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Possibilities for the Estimation of Electrospun Nanofibre Diameter Distribution by Normal (Gaussian) Distribution

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Abstract
Fiber diameter and its distribution are one of the most important parameters related with the quality of electrospun web. The diameter of electrospun nanofibres is not uniform and the histograms of values are usually distributed differently.Analysis of literature sources showed that sometimes the distribution curves are similar to normal (Gaussian) distribution. Sometimes the distribution shapes are sophisticated and do not resemble this one. The problem arises because it is impossible to compare the average values of different measurement distributions, and a method with possibilities to compare the different results for estimation of nanofibre web quality is necessary. Yet, till now no standardized method for measurement of the diameter and evaluation of the distribution has been created. In this article, various distributions from literature sources and those of nanofibre diameter obtained during own experiments were analysed. Values of the nanofibre diameter are described by normal (Gaussian) and compound mathematical distributions. It was decided that the skew of distribution along with other characteristics can be used as criterion for web estimation and the diameter of nanofibres can be described by skewed normal (Gaussian) distribution when the coefficient of skewness is less than 0.5. The possibilities of estimation by compound distribution from a few normal distributions must be checked when the coefficient is higher.

Key words: *electrospinning, nanofibres, diameter, distribution, quality.*

Introduction

Nanofibres can be prepared by different methods including the following: drawing out [1], template synthesis [2], phase separation [3], self-assembly [4], melt blowing, [5, 6] and electrospinning [7, 8]. Electrospinning is a versatile, convenient and unique process used to produce continuous and ultra fine fibres with a diameter range from a few nanometers to over 1 μm [8]. Electrospinning technology can be divided into needle electrospinning and roller electrospinning.

The history of electrospinning dates back to 1900, when John Francis Cooley filed the first electrospinning patent, where several types of indirectly charged spinning heads were proposed. From literature sources it is known that a little later William James Morton and John Zeleny developed it too. Anton Formhals made a significant contribution to the development of electrospinnin during 1931 - 1944, and published a series of patents on electrospinning theory. After Formhal's studies, various research studies and patents on the electrospinning method were recorded. Nikolai Albertowich Fuchs, Igor' Vasil'evich Petryanov-Sokolov, Natalya D Rosenblum, Sir Geoffrey Ingram Taylor, Reneker and other researchers distinguished themselves in this area too. Geoffrey Ingram Taylor modelled the shape of the Taylor cone created by a fluid droplet under the influence of an electric field (from 1964 to 1969) [9 - 12]. Nevertheless electrospinning became more widely used only at the end of the 20th century, when the ability to manufacture fibres on a nano-scale emerged.

Nanofibre production has become very important for scientists in recent years. Various polymers have been successfully electrospun into fibres [13]. Polyvinyl alcohol (PVA), polyamide (PA), thermoplastic polyurethane (TPU), polyurethane (PU), polycaprolactone (PCL), Poly(Ethylene Oxide) (PEO), water-soluble polymers and others are the most often used in this process [14-19]. A very small diameter, large surface area, and small pore size are the important properties of nano size fibres. Due to these characteristics, nanofibres have been used in a number of applications, such as bio-medicine, tissue engineering, drug delivery, bio-sensors, filtration, protective clothing, reinforcement in composite materials, microelectronics (battery, transistors, super capacitors, sensors, and display devices), photonics, and others [6, 20, 21].

Varying diameter is one of the most common problems which may be appear in a electrospun web, which may be obtained when a jet of polymer is split into multiple jets when the solution jet is traveling from the Taylor cones to the upper collector. Nanofibres sticking on the web after reaching the web, not being finally dried can be the other reason for such a problem.

