Equipment for Obtaining Polimeric Nanofibers by Electrospinning Technology

I. Constructive and functional elements of the Computerized electrospinning equipment

LILIANA ROZEMARIE MANEA[1*] BOGDAN CRAMARIUC[2], VASILE CAUNII[3], ION SANDU[4, 5*]

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

[2]IT Center for Science and Technology, Av. Radu Beller 25, 011702, Bucharest, Romania

[3] "Gheorghe Asachi" Technical University Iasi, Faculty of Mechanics, 32 Dimitrie Mangeron Blv., 700050, Iasi, Romania

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

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

The present context of the international studies identifies the electrospinning process as one of the key technologies for obtaing nanofibers. The dynamics of the studies connected to the implementation of this technology is given by the great diversity of the applications of the obtained nanofibers which start from filtering mediums and separating membranes to sensors and actuators, implants, structures for the controlled release of drugs up to the molecular photonics. This paper aims to present a new equipment for obtaining the nanofibers from polymer solutions by means of the electrospinning technology. The suggested equipment accomplishes the computerized control of the electrospinning technological parameters on starting from the idea of modularity and automatic control of the electrospinning process. The equipment allows the real time control for the electrospinning process based on determination of both jet characteristics and the obtained nanofibers properties.

Keywords: nanofibers, equipment, technology, modular conception, electrospinning

Over the last years, research and development in the field of nanomaterials, which are materials with structural units on a nanometer scale in at least one direction, have enjoyed unprecedented support, nanomaterials becoming the fastest growing area in materials science and engineering [1-7]. Nanofibers (fibers that have diameter equal to or less than 100 nm), especially organic nanofibers, are among the most interesting and promising class of one-dimensional (1-D) nanomaterials [8-14].

Several techniques were reported in recent years for the laboratory preparation of nanofibers, such as electrospinning, phase separation, drawing of polymer melts, template synthesis, self-assembly of polymers under certain conditions, etc. [11]. Electrospinning is currently the most effective technique to fabricate nanofibers [15], as none of the other methods can match electrospinning in terms of versatility, flexibility and ease of fiber production [16-21].

Electrospinning is a processing technique which generates polymer, ceramic, metallic, and composite fibers with diameters ranging from tens of nanometers to a few micrometers from solutions, dispersions, or melts as a liquid jet accelerates through an electric field [2 - 4].

The electrospinning process is a complex and efficient one, capable to produce long, continued, control-directed nanofibers, being adjustable to various types of polymers. Electrospinning allows the obtaining of spatially-directed ultra-fine fibers, with an especially great specific surface, with small-sized interconnected pores and a good control of their geometry at the nanofibers level, a high elasticity modulus, a high surface/volume ratio, all these being defining characteristics for their implementation in various application domains [3].

Despite the large number of electrospinning equipment types, there are still many problems related to the process control and the electric field instability. These problems influence fiability and reproductibility of structural characteristics for the obtained nanofibers, being an obstacle for exploiting electrospinning at its full potential.

The Electrospinning Equipment

The designed and accomplished electrospinning equipment which allows the obtaining of the nanofibers from polymer solutions has the following constructive elements: feeding device, delivery device, collecting device, operating and control device for the electrospinning process (SCC) and high-voltage power supply [22, 23].

The general view of the designed and accomplished computerized electrospinning equipment is shown in figure 1. This equipment fulfils the following functions [1, 4, 15, 17-19]:

a. the electrical power supply of the equipment components;

b. the high-voltage supply of the electrospinning electrodes;

c. the operating and control of the electrospinning process technological parameters;

d. the control of the dimensional characteristics of the obtained nanofibers.

