EQUIPMENT FOR OBTAINING NANOFIBERS THROUGH THE ELECTROSPINNING SYSTEM

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Abstract: From a large and international perspective the paper suggests a new design of the process equipment: modularity and automatic controllability. Modularity has been implemented so that it should allow the systematic variation of a large number of parameters influencing the electrospinning process. The existing modular designs usually allow the variation of a small number of parameters. In addition, there is to our knowledge no reported system in which the position of the collector can be adjusted to be both vertical and horizontal. Automatic controllability allows the real time control of the parameters and the simultaneous recording of the changes in the fluid jet and macroscopic nanofiber morphology. Again, there is to our knowledge no electrospinning device incorporating all these features.
Key words: equipment, electrospinning, parameters, nanofiber, system, process

1. INTRODUCTION

The electrospinning technology allows the obtaining of continuous fibers with diameters up to a few nanometers by processing different polymers (synthetic, natural, polymers loaded with nanoparticles or active agents, metals, ceramics) for obtaining nanofibers with various degrees of complexity and arrangement, architecture (structures), respectively.
The electrospun fibers are considerably finer than either the human hair or a pollen grain (Fig. 1).

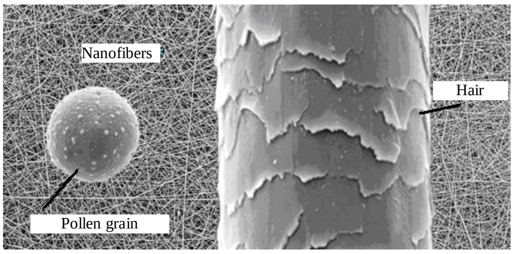


Fig.1. SEM image of a hair vs. pollen grain vs. nanofibers

Numerous technologies for obtaining nanofibers have been developed up to the present day. Among these, the most efficient from the view point of the productivity and efficiency is the electrospinning technology. At the same time, one has to underline the fact that the electrospinning process is an efficient one, capable of producing long and continuous fibers with controlled arrangement, being adaptable to various types of polymers. This process is nowadays the only one which allows the obtaining of continuous fibers with diameters up to a few nanometers.
On having in view the functional aspect and the range diversity of the nanofibers accomplished for various applications, numerous electrospinning equipments built up throughout the years have had various constructive modifications. The most significant modifications brought to the electrospinning equipments, on starting from first patent (Formhals, 1934) are: the multijet electrospinning (with more syringes) (Fang et al., 2004), the electrospinning system by means of the UV irradiation (Wilkes & Gupta, 2003), the multilayers electrospinning system (polymers consecutively-electrospun on the same collector surface), the mixed electrospinning system (usage of various syringes for the simultaneous deposition of the various polymers upon the collector surface) (Kwon et al., 2005), the electrospinning of the nanofibers without the usage of the needles for eliminating the drawbacks connected with the clogging of the tips of the syringe needles (Yarin et.al., 2006), the coaxial electrospinning system (Wang et al., 2005).
Industry researchers have identified the electrospinning processing as one of the key technologies of the future to be capable of ensuring the obtaining of innovative materials with implementation in the most various domains of application. The characteristics of the nanofibers resulted from the electrospinning technology make them attractive for various implementation domains, namely, the biomedical usage (tissue engineering, bandages, dressings, controlled-released textiles,

medical prosthesis, etc), composites, materials for electrodes, electronic and optical devices, membranes for filtering media, smart clothes, supports for catalysts and enzymes, sensors, aeronautical materials with high strength to weight ratios.

The aim of this paper is to present a computerized electrospinning equipment, accomplished by means of our own researches and used for obtaining nanofibers.

The ever-increasing interest concerning the technology for obtaining nanofibers through the electrospinning method at the world level and the high potential of the application areas, respectively, are demonstrated by numerous other aspects such as, for instance, the special importance given to the accomplishing of smart systems which include new innovative knowledge with a view to control and monitor the industrial technological processes. The obtaining of nanofibers with pre-established morphologies and characteristics is desired in the future. This thing can be possible by introducing some new collectors, by micro-electro-mechanical systems (MEMS) at the capillary or by using auxiliary electrodes for the stabilization of the polymeric jet.

With a view to substantiate the electrospinning process and to understand the existing correlations, one suggests to infer all the factors (raw material, equipment, environment) which, when brought together, lead to the obtaining of some nanofibers whose structures and characteristics correspond to the requirements imposed by the destination (Fig. 2) (Manea et. al., 2011).