Geometrical properties of electro-spun nanofibres, such as fibre diameter and uniformity, diameter distribution, fibre orientation and fibre morphology depend on the polymer type, its molecular weight, solution properties (conductivity, surface tension, viscosity, temperature) environmental properties (temperature, humidity, pressure) and equipment parameters (voltage, distance between electrodes and also coating time on basic material) [7, 15, 16, 22]. An analysis of various works showed that the conclusions of different researchers' studies where the influence of the same factor on the structure of nanofibres is analysed are different in some cases. The mismatches of the results arise because very often not all parameters are described in the articles. Probably the mismatches of the results occur because of there not being common methodology for characterisation of the structure of nanofibres. Quite often only the average diameter of nanofibres is used for estimation of web quality, while the distribution of measurements is analysed even more rarely. According to changes in the average value, the conclusions of various authors are made about the influence of the parameters on the web structure, which can be one of the reasons for controversial results. The average diameter is very important for estimation of the structure, however only the average value cannot be used for characterisation of the web.

From various literature sources, it has been observed that the diameter of nanofibres always distributes differently [5, 8, 18, 19, 23 - 29], and it is very difficult to compare the average values when the distributions of the diameter are different. Very often the changes in the average value do not imply the same changes in the distribution skew or in the width of the distribution. Therefore it is necessary to characterise webs mathematically and evaluate the shape of the distribution obtained.

Sometimes the distributions of the diameter are close to the log normal distribution, and various authors have stated that fibre diameter distributions are described well by log-normal functions [5, 19, 27, 28, 30]. However, it is not correct to characterise the diameter of nanofibres by log normal distribution because all nanofibres are made simultaneously in the web.

According to classical theory, usually the values of the fibre diameter of various textile materials are spread by normal (Gaussian) distribution [31]. Some authors state that the distribution of nanofibre diameters could be described exactly as normal distribution [23, 24, 32]. The shape of the normal distribution can be described as symmetrical, also right or left skewed. Skew normal distribution is not symmetrical about a central value. The data presented in various literature show that sometimes it is very difficult to comment on the normal and abnormal distribution of diameter values. For this reason, the use of the skewness coefficient as one of the criterion for estimation of web structure is proposed.

As was mentioned in previous works [29, 33 - 35], in reality the histograms of fibre diameter are very often far from the normal distribution. Furthermore the measurements are distributed by very complex distributions. The problem arises when it is described as a normal one distribution. The method of compound distribution for estimation of diameter distribution with several peaks was proposed in previous works [29, 33 - 35].

Analysis of various distributions of nanofibre diameter as well as comparison and evaluation of the structure of the web by mathematical criteria, but not the causes of results received, are the main goals of the present article. The results presented in this paper, along with previous ones [29, 33 - 35], allow us to evaluate the structure of the web more accurately.

Materials and methods

Some data from literature sources were used for analysis of the present work. The distribution of nanofibres prepared with a combination of poly(D,L-lactide) (PDLLA) and poly(ethylene oxide) (PEO) and that of the cd-PVA nanofibre diameter were chosen for our analysis [18, 25]. The diameter distribution of poly(succinimide) (PSI) nanofibres, distribution of keratin/poly(ethylene oxide) (PEO) nanofibres and that of polyurethane (PU) nanofibres formed by electrospinning and other distributions were used for our investigations [23, 24, 36]. Some parts of the experiments were carried out in our laboratory. The first experiment was carried out for polyvinyl alcohol PVA, where an aqueous PVA solution was diluted using distilled water in order to get 8% concentration of the solution. The distance between electrodes during the experiment was 13 cm and the voltage applied was 65 kV. The second experiment was carried out for polyamide 6 (PA6). A 15% solution of PA6 in 85% formic acid was gently stirred at ambient temperature for 8 hours. The distance between electrodes during the experiment was 13 cm and the voltage applied was 70 kV.

A nonwoven web from PVA and PA6 nanofibres was formed using electrospinning - equipment „Nanospider™“ (Elmarco, Czech Republic), which is an electrospinning technique requiring the use of a high-voltage electrostatic field in order to create an electrically charged stream of polymer solution. The spinning head in this equipment is a rotating roller partially submerged in a polymer solution. Taylor cones with increasing electrostatic forces are created on the surface of this roller. A jet of polymer solution is ejected from the Taylor cones when electrostatic forces overcome the surface tension, and is directed up to the support material, which is covered by a layer of nanofibres.

