Analysis of Electrospun Nanofibers Flaws from Polymeric Solution of Polyetherimide

LILIANA ROZEMARIE MANEA[1,2*], ROXANA SCARLET[1], ION SANDU[2,3*]

[1] "Gheorghe Asachi" Technical University Iasi, Faculty of Textile and Leather Engineering and Industrial Management, 29 Dimitrie Mangeron Blvd., 700050, Iasi, Romania

[2]Romanian Inventors Forum, 3 Sf.P.Movila Str., L11, III/3, 700089, Iasi, Romania

[3]"Alexandru Ioan Cuza" University of Iasi, ARHEOINVEST Interdisciplinary Platform, 11 Carol I Blvd., Corp G, 700506, Iasi, Romania

Any activity dedicated to the evaluation and amplification of qualitative level is based on a deep and systematic analysis of flaws. This represents in fact an analysis of quality by means of non-quality. Utilization of a new polymer with a specific structure as a raw material needs a study on the flaws appeared on the surface of fibrous deposits. The analysis of non-quality is related to the determination and characterization of the appeared flaws and the analysis of the causes which generate them, such as: the raw material through its characteristics, the equipment by its technological and constructive parameters, environmental parameters, as well as lack of correlation between the specified parameters and characteristics. The present paper contains a study related to a series of flaws appeared through electrospinning of a polymeric solution of polyetherimide (PEI) with a concentration of 12%, using as solvent a mixture of dimethylacetamide/tetrahydrofuran (DMAC/THF), 1:1 ratio. Fibers obtained through electrospinning were studied by Scanning Electron Microscope method to analyze the appeared flaws in correlation with their technical and technological causes.

Keywords: *electrospinning, nanofibers, polyetherimide, flaws, SEM*

The electrospinning process and equipment, respectively, are relatively simple, efficient and capable of producing long, continuous, exceptionally long and direction-controlled fibers. Nowadays, the electrospinning technology is the only one capable of leading to the forming of continuous filaments with diameters up to a few nanometers [1-7]. The ever increasing world interest in the electrospinning process and the high potential of the applications areas are demonstrated by some other numerous aspects; as outlined below [8, 9]:

- the necessity of integration into the European development strategy regarding the development of high precision non-conventional technologies which lead to nanomaterials with applications in medicine, biomedicine, molecular electronics, molecular photonics, catalytic chemistry, filtering etc.
- the special importance given to the accomplishment of the state-of-the-art smart systems and materials which include new and innovative knowledge for controlling and monitoring the industrial technological processes;
- the large number of papers published in the most known databases.

Therefore, this nanotechnology is one of the most promising areas to be developed in the future in numerous domains of applicability (technical applications, filters, cosmetics, protection, medical dressings, tissue engineering, artificial organs, electronics and nanosensors, drugs, textile carriers for drugs etc.) [10-14]. The stages of electrospinning fibers formation are: jet initiation, its extension and finally nanofiber formation [15-19].

Initiation of electrospun polymer jet

Electrospinning is described as the dynamic equilibrium between the repulsive Coulombian forces and the surface tension of the processed polymeric solution. When the polymer viscosity is adequate, the drops of polymeric solution are transformed in electrically charged jets which solidify in polymeric filaments under the existence of the dynamic drawing equilibrium, electric field and gravity forces [20-25]. Initially, the drop of electrospun polymeric liquid is at equilibrium with gravity force, impeding the solution to flow from the needle point. By applying an alternative potential to the polymeric solution, as the potential difference between the two electrodes increases, the electric forces which act on the charges induced in solution overbalance the associated forces with the value of the surface tension, thus determining the jet initiation.

Jet extension

The polymeric solution drop formed by electrospinning remains in suspension at the needle point, being sustained by superficial forces. Simultaneous with physical electrospinning process it takes places the solvent evaporation, which determines nanofibers solidification on collector surface. As the electric charge exceeds the surface tension, the polymeric solution split off in small drops [26-29]. Under the stretching effect of the electric field, the electrostatic forces manifest a deformation dynamics. Subsequently, after the polymeric filament goes out of the Taylor zone, under the action of the field, with a quasi-linear shape, the delivered jet is subjected to a complex bending strength, meanwhile the electrostatic forces stressing and thinning the jet. Once the polymeric jet is out of the Taylor cone, it is thin, shaped as a continuous filament. The jet is electrically charged and its distribution is intensified in the presence of the electric field generated by the electric potential and the electric charge of polymeric solution [30-32].