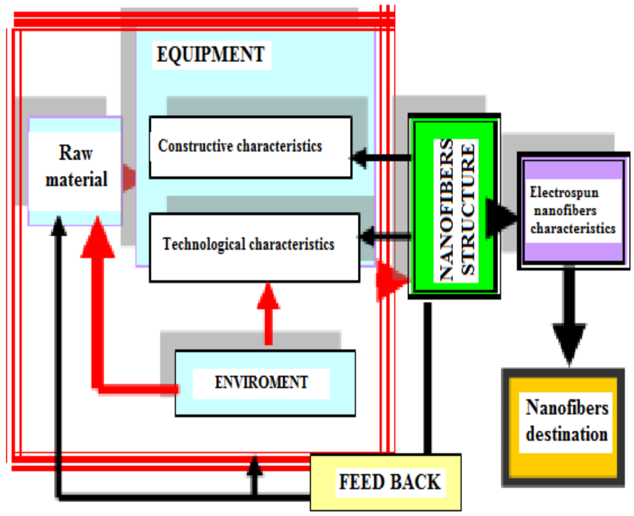


Fig. 2. Factors which influence the characteristics of the electrospun nanofibers, [8]

2. THE ELECTROSPINNING EQUIPMENT

The accomplished electrospinning equipment includes a pair of electrospinning electrodes, a high-voltage power supply, an acquisition unit, self-contained power supply units, computation unit for operating and controlling the electrospinning process (Fig. 3).

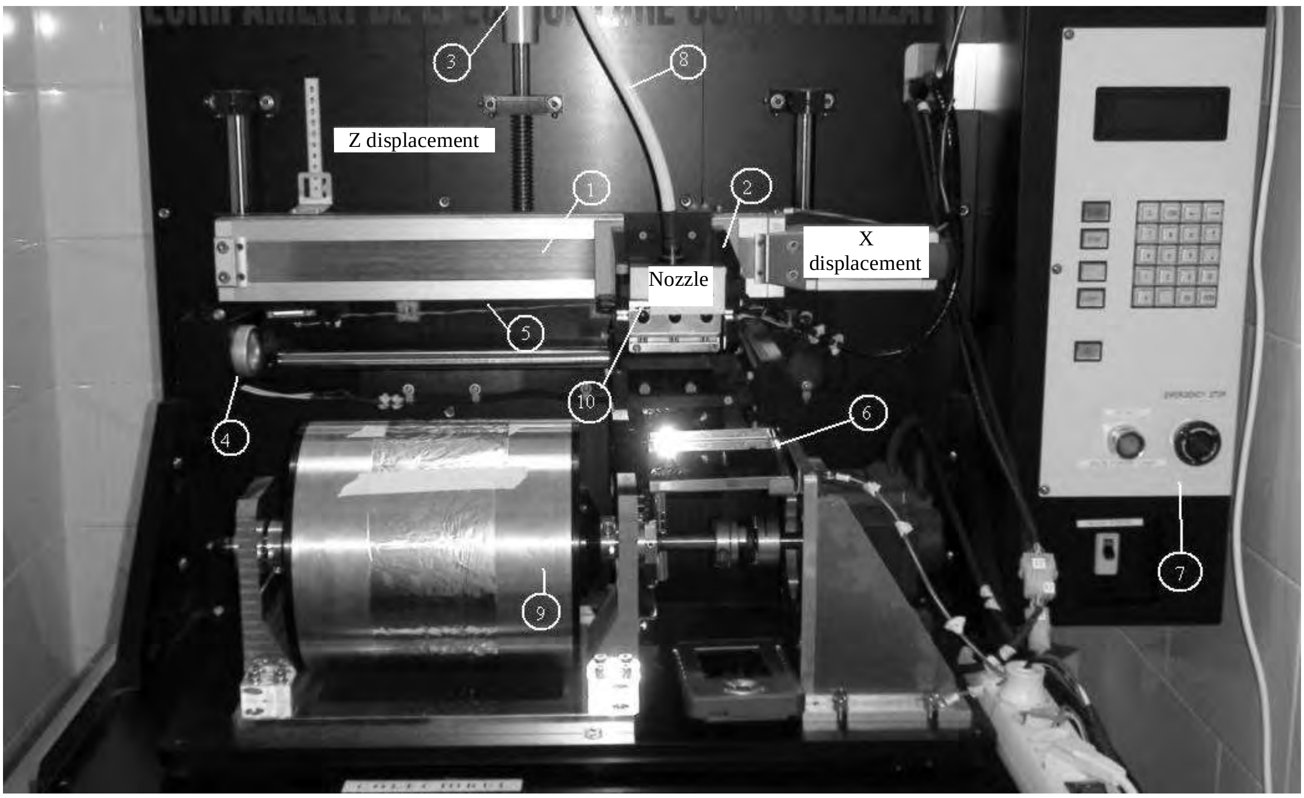


Fig.3. Computerized electrospinning equipment, [10]
1. slider; 2. slider arm; 3. slider handle for vertical movement ; 4. lamp; 5. nozzle holder
6. cleaning device; 7. control panel; 8. high-voltage cable; 9.cylindrical collector; 10. nozzle

