

The influence of solution parameters on the electrospinning intensity from foamed surface

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ABSTRACT: Electrospinning is an efficient process for producing polymeric and hybrid nanofibers. There is, however, a lack of understanding concerning scalability of the process and in particular the production rate optimization. The electrospinning mass transfer intensity depends predominately on solution parameters, process parameters and the design of the equipment. These parameters influence the deposition intensity of the spinning process differently, but it is not known which factors dominate. The e-spinning deposition intensity of polyethylene oxide, polyvinyl alcohol and their mixtures was investigated using a bubble foamed polymer solution surface to promote high mass deposition. Based on the measured properties of the solutions, a mathematical criterion was developed which made it possible to predict the electrospinning intensity of a given polymer solution. The proposed formula agrees with the experimental data and confirms that spinning intensity can be predicted from pre-determined solution parameters. Using computer modeling, the weighting coefficients of the solution parameters have been determined, showing which parameter is the most important for the process intensity. The criterion and the same weighting coefficients were applied to the analysis of published data and it was found that they can be applied not only for electrospinning from the foamed surface but also from the free surface. A physical explanation of the criterion is proposed. © 2015 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 42034.

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INTRODUCTION

Electrospinning (e-spinning) is an efficient, simple, versatile, and cost-effective technique for producing nanofibers where the fiber diameters are in the range from a few tens of nanometers to a few microns. The technique is very promising and dynamic since it enables the production of multifunctional nanofibers from polymers, polymer blends, sol-gels, composites for different fields. Nanofibers with a high surface area in combination with different functionalities make them interesting candidates for many applications such as filter production,¹ tissue engineering,² drug delivery,^{3–5} protective clothing,⁶ the production of advanced biomaterials,^{1,7} and biosensors.⁸

E-spinning of many polymers has been thoroughly investigated,^{9–14} and it has been found that the nanofiber formation and nanofiber properties depend on the polymer solution viscosity,^{15,16} the solution concentration,^{16,17} the surface tension,¹⁶ the electrical conductivity,^{17,18} and the dielectric permittivity or

polarizability of the solvent.¹⁹ The process is further influenced by the solution feed intensity,^{10,20} equipment features, and the e-spinning method,^{16,21–23} and particularly by the strength of the electric field.^{15,24} Nevertheless, there is a lack of understanding of the most important process variables for controlling fiber morphology and deposition intensity. In most publications is investigated the process of fiber formation and data about the process intensity (material transfer rate) are not presented.

The theoretical investigations of the e-spinning process have been devoted mainly to a modeling of the jet formation. These works include modeling of the criteria for jet initiation, modeling of the jet's straight part and modeling of the entire jet. The results of the theoretical modeling make it easier to understand the physics of the e-spinning process. In some cases, theoretical modeling supports conclusions regarding the adjustment of the solution and process parameters. For example, in Refs. 20, 25, and 26, operation diagrams that were developed are useful for adjusting the solution properties for the needle e-spinning, but

questions concerning the process intensity remain unanswered. The experimental investigations of the jets and fiber formation reviewed by Reneker and Yarin²⁷ do not address the question of the dominant variables affecting e-spinning intensity.

The problem of developing a reliable method to predict the possibility of e-spinning the polymer in a particular solution and/or the intensity of the material transfer on the basis of a certain set of solution parameters thus remains unsolved. Such a method is of interest because the fields of nanofiber applications are permanently enlarged and more new e-spinnable polymers of different classes are continuously required. With some exceptions, the ability of a polymer to form a Taylor cone and to be e-spun is determined by testing a polymer solution in the process and adjusting the solution formulation and e-spinning parameters thereafter. This “trial and error” procedure is not simple and requires e-spinning equipment.

One of the limitations of the e-spinning technology is its low productivity or low deposition intensity. E-spinning from a single jet is commonly used to produce very fine polymer fibers, but the production rate with such a system is relatively low. Several methods of producing e-spun nanofibers with a higher production rate have been investigated using different alternatives for feeding and depositing nanofibers, such as multiple needle systems and so on. A commercial example is the NanospiderTM equipment²⁸ which liquid jets formed from surfaces of rotating cylinders or wires lead to a productivity more than ten times greater (9.42 g/h) than that of the needle e-spinning system (0.2 g/h) reported for spiral coil spinnerets.²⁹ Such an increase in the production rate is due to the formation of innumerable polymer jets from a sufficiently large surface. The jets are formed from the free surface due to the electrodynamic instability of surface waves.³⁰ The NanospiderTM principle was used in the investigation of the e-spinning of some polymers, for example polyvinylbutyral³¹ and polyurethane.³² In these publications, the process productivity was determined and the results were interpreted on the basis of solution parameters but their role is not clearly established. He and coworkers^{33,34} investigated bubble e-spinning as a method of high productivity but the productivity data were not reported.

In all e-spinning methods, the process is controlled by adjusting the solution properties and process parameters. The polymer concentration, solvent, additives, etc. are changed empirically and it is important to have a method to predict the result. This means that it is necessary to know which solution parameter affects the e-spinning process more than the other parameters.