* email: manearozemarie@yahoo.com, Tel.: +40-721-334034; sandu_i03@yahoo.com, Tel.: +40-744-431709

Hydrodynamic instability of electro statically delivered jet.

In the presence of the electric field, the polymeric jet manifests the phenomenon of hydrodynamic instability just after leaving the needle point [33-36]. Once appeared the jet bending, this generates a series of helical sequences as the result of the interaction between the rejection forces between the electric charged captured by the jet, and the stabilizing forces due to superficial forces, viscoelasticity (if any) and the applied electric field. The diameter of the generated loops described by the jet trajectory reaches some centimeters, and the mean speed with which they descend to the collecting surface remains reduced (~1m/s). That is why the elongation forces acting for some fractions of second on the jet in its motion toward the collecting surface determine the appearance of a thinning of electrospun jet diameter. Experimental studies [8, 9, 37-45] have revealed the presence of two distinctive phases in jet formation (fig.1), namely:

- hydrodynamic stability phase or "stable jet" phase, when the electrospun polymer particles which left the capillary are accelerated and slightly stretched by the electrostatic forces in the longitudinal direction within the area between the two electrodes; within this area the electric field is uniform and constant and is not affected by the electric charges carried by the polymer jet (this phase directly contributes to the thinning of the polymeric solution jet, at the same time creating conditions for the phenomenon of hydrodynamic instability);
- the phase of hydrodynamic instability which is present at a certain distance from the needle point (unstable jet) - the experiments confirm that the very thin fibers are obtained from electrospun polymeric jets which present the instability phenomenon, while the nanofibers with larger diameters are obtained from hydro dynamically stable jets.

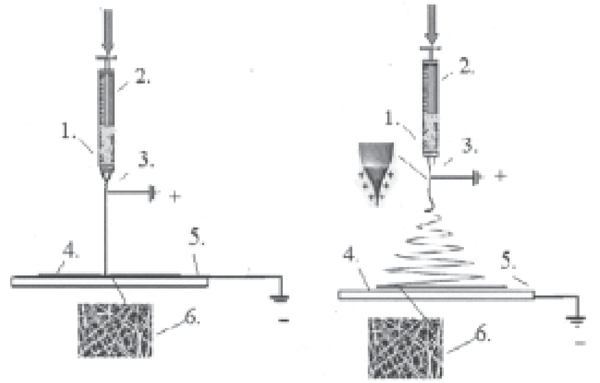

Fig. 1. Typologies of jets delivered during electrospinning process (a. stable jet; b. instable jet): 1- polymeric solution; 2 - syringe; 3 - needle; 4 - fibrous surface; 5 - collector; 6 - electrospun fibers

Because the lateral disturbance of the jet increases as the response to repulsive force between the jets, the unstable jets bend, forming loops in this spiral motion, after which the jet is dispersed in multiple filaments, generating nanofibers. The longitudinal distortion of the delivered jet determined by the electric field stabilizes the axially pulverized jets, such that unique jets (thermodynamically stable) are subjected to a mechanical elongation in the area comprised between the needle and the collector, thus leading to nanofibers generation.

The surface tension of the polymeric solution influences the jet instability. This can be diminished by adding surfactants in polymeric solution. Even small amounts of surfactants can modify viscoelasticity, thus influencing the electrospun jet stability.

Nanofibers formation occurs in the presence of a high electric field when the external forces exceed the surface tension. It is hard to specify the exact trajectory of the jet in the presence of disturbances appeared in the polymeric jet. The assessments realized until now [8, 9, 37-40] are based on the distribution of the electric field which leads to Taylor cone formation, in the case of plane, symmetrical, axially distributed collecting mechanisms, and the uniform distribution for the radial symmetric electrode, respectively multiple cones for drum collecting mechanisms, with significant differences in electric field distribution. At the capillary outlet the geometric shape can be of drop, spindle or jet; the most important characteristic for get characterization in the area of instability phenomenon generation is the conductivity.

The basic criteria for the classification of various modalities of electro hydrodynamic spraying are: geometrical shape of the liquid drop when leaving the capillary, and the instability type (the way in which the jet is disintegrated in drops). The characterization of electrospinning process, either theoretically or experimentally, is not simple. Electrospinning process is based on a complex combination of phenomena from electrostatics, fluid mechanics and polymer science. Both the properties of polymeric solution and the process variables can significantly affect the electrospinning process; until now, there is no complete description of its basic mechanism [40-47]. The variables which influence, as decision factors, upon the morphological structure of the nanofibers are grouped, as shown in figure 2.

