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T.M. Shevchuk[1], M.A. Bordyuk[2], V.A. Mashchenko[3], V.P. Kvasnikov[4], V.V. Krivtsov[1]

Percolation characteristics of filled polyurethane auxetics

[1]*Rivne State University for the Humanities, labor.relax@gmail.com*
[2]*Rivne Medical Academy, bordiuk57@ukr.net*
[3]*National University of Water and Environmental Engineering, Scientific and manufacturing company “PRODECOLOGIA”, Rivne, Ukraine, v.a.mashchenko@muwn.edu.ua*
[4]*National Aviation University, Kyiv, Ukraine, kvp@nau.edu.ua*

According to the experimental values of the propagation velocities of longitudinal and transverse ultrasonic waves, the Poisson's ratio of polyurethane systems filled with metal particles was determined. For such systems, the Poisson's ratio is negative. Its value for metal-filled polymer auxetics with polyurethane matrix allowed to determine fractal dimensions and critical percolation indices. This approach made it possible to clarify the features of structure formation in polymer auxetics. It is shown that the fractal-percolation characteristics of these systems are determined by the type of metal filler and the size of its particles.

Keywords: polymer auxetic, Poisson's ratio, fractal, critical percolation indices, percolation cluster, coordination number, macrolattice.

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Introduction

The growth of the use of polymeric materials in various areas of human life causes the expansion of their production [1]. The development of modern science of polymers makes it possible to create a variety of polymer systems. The development and achievements of modern science of polymers open opportunities for the creation of polymer composite materials for multifunctional purposes using one of the most effective methods of modifying their structure and properties – filling [2, 3].

Among such materials a special place belongs to polymer auxetics [4–6], which find prospects for application in various fields of science, technology, production [7, 8]. Various models are used to analyze the structural features of polymer systems with a negative Poisson's ratio and their auxetic behavior [9, 10]. Peculiarities of structure formation in polymer auxetics indicate thermodynamic nonequilibrium of these processes and their nonlinearity. This makes it possible to use, as for other polymers, fractal and percolation approaches [11–14].

The aim of this work is to determine the percolation and structural parameters of auxetic polyurethane compositions and their interpretation based on a model of a polymer with a critical filler content.

I. Materials and methods of research.

Thermoplastic polyurethane (TPU) synthesized on the basis of 4,4-diphenylmethane diisocyanate, 1,4-butanediol and polyoxytetramethylene glycol with MM = 1500, filled with fine powders of iron (Fe), molybdenum (Mo), copper (Cu). The size of the particles was 0.3 to 1.0 μm. The compositions were obtained by direct mixing in T-p mode. The volume concentration of filler for TPU systems corresponded to the critical in the system and, respectively, for Mo, Fe, W, Cu is 48; 43, 52, 50 vol. % was used for the research [15, 16].

Determination of the propagation velocity of the longitudinal υ_l and the transverse wave υ_t of the ultrasonics waves was performed at a frequency of ω = 1,884 MHz and by a method based on comparing the results of direct measurements of the propagation time of the probe pulse through the immersion fluid in the absence of a sample (τ) and in the presence of a sample (τ_l) between the emitter and the signal receiver [17, 18]. The value of υ_l is determined by the ratio:

$$\upsilon_l = \frac{\upsilon_{liq} d}{d - \Delta\tau_l \upsilon_{liq}}. \tag{1}$$

Where υ_{liq} – the speed of propagation of the ultrasonic wave in the immersion fluid; d – the thickness of the sample material with strictly parallel surfaces, $\Delta\tau_l$ – the difference in time of the probing pulses ($\Delta\tau_l = \tau - \tau_l$).

The value of the υ_t is calculated by the ratio:

$$\upsilon_t = \frac{\upsilon_{liq}}{\sqrt{\sin^2(\theta_{kr}) + \left(\cos(\theta_{kr}) - \frac{\upsilon_{liq}\Delta\tau_t}{d}\right)^2}}. \tag{2}$$

Where $\Delta\tau_t$ – the difference between the time of passage of the probing pulse in the absence of the sample and in the presence of the sample (τ_t), placed at an angle $\theta_{kr.}$ to the direction of fall.

At known values υ_l, υ_t the Poisson's ratio of the polymeric auxetic is calculated by the ratio:

$$\nu = \frac{2 - \left(\frac{\upsilon_l}{\upsilon_t}\right)^2}{2\left(1 - \left(\frac{\upsilon_l}{\upsilon_t}\right)^2\right)}. \tag{3}$$

The results of determining the propagation velocities of ultrasonic waves, the Poisson's ratio of TPU-systems are presented in Table 1.