All mechanisms of the equipment are connected to a computerized command system (SCC), having a

* email: rozemariemanea@yahoo.com; sandu_i03@yahoo.com; Tel.: +40-744-431709

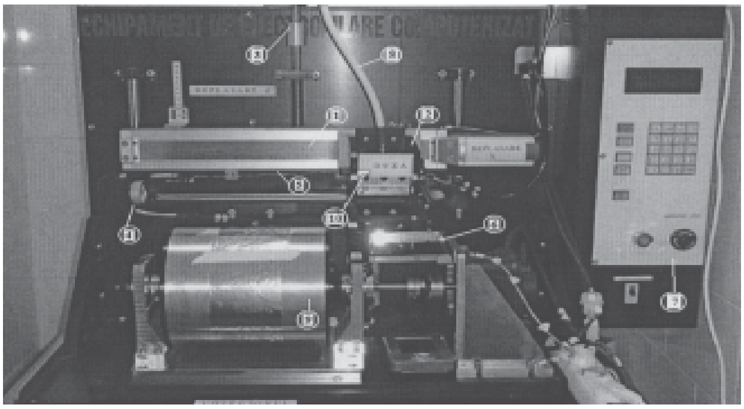


Fig.1. Computerized electrospinning equipment
1. slider; 2. slider arm; 3. slider handle for vertical movement ; 4. lamp; 5. nozzle holder; 6. cleaning device; 7. operating and control device for the electrospinning process (SCC); 8. high-voltage cable; 9. cylindrical collector; 10. nozzle [1]

command and control software for entire process, which assures the real-time control of the process.

Constructive Characteristics of the Computerized Electrospinning Equipment

The feeding device is made up of a KD Stientific proportioning pump of the KDS100 series for a simple, precise and efficient injection, having the role of maintaining and delivering with a constant pressure the polymer solution existing in the syringe.

The feeding device allows the usage of the plastic or glass syringes with a 10-60μL capacity. The technical running specifications are: temperature: 20-30°C, humidity: 30 -70%, voltage/frequency: AC 170 ~250V, 50Hz, ventilation maximum value 0.1m^3/min. The optimum variation domain of the main parameters for the proper running of the feeding device for the electrospinning equipment is the following: applied voltage: 5-30kV; solution volume: 0.1-10mL/h; solution feeding degree: 0.1-99.9mL/h; syringe diameter: 5-30mm [1, 4, 15, 17].

The electrospinning force is given by the high-voltage power supply by means of a cable dipped into the polymer solution. The high-voltage source generates up to 50kV with positive or negative polarity. The adjustment of the liquid flow and the electrical field strength control the electrospinning speed.

Figure 2 shows the syringe and the component elements of the feeding pump and of the syringe which allow the loading and the feeding of the polymer solution to be electrospun. To make easier the coupling of the syringe to the feeding pump, the pushing unit (1) can be disengaged from the main cylindrical rod and manually moved along the guide bar to adjust the syringe position. By operating the release button (2) from the side of the pushing unit (1) for releasing the unit from the rod, the spring of the fastening button (3) is raised and unscrewed from the syringe case.

The equipment allows the gauging of the feeding pump, the proportioning of a volume and of a constant flowing ratio at pre-established values by the direct setting of the value of the inner diameter of the syringe, a value used by the program of the operating and control block of the computerized electrospinning equipment. The value of the syringe diameter is also used for the automatic setting of the volume units as well as of the flow rate. The syringe diameter can be directly introduced or the syringe can be identified from a value list of the syringe characteristics, a list introduced into the memory of the software employed for the operating and control block of the accomplished electrospinning equipment [24-30].

In what the running conditions of the feeding device are concerned, this equipment allows: (a) the discontinuous running (the feeding pump controls the feeding and the proportioning of the volume of the polymer solution from the syringe, automatically shutting-down the equipment when the initially-set value of the liquid is reached); (b) the continuous running (the feeding pump creates the pressure on the syringe at the pre-established value at which the pump runs at the established rates up to the moment when the equipment is manually turned-off) [31-36].

In the case of some constructive types of glass syringes for which the corners of the syringe flanges are rounded off, there appears the tendency of the syringe to slide out of its holder. To prevent this sliding, the feeding pump is endowed with a circular metallic ring (sleeve) which ensures an optimal fastening. Depending on the type of the employed syringe/needles, the equipment allows the monoaxial or coaxial electrospinning [37-42].