The nanofibres web morphology received was analysed by Scanning Electron Microscopy (SEM) – SEM - FEI Quanta 200 (Netherlands). Values of the nanofibre diameter were measured from SEM images by a LUCIA Image 5.0 programme with an accuracy of ± 0.01 nm. The mathematical statistic parameter, i.e. the coefficient of skewness |a|, was used in this article for estimation of the web [31]. It was calculated using the formula:

$$A = \frac{\sum_{i=1}^{n} \left(\bar{x} - x_i\right)^3}{n \times s^3}$$

where, $\bar{x}$ is the average diameter of nanofibres, x_i the value of the diameter, n the number of measurements, and s is the standard deviation.

This coefficient shows the skew of distribution and characterises the shape of the distribution obtained. It is considered that the distribution of measurements is very symmetric when $|A| \leq 0.1$, and it is considered that the distribution of measurements is very asymmetric when $|A| \geq 0.5$. In this case, it is necessary to check the suitability of the normal distribution for estimation of the web structure [31].

The method of dividing the compound distribution into several normal distributions is presented in previous works [29, 33 - 35]. All normal distributions were calculated according the empirical values, and the compound distribution was calculated by summarising the values of all normal distributions. A high correlation between empirical and calculated values using a compound distribution was established in previous investigations [29].

Results and discusion

There are many studies where empirical distributions of electrospun nanofibres are analysed, some of which are similar to normal (Gaussian) distributions. However, there are studies with a sophisticated distribution of the fibre diameter and types of fibres with an unclear distribution.

The diameter distribution of PSI nanofibres from literature sources is presented

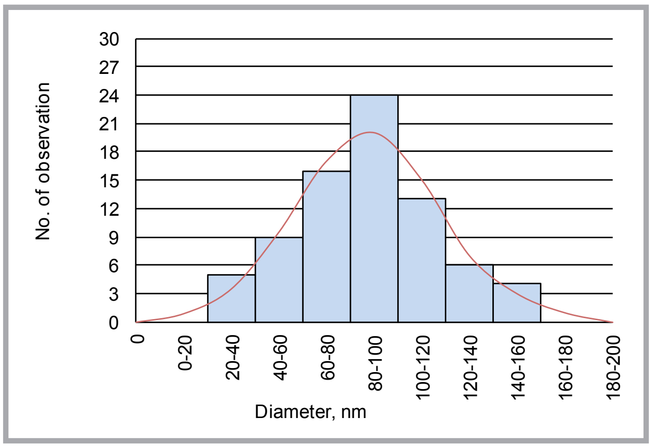

Figure 1. *Distribution of diameter from PSI nanofibres (results from source [23]).*

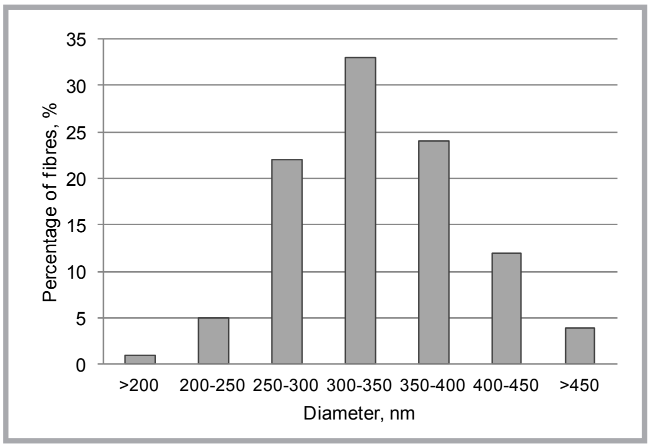

Figure 2. *Distribution of the diameter from a blend of PDLAA and PEO nanofibres (results from source [18]).*