The aim of this work is to experimentally investigate the e-spinning deposition intensity from the foamed surface and, on the basis of the experimental data, to develop a model which will make it possible to determine which polymer solution characteristic dominates in the e-spinning process and to predict spinning intensity on the basis of solution parameter values without performing an e-spinnability test. Such a method of computer modeling will also provide new insight into the mechanisms of e-spinning from a foamed surface and information about the dominating factors in the spinning process.

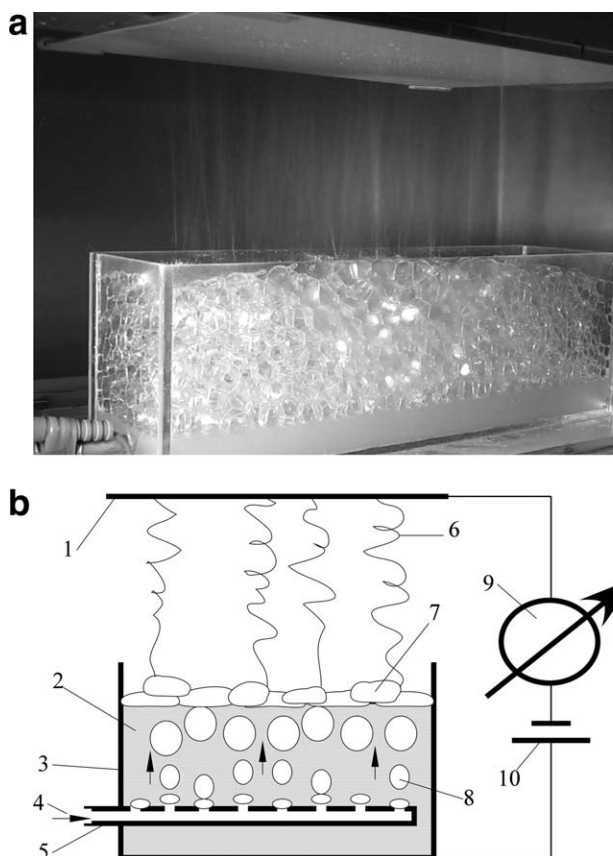


Figure 1. E-spinning apparatus (a) and scheme (b): (1) collector; (2) polymer solution; (3) vessel on the electrode; (4) compressed air; (5) tube with holes; (6) nanofibers; (7) foam bubbles; (8) bubbles in the solution; (9) ammeter; (10) high voltage source (60 kV).

MATERIALS AND METHODS

Equipment

The deposition intensity with the e-spinning equipment was investigated using a foamed surface (Figure 1) according to a similar process reported by He *et al.*^{33,34} Compressed air (pressure 0.1–0.5 Mpa) was fed into a tube equipped with holes, which then created foamed bubbles from which Taylor cone and subsequently e-spinning occurred. Nanofibers were deposited on a metal collector. Depending on the process parameters, fibers of different morphology can be formed (Figure 2). The process intensity was estimated as the amount of dry material transferred material from 1 m² of polymer solution surface per second (g/s m²). The mass of the transferred material was determined as the difference between the target weight before e-spinning and the weight after the drying of the deposition. The process intensity was calculated by dividing the deposited weight by the vessel surface area and process time. The quality of the fibers was not investigated.

Materials

The polymers used were polyethylene oxide, PEO, [(Sigma-Aldrich) four grades with molecular weights 600,000, 900,000, 2,000,000, and 4,000,000 g/mole], polyvinyl alcohol, PVA, (Mowiol 15–99, viscosity of 4% water solution 12.5–17.5, Kuraray) and a mixture of PEO and PVA. PEO with different molar

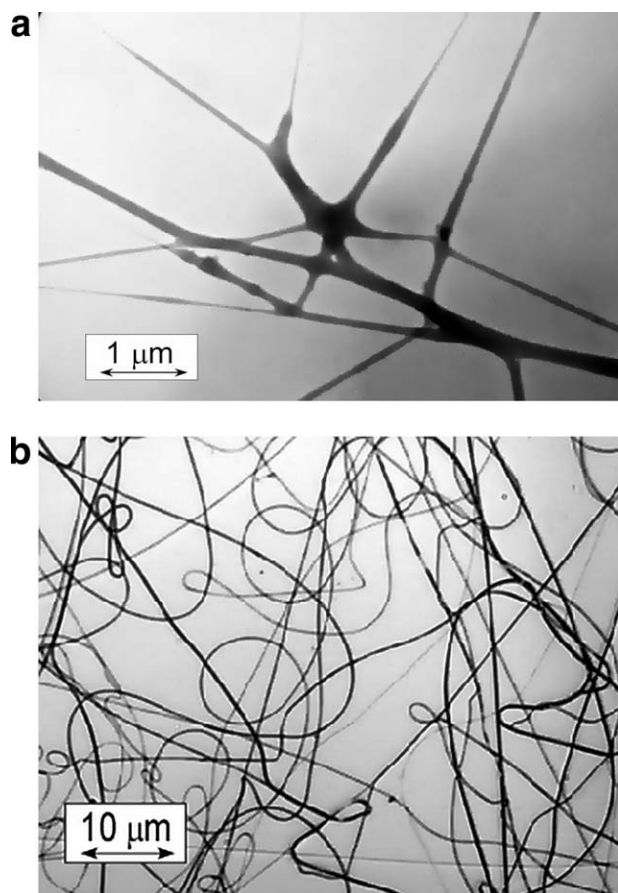


Figure 2. PEO fibers formed from the foamed surface: (a) PEO, MW 600,000, EM image (transmission electronic microscope EVM-100B), (b) PEO, MW 900,000, optical microscope image.

weights (four grades with molecular weights 600,000, 900,000, 2,000,000, and 4,000,000 g/mole) were selected to test the polymers with different properties (viscosity, surface tension) but chemically very similar. PVA and PEO solutions were prepared by dissolving the polymer in distilled water under stirring at an elevated temperature (up to 90°C).

