

Two more peaks are also observed in Fig. 5 in the vicinities of fractal dimensions of 2.4 and 1.7. Those values can be associated with the mechanisms of diffusion-limited single-particle and cluster-cluster aggregations, respectively. Thus, the aggregation kinetics is governed by such external factors as the temperature, pressure, and electromagnetic field, as well as the physico-chemical parameters of the nanoparticle surface. At the same time, changes of the kinetics type – e.g., the diffusion-limited or the reaction-limited mode – lead to differences in the structuring from the viewpoint of fractal parameters. The value

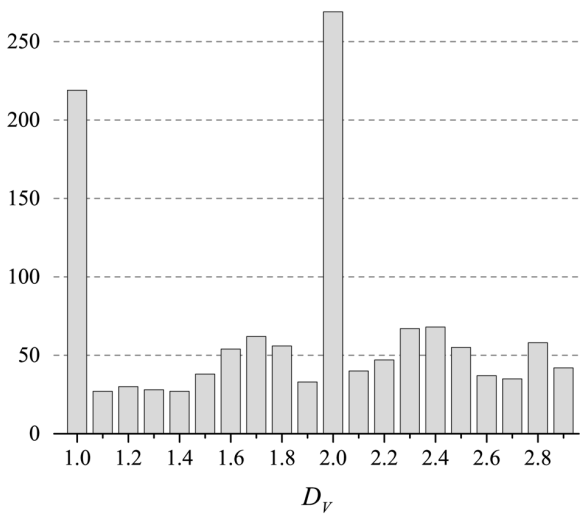

Fig. 5. Distribution of nanosystems with volume fractals over the dimensions of the latter

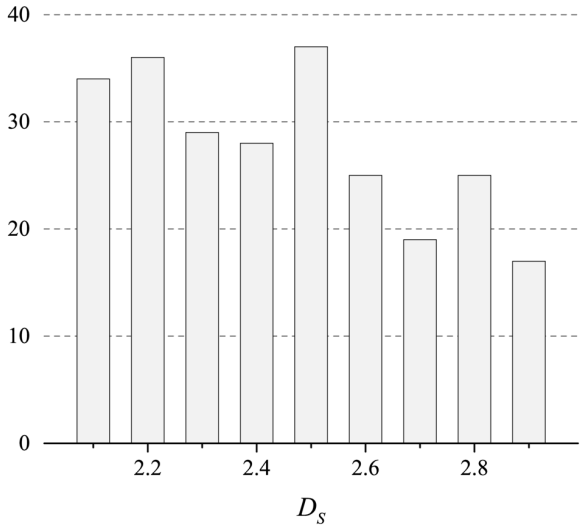

Fig. 6. Distribution of nanosystems with surface fractals over the dimensions of the latter

$D_V \approx 2.4$ is typical of the clusters of detonation nanodiamonds in polar solvents [5, 130, 147, 196, 230, 351], aqueous suspensions of fullerene aggregates [311, 362], polymer–surfactant mixtures [315], and others. The value $D_V \approx 1.7$ can be observed for aggregates in some magnetic colloids [91, 308, 367], proteins in mesoporous silicates [326], aluminum oxyhydroxide aerogel [236], and others.

On the contrary, the distribution of nanosystems over the surface fractal dimension (Fig. 6) is rather uniform, with a small decrease far from a smooth