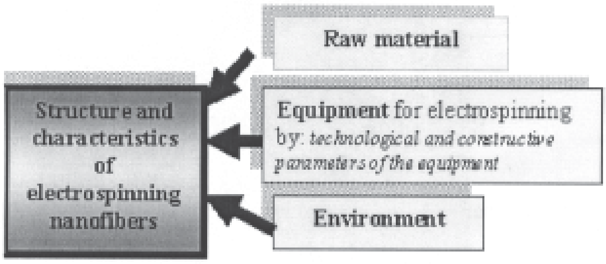

Fig. 2. Groups of factors which influence the structure and the characteristics of the electrospinning nanofibers

The present paper proposes an analysis of non-quality of electrospun fibers obtained from polyetherimide (PEI) solution with the concentration of 12%, using as solvent a mixture of dimetylacetamide/tetrahydrofuran (DMAC/THF) 1:1 ratio, which implies the characterization of the appeared defects and the analysis of the causes of these defects. In order to eliminate the not quite negligible influence of the polymeric solution characteristics on the formation of fiber structural defects, the work proposes to analyze the defects and their causes for the same polymeric solution.

Experimental part

Materials and methods

The electrospun polymer solutions were made of polyetherimide with molecular weight M_w = 39000g/mol, and the solvent mix DMAC/THF, 1:1 ratio. The characteristics of polyetherimide used in experiments in concentration of 12% [2, 3, 13, 14, 21] were: tensile strength up to 200°C, elongation 56%, bending modulus $3.3\cdot10^3$MPa, elasticity modulus $3.4\cdot10^3$ MPa, specific weight 1.3N/m^2, shearing strength 10^3MPa, Poisson coefficient 0.36 and dielectric constant 3.2. The solvents used in polymeric solutions have the following characteristics: DMAcA: molar mass 87g/mol, density at 20°C 0.94g/mL, boiling point 166°C, melting point 20°C, viscosity $1.956\cdot10^{-3}$ Pa·s;

Table 1
ELECTROSPINNING CONDITIONS

parameters	Values of the parameters / constructive types	
parameters maintained constant	feeding mechanism	syringe volume = 3mL, number of attached syringes = 3, inter-nozz distance 2.5mm; interval of displacement along the *Ox* axis, x 100mm; interval of displacement along the *Oz* axis, z = 80mm;
	environment parameters, 20°C, RH = 40% under normal atmospheric pressure;	
constructive and technological parameters variable	inner diameter of the needle, Φ = 0.2 - 0.4mm;	
	constructive type of collector: rotating cylinder, cylinder rotation speed v = 1000rp respectively v = 0rmp;	
	the distance between needles and collector: D = 45mm, D = 70mm, D = 120mm, D 130mm;	
	flow rate Q, mL/min 0.05mL/min, 0.075mL/min, 0.1mL/min, 0.15mL/mi 0.2mL/min;	
	Voltage U = 15kV, U = 20kV, U = 25kV, U = 30kV, U = 35kV.	

THF characteristics: molar mass 72g/mol, density at 20°C 0.889g/mL, boiling point 66°C, melting point 110°C, viscosity la 25°C, 0.48Pa·s [2, 3, 13, 14, 21, 48-54].

Preparation of polymer solution

The polymer was dried at 100°C for 2h, and then solved at 50°C for 24h in the mix DMAC/THF, 1:1 ratio [55-60].

Nanofibers electrospinning device was a computerized electrospinning equipment with needle, with collecting mechanism type rotary cylinder [2, 3, 61-64] which carries out electrospinning from polymeric solutions.

Electrospinning conditions

The present work is the result of an ample study concerning PEI processability through electrospinning. The parameters maintained constant and constructive and technological parameters variable during the entire process are presented in table1.

Experimental strategy implied that, at the same values of the voltage U, kV, to carry out experiments for the four values of the inter-electrodes distance, D, mm. Each of these experiments were performed at the four values of the flow rate Q, mL/min.

Results samples were analyzed by the method of Scanning Electron Microscope. From samples made the randomly selected samples were subjected to a gold plating procedure (with Phenom G2 pro equipment) to increase the sharpness of SEM images [13, 14, 21, 37].

