



Research Article

A novel formula derived by using ABC algorithm for calculation of the average fiber diameter of electrospun poly (ϵ -caprolactone) scaffolds

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ABSTRACT

The characteristics of a scaffold that is the basic component of tissue engineering are considerably influenced by the fiber diameter of the fibrous scaffolds. Since the significant effect of the fiber diameter on the scaffold properties, many researchers have focused on estimating the fiber diameter based on the electrospinning parameters. With similar motivation, in this paper, a new and simple closed-form expression, which can help researchers in fabricating the electrospun poly (ϵ -caprolactone) (PCL) scaffold with desired fiber diameter, is presented. In order to construct the expression proposed, an experimental study has been performed to obtain the data set, in which 25 experimental data including average fiber diameter (AFD) values dependent on different combinations of parameters such as voltage, solution concentration, tip to collector (TTC) distance, and flow rate. Then, an expression has been constructed that is used to estimate the AFD of the electrospun PCL, and the coefficients of the expression were determined by using the artificial bee colony (ABC) algorithm. In order to validate the estimation ability of the expression, the metrics such as mean absolute error (MAE) and mean absolute percentage error (MAPE) have been used, and the optimization and test errors were respectively obtained as 3.30% and 1.27% in terms of MAPE. In addition, the results obtained were compared with those reported in the literature. Results show that our new expression can be successfully used to estimate the AFD of electrospun PCL prior to the electrospinning process. Thus, the number of test repetitions could be reduced by using the expression proposed, and time, cost, and labor could be saved in this way. This study contributes to the literature because there have been only a limited number of studies that focus on estimating the AFD of PCL nanofiber despite many studies about various polymers.

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1. Introduction

Electrospinning derived from “electrostatic spinning” is a well-known and widely used technique for producing fibrous structures with ultrafine fiber diameters ranging from hundreds of nanometers to several micrometers [1]. In a classical electrospinning procedure, a polymer is forced to pass through a thin nozzle under a high electric field, and thus continuous and uniform polymeric fibers are obtained [2]. The most important advantages of the electrospinning method over other fiber production techniques (phase separation and self-assembly) are to be

relatively fast, simple and economical in fabricating fibrous and porous materials in a variety of shapes, topographies, and sizes. Another notable important feature of the electrospinning process is its versatility which allows nanofibers' production of diverse compositions with controlled structures. The morphological network, particularly the diameter of the fibers, can be influenced by several parameters, including viscosity, conductivity, temperature, humidity, electric field, TTC distance, and nozzle/needle size. All these factors affecting the fiber diameter are grouped under three main headings: solution characteristics (conductivity, viscosity, surface tension,

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concentration, molecular weight, and dielectric coefficient of the solvent), processing parameters (applied voltage, feed rate, type of collector, and TTC distance) and ambient conditions (humidity, temperature, and airflow rate) [3-5].

Due to unique features such as a high surface area to volume ratio, controllable membrane thickness, tensile strength, adjustable porosity, and extensibility of the materials produced from electrospun fibers, several applications in protective clothing [6], sensors [7], food packaging [8], water filtration [9], drug delivery [10] and tissue engineering [11] have been proposed and investigated in the literature. Particularly in tissue engineering, electrospun polymeric materials have been of great interest. The fibrous and porous structures, which are similar to an extracellular matrix of natural tissues, serve as scaffolds for supporting cell adhesion and delivering growth factors [12]. The architecture of an electrospun scaffold, such as fiber diameter, porosity, and surface topography, is related to the characteristics and the biological response of the scaffold, which affects many vital functions of the cells like cell-to-cell communication, cell growth, cell adhesion, proliferation, differentiation and migration [13]. Moreover, the mechanical stability of the scaffold is associated with its fiber size and density [14]. Therefore, preparing fibrous scaffolds in the targeted fiber diameter by optimization of the electrospinning parameters is highly desirable.

The significant effect of the fiber diameter on the properties of the scaffold has motivated researchers to investigate the effect of electrospinning parameters on the fiber diameter. However, since electrospinning is a very complex and nonlinear process, it is quite difficult to determine precisely the diameter of the electrospun fibers. Therefore, different modeling techniques are used that capable of giving convergent results even if they don't give precise results for estimating the diameter of the electrospun fibers. When the studies conducted to estimate the AFD are examined, it is seen that response surface methodology (RSM) and artificial neural network (ANN) are used mostly. RSM is a practical modeling technique using together statistical and mathematical techniques to establish a correlation between the electrospinning parameters and the AFD [15-17]. Since ANN is a simulation of the biological neural system is an effective method that can be modeling highly complicated functions and automatically learning the structure of data, it is used to estimate the fiber diameter of the mats fabricated by electrospinning, which is a complex process [18, 19]. In spite of the fact that ANN can generally estimate the AFD successfully, its disadvantage is a black box structure that is not giving any explicit expression. On the contrary, in RSM, although the polynomial expression used for estimating can be accessible, high accuracy to estimate AFD may not be achieved. Studies on the estimation of