The delivery device of the computerized electrospinning equipment is made up of the syringe and the metallic electrode. The polymeric solution gets from the feeding pump to the nozzles holder connected to the high-voltage supply source (fig. 3). The equipment is endowed with a three nozzles holder, thus allowing the simultaneous feeding of three syringes filled either with the same polymer solution or with different polymers. By the existence of more delivery tips, this equipment allows either the unijet electrospinning or the multijet one (the parallel assembly of three syringes on the same nozzle holder).

The nozzle holder is manufactured in such a way so that it could ensure the displacement movement on the X, Y, Z directions (fig. 4). The displacement on the X direction

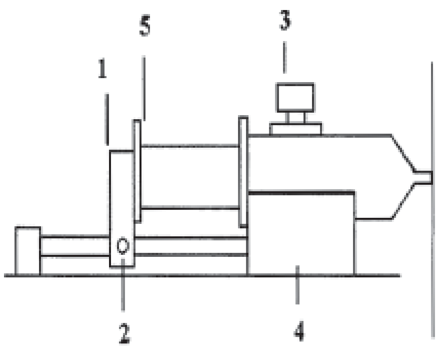


Fig. 2. The diagram of the feeding device

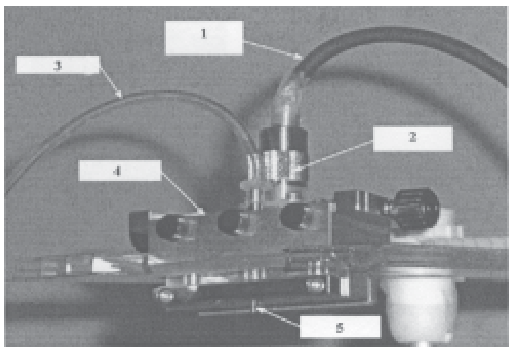
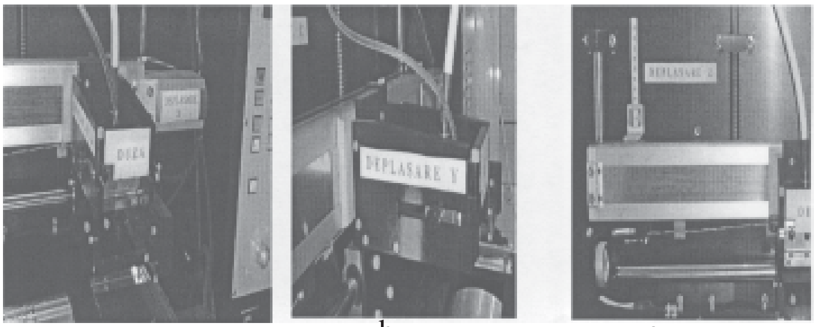
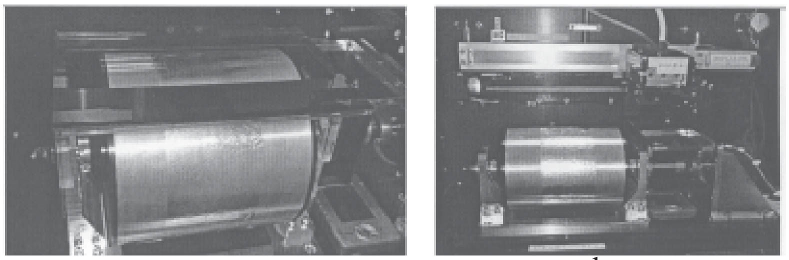


Fig. 3. Delivery device of the computerized electrospinning equipment



a b c

Fig. 4. Holder of the delivery nozzles:
a. displacement of the nozzles holder on the direction of the X axis;
b. displacement on the direction of the Y axis;
c. displacement on the direction of the Z axis



a b

Fig. 5. Collecting device:
a.plane collecting; b. rotary drum collecting

and the setting of the value of the nozzle displacement speed, respectively, is done directly from the operating panel from the operating and control block of the equipment (fig. 4a). The displacements on the Y and Z directions as well as the setting of the displacement values of the nozzles displacement on these directions, respectively, are manually done before the start of the electrospinning process or during it (figs. 4b and 4c). The displacement on the Z direction of the nozzles holder is manually done by means of a screw-nut mechanism.