II. Results and discussion.

In [15], TPU-systems were considered on the basis of a cluster model [19], for which the structural-dimensional characteristics of clusters (ordered regions) and intercluster (non-ordered regions) were determined on the basis of a fractal approach.

Depending on the content of filler in the polymer matrix, there may be spatial-planar structural organizations of two types. If the filler content is less than critical, in the absence of particle segregation, they form a quasi-one-dimensional non-periodic lattice (with unequal distances between particles), in which there is no cooperative movement of nodes. The intensity of their individual movement will be determined by the temperature of the thermostat, particle size, viscosity of the polymer matrix. Under the action of external forces, the filler particles will move independently of the neighboring ones, like colloidal particles in a medium with high viscosity. The critical content of filler in the polymer matrix leads to structural changes in the system, namely to the transition of the polymer matrix in the interfacial state, its properties will differ from the properties of the unfilled polymer. The filled polymer under such conditions is considered as two-component: interfacial layer-filler. In this case, the system of filler particles is ordered, and the movement of its nodes immersed in the interfacial layer depends on the state of the neighbors. Such a structurally ordered system is considered as a periodic macro lattice [20].

The use of such modeling of the structural organization of TPU-systems allows to analyze it on the basis of the percolation approach [21]. In such a system, the structures are characterized by critical percolation indices. Middle-order regions in the polymer matrix and the interfacial layer form the framework of the percolation cluster, and such a subset is described by the critical percolation index β. Another subset in the structural organization of the polymer consists of disordered regions characterized by a critical percolation index ν_0. At the critical content of the filler, its particles form endless chains connected by macromolecular elements and a framework. Their behavior is described by the critical indices γ, t. Critical indices of percolation were determined by the values of the fractal dimension of the structures of the filled polymers:

$$\beta = \frac{1}{d_f}; \quad \nu_0 = \frac{2}{d_f}; \quad \gamma = \frac{3}{d_f}; \quad t = \frac{4}{d_f}. \tag{4}$$

To find the fractal dimension of the structures of such systems used a synergistic approach, in which:

$$d_f = (d - 1)(1 + \nu), \tag{5}$$

where $d = 3$ – dimension of Euclidean space.

The values of the critical percolation indices (Table 2) indicate a significant difference in the structural organization in the unfilled polymer and TPU-systems. This difference in the values of the critical percolation indices indicates the different behavior of the structures of TPU-systems during the transfer of different types of energy excitations (mechanical, thermal, electrical). These values for polyurethane auxetics at a critical filler content significantly exceed the corresponding values for traditional filled systems (for example for systems based on polyvinyl chloride and polyvinyl butyral with a critical filler content) [21].

For filled polymer auxetics, the structure of the percolation cluster can be conditionally taken as two-dimensional ($d = 2$) or, taking into account the volume

Table 1

Characteristics of TPU-systems.

Compositions	ρ_p, kg/m^3	$\rho_{fill.}$, kg/m^3	$\varphi_{fill.}$, vol. %	υ_l, m/s	υ_t, m/s	$r_{fill.}\cdot 10^{-7}$,m	ν
TPU	1125			1635	1415	3.0; 4.0; 5.0; 6.0; 9.0; 10	– 0.99
TPU+48 vol. % W	1125	19000	0.48	1755	1405	3.0; 4.0; 5.0; 6.0; 9.0; 10	– 0.39
TPU+43 vol. % Mo	1125	9010	0.43	1700	1355	3.0; 4.0; 5.0; 6.0; 9.0; 10	– 0.38
TPU+52 vol.% Fe	1125	7870	0.52	1775	1370	3.0; 4.0; 5.0; 6.0; 9.0; 10	– 0.24
TPU+50 vol. % Cu	1125	8900	0.50	1750	1500	3.0; 4.0; 5.0; 6.0; 9.0; 10	– 0.89

Table 2

Critical percolation indices for TPU-systems.