AFD of various polymers using ANN and RSM techniques are given in Table 1. Although ANN and RSM are predominantly used in studies on the estimation of AFD, as seen in Table 1, there are studies in which different techniques such as Adaptive Neuro-Fuzzy Inference Systems (ANFIS), Support Vector Machines (SVMs), and Gene Expression Programming (GEP) methods are used. Nurwaha et al. [20] used methods of ANFIS, SVMs, and GEP to estimate the AFD of electrospun PEO. They reported that the SVMs model was better predictive power in comparison with both ANFIS and Gene Expression Programming models. In the study conducted by Khatti et al. [21] in 2019, they stated that although there were studies to model the electrospinning process of the various nanofiber, no study had been performed on the PCL. In the current literature review, no new study was found for the estimation of AFD of electrospun PCL other than the Khatti's et al. [21] study. Therefore, the validity of the presented study has been ensured by comparing it with the results of Khatti's study.

Khatti et al. [21] had been tried to estimate the AFD of electrospun PCL using ANN and RSM techniques in their study. While the Box-Behnken design was used in RSM, which was used to develop a mathematical model, the Levenberg-Marquardt backpropagation algorithm was used to develop the artificial neural network. They reported that although both models had good agreement with experimental data, the RSM model had a slightly lower error than the ANN model. The comparison of the results of the presented study with the results found by Khatti et al. [21] has been given in section 3.

In this study, a simple and novel expression has been derived for accurately estimating the fiber diameter, and the unknown coefficients of this expression have been optimally determined by the ABC algorithm that is one of the swarm-based algorithms.

Table 1. Summary of studies on the estimation of AFD

Nanofiber	Year	Method	Reference
PCL	2019	ANN, RSM	[21]
PVDF	2018	ANN	[22]
Kefiran	2017	ANN	[23]
PVA	2018	ANN	[24]
Ferrofluid/ polyvinyl alcohol	2020	ANN	[18]
Chitosan-Collagen	2018	RSM	[16]
Titanium Oxide	2017	RSM	[17]
PET-PVP	2018	RSM	[25]
PEO	2019	ANFIS, SVMs, GEP	[20]

Swarm intelligence algorithms that can produce solutions for many different optimization problems have become increasingly popular in recent years. These algorithms, which are inspired by various social behaviors performed by animal groups, do not guarantee the exact solution to the problem, but they can produce very successful solutions in a reasonable time. The ABC algorithm, which introduced by Karaboğa in 2005 [26]. ABC algorithm has been developed by inspiring honey bees' cooperative feeding behavior. Because of its simple structure and good performance, ABC has become a widely used optimization algorithm. Details on the ABC algorithm and its engineering application can be found in [26-32].

This study aims to derive a simple expression by using the ABC algorithm to estimate the average diameter of electrospun fibers that have the potential to be utilized in tissue engineering. To fabricate the fibrous scaffolds using electrospinning, PCL, which is a Food and Drug Administration (FDA) approved synthetic polymer, was used due to its biocompatibility, biodegradability, good processability, and high mechanical properties [10, 33]. Another reason is that there are not enough studies that can be used to estimate the AFD of electrospun PCL. The effects of main factors such as TTC distance, voltage, polymer concentration, and flow rate on the AFD of PCL were studied. The number and variety of experiments were determined using RSM. Morphological characteristics of fibers fabricated at the different process and solution parameters were examined using scanning electron microscopy (SEM). The AFD of the scaffolds as experimental results were calculated using SEM images that have been used for the optimization process. Then, a closed-form expression, determined the unknown coefficient of the derived expression using the ABC algorithm, was derived for calculating AFD of the electrospun PCL scaffolds.

The article is organized into the following sections: Firstly, the materials and methods used are described in Section 2. Explanations about the preparation of fibers, planning the experiments using RSM, measurement studies of AFD are given in this section. Section 3 discusses the numerical AFD results from SEM images and the derivation of the AFD expression to be used in estimating fiber diameter. In addition, it discusses the comparison of the results of the presented study with the results found by another study with similar motivation. Finally, the results from the study are summarized in Section 4.