For the optimal running of the electrospinning equipment, the adjustment domain of the constructive parameters from the delivery area is the following: the displacement width of the nozzle holder on the direction of the X axis: 0 - 200mm; the distance between the capillary tube and the collecting device: 40 – 170mm; the displacement speed of the nozzle holder: 0 - 300mm/s.

Collecting Device. The accomplished electrospinning equipment allows the usage of three constructive types of collectors, namely, the plate-type plane collecting device and the rotary drum-type collecting device, respectively.

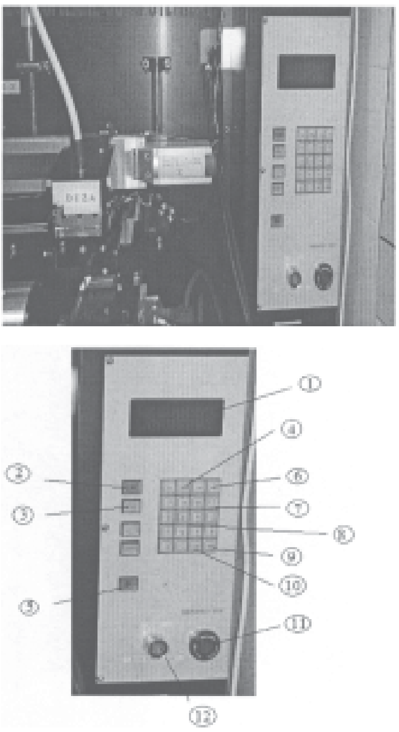
Two out of these three collecting possibilities are presented in figure 5. In the case of the rotary drum-type, the following technical specifications should be done: rotation speed of the rotary collector: 0-2,500rot/min; diameter of the collecting drum: 200mm; width of the collecting drum: 220mm.

The operating and control device (SCC – fig. 6*)* is made up of:

-VFD fluorescent display panel to visualize the values of the electrospinning technological parameters as well as the running conditions;

- keyboard (fig. 6a);
- hardware and software.

By comparison with the conventional electrospinning equipment, the designed and obtained equipment, through its hardware and software components, allows an improved control for the electrical field, both in the stage of nanofibers forming and in the next stage of their elongation in the electrostatic field.



a

b

Fig. 6. Operating and control block with incorporated software for the operation and control of the parameters of the electrospinning process: a. operating block, close-up view; b. operating block, operating panel, general view;

1 - Display - numerical values displaying; 2 - start of the electrospinning process; 3 - STOP shut-down of the electrospinning process; by pressing the key twice, the nozzle holder returns to its initial, starting position; 4 - ENTER validation of the introduced data (values); 5 - ensurance of the electrospinning area lighting; 6 - displacement of the syringe rod to introduce the syringe; and displacement of the nozzles holder to the right or to the left in the moment of establishing the parameters values, namely, the distance the nozzle holder is covering and the displacement speed of the nozzle holder; 7 - Keys with up/down arrows, the menu displayed by the parameters operates as a continuous loop, these keys being used for the displacement along the loop; 8 - Keys from 1 to 10, digital keys allowing the introduction of the figures (values of the process parameters); 9 – STORE, permission to memorize the values of the process parameters; 10 - CL" with double function (to allow the cleaning of the needle tip during the resting period and during the electrospinning process and allow the displacement of the arm of the nozzle holder in the desired position); 11 - EMERGENCY STOP, general shut-down of the equipment

The obtained nanofibers are qualitative superior compared with those created by conventional electrospinning equipment, a great advantage being that exists the posibility to get nanofibers with controlled characteristics, high orientated and good uniformity for dimensional properties, smaller diameters etc. [1-3, 15, 17-21]. The hardware and software components installed on this equipment allows the command and control for the following parameters:

- the distance between the nozzle and collector, the nozzle and collector moving speeds, the jet width obtained through scanning, the polymer solution flow from the seringe;
- the feeding tensions, total charge for the jet;
-atmosperic temperature and humidity.