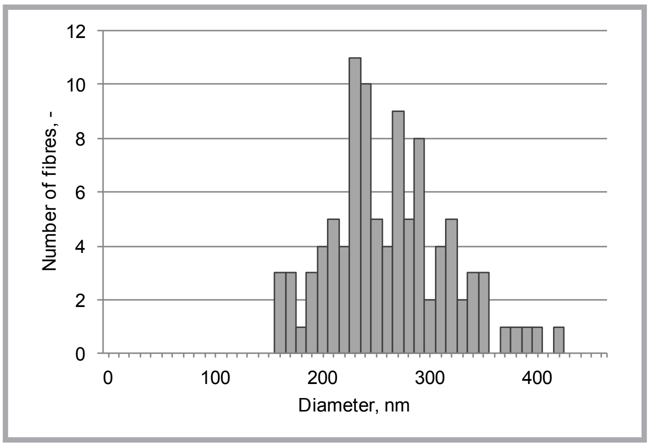

Figure 3. *Diameter distribution of keratin/PEO nanofibres (results from source [24]).*

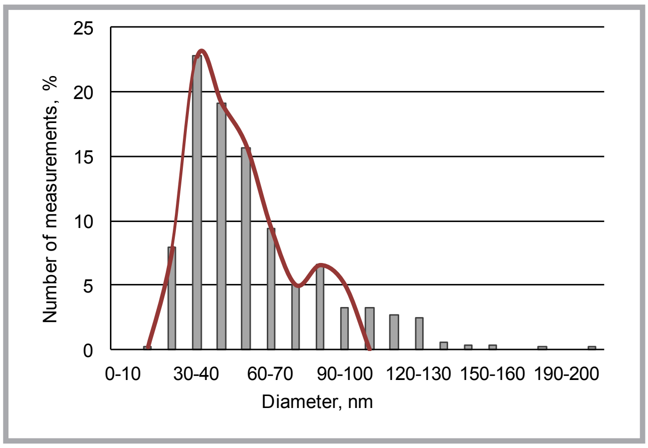

Figure 4. *Diameter distributions of PVA nanofibres with compound distribution.*

in ***Figure 1*** [23]. The histogram has only one peak, with an. average value of $d_{average}$ = 88 nm and modal value of about 90 nm. It is not difficult to observe that the histogram of the fibre diameter is similar to a normal distribution. However, it is not correct to evaluate the diameter only from the shape of the histogram. Consequently the coefficient of skewness |A| = 0.099 was calculated. It can be said that the diameter of PSI nanofibres is distributed very symmetrically and their distribution can be described by normal (Gaussian) distribution. The coefficient of skewness along with the average value of the diameter can be used as a criterion for evaluation of the web structure.

The histogram presented in ***Figure 1*** can be compared with another histogram from the literature (***Figure 2***) [18] which has also only one peak, and it can be noticed as well when the diameter of nanofibres is about 325 nm. This value is also the modal value of this distribution. The average diameter of fibres is about 360 nm. The diameter distribution according to the data presented in ***Figure 2*** can be compared with the normal distribution. A coefficient of skewness of |A| = 0.085 was found in this case. The coefficient |A| shows that the diameter of nanofibres is distributed there by symmetrical normal distribution too, and it is also not necessary to calculate the compound distribution. The measurements can be described as normal (Gaussian) distribution and the structure of the web along with the average value can be estimated by the coefficient of skewness.

While analysing the literature [24], the following distribution where the shape of the diameter is very sophisticated and not similar to the normal distribution was found (***Figure 3***). This histogram has several peaks, and it is difficult to compare this histogram with well-known statistical distributions. The first peak is around 230 - 240 nm, while the second is around 270 - 290 nm. However, the authors in [24] state that the distribution can be fitted by a Gaussian band. Therefore the statement about the normal distribution was checked, and the coefficient of skewness |A| = 0.43 was calculated. The results confirm the hypothesis about the skewed normal distribution despite the fact that it is not simple. Similar results of calculations where the diameter of nanofibres can be analyzed as a skewed Gaussian distribution were found in other works too [32, 37 - 42]. The coefficient of skewness which we calculate from results presented in these papers was no higher than 0.5 in all cases.