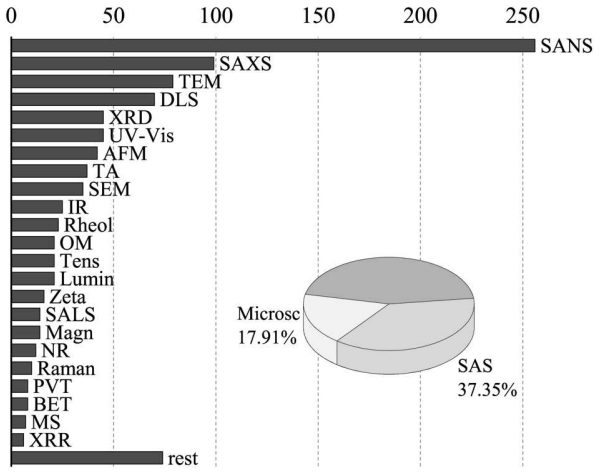

Fig. 7. The frequency of applications of the methods used in the researches of nanosystems. Notations: SANS – small-angle neutron scattering; SAXS – small-angle X-ray scattering; TEM – transmission electron microscopy; DLS – dynamic light scattering; XRD – X-ray diffraction; UV-Vis – light absorption spectroscopy in the ultraviolet and visible spectral intervals; AFM – atomic force microscopy; TA – thermal analysis, including differential scanning calorimetry and thermogravimetric analysis; SEM – scanning electron microscopy; IR – infrared spectroscopy; Rheol – rheological studies; OM – optical microscopy; Tens – tensiometry; Lumin – luminescent analysis; Zeta – electrokinetic potential measurements; SALS – small-angle light scattering; Magn – magnetometry; NR – neutron reflectometry; Raman – Raman scattering; PVT – determination of the thermal equation of state; BET – determination of the specific surface by low-temperature adsorption (the Brunauer–Emmett–Teller method); MS – mass spectrometry; XRR – X-ray reflectometry; rest – other methods inserting a small contribution to the given statistics. The insert demonstrates the cumulative fractions of small-angle radiation scattering methods, including neutrons, X-rays, and visible light (SAS), as well as microscopy methods, including electron, atomic force, and optical microscopies (Microsc)

surface at $D_S = 2$. At first glance, this is a result of minimizing the corresponding thermodynamic potential by reducing the free surface. Nevertheless, this issue has to be studied more thoroughly, because the examined processes are actually strongly nonequilibrium. The only exception is the value $D_S = 2.5$, which points to an analogy with the bulk variant of the diffusion-limited single-particle aggregation.

Various modern structural methods were used to study the properties of nanosystems. In Fig. 7, a statistical distribution of the application frequency of structural diagnostic methods in the works covered in this review is depicted. It was taken into account that several methods could be used simultaneously in the same work. According to the diagram, the methods of small-angle scattering of neutrons, X-ray radiation (including synchrotron radiation), and visible light are the most suitable for studying the fractal characteristics: together, they comprise more than 37%. It is also worth noting the share of microscopy methods. In particular, the optical, electron, and atomic force microscopy methods were used in almost 18% of the studies of fractal nanosystems in the direct space.

4. Conclusions

Hence, the concept of fractals has found its wide application in structural studies of nanosystems. By an example of the results obtained by L.A. Bulavin's scientific school, the tendency to a significant growth of applications of the fractal analysis of matter in various physical states was demonstrated. It was shown that a substantial number of nanosized formations reveal scale-invariant structural properties in the bulk or at the surface. The distribution of nanosystems over their dimensions is considerably non-uniform, which testifies to the predominance of certain growth mechanisms on the nanoscale. The important role of the microscopy and small-angle scattering methods in the framework of complex structural studies of nanosystems with fractal properties is emphasized. The presented results can be extrapolated, to a large extent, to the current state of nanophysics in whole.

Despite the significant progress in the examined issue, the mathematical concept of fractals still remains at the stage of its genesis. Its further development will be associated, first of all, with precision experiments aimed at determining the structure of not only self-similar but also self-affine systems, and at evaluating their anisotropy and lacunarity. An active application of this tool can help to deepen our understanding of the nanoworld.

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ФРАКТАЛЬНА КОНЦЕПЦІЯ
В СТРУКТУРНОМУ АНАЛІЗІ НАНОСИСТЕМ:
РЕТРОСПЕКТИВНИЙ ПОГЛЯД І ПЕРСПЕКТИВИ

Р е з ю м е

Концепція фракталів широко використовується в різноманітних галузях науки. На прикладі результатів наукової школи Л.А. Булавіна була продемонстрована тенденція до все активнішого застосування фрактального аналізу в структурних дослідженнях наносистем. В роботі показано, що особливості розподілів наносистем за фрактальними розмірностями пов'язані з механізмами росту і агрегації дисперсної фази. Важливим аспектом є саме кінетика процесу під дією різних чинників. Підкреслена провідна роль методів малокутового розсіяння (аналіз в оберненому просторі Фур'є) в поєднанні з методами мікроскопії (аналіз в прямому просторі) при дослідженні актуальних наноструктурованих матеріалів в різних агрегатних станах.