The determination of nanofibers characteristics depends on the properties specific to processed polymer solution: molecular mass, the distribution of the molecular mass, viscosity, conductivity, dielectric constant for the solution, superficial tension, density of the polymer solution.

In the case of the conventional electrospinning equipment the values of technological parameters, design parameters, and environmental parameters are set manualy when the processing begins and they are kept unchanged during the entire process.

The fundamental diagram of the equipment for obtaining the nanofibers by means of the computerized electrospinning system (fig. 7).

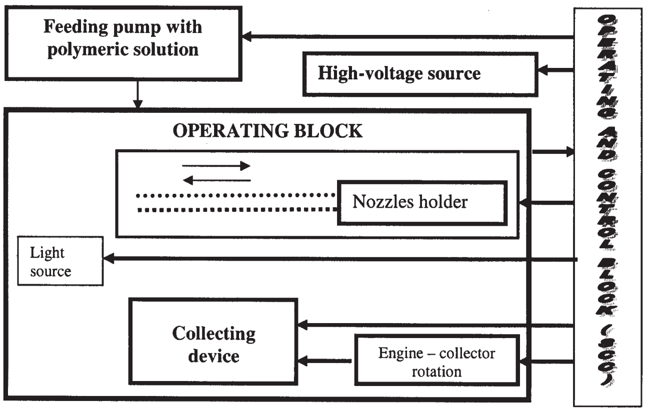


Fig. 7. Fundamental diagram of the computerized electrospinning equipment

Innovative elements involved in the operation of equipment

The accomplished computerized electrospinning equipment allows the obtaining of nanofibers from polymer solutions. The accomplished equipment allows, by its characteristics, the systematic variation of a great number of parameters which influence the electrospinning process as well as the structure and characteristics of the obtained nanofibers.

This equipment is an innovative one because, by means of its hardware and software components, allows the operation and control of the following parameters:

-the distance between the capillary tube and the collector;
- the displacement speed of the nozzle and of the collecting screen, respectively;
-the width of the jet released by the nozzle as well as the rate and the volume of the polymer solution from the syringe, respectively;
-the voltage of the electric field, the total current of the jet, the cleaning speed of the needle tip;
- the environmental parameters (the temperature of the environment, the atmospheric humidity, the air speed within the environment).

The determining of the characteristics of the nanofibers obtained by means of the electrospinning process implies the beforehand knowledge of the following characteristics belonging to the processed polymer solution: gram-molecular weight, distribution of the gram-molecular weight, viscosity, conductivity, dielectric coefficient of the solution, surface pressure, density of the polymer solution.

The computerized electrospinning equipment has the following advantages: by its construction (software and hardware) it allows the obtaining of nanofibers, nanotubes and meshes with controlled characteristics, with good orientation and high levelness for a multitude of medical applications and filtering mediums. This equipment allows adjustments for various constructive types of collecting systems with the usage of a large range of polymeric solutions.

The computer controls by its hardware and software components all the sub-assemblies and ensures the programming and controlling of the electrospinning process with the following main parameters: high-voltage power source (applied voltage), pumping syringe (flow rate), engine of the rotary collector (collector speed), engine of the movable nozzle (displacement speed of the nozzle).

This new equipment ensures: a. complex functions / easy usage; b. flexible monitoring conditions; c. the fiability increases the possibility of expanding the basic functions with some new ones depending on the necessities imposed by the destination of the obtained nanofibers.

Conclusions

This equipment allows, by its construction (software and hardware), the obtaining of nanofibers, nanotubes and meshes with controlled characteristics, with good orientation and high levelness for a multitude of medical applications and filtering mediums. The system allows the adjustment of the equipment for various constructive types of collecting systems, thus allowing the usage of a large range of polymeric solutions. Among the advantages of the suggested electrospinning equipment one can mention the following:

- drastic reduction of the processing time;
- computerized control of the process;
- 55% diminishing of the power supply.

The technology for obtaining the nanofibers by means of the electrospinning process will be extended for different types of polymers.