The distribution of fibres from PVA was analysed during the next step of our investigations (***Figure 4***). The interval of measurements was distributed by 50 nm, which is only one peak in this case, where a diameter of about 150 nm can be observed. The histogram presented is similar to the log normal distribution at first sight, but as was mentioned previously,

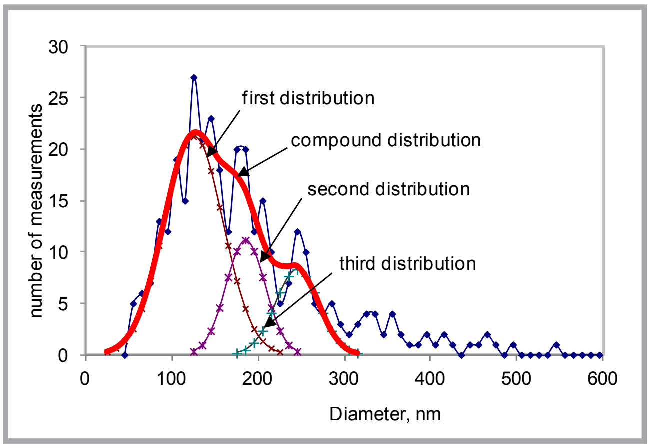

Figure 5. *Calculations of PVA compound distribution [29].*

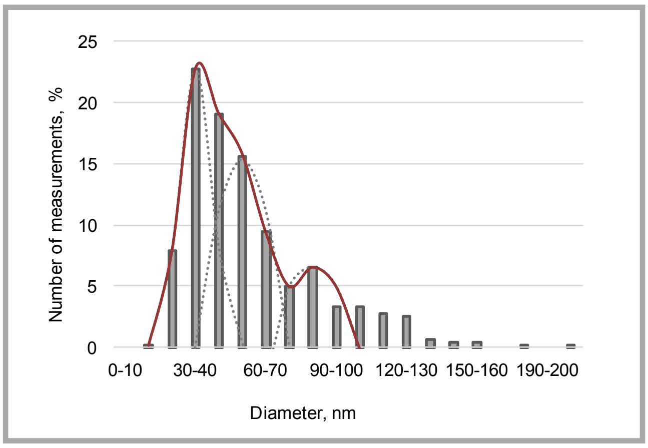

Figure 6. *PA 6 nanofibre diameter distributed by compound distribution.*

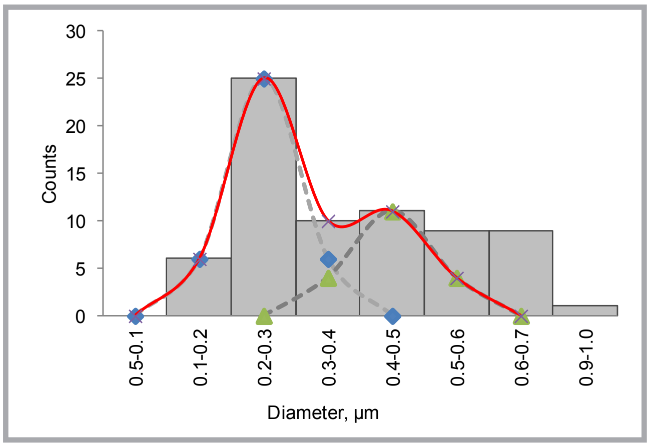

Figure 7. *Distribution of cd-PVA fibre diameter distributed by the compound method (results from source [25]).*