Solution Properties

The viscosity, surface tension, and electrical resistivity of each polymer solution were measured. The surface tension was measured by the drop volume or weight method,³⁵ the viscosity was measured using a Zahn viscosity cup,³⁶ and the DC electrical resistivity of the polymer solution was measured in a cell consisting of two metal 5 × 5 mm plates to which a voltage was applied. The resistivity of the polymer solution was calculated from the electrical current, which was measured using a Wayne Kerr 640B LCDR meter.

RESULTS AND DISCUSSION

Criterion Development

Polymer solution concentration influences the e-spinning process and morphology of fibers (example on Figure 2). The polymer concentration has also a significant impact on the e-spinning intensity and there is usually an optimal polymer concentration region, which was also observed in our experiments.

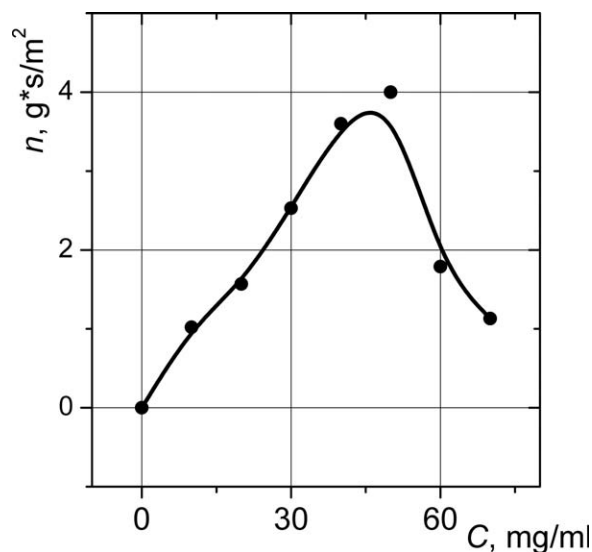


Figure 3. E-spinning intensity versus polymer concentration (PEO, M_w 900,000 g/mole).

Figure 3 shows an example of the PEO e-spinning intensity dependence on the concentration with an initial linear increase up to a concentration of 45 g/L (4.5 wt %), after which the deposited amount decreased rapidly. At zero polymer concentration, solvent alone is electrostatically sprayed. With increasing polymer concentration, the material transfer increases, but it begins to decrease when the solution viscosity becomes too high and the electrostatic forces are unable to create a Taylor cone and form elongated nanofibers.

The concentration of the solution affects not only the viscosity but also the other measured polymer solution parameters. Figure 4 shows the effect of polymer concentration on viscosity, surface tension, and electrical resistivity and it is evident that a prediction of the e-spinning deposition intensity from these parameters is not obvious. Therefore, the mechanism for

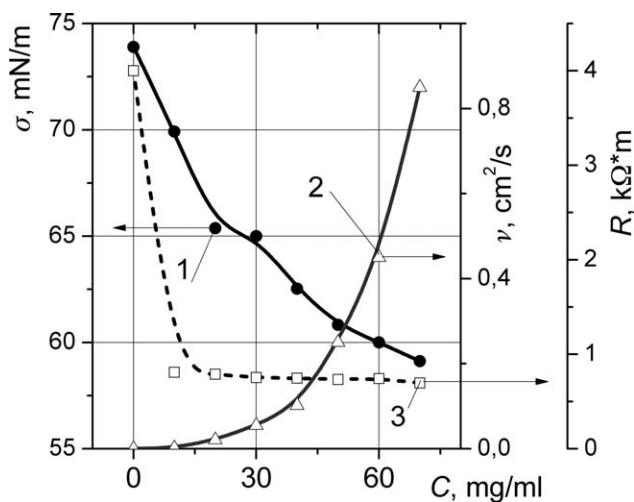


Figure 4. The dependence of (1) the PEO (M_w 900,000 g/mole) solution surface tension, (2) kinematic viscosity, and (3) resistivity on the polymer concentration.

achieving a high deposition rate needs to be clarified and developed, with a particular emphasis on the dominating polymer solution parameters. Here, a novel solution is proposed with a criterion based on a mathematical formula and computer modeling, which takes into account the solution properties.