References

1.SCARLET, R., MANEA, L.R., CRAMARIUC, B., Modern Technologies, Quality and Innovation, Modtech, **2,** 2011, p. 977.

2.MANEA, L.R. SCARLET, R., CRAMARIUC, B., Tehnologii de obţinere a nanofibrelor prin electrofilare, Ed. Tehnopress, Iasi, 2012.

3.SCARLET, R., MANEA, L.R., CRAMARIUC, O., Modern Technologies, Quality and Innovation, Modtech, **2,** 2011, p. 981.

4. CRAMARIUC, B., MANEA, L.R., SCARLET, R., CRAMARIUC, R., Journal of Electrostatics (http:/dx.doi.org./10.1016/i.elstat.2012.12.018), **1**, no. 10, 2013, p. 289.

5. BRAGHIROLLI, D.I., STEFFENS, D., PRANKE, P., Drug Discovery Today, **4**, 2014, p. 345.

6. KHAN, W.S., ASMATULU, R., CEYLAN, M., JABBARNIA, A., Fibers and Polymers **14**, no.8, 2013, p. 1235.

7.SU, C.-I., LAI, T.-C., LU, C.-H., LIU, Y.-S., S.-P. WU, Fibers and Polymers, **14**, no.4, 2013, p. 542.

8.ACATAY, K., ŞIMŞEK, E., AKEL, M., MENCELO LU, Y.Z., Electrospinning of Low Surface Energy-Quaternary Ammonium Salt Containing Polymers and their Antibacterial Activity, in GUCERI, S., GOGOTSI, Y., (editors), Nanoengineered Nanofibrous Materials, Kluwer Academic Publishers, Dordrecht, The Netherlands, 2003, p. 97.