2. Materials and Methods

2.1 Materials

PCL in pellet form with an average molecular weight of 80 kDa was supplied from Sigma-Aldrich (United

Kingdom). Chloroform and dimethylformamide used as solvents were obtained from Tekkim (Turkey) and Carlo Erba Reagents (France), respectively. All reagents have analytical purity and were used as received, without further purification.

2.2 Fabrication of PCL Nanofibers Using Electrospinning Technique

Fibrous membranes were first produced under different conditions to estimate the AFD of the nanofibers to be produced by changing the electrospinning parameters (polymer concentration, applied voltage, tip-to-collector distance, and flow rate).

For the experiments in this study, a horizontal electrospinning set-up was used with a high voltage power supply (Gamma, ES40P, USA) and a microfluidic syringe pump (New Era, NE-1002X, USA), as illustrated in Figure 1.

In the premise of ensuring the quality of the electrospun fiber membranes, the parameter values of various electrospinning factors were determined at the maximum and minimum ranges as a result of the preliminary optimization studies carried out by Bölgen et al. [34]. Considering that study, the value interval for the setup parameter such as the concentration of PCL, voltage, TTC distance, and the flow rate was used as 11-15 (% w/v) 11-15 (kV), 8-12 (cm) and 1-3 (mL/h), respectively.

To prepare the different concentrations of polymer solution (11, 13, and 15%, w/v) for electrospinning, a calculated amount of PCL was firstly dissolved into 3 mL of chloroform. After PCL was completely dissolved, 7 mL of dimethylformamide was added to the PCL-chloroform solution. The PCL-chloroform-dimethylformamide solution was stirred for 2 hours until a homogeneous mixture was obtained. The prepared solution in a 2 mL portion was loaded in a plastic syringe with a metal capillary needle. The syringe was placed in the syringe pump to ensure a controlled flow rate. The needle tip was connected to the high voltage power supply operating at 11, 13, and 15 kV. The polymer solution was fed at different rates of 1, 2, and 3 mL/h. The voltage was applied for 15 min to fabricate electrospun fibers. TTC was varied from 8, 10, and 12 cm. All experiments were performed in triplicate at room temperature ($27\pm 1^\circ\text{C}$) with relative humidity at about 45%.

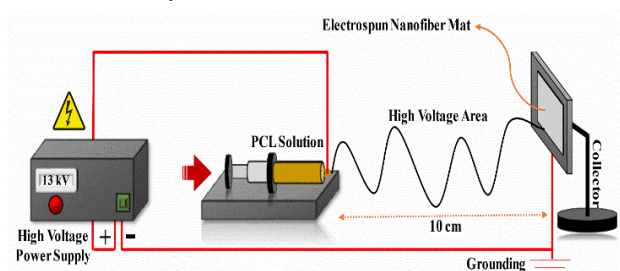


Figure 1. Schematic representation of the horizontal set-up of electrospinning apparatus

2.3 Morphological Observations and Fiber Diameter Analysis

The morphological structure of the produced nanofibers at the different processes and solution conditions was evaluated by using SEM. Before analysis, the samples were sputter-coated with platinum. Images of the samples were taken at 20.00 and 50.00 KX magnifications. The diameters of individual fibers were measured using Image-J Software from SEM images. The average diameter of fibers was calculated using at least 50 measurements per group. The standard deviation was determined and used as a measure of the uniformity of the collected fibers.

3. Results and Discussion

3.1 Derivation of the AFD Expression

In order to derive the expression that calculates AFD of the electrospun PCL scaffolds, 25 experimental data given in Table 2 have been used. The fiber sizes of each electrospun PCL membrane were measured using Image-J Software. Several SEM images yielded with the experiments, which have different setup parameters resulting in different fiber diameters, are shown in Figure 2.

In the optimization process, while 22 samples are used to construct the AFD expression, the remaining 3 ones are used for the verification. The experimental results, calculated using the SEM images, for the fiber diameter of the electrospun PCL scaffolds are presented in Table 2.