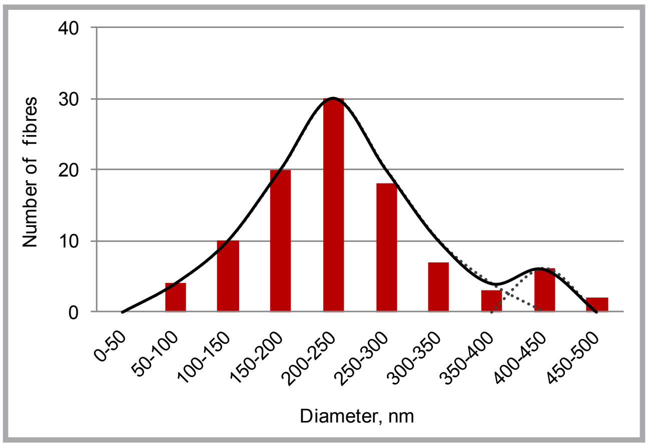

Figure 8. *Distribution of PU nanofibre diameter distributed by compound distribution (results from source [36].*

the log normal distribution does not make theoretical sense. Hence it is possible to make the assumption that the diameter of nanofibres is distributed by a right-skewed normal distribution. A coefficient of skewness of |A| = 1.10 was established for estimation, which means that it is impossible to compare this histogram with the normal (Gaussian) distribution. The following assumption that the diameter is distributed by a compound distribution consisting of several normal distributions can be made. The dispersion of values must be analysed more comprehensively in order to ensure the compound distribution. In such a case it is not enough to evaluate the structure of the web with the average diameter and coefficient of skewness. In previous works the diameter of PVA nanofibres was estimated exactly by the compound method [29, 33 - 35]. The interval of the diameter was distributed not by 50 nm, as in ***Figure 4***, but by 10 nm (***Figure 5***). Three obvious peaks were received when the interval of the diameter was divided by 10 nm. ***Figure 4*** shows that the compound distribution corresponds to empirical values very well. The high accuracy of the method proposed confirmed the correlation of the empirical distribution with the compound curve [29, 33]. When the diameter of fibres has a compound distribution, the criterion presented in previous works (the modal value and percentage quantity of the first distribution) along with the average value can be used for estimation of the web structure.

The empirical histogram presented in ***Figure 4*** is very similar to histogram with one obvious peak for PA6 nanofibres (***Figure 6***). The web was manufactured by electrospinning from PA6 solution of 15% concentration. Empirical values were attributed by 10 nm. The distribution was analysed as a right-skewed normal distribution. The coefficient of skewness showed that the values of measurements in this case are also distributed very asymmetrically |A| = 1.31. This distribution of values also cannot be described as a right-skewed normal distribution as the coefficient is very high. In order to make sure that the diameter of nanofibres is spread by another distribution, empirical measurements were analysed by the same method as in a previous case (by compound method). The distribution was divided into several normal distributions, and it was noticed that empirical values are distributed by a compound distribution from three normal distributions. The curve of the compound distribution corresponds to the empirical distribution very well (***Figure 6***).

The similar distributions can be found very often in literature sources [25]. The distribution of cd-PVA nanofibres is presented in ***Figure 7***. Unfortunately only 6 columns in this figure were presented by authors. The accuracy of calculations would be higher if the results were presented in a higher number of columns, but authors of this paper did not do it, despite the fact that it was possible to analyse the structure of the web from the data presented in this histogram. According to [25], the average diameter of this distribution is 0.39 μm. However, only the average value cannot characterise the structure of the web, therefore the distribution of measurements was analyzed by the compound method. The coefficient of skewness was calculated (|A| = 0.9) and the compound distribution was used for web presented. Taking into account the results of the calculations, similar conclusions about the compound distribution were obtained. Deeper analysis of works from literature showed that it is the absolutely wrong way to describe the diameter of nanofibres by normal distribution when the coefficient of skewness is higher than 0.5 [5, 32, 40, 43 - 45]. Such distributions must be analysed by the method of compound distribution.