The general dependence of the criterion on the solution concentration should be similar to the dependence of the e-spinning intensity on the solution concentration. If the criterion is constructed properly, both dependences should coincide not only for a particular polymer solution but also under different conditions and for different polymer solutions. The general criterion is a combination of the individual criteria which are solution properties such as surface tension, viscosity, etc. For the construction of the general criterion, individual criteria must be expressed in a special dimensionless form. The solution property in the individual criteria will be presented with respect to some constant and it is most convenient to choose as a constant the corresponding property of the pure solvent (in our work water). There are two ways of creating the general criterion: as the sum of the individual criteria or as their algebraic product. Attempts to create a general criterion as the sum of the individual criteria were unsuccessful because the maximum in the dependence on the solution concentration could not be obtained. The general criterion calculated as the algebraic product gave encouraging results but required modifications. Each individual criterion must vary between one and zero, but it can never reach zero because the general criterion will then be zero or infinity. For convenience, it is reasonable to rearrange the criterion so that the maximum value of the solution property (for example, the surface tension) is equal to the same property of the solvent. So, if the property value increases with increasing polymer concentration, the inverse values of the individual criterion will be taken. For example, the solution kinematic viscosity, ν , which increases with increasing polymer concentration, will be presented in the general criterion as the inverse value $1/\nu$. Also, instead of solution electrical conductivity, the solution electrical resistivity R is used. The relative importance of each solution parameter in the e-spinning process can be taken into account by introducing different weighting factors k_s for the individual criteria. The individual criterion will then be expressed as:

$$C_s = 1 - \frac{S_{\text{sol}} - S}{S_{\text{sol}}} \times k_s, \quad (1)$$

where S is the measured certain solution property, S_{sol} is the value of this property for the solvent (water), and k_s is the weighting factor of this solution property.

Using these individual criteria, there are some limitations for the weighting factor. It must be $0 \leq k_s < S_{\text{sol}}/(S_{\text{sol}} - S)$ because C_s cannot be negative. The number of the individual criteria included in the general criterion is not limited but the problem is how to construct it. Individual criteria can be placed in the general criterion in the numerator or in the denominator. There is no explicit rule because *a priori* the role of the parameter is not known. We construct the general criterion using a computer-modeling program. This criterion takes into account

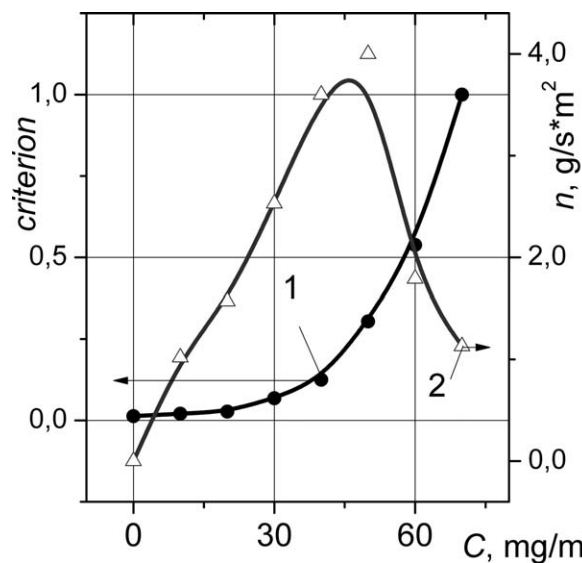


Figure 5. The dependence of the e-spinning intensity (1) and of the general criterion (2) on the PEO (M_w 900,000 g/mole) concentration. All weighting factors are equal to 1. The correlation coefficient between the e-spinning intensity and the criterion is -0.031 .

the solution parameters mostly used in the investigations of e-spinning, like electrical conductivity, surface tension, and viscosity. For our experimental data, the general criterion is:

$$\text{Criterion} = \frac{1 - \frac{(\sigma_{\text{sol}} - \sigma)}{\sigma_{\text{sol}}} \times k_{\sigma}}{\left(1 - \frac{(1/\nu_{\text{sol}} - 1/\nu)}{1/\nu_{\text{sol}}} \times k_{\nu}\right) \times \left(1 - \frac{(R_{\text{sol}} - R)}{R_{\text{sol}}} \times k_R\right)}, \quad (2)$$

where σ is the surface tension of the solution, ν is the viscosity, R is the resistivity, and k_s is the weighting factor of the solution property. Computer modeling is based on the fitting of the dependence of criterion on the solution concentration to the e-spinning intensity dependence by adjusting the weighting coefficients. Computer modeling was performed using standard MS Office Excel Visual Basic software.

It must be noted that more parameters for example dielectric permittivity, polarizability etc., can be introduced into the criterion without restriction.

Criterion Verification

This general criterion was used to analyze the dependences of the e-spinning intensity on the polymer concentration. First, the criterion with all weighting factors equal to 1 was used to determine the dependence of the e-spinning intensity on the concentration of PEO (M_w 900,000 g/mole) in the solution (Figure 5). At low PEO concentrations (up to 40–50 mg/mL), both the e-spinning intensity and the general criterion increase with increasing concentration, although the trends differ. Similar dependences were obtained for the PEO with a higher molar mass (Figure 6), but the concentration at which the maximum e-spinning intensity was observed was lower in the latter case.