Compositions	β	ν_0	γ	t
TPU+48 vol. % W	0.89	1.63	2.45	3.27
TPU+43 vol. % Mo	0.8	1.61	2.41	3.22
TPU+52 vol.% Fe	0.66	1.31	1.97	2.63
TPU+50 vol. % Cu	4.55	9.09	13.6	18.18
TPU	50	100	150	200

effects, three-dimensional with $d = 3$. The value of critical percolation indices allows in such a model to determine the maximum size of a finite cluster:

$$\Delta = d\nu_0 - \beta = \frac{2d-1}{d_f}. \quad (6)$$

In case of detection of planar effects $\Delta = \frac{3}{d_f}$, and volumetric – $\Delta = \frac{5}{d_f}$.

The properties of the percolation cluster in the self-similar mode characterizes the parameter *S* and describes the rate of growth of the fractal resistance with increasing spatial scale. The value of anomalous diffusion Θ in the analysis of the sorption properties of filled polymer systems indirectly allows to take into account the influence of scale on the diffusion coefficient. Relationships were used to calculate these characteristics:

$$\Theta = \frac{t-\beta}{\gamma}; \quad S = \frac{t}{\nu_0} + d - 2 \ \text{(for } d = 2 \text{ and } d = 3\text{)}. \quad (7)$$

The calculated values of the corresponding characteristics of the percolation cluster for TPU-systems are given in Table 3.

Table 3

An indicator that determines the maximum cluster size, an indicator of the properties of the percolation cluster in the self-similar mode and the characteristics of anomalous diffusion for TPU-systems.

Compositions	Δ		*S*		Θ
	$d = 2$	$d = 3$	$d = 2$	$d = 3$	
TPU	150	250	2	3	1
TPU+48 vol. % W	2.37	4	2	3	1
TPU+43 vol. % Mo	2.42	4.03	2	3	1
TPU+52 vol. % Fe	1.96	3.27	2	3	1
TPU+50 vol. % Cu	4.58	13.67	2	3	1

In this model, the skeleton of an infinite percolation cluster of fractal structure in filled polyurethane composites is characterized by the fractal dimension d_b taking into account the critical index β_b, which describes the strength of the cluster skeleton (for $d = 2$ index $\beta_b = 1.67$ and for $d = 3$– $\beta_b = 1.68$), as well as the fractal dimension of wanderings d_w, the internal dimension of wanderings $d_{w'}$ and spectral dimension d_s [22]:

$$d_b = d - \frac{\beta_b}{\nu_0}; \ d_w = \frac{d_f}{2+\Theta}; \ d_{w'} = 2 + \Theta; \ d_S = \frac{d_f}{2+\Theta}. \quad (8)$$

During the formation of the percolation cluster there is a resistance (ε), which is characterized by the relationship between the resistance of the cluster and its resistivity (d_t), the dimension of the moments of time at which wandering returns to the starting point and the dimension of the outer boundary of the cluster (d_h)

$$\varepsilon = d_{w'} - d_f; \quad d_t = 1 - \frac{d_s}{2}; \quad d_h = 1 + \frac{1}{\nu_0}. \quad (9)$$

The above parameters that characterize the processes of structure formation of the percolation cluster are presented in Tables 4, 5.

Within the percolation approach for polymer auxetics, the dependence of the Poisson's ratio on the structures of the matrix and filler was used. According to [23], there is a relationship between ν and the coordination number (z) of the percolation lattice:

$$\nu = \frac{d - \frac{z}{4}}{d(d-1) + \frac{z}{4}}. \quad (10)$$

The values of ν for TPU-systems determine the coordination number for two cases:

$$z = 8 - 16\frac{\nu}{\nu+1} \quad \text{(for } d = 2\text{)};$$

$$z = 12 - 36\frac{\nu}{\nu+1} \quad \text{(for } d = 3\text{)}; \quad (11)$$

As follows from the data presented in Table 5, z differs significantly for TPU-systems from filled polymers, for which $0 < \nu < 0.5$. This indicates that when