The equipment carries out the following functions: (a) power supply for the system components; (b) high-voltage supply for the electrospinning electrodes; (c) control of the electrospinning process and of the nanofibers sizes; (d) ensuring of the computerised control of the process; (e) ensuring of the environmental conditions in the electrospinning chamber and on the environment.
The running limits have been experimentally determined for adequate running technological conditions, (Table 1).

Table 1. Process parameters and running limits

Parameters values	***Adjustment domain***		***Resolution***
	Min.	***Max.***	
Applied voltage	0.5 kV	50 kV	0.5 kV
Solution volume	0.1 ml	10 ml	0.1 ml
Feeding speed	0.1 ml/h	99.9 ml/h	0.1 ml/h
Syringe diameter	5 mm	30 mm	0.1 mm
Revolution speed for a rotary drum-type collecting device (diameter; 200 mm; width: 220 mm)	0 rev/min	2500 rev/min	50 rev/min
Displacement width of the nozzle holder	0 mm	200 mm	1 mm
Displacement speed of the nozzle holder on the X direction	0 mm/s	300 mm/s	1 mm/s
Cleaning frequency of the needle tip	5 s	60 s or 60 min	1 s
Ventilation	0%	100%	10%

3. CONSTRUCTIVE CHARACTERISTICS OF THE ELECTROSPINNING EQUIPMENT

The electrospinning equipment has the following constructive elements: the feeding device, the delivery device, the collecting device, the control device, the high-voltage supply source.
The solution feeding device is made up of a KD scientific proportioning pump of the kds100 series which is a syringe pump for a simple, precise and efficient injection, designed for glass or plastic syringes from 10 microliters to 60 microliters (Fig. 4, el.). The feeding device runs with some technical specifications, namely, 20-30ºC temperature, 30% - 70% humidity, AC 170 ~250V, 50Hz established voltage / frequency, max 0.1 mc/min. ventilation. The optimum variation domain for the electrospinning equipment is the following: 5-30kV high-voltage, 0,1-10 ml/h solution volume, 0.1-99.9 ml/h solution feeding degree, 5-30 mm. syringe diameter. The maximum displacement rate is 400 ½ steps/second with a speed variation of 12000:1 and a flow variation of 0.1µl/h for the 10 µl syringe) and of 506ml/h for the 60 ml. syringe.

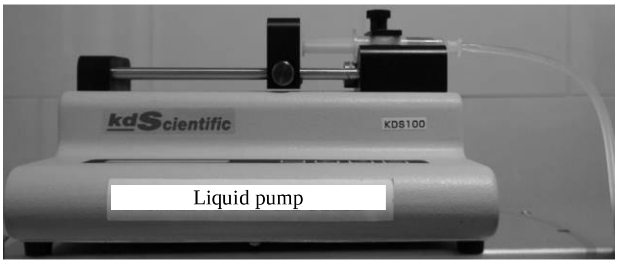


Fig. 4. Delivery pump, [10]

To calibrate the pump and to dose a selected volume and flow ratio, it is important the setting of the inner diameter of the syringe, a value used by the control program of the equipment. The value of the syringe diameter is also used to automatically set up the volume and flow ratios, respectively. The syringe diameter can be directly introduced or the syringe can be identified from a syringe list kept in the memory of the software used by the accomplished electrospinning equipment.
Concerning the pump distribution there are two accomplishing methods, namely, (a) the volume distribution one in which the pump controls the distribution of the volume and automatically stops when the initially established volume is reached; (b) the continuous one in which the pump runs at the established parameters until it is manually stopped.
In the case of certain glass syringes the corners of the syringe flanges are rounded off and due to this fact there appears the tendency of the syringe to come out of its holder. For a better fastening of the syringe in its seat one can attach a circular ring or a metallic sleeve upon the syringe so that this should push it towards its seat.