This discrepancy between the general criterion and the e-spinning intensity is related to the fact that all the solution parameters were considered to be equally important and the weighting factors being set at 1. Using the computer modeling, new weighting

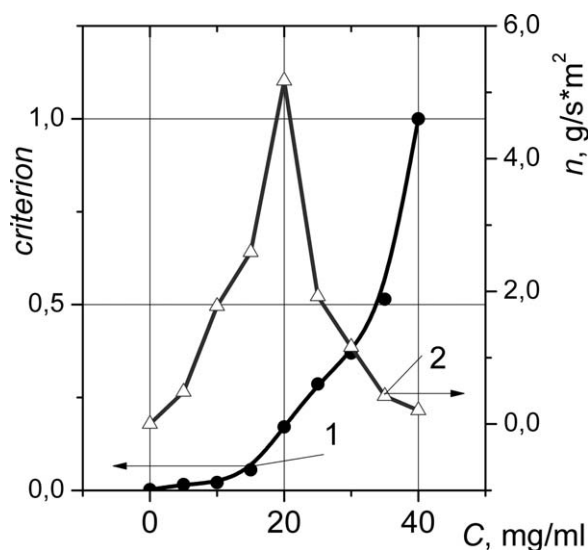


Figure 6. The dependence of the e-spinning intensity (1) and of the general criterion (2) on the PEO (M_w 2,000,000 g/mole) concentration. All weighting factors are equal to 1. The correlation coefficient between the e-spinning intensity and the criterion is -0.292 .

factors for the PEO ($M_w = 900,000$ g/mole) were obtained. The result obtained with the weighting factors $k_\sigma = 5.1$, $k_v = 0.93$ and $k_R = 0.96$ is presented in Figure 7, showing that the curves for the e-spinning intensity and the general criterion here agree quite well. The discrepancy at the low concentration is probably due to scatter in the solution parameter values.

The general criterion is then:

$$\text{Criterion} = \frac{1 - \frac{(\sigma_{\text{sol}} - \sigma)}{\sigma_{\text{sol}}} \times 5.1}{\left(1 - \frac{(\frac{1}{v_{\text{sol}}} - \frac{1}{v})}{\frac{1}{v_{\text{sol}}}} \times 0.93\right) \times \left(1 - \frac{(R_{\text{sol}} - R)}{R_{\text{sol}}} \times 0.96\right)} \quad (3)$$

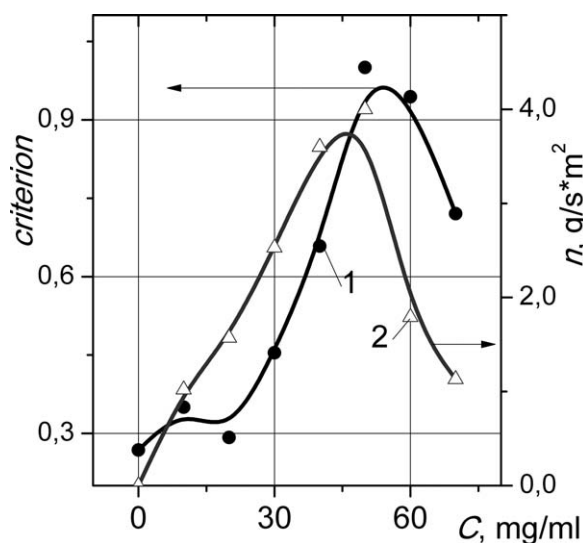


Figure 7. The dependence of the e-spinning intensity (1) and of the general criterion (2) on the PEO (M_w 900,000 g/mole) concentration with weighting factors: $k_\sigma = 5.1$, $k_v = 0.93$, and $k_R = 0.96$. The correlation coefficient between the e-spinning intensity and the criterion is 0.811 .

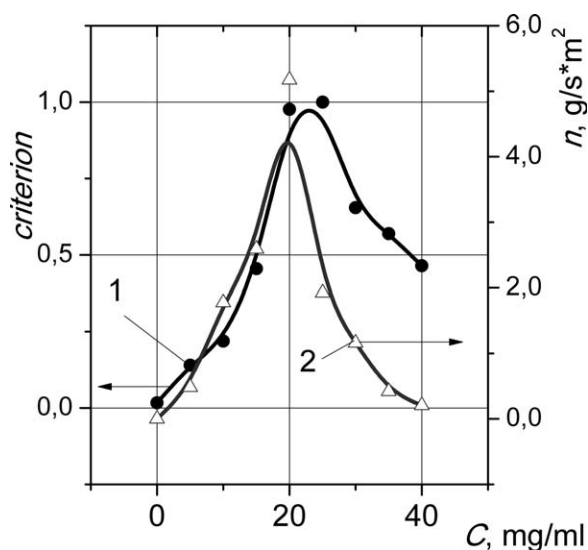


Figure 8. The dependence of the e-spinning intensity (1) and of the general criterion on the PEO concentration (M_w 2,000,000 g/mole). Weighting factors: $k_\sigma = 5.1$, $k_v = 0.93$, and $k_R = 0.96$. The correlation coefficient between the e-spinning intensity and the criterion is 0.812 .

This criterion was used to model the e-spinning of PEO solutions with different molecular weights (Figures 8, 9, and 10). In all cases, the dependence of the general criterion on the PEO concentration agrees quite satisfactorily with the measured dependence of the e-spinning intensity on the polymer concentration. This means that the criterion with these weighting factors takes into account not only the parameters of the particular polymer solution but also some general features of e-spinning from foamed surface features.