Table 4

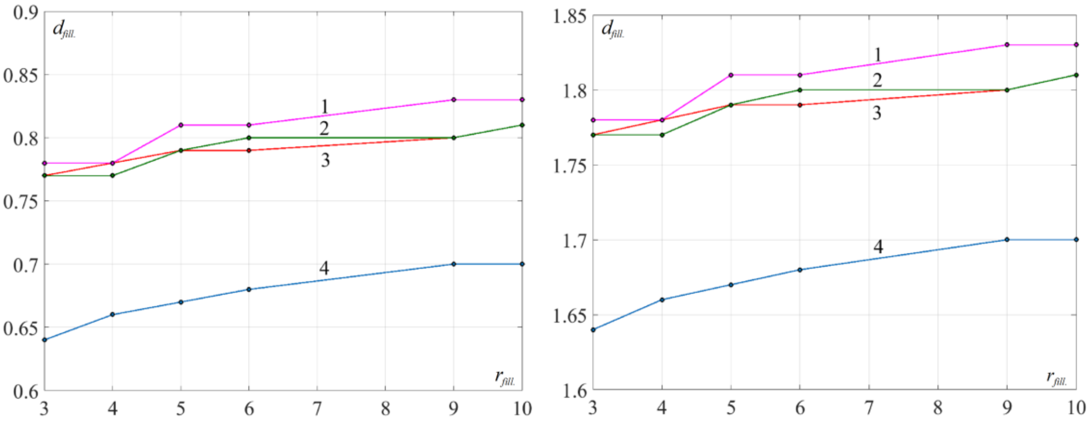
Dimensionality of percolation clusters, fractal dimension of wanderings, internal dimension of wanderings and spectral dimension of the system for TPU-systems.

Compositions	d_b; $d = 2$; $\beta_b = 1.67$	d_b; $d = 3$; $\beta_b = 1.68$	d_w	$d_{w'}$	d_s
TPU	2.98	1.98	0.007	3	0.13
TPU+48 vol. % W	0.97	1.97	0.41	2,97	0.82
TPU+43 vol. % Mo	0.96	1.96	0.42	3	0.83
TPU+52 vol. % Fe	0.72	1.72	0.5	3	1
TPU+50 vol. % Cu	0.82	2.82	0.07	3	0.15

Table 5

The relationship between the resistance of the cluster and its resistivity, the dimension of the moments of time t at which the wander returns to the starting point and the dimension of the outer boundary of the cluster for TPU-systems**.**

Compositions	ε	d_t	d_h	z	
				$d = 2$	$d = 3$
TPU	2.98	0.94	1.01	1592	3572
TPU+48 vol. % W	1.75	0.59	1.61	18.2	35
TPU+43 vol % Mo	1.76	0.58	1.62	17.76	34
TPU+52 vol. % Fe	1.48	0.5	1.76	14.12	23.5
TPU+50 vol. % Cu	2.78	– 0.98	1.11	137.6	303.6



a) b)

Fig. 1. Dependence of $d_{fill.}$ on the particle size of the fillerfor 1 – TPU+Fe, 2 – TPU+Cu, 3 – TPU+Mo, 4 – TPU+W: a – for a two-dimensional model; b – for a three-dimensional model.

applying external loads, the deformation of the percolation lattice occurs according to different patterns than for traditional filled polymer systems.

In the macrolattice model, the surface of the filler particle was considered as a fractal of dimension $d_{fill.}$. The fractal relationship between the specific surface $S_{fill.}$ and the particle diameter of the filler $D_{fill.}$ is expressed by the ratio [24]:

$$S_{fill.} = 410\left(\frac{D_{fill.}}{2}\right)^{d_{fill.}-d}. \qquad (12)$$

From where, we get:

$$d_{fill.} = d + \frac{lnS_{fill.} - ln\,410}{lnr_{fill.}}, \qquad (13)$$

where $r_{fill.}$ – the radius of the filler particle (nm). Since the surface of the metal filler is a source of potential field determined by the number of contacts: the structural element of the macromolecule-force center of the filler will have that the number of contact points depends on the dimension available for such contact surface of the particle d_u, which is defined as follows:

$$d_u = d_{fill.} + -1 + \frac{d - d_{fill.}}{d_{wf}}, \qquad (14)$$

where d – dimension of the Euclidean space in which the fractal is considered (in our case $d = 2; 3$), d_{wf} – dimension of random wandering on a fractal, which can be estimated by the Aaron-Stauffer ratio:

$$d_{wf} = d_{fill.} + 1. \qquad (15)$$

The effective value of this dimension d_u^{eff} decreases with increasing nominal value. The observed behavior is described by the following equation [24]:

$$d_u^{eff} = 2 - d_u. \qquad (16)$$

The dependence of these characteristics on the particle sizes of the metal filler for TPU-systems are presented in Figures 1–4 for two- and three-dimensional models $d_{fill.}$