Results and discussions

The structural defects of electrospun fibers studied in this work include: bead structural defects, necking patterns, fibers inlaid with polymeric solution film, secondary jets, branches, nanodrops. The work does not discuss the appearance of fiber non-uniformities (coefficient of variation of fiber diameter values), as this represents the subject of previous studies [10].

The appearance of structural defects of electrospun fibers is caused by polymeric solution processing at inadequate values of technological, constructive or environmental parameters. There are no strict intervals, generally valid, of the values of polymeric solution characteristics or technological, constructive or environmental parameters, for which one can obtain from whatever polymeric solution, dry uniform long fibers with small fibers diameters or small coefficients of diameters variations. The structure and morphology of the electrospun fibers depend on all the factors which influence the electrospinning process [11-14].

Even if the electrospinning process is controlled by this multitude of characteristics (of the polymeric solution) and parameters (constructive, technological and environmental), many of these only have an irrelevant influence on the values of mean fibers diameters and their non-uniformities, or on the possibility of fiber defect appearance [48]. That is why in most of the theoretical and experimental studies [1, 7, 48-51] are taken into account only the influence of certain characteristics and parameters, such as viscosity, molecular weight, solution conductivity, surface tension, solvent volatility, solution feeding rate, applied voltage, spinning distance nozzle orifice diameter, inter-nozzle distance etc.

The integrity of fibrous structure deposited on the collector surface depends on: overlapping degree of delivered nanofibers, interactions developed between delivered fibers. The topics of structural defects generation in electrospun fibrous deposits can be approached in different ways: starting from their causes or plurality of causes (correlated treatment of the causes which influence the appearance of certain defects) or starting from defects (treating the defect produced by various individual or cumulated causes). The fact that the same structural defect can be determined by several causes (several characteristics of polymeric solution and several parameters with uncorrelated values) leads us to an adequate approach starting from the cause to the effect.

Polymeric jet instability

For the same values of the spinning distance, the increase of the applied voltage leads to the increase of electric field intensity which, in fact, will favour the generation of thinner fibers [1, 7]. Contrary to this logics, when processing the polyetherimide solution with concentration of 12% using as solvent a mixture of DMAC/THF 1:1 ratio (with previously presented characteristics), when increasing the applied voltage for the same value of the spinning distance, less finer electrospun fibers were obtained. This can be explained by the decrease of the polymeric jet stability zone and the appearance of electro-hydrodynamic instabilities respectively. The increase of the applied voltage has as a secondary effect the increase of the solvent mix evaporation rate, resulting in the increase

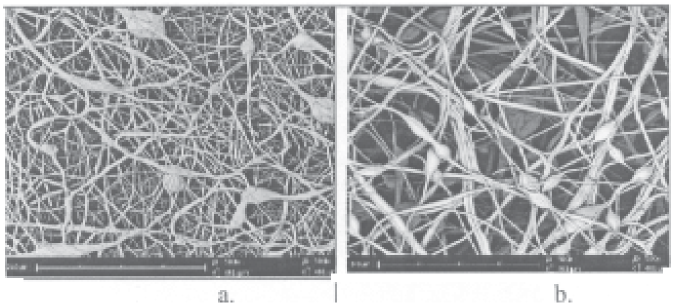

Fig. 3. SEM images for fibers with bead structural defects from 12 % PEI solution in DMAC/THF (1:1 ratio), processed on electrospinning equipment at flow rate Q = 0.175mL/min: a. U = 30kV and D = 100mm; b. U = 35kV and D = 100mm

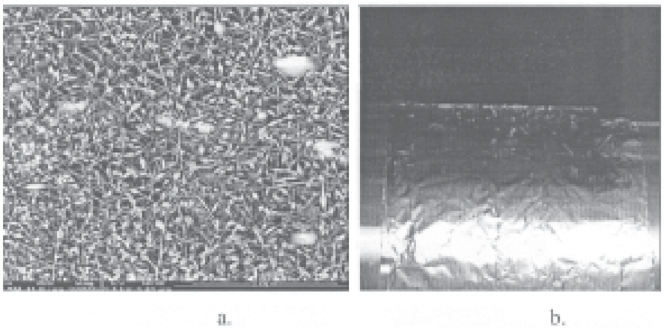

Fig. 4. Drop of polymer solution from PEI 12% (DMAC/THF) 1/1 ratio: a. drops interlaid between collected fibers, Q = 0.175mL/min, U = 35kV, D = 130mm; b. drops on collector surface, Q = 0.2mL/min, U = 35kV, D = 130mm