The successful application of the same weighting factors for PEO having different molecular weights suggests that the

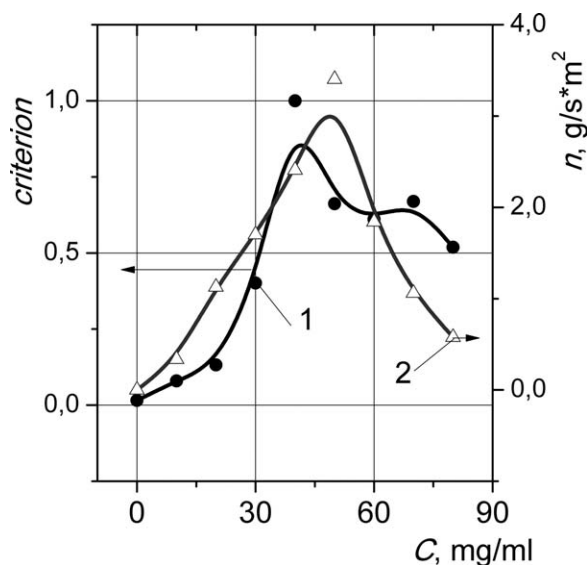


Figure 9. The dependence of the e-spinning intensity (1) and of the general criterion (2) on the PEO concentration (M_w 600,000 g/mole). Weighting factors: $k_\sigma = 5.1$, $k_v = 0.93$, and $k_R = 0.96$. The correlation coefficient between the e-spinning intensity and the criterion is 0.760 .

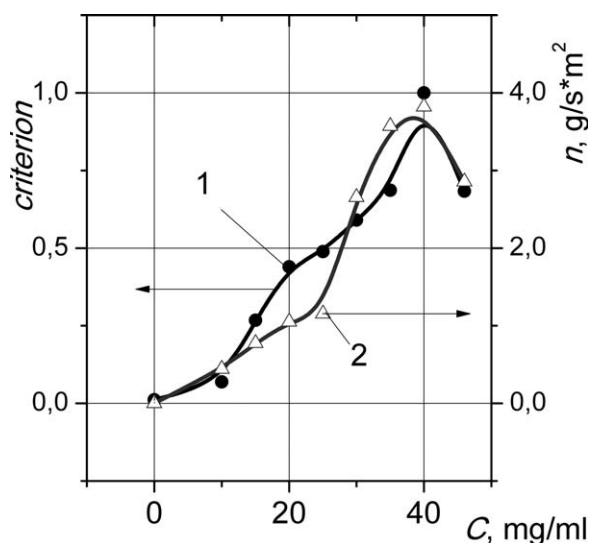


Figure 10. The dependence of the e-spinning intensity (1) and of the general criterion (2) on the PEO concentration (M_w 4,000,000 g/mole). Weighting factors: $k_\sigma = 5.1$, $k_v = 0.93$, and $k_R = 0.96$. The correlation coefficient between the e-spinning intensity and the criterion is 0.861.

criterion with the same weighting factors may be used for the other polymers. E-spinning from the foamed surfaces of PVA and of a blend of PVA and PEO solutions was investigated. Figures 11 and 12 show that the curves for the criterion and the e-spinning intensity again coincide quite satisfactorily.

An analysis of the dependences of the e-spinning intensity on the polymer solution parameters using the proposed criterion revealed information about the e-spinning from the foamed surface physical processes. The interpretation of the results shows that the formation of the foam bubbles on the polymer solution surface is dependent on the surface tension with the participa-

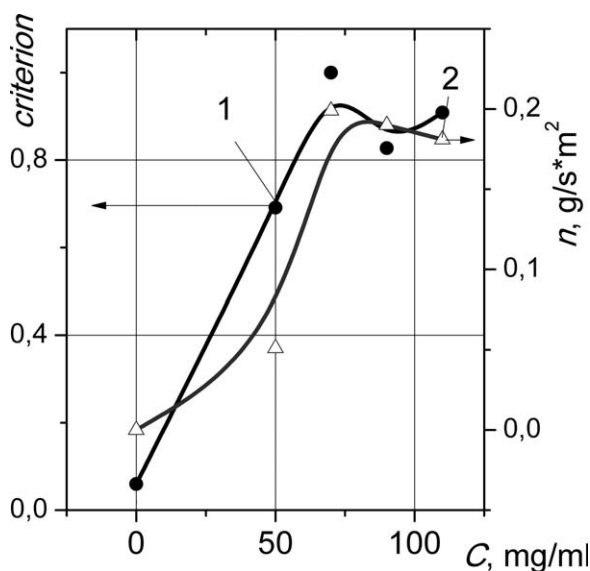


Figure 11. The dependence of the e-spinning intensity (1) and of the general criterion (2) on the PVA concentration (Mowiol 15-99). Weighting factors: $k_\sigma = 5.1$, $k_v = 0.93$, and $k_R = 0.96$. The correlation coefficient between the e-spinning intensity and the criterion is 0.860.