Fig. 5. Position of the syringe on the delivery pump, [10]

A diagram of the feeding pump functions is shown in Fig. 6.

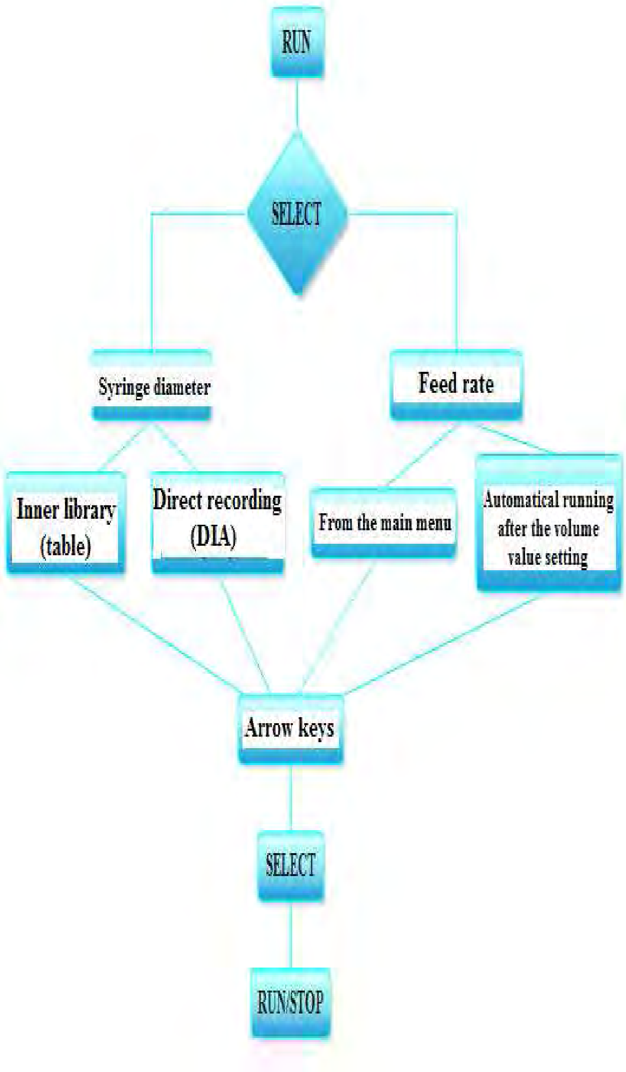
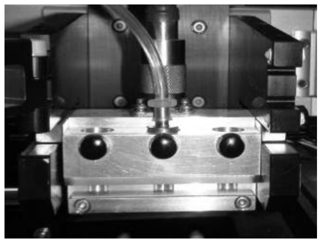


Fig. 6. Diagram of the feeding pump, [10]

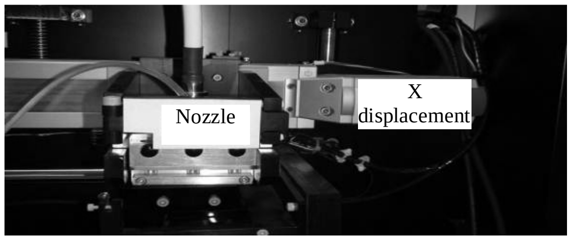
The polymer solution is transmitted from the delivery device to a nozzle coupled to a power supply (Fig.2, el. 10). The nozzle can accomplish displacement movements on the X direction (the setting of the displacement speed is done on the operating panel), on the Y and Z direction (the setting is done manually at the beginning or during the electrospinning process) (Fig.7).

The setting domain for the optimum running of the electrospinning system is for a displacement width of the nozzle holder of 0-200 mm, for a displacement speed of the nozzle holder of 0-300 mm/s, at a cleaning frequency ranging between 5-60s or 60 min.

The operating and control panel is made up of a panel with a VFD fluorescent display which shows the electrospinning technological parameters and the operating conditions, as well as of a keyboard which uses the computer notations (Fig.2, el.7). Among the electrospinning technological parameters which can be changed by means of the control panel one can mention the supply voltage, the feeding ratio, the rotation speed of the drum collector, the displacement speed of the nozzle.



a.