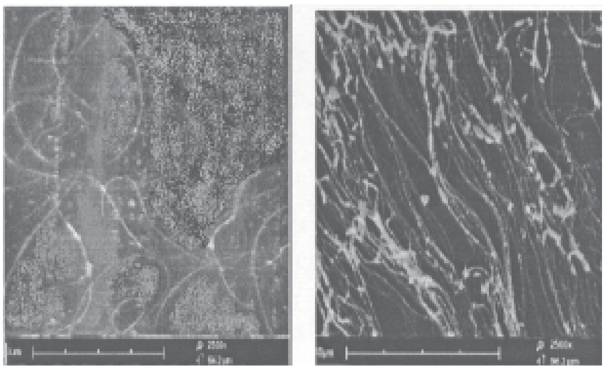

Fig. 5. Effect of polymer instability on fibrous deposits: a. different shapes of deposits on collector surface; b. irregularly spread nanodrops

of fibers diameter values. The structural defects determined by this multitude of factors which result together to the appearance of polymeric jet instability are: beads and necking patterns, linked fibers.

Bead structural defects represent a frequent fiber defect in the case of electrospinning processing of any polymer type. The specialized literature specifies that for each polymer solution characteristic (concentration, viscosity, molecular weight, solution conductivity, surface tension, solvent volatility) there are critical values starting with which bead structural defects appear under the condition of keeping constant all the other process and environmental parameters [1, 7], and these critical values are specific for each polymeric mixture. Figure 3 presents the SEM images for fibers with bead structural defects from PEI solution in DMAC/ THF (1:1 ratio) processed on electrospinning equipment at flow rate Q = 0.175mL/min.

One can notice that the frequency of bead structural defects significantly increases with the increase of the applied voltage, due to the increase of electro hydrodynamic instability. One can also see that the shape of bead structural defects is not perfectly spherical, but elongated (necking pattern); this can be the effect of the sufficient high values of polymeric solution concentration and feed rate respectively.

Big electrohydrodynamic instability also leads to the appearance of deposits under the form of *nanodrops* (fig. 4a).

These nanodrops appear irregularly spread, like some points distributed on the fibrous deposit surface. If the feed rate is excessively large (an inadequate feed rate value for polymeric solution processing), only drops are deposited from the polymer jet, without being able to form electrospun fibers. This is the case of processing the 12% PEI solution in DMAC/HF (1:1 ratio) on electrospinning equipment at a flow rate Q = 0.2mL/min (fig. 4b).

Figure 5 presents fibrous deposits with random arrays (*worms*) determined by a large jet instability.

Viscoelastic behaviour of polymeric jet delivered in the electrospinning process depends on the flow rate. The lack of control of solvent evaporation and solidification processes during the experiments determines the appearance of bending instabilities and implicitly of linked fibers (fig. 6).

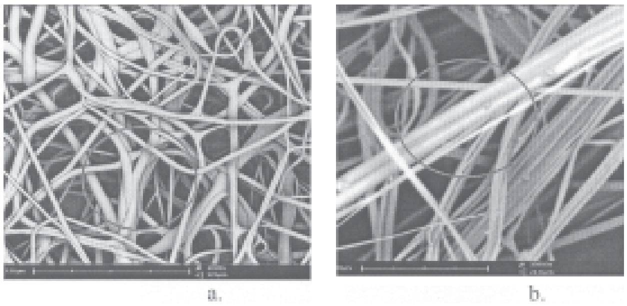

Fig. 6. SEM images for linked fibers from 12% PEI solution in DMAC/THF (1:1 ratio) processed on electrospinning equipment at a flow rate Q = 0.1mL/min: a. fibers with drops (U = 30kV and D = 100mm); b. linked fibers (U = 30kV and D = 100mm)

The phenomenon appears at the contact points between the electrospun fibers deposited on the collector surface in the case when they reach the collector surface in liquid state and do not have enough time to dry. A large flow rate of the polymer under the action of a small applied voltage, on the background of environmental conditions (temperature and humidity) inadequate to the characteristics of processed polymeric solution leads to the appearance of linked fibers in the collected fibrous deposit.

High flow rate values adopted at processing polymeric solutions, for which the evaporation rate is small or the spinning distance is too small can result in the generation of fibrous deposits with a random character, namely solution deposits under the form of films interlaid with fibers (fig. 7).