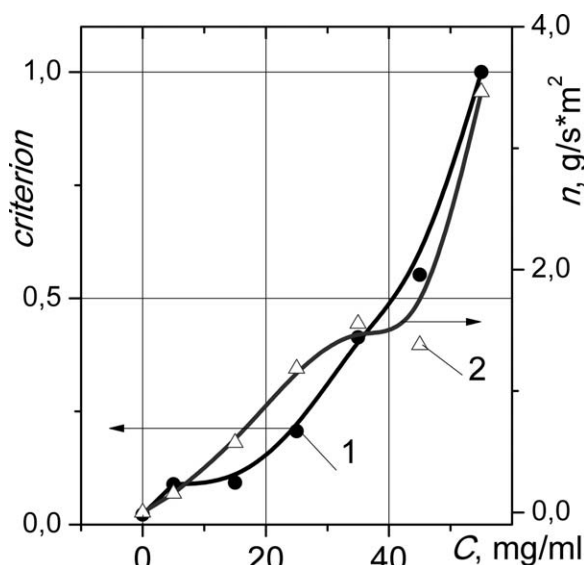


Figure 12. The e-spinning intensity (1) and the general criterion (2) plotted versus the polymer concentration for PVA (Mowiol 15-99) and PEO (M_w 4,000,000 g/mole) blend. Weighting factors: $k_\sigma = 5.1$, $k_v = 0.93$, and $k_R = 0.96$. The correlation coefficient between the e-spinning intensity and the criterion is 0.965.

tion of the viscoelastic flow of the fluid and the air pressure in the bubble. At a constant air pressure in the compressed air supply system, the surface tension mainly determines the bubble formation. Small bubbles are formed at high surface tension and the bubble concentration on the solution surface is high. When the solution surface tension becomes lower due to the increase in polymer concentration, the bubbles formed are larger, the curvature of the bubble surface becomes smaller and number of bubbles per unit area decreases (Figure 13). The formation of the Taylor's cone followed by the jet occurs from the bubble surface and, at a lower bubble concentration, fewer e-spun jets can arise. The decrease in the solution surface tension, on the other hand, facilitates Taylor's cone formation. Thus, the surface tension plays a double role in the e-spinning from the foamed surface. A decrease in surface tension reduces the e-spinning intensity due to the lower bubble concentration and lower bubble surface curvature and it simultaneously enhances the process because jet formation becomes easier.

The structure of the criterion and modeling shows that an increase in the solution viscosity should increase the e-spinning intensity from the foamed surface. The solution viscosity, which determines the viscoelastic fluid flow, is usually considered to hinder the formation of the Taylor's cone and jet. This cannot be disclaimed, but it must be taken into account that a more viscous solution has a higher polymer concentration. Modeling using the proposed criterion showed that this is more important for the e-spinning intensity than the solution flow deceleration. The solution viscosity also influences the foam bubble formation but the criterion does not provide information to show its role in foam formation.

Due to the electrical conduction of the polymer solution, the applied electric field results in the appearance of charges on the

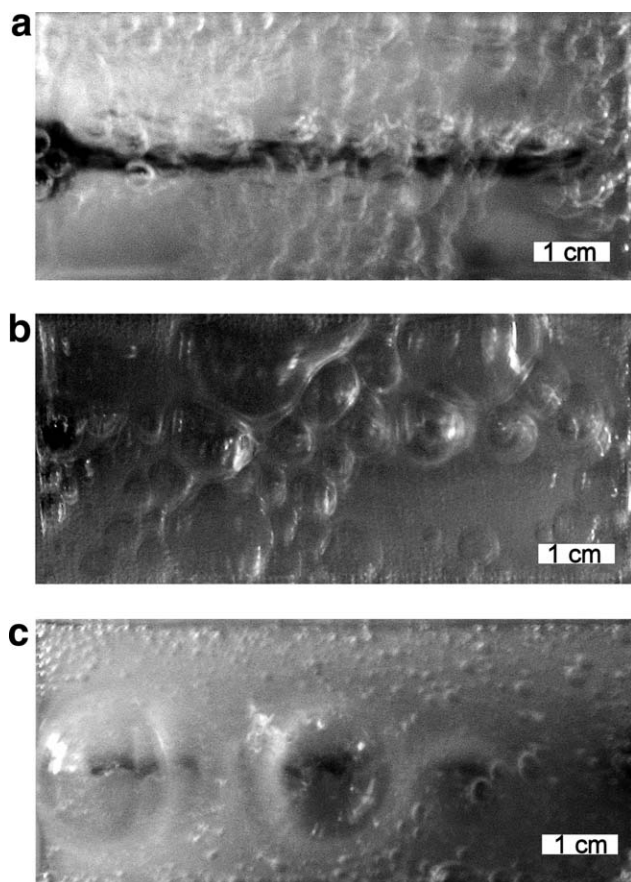


Figure 13. Foam bubbles on the surface at different PEO (M_w 600,000) concentrations: (a) 0 mg/mL; (b) 40 mg/mL; (c) 60 mg/mL. Scale 1 : 1.

bubble surface which are bound to the polymer and/or solvent molecules. Interaction of the electric field with these charges leads to a force, which pulls them to the target having opposite polarity. Results achieved using the proposed criterion and computer modeling do not provide sufficient information to make decisions concerning the role of the electrical conductivity, but it is clear that the electrical conductivity is less important than the surface tension and viscosity at least in the cases analyzed. The weighting factor of the conductivity is very close to the weighting factor of the viscosity, but the viscosity has a much greater influence because it changes significantly with concentration.