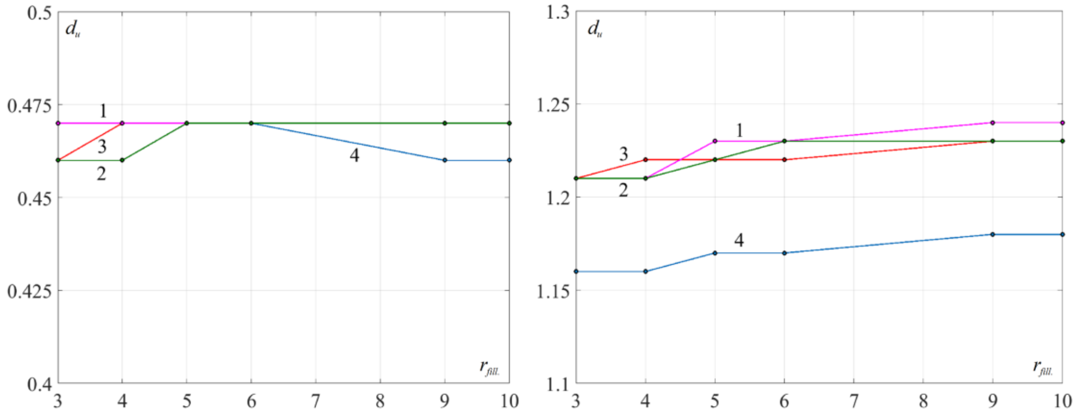
As evidenced by the results of calculations of quantities $d_{fill.}$ and d_w (Fig. 1, 3) the increase in the particle size of the filler causes a non-monotonic growth of the corresponding fractal characteristics for the introduced metal ingredients. However, the increase in particle size of the metal filler does not lead to significant changes in the dimensions of the available points of contact of the structural elements of TPU macromolecules with the active centers of the surface (Fig. 2, 4).

For the framework of the filler particles, the fractal dimension $D_{fp.f.}$ was determined according to the ratio:

$$D_{fp.f.} = d - d_f. \qquad (17)$$

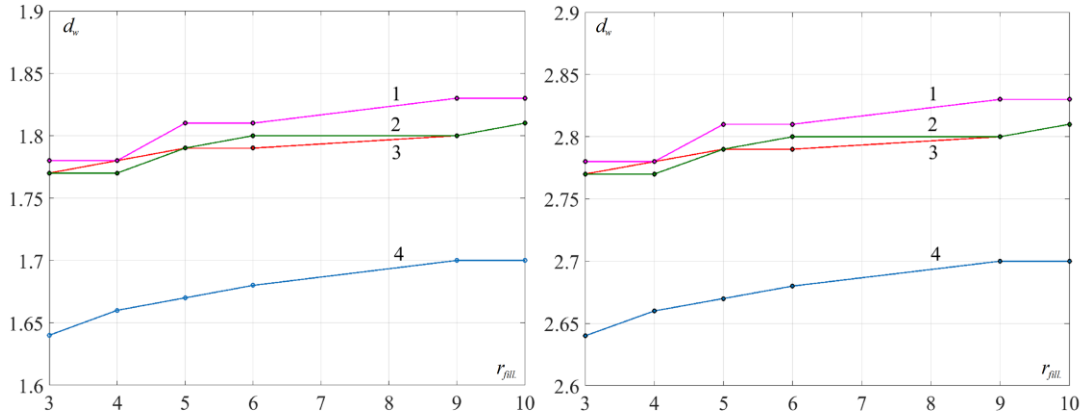
The results of the calculations indicate that under volume defects ($d = 3$), the particles of fillers during deformation are characterized by linear behavior.

Therefore, in the model of filled polymer auxetic at the critical content of the filler of its structure form film structures.



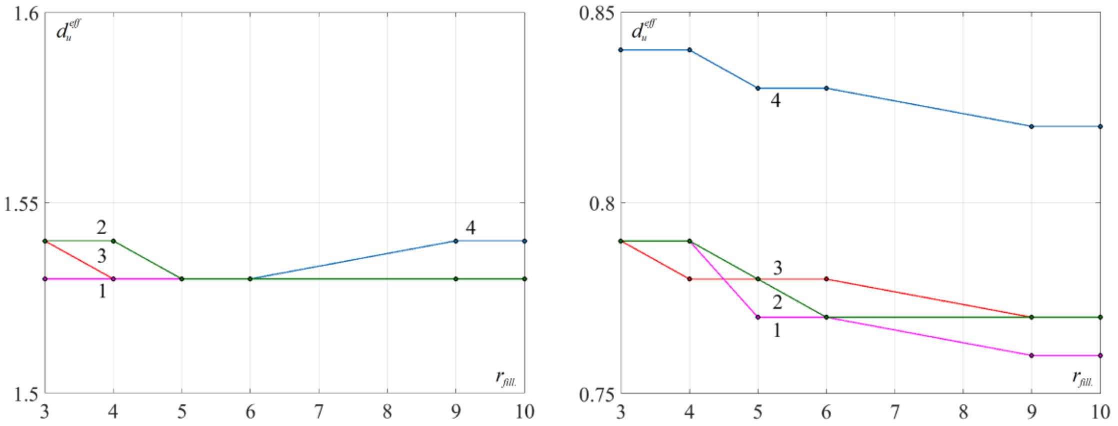
a) b)