High evaporation rate of the solvent or solvent mix in the present case, together with big surface tensions,

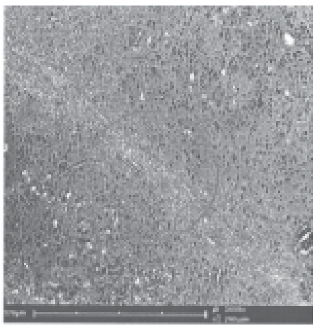

Fig. 7. SEM images with fibers and areas of randomly deposited polymer films from 12% PEI solution in DMAC/THF (1:1 ratio), processed on electrospinning equipment at flow rate Q = 0.2mL/min, U = 35kV, D = 45mm

Fig. 8. SEM images for fibers with agglomerations of polymer solution of 12% PEI in DMAC/THF (1:1 ratio), processed on electrospinning equipment at flow rate Q = 0.175mL/min, U = 15kV, D = 130mm

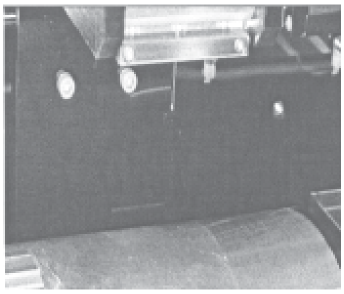

Fig. 9. Twiggy jet from 12% PEI solution in DMAC/THF (1:1 ratio), processed on electrospinning equipment at flow rate Q = 0.175mL/min, U = 35kV, D = 130mm

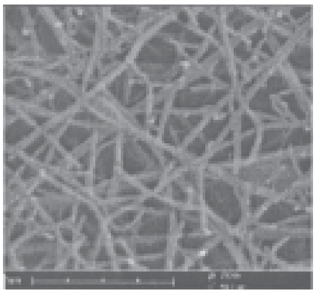

Fig. 10. Generation of secondary jets from 12% PEI solution in DMAC/ THF (1:1 ratio), processed on electrospinning equipment at flow rate Q = 0.2mL/min, U = 35kV and D = 130mm

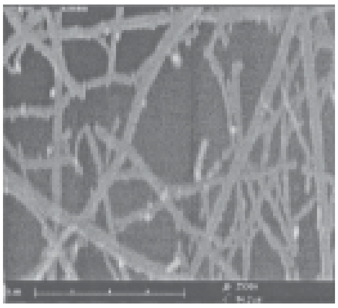

Fig. 11. Generation of deposits formed from 12% PEI solution in DMAC/THF (1:1 ratio), processed on electrospinning equipment at flow rate Q = 0.2mL/min, U = 35kV and D = 130mm

spinning distances and environmental conditions (temperature and humidity) inadequate for the characteristics of processed polymeric solution can result in deposits which contains fibers with agglomerations of polymeric solution (fig. 8).

The characteristics of electrospun polymeric solution (polymer concentration, solvent evaporation rate) have a relevant influence on the surface tension. Under the condition of keeping constant all the technological and constructive parameters, the surface tension will determine the technologically established stability domain.

Utilization of a large value of the applied voltage can determine the generation of twiggy jets. Figure 9 presents the formation of a twiggy jet as the result of electrospinning a PEI 12% solution with DMAC/THF (1:1 ratio) at flow rate Q = 0.175mL/min, U = 35kV, D = 130mm (fig. 9).

Non-uniform nanofibrous structures are obtained together with the appearance of secondary jets derived from the primary jet (secondary jets, branches or of another deposits formed upon nanofibers (figs. 10 and 11).

Secondary or branched jets can appear at larger values of the applied voltage or due to spraying manner.

Conclusions

The structural defects of electrospun fibers studied in this work include: bead structural defects, necking patterns, fibers inlaid with polymeric solution film, secondary jets, branches, nanodrops. In order to eliminate the not quite negligible influence of the polymeric solution characteristics on the formation of fiber structural defects, the work takes in analysis the defects and their causes for the same polymeric solution.

Getting fibers with pre-established quantitative and qualitative characteristics (reduced average diameters, uniform dry flawless fibrous deposits) resulted from a stable continuous technological process is based on pluri-disciplinary theoretical knowledge, as well as on vast experimental studies. An accurate and correlated selection of all the parameters (polymeric solution concentration, solvent volatility, applied voltage, feed rate, spinning distance) can significantly reduce the appearance of structural defects on electrospun fibers.

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