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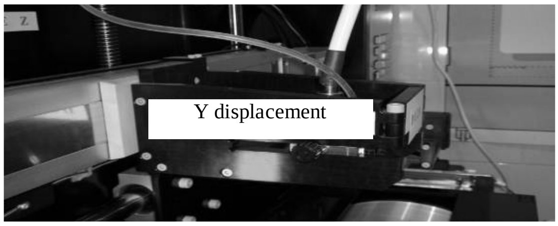
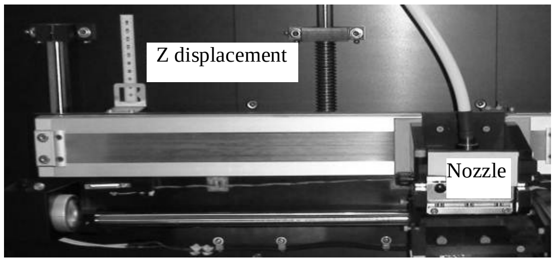


c.



d.

Fig. 7. Image of the delivery nozzle
a. Nozzle with 3 delivery areas; b. Displacement on the X axis; c. Displacement on the Y axis; d. Displacement on the Z axis, [11]

The component parts which are operating and which are directly controlled from the control panel are shown in Fig. 8.

Fig. 8. Operating and control panel, [10]

The diagram for introducing the technological parameters which can be set by means of the operating and control panel is shown in Fig. 9.

The diagram for setting the technological parameters on the ELECTROSPIN equipment is presented in Fig.11.

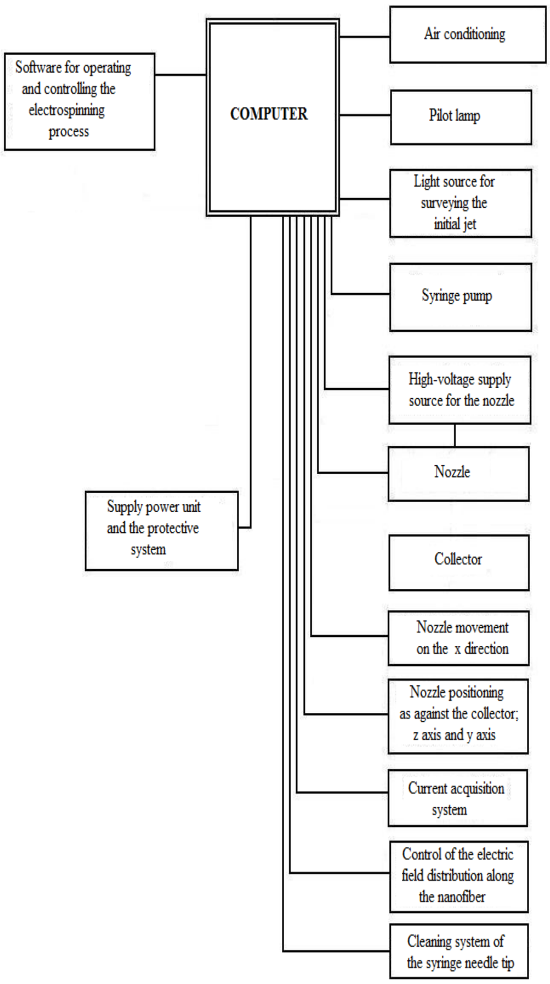


Fig.9. Diagram of the computerized components, [9]

The collecting device is of the rotary drum type (Fig.10) being made up of a brushless intermittent current motor with high and small torque, high precision encoder and driver. This is a combined device equipped with a special transmission gear case, especially fitted for running at a high torque by reducing the gear speed, as well as with a circular rod, an optimum device when high speeds are necessary. This type of collector has major advantages such as depositions with high degree of arrangements and easy handling.

The adjustment domain for the optimum running of the electrospinning equipment is for a rotation speed of 0-2,500 rev/min, at a diameter of 200 mm and a width of 220 mm.

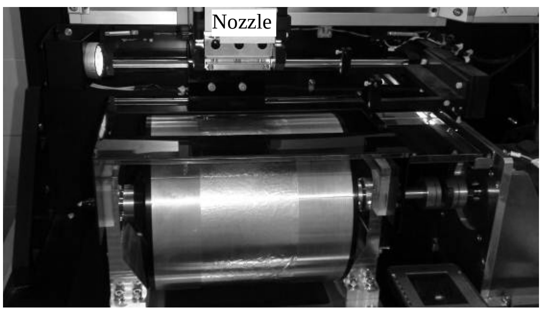


Fig. 10. Collecting device, [9]