Fig. 2. Dependence d_u from the sizes of particles of a filler for 1 – TPU+Fe, 2 – TPU+Cu, 3 – TPU+Mo, 4 – TPU+W: a – for two-dimensional model; b – for a three-dimensional model.



a) b)

Fig. 3. Dependence d_w from the sizes of particles of a filler 1 – TPU+Fe, 2 – TPU+Cu, 3 – TPU+Mo, 4 – TPU+W: a – for two-dimensional model; b – for a three-dimensional model.



a) b)

Fig. 4. Dependence d_u^{eff} from the sizes of particles of a filler 1 – TPU+Fe, 2 – TPU+Cu, 3 – TPU+Mo, 4 – TPU+W: a – for two-dimensional model; b – for a three-dimensional model.

Table 6

Fractal dimension of the frame of the filler particle.

Compositions	$D_{f\,p.f.}$	
	$d = 2$	$d = 3$
TPU+48 vol. % W	0.78	1.78
TPU+43 vol. % Mo	0.76	1.76
TPU+52 vol. % Fe	0.48	1.48
TPU+50 vol. % Cu	1.78	2.78

Consolations

The use of percolation approaches to TPU-systems has shown that their structural parameters differ from the corresponding characteristics of traditional filled systems, in particular on the basis of polymers of the vinyl series [21]. Percolation parameters of clusters of thermopolyurethane auxetics are determined by the type of metal filler and their size. Consideration of the polymer matrix, filler particles as fractals makes it possible to analyze the processes of structural organization in polymer auxetics.

The determined percolation parameters allow to analyze the behavior of TPU-systems in power (mechanical and electric) and thermal fields.

Shevchuk T.M.* –** Ph. D, associate professor of the department of physics;***

Bordyuk M.A.* –** Ph. D, associate professor of the department of fundamental disciplines;***

Mashchenko V.A* –** Ph. D, associate professor of the department of automation, electrical engineering and computer-integrated technologies;***

Kvasnikov V.P.* –** doctor of technical sciences, professor, head of the department computerized electrotechnical systems and technologies;***

***Krivtsov V.V.* –** Ph. D., associate professor of the department of physics, astronomy and teaching methods.

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Т.М. Шевчук[1], М.А. Бордюк[2], В.А. Мащенко[3], В.П. Кваснiков[4], В.В. Крiвцов[1]

Перколяційні характеристики наповнених поліуретанових ауксетиків

[1]*Рівненський державний гуманітарний університет, Україна, labor.relax@gmail.com*
[2]*Рівненська медична академія, Україна, bordiuk57@ukr.net*
[3]*Національний університет водного господарства та природокористування, Науково-виробнича фірма „ПРОДЕКОЛОГІЯ”, Рівне, Україна, v.a.mashchenko@nuwn.edu.ua*
[4]*Національний авіаційний університет, Київ, Україна, kvp@nau.edu.ua*

За експериментальними значеннями швидкостей поширення поздовжніх і поперечних ультразвукових хвиль визначено коефіцієнт Пуассона поліуретанових систем наповнених частинками металу. Для таких систем коефіцієнт Пуассона є від’ємним. Його значення для металонаповнених полімерних ауксетиків з поліуретановою матрицею дозволило визначити фрактальні розмірності та критичні індекси перколяції. Такий підхід дав можливість з’ясувати особливості структуроутворення в полімерних аукетиках. Показано, що фрактальні-перколяційні характеристики таких систем.

Ключові слова*:* полімерний ауксетик, коефіцієнт Пуассона, фрактал, критичні індекси перколяції, перколяційний кластер, координаційне число, макрогратка.