9.KO, F., Nanofiber technology: Bridging the Gap between Nano and Macro World, in GUCERI, S., GOGOTSI, Y., (editors), Nanoengineered Nanofibrous Materials, Kluwer Academic Publishers, Dordrecht, The Netherlands, 2003, p. 128.
10.HUANG, Z.-M., ZHANG, Y.-Z., KOTAKI, M., RAMAKRISHNA, S., Composites Science and Technology, **63**, 2003, p. 223.
11. ANDRADY, A. Science and technology of polymer nanofibers, John Wiley & Sons, Inc., Hoboken, New Jersey, 2008, p. 458.
11.GOGOTSI, Y., Nanotubes and Nanofibers, CRC Press, Taylor & Francis Group, Boca Raton, 2006, p. 245.
12.HAGHI, A. K., ZAIKOV, G. E., Advances in Nanofibre Research, Smithers, Shropshire, UK, 2011, p. 328.
13.HAGHI, A. K., ZAIKOV, G. E., Advances in Nanofibre Research Vol. 2, Smithers Rapra Technology Ltd, Shropshire, UK, 2012, p. 169.
14.SCARLET, R., MANEA, L.R., SANDU, I., CRAMARIUC, B., SANDU, A.V., Rev. Chim. (Bucharest), **63**, no.8, 2012, p. 777.
15.DELIU, R., SANDU, I., BUTNARU, R., SANDU, I.G., LUKAS, D., Rev. Chim. (Bucharest), **63**, no. 6, 2012, p. 603.
16.HUANG, Z., M., ZHANG, Y.Z., KOTAKI, M., RAMAKRISHNA, S., Composites Science and Technology, **63**, 2003, p. 223.
17.SCARLET, R., MANEA, L.R., SANDU I., MARTINOVA, L CRAMARIUC O., SANDU, I.G., Rev. Chim. (Bucharest), **63**, no. 7, 2012, p. 688.
18.DELIU, R., SANDU, I.G., BUTNARU, R., LUKAS, D., SANDU, I., Mat. Plast., **51,** no. 1, 2014, p. 62.
19.CALIN, M.A., KHENOUSSI, N., SCHACHER, L., ADOLPHE, D., MANEA, L.R., GRADINARU, I., ZETU, I., STRATULAT, S., Mat. Plast, **50,** no. 4, 2013, p. 257.
20.TURA, V., TOFOLEANU, F., MANGALAGIU, I., MINDRU, T.B., BRINZA, F., SULITANU, N., SANDU, I., RAILEANU, D., IONESCU, C., Journal of Optoelectronics and Advanced Materials, **10**, no. 12, 2008, p. 3505.
21.TOFOLEANU, F., MINDRU, T.B., BRINZA, F., SULITANU, N., SANDU, I.G., RAILEANU, D., FLORISTEAN, V., HAGIU, B., IONESCU, C., SANDU, I., TURA, V., Journal of Optoelectronics and Advanced Materials, **10**, no. 12, 2008, p. 3512.
22.CRAMARIUC, B., CRAMARIUC, O.T., CRAMARIUC, R., LUPU, I.G, MANEA, L.R., Technology for producing nanofibres using a computerized electrostatic spinning system, Patent Number: RO126974-A2, 2012.
23.CRAMARIUC, B., CRAMARIUC, O.T., CRAMARIUC, R., LUPU, I.G, MANEA, L. R., Equipment for producing nanofibres by using a computerized electrostatic spinning system, Patent Number: RO126949-A2, 2012
24.POPESCU, V., MANEA, L., AMARIEI, N., Mat. Plast, **46**, no.1, 2009, p.95.
25.POPESCU, V., MANEA, L.R., CURTEZA, A., VASLUIANU, E., Tekstil, **60**, no. 7, 2011, p. 306.
26. GHERASIMESCU, C., LEVA, M., BUTNARU, R., MURESAN, A., MANEA, L.R., Industria Textila, **62**, no. 1, 2011, p. 19
27. DIACONU, M., CRETESCU, I., LUCA, F., MANEA, L., POHONTU, C., Environmental Engineering and Management Journal, **9**, no.1, 2010, p. 67.
28. NEJNERU, C., NICUTA, A., CONSTANTIN, B., MANEA, L., TEODORESCU, M., AGOP, M., Journal of Applied Mathematics, **2013**, no. 137056, 2013.
29. MANEA L. R.,. NEJNERU, C., MĂTĂSARU, D., AXINTE, C., AGOP, M., Journal of Modern Physics, **4**, no.7, 2013, p. 1013.
30. MANEA, L., GRIBINCEA, V., SUFITCHI, P., Rivista della Tecnologie Tessili, no.7, 2000, p. 112.
31. MANEA, L., MOISESCU, E., SUFITCHI, P., Industria Textila, **40**, no.4, 1998, p. 234.
32. MOISESCU, E., MANEA, L., Revista Romana de Textile-Pielarie, no. 3-4, 1999, p. 75.
33. MOISESCU, E., MANEA, L., SUFITCHI, P., Industria Textila, **50**, no. 1, 1999, p. 21.
34. MOISESCU, E., MANEA, L., SUFITCHI, P., Industria Textila, **49,** no. 3, 1998, p. 179.
35. SECULA, M.S, CRETESCU, I., CAGNON B., MANEA, L.R., STAN, C.S., BREABAN, I.G., Materials, **6,** no.7, p. 2723
36. POPESCU, V., RADU, C. D., MANEA L.R., Industria Textila, **61**, no.1, 2010, p. 23.
37. COMANDAR, C., ANDRIUTA, M.,MANEA, L., Texsci 2000, 2001, p. 91.
38. MANEA, L., MOISESCU, E.,COMANDAR, C. Texsci 2000, 2001 p. 278.
39. POPESCU, V., MANEA L.R., AMARIEI, N., Mat. Plast. (Bucharest), **46**, no. 1, 2009, p. 95.
40. CHIRITA, M., GRIBINCEA, V., MANEA, L.,Industria Textila, **48**, no. 2, 1997, p. 82.
41. GRIBINCEA, V., CHIRITA, M., MANEA, L.,Industria Textila, **48**, no. 1, 1997, p. 20.
42. GRIBINCEA, V., CHIRITA, M., MANEA, L.,Industria Textila, **48**, no. 2, 1997, p. 79.

Manuscript received: 15.10.2014