The next step of our investigation was to analyse the distribution of PU nanofibres (***Figure 8***) [24]. At first sight, the distribution presented in ***Figure 8*** can be fitted with a Gaussian distribution. Verification of the correspondence of normal (Gaussian) distribution was checked. A coefficient of skewness of |A| = 0.62 was obtained, which, being higher than 0.5, means that it is not accurate to estimate the structure of the web by normal distribution. The compound distribution from two normal distributions was obtained by the method presented in literature [29, 33]. According to the results, it is possible to state that the measurements of the diameter are distributed not by normal distribution but by a compound distribution.

The results presented in this work showed that the normal distribution is not always suitable for web structure evaluation. It is possible to divide the distribution into few normal distributions if the coefficient of skew |A| is higher than 0.5. In this case the diameter can be distributed with a compound distribution from a few normal distributions, which means that sometimes deeper statistical analysis is necessary when the structure of the web is evaluated. The normal (Gaussian) distribution can be used when the |A| is lower 0.5 ,and it can be evaluated by the coefficient of skewness.

A summary of the method developed in the present study is presented in ***Figure 9***. The algorithm presented describes how the web structure should be evaluated in practice. Using this method, it is possible to compare the results of various authors.

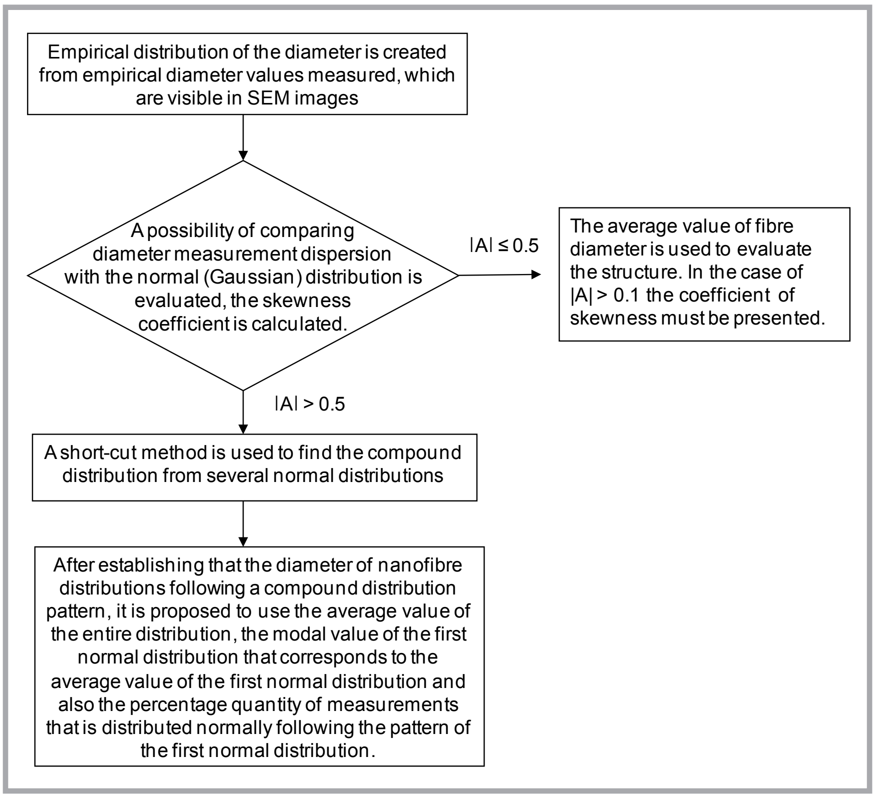

Figure 9. *Algorithm for estimation of the electrospun nanofibre web.*

Conclusions

- The diameter of nanofibres is usually distributed differently, and the shape of the distributions can be described as a normal or compound one consisting of several normal distributions.
- A normal (Gaussian) distribution cannot be used for estimation of the web structure when the coefficient of skewness is more than 0.5, and the dispersion of the diameter must be analyzed more comprehensively. The values of the diameter must be analysed by a compound distribution.
- The skewness of the distribution along with the average diameter of nanofibres can be used for estimation of the web structure when the diameter of nanofibres is distributed with a normal (Gaussian) distribution.

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