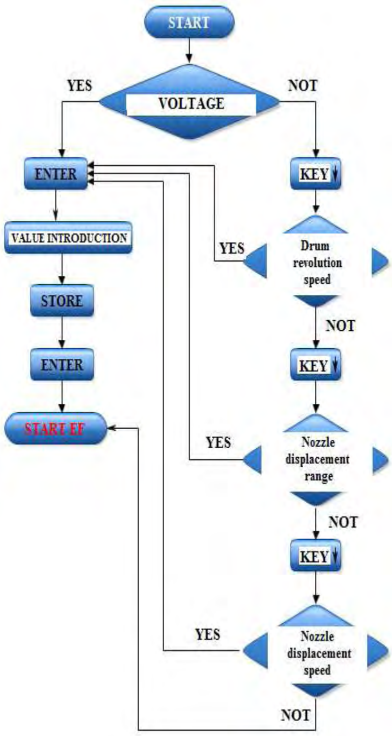


Fig. 11. Diagram for setting the technological parameters on the ELECTROSPIN equipment, [10]

The high-voltage supply source has an HV ultra-compact, little noise highly performant module. The HV sources are modular, mobile, used for a great variety of applications, including the mass spectrophotometry and electronic microscopy. These sources are excellent choices when a high secure/ stability outlet is needed. The outlet is controlled by means of the operating and control panel by employing an externally-controlled voltage.

Regarding the testing of the viability of the accomplished electrospinning equipment, our experimental researches have aimed at drafting an experimental plan which firstly assumed the following steps: the feeding of the syringe with a polymeric solution, the setting of the polymeric solution feeding speed, the setting of the technological parameters (voltage, nozzle displacement speed, drum revolution speed, needle – collector distance, nozzle displacement range on the X direction), the setting of the constructive parameters. The accomplished experimental researched have aimed at adopting successive values in certain intervals most favourable from a technological view point, namely, 15-35 kV voltage (V), 45-120 mm needle – collector distance (d), 0.1-0.4 ml/h feeding speed (Q). The influences of the considered parameters upon the way of deposition and upon the characteristics of the nanofibers obtained with the ELECTROSPIN equipment have been studied. The accomplished experimental researches have made us come to the following conclusions: a. the ELECTROSPIN equipment, devised and accomplished for obtaining nanofibers by electrospinning, allows the production of nanodimensioned fibres under a stable process; b. the computerized electrospinning equipment allows the obtaining of nanofibers from polymeric solutions by the systematic variation of a great number of parameters which influence the electrospinning process as well as the structure and the characteristics of the obtained nanofibers, respectively; c. the determination of the characteristics of the nanofibers obtained after the electrospinning process on the ELECTROSPIN equipment implies the previous knowledge of the following characteristics belonging to the processed polymeric solution, namely, gram-molecular weight, distribution of the gram-molecular weight, viscosity, conductivity, dielectric constant of the solution, surface tension, density of the polymeric solution; d. the computer integrated in the operating and control panel of the ELECTROSPIN equipment operates, by its hardware and software components, all the subassemblies and ensures the programming and controlling of the electrospinning process.

4. CONCLUSIONS

The electrostatic spinning, known as ”electrospinning”, has become during these last years the most viable variant for producing continuous fibers, fibers networks and fibrous depositions with diameters of the fibers smaller than 1 μm to be used for a large range of materials from biopolymers to ceramics. Thus, the development of the nanotechnologies and equipments for obtaining nanofibers is one of the most promising field to be extended in the future in various applicability domains (technical applications, filters, cosmetics, protection, medical bandages, tissue engineering, artificial organs, electronics and nanosensors, medicines, textile supports for medicines, etc.)

The electrospinning process implies knowledge of electrostatics, fluid rheology and properties of the polymer solution such as the solvent evaporation, surface stress, solution conductivity. These fundamental properties interact constantly and are influenced reciprocally during the electrospinning process. Fibers with different morphologies, made up from various materials, can also be obtained, directly or indirectly, by electrospinning. Consequently, various polymers and mixtures can be used in obtaining fibers for specific applications. Nanofibers with different morphologies can be obtained only by varying the electrospinning parameters. As a result of the experiments carried out on the electrospinning equipment presented in this paper one can estimate that this equipment is capable to produce fibers with various morphologies and applications. The equipment provides adequate means to produce polymer fibers, ceramic fibers, composite polymer/ceramic fibers with diameters ranging from 10 nm to some microns. The equipment consists mainly of a body, a control unit and a spinning unit. Depending on the user requirements, the electrospinning unit can be made up of more component elements, a fact which allows the choosing of certain nanofibers morphologies. The equipment provides the security level for the user when handling the high-voltage sources, the chemical organic solvents and the nanofibers.

ACKNOWLEDGEMENTS

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