The importance of the solution property in particular is indicated by the weighting coefficients determined by modeling studies. Whereas the viscosity and electrical conductivity weighting coefficients are only slightly less than 1, the surface tension has a weighting coefficient over 5, which means that, despite the relatively little change in the surface tension, the changes in bubble formation influence the e-spinning intensity from the foamed surface much more.

These mechanisms of e-spinning from the foamed surface suggest that the developed criterion can be used with the Nanospider™ equipment. In similar feeding and deposition technologies, the process intensity depends on the number of the jets formed due to the electrodynamic instability of the

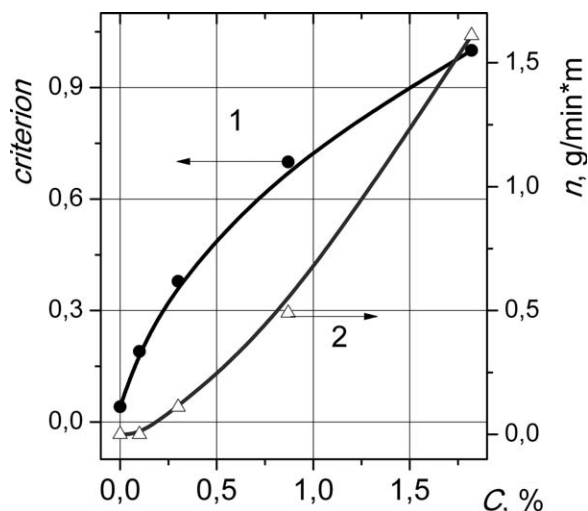


Figure 14. Polyurethane (data from Ref. 32). The dependence of the e-spinning intensity (1) and of the general criterion (2) on the polymer concentration. Weighting factors: $k_\sigma = 5.1$, $k_v = 0.93$, and $k_R = 0.96$.

surface waves.³⁰ Here, the deposition system is similar to the e-spinning occurring from the foamed surface, where the process intensity and production rate depend on the number of bubbles. A further analysis of published results based on the roller feeding in an electrospinning process of, for example, solutions containing polyurethane with salt addition³² or polyvinylbutyral³¹ are presented in Figures 14 and 15. The data obtained from the publications are limited, but it was possible to evaluate the application of the criterion without any change in the weighting factors. The results confirm that the criterion can be used for the commercial electrospinning system based on the roller solution feeding system. The agreement between the criterion and process productivity is not as good as in the case of foamed surface e-spinning, but it can be assumed that additional parameters and the exact process conditions for the

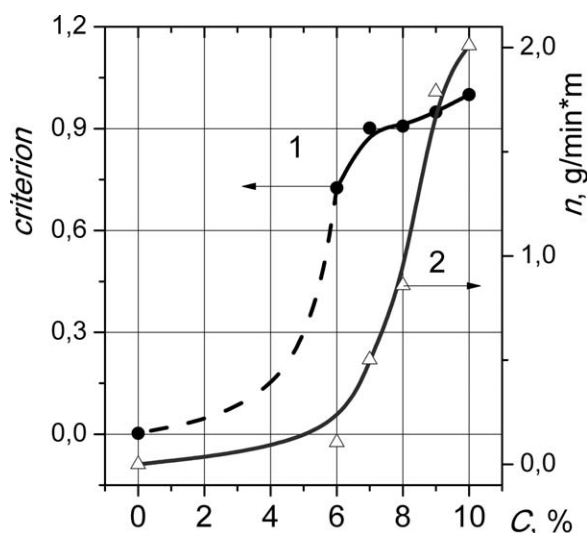


Figure 15. Polyvinylbutyral (data from Ref. 31). The dependence of the e-spinning intensity (1) and of the general criterion (2) on the polymer concentration. Weighting factors: $k_\sigma = 5.1$, $k_v = 0.93$, and $k_R = 0.96$.

evaluated technology limit the accuracy. It must be noted, however, that the criterion developed cannot be applied to the needle e-spinning because of the differences in jet formation.

CONCLUSIONS

The results presented in this work show that the criterion developed for the prediction and analysis of the e-spinning process can be used successfully at least with regard to the e-spinning intensity. Developed criterion makes it possible to find the optimal polymer concentration to obtain the highest process productivity without performing an e-spinnability test.

The proposed method and criterion make it possible to determine the role of solution parameters in the e-spinning process from a foamed surface. The role of surface tension and viscosity of the solution are different in e-spinning from a foamed surface and from e-spinning using the spinneret system, which is ascribed to bubble formation peculiarities. For the investigated polymers (PEO with different molecular weights, PVA and a blend of PVA and PEO), the solution surface tension played a major role because together with the viscosity it governs the bubble formation. Experiments showed further that the weighting factors of the particular polymer solution parameters were the same in the case of all investigated non-ionic polymers and mixtures. The criterion with the same weighting factors was tested on similar commercial electrospinning processes with encouraging results. The validity of the weighting factors in a wider concept requires more experiments with different polymers and solvents. Nevertheless, the proposed approach should be valuable for optimizing the e-spinning process and in the understanding of process rate optimization.

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