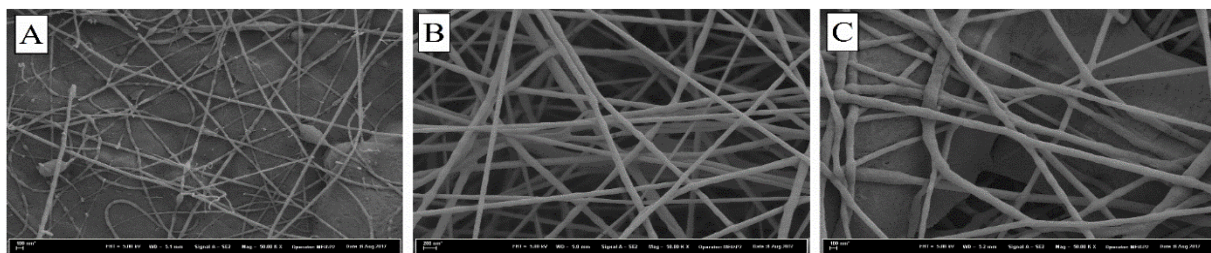


Figure 2. SEM image samples belonging to the electrospun PCL nanofibrous scaffolds fabricated at different electrospinning parameters: A) PCL concentration: 11%, Voltage: 11 kV, TTC distance: 12 cm and Flow rate: 1 mL/h; B) PCL concentration: 13%, Voltage: 13 kV, TTC distance: 10 cm and Flow rate: 2 mL/h; C) PCL concentration: 13%, Voltage: 13 kV, TTC distance: 8 cm and Flow rate: 2 mL/h

Table 2. Electrospinning setup parameters corresponding to the AFD values

Exp. No	Process and solution parameters				AFD	
	P ₁ PCL concentration (%, w/v)	P ₂ Voltage (kV)	P ₃ TTC Distance (cm)	P ₄ Flow rate (mL/h)	Mean (nm)	Std. dev. (nm)
1	11	11	12	1	45	7.9
2	15	11	8	3	106	8.8
3	15	11	8	1	103	9.9
4	13	13	8	2	141	14.1
5	11	15	12	1	37	5.3
6	11	15	8	3	74	7.2
7	15	13	10	2	115	11.6
8	13	13	10	3	138	31.6
9	13	13	10	1	99	15.1
10	11	13	10	2	63	9.3
11	15	15	8	1	87	3.8
12	11	15	12	3	53	7.3
13	13	13	12	2	119	16.2
14	11	11	12	3	67	9.2
15	13	15	10	2	99	10.4
16	15	15	12	3	75	10.3
17	15	15	12	1	68	6.9
18	15	11	12	1	89	13.0
19	15	15	8	3	89	13.1
20	11	11	8	1	69	6.5
21	11	11	8	3	90	11.3
22	13	13	10	2	105	12.4
23	13	11	10	2	123	12.6
24	11	15	8	1	59	3.1
25	15	11	12	3	93	9.8

Table 3. The optimization parameters and the values associated with these parameters used in the ABC algorithm

Parameters	Assigned values
Number of dimensions (D)	11
Population size (NP)	50
Maximum iteration number	3000
Trial number	NP*D

For simplicity in expression, the concentration of PCL, the voltage, the TTC distance, and the flow rate is denoted as P₁, P₂, P₃, and P₄, respectively. ABC is a flexible algorithm that provides to optimize according to the different metrics. Therefore, the metrics are given below, including MAPE and MAE, have been utilized.

$$MAPE = \frac{1}{n} \sum_{i=1}^n \left| \frac{AFD_M - AFD_C}{AFD_M} \right| \times 100 \quad (1)$$

$$MAE = \frac{1}{n} \sum_{i=1}^n |AFD_M - AFD_C| \quad (2)$$

where AFD_M and AFD_C are the measured and computed AFD values, respectively. The number n indicates the experiment number. Optimization parameters of the ABC algorithm used in this study and the values associated with these parameters are given in Table 3.

To achieve the best model corresponding to the AFD, several trials are performed. The following AFD expression, which is giving satisfactory results, is obtained Equation (A.1) (in Appendix).

Note that the AFD expressions that are simpler and more complex according to the one proposed have also been tried. Simpler ones showed poor concordance with the experimental results, while more complex ones provided minor improvement in error values.

In Table 4, the coefficient values obtained for considering 22 experiment samples are listed. The coefficients of the expression are optimally determined by the agency of the ABC algorithm so as to minimize the error between the calculated results and the experimental ones.

3.2 Numerical Results

The AFD expression obtained by substituted x values (Table 4) into Equation (A.1) (in Appendix) is presented. The values of AFD calculated using the expression proposed given by (A.2) (in Appendix) are presented in Table 5.

When this table, which also includes the experimental results of the AFD of the electrospun PCL scaffolds, is analyzed, it is clearly seen that the experimental results agree well with the calculated using the expression proposed ones. MAPE and MAE were achieved as 1.27% and 1.14, respectively, for 22 experiments. This good agreement supports the accuracy and reliability of the AFD formula proposed in this work. Results are also given

graphically in Figure 3 and Figure 4 to show the performance of the expression proposed. When Figure 3 is examined, it can be seen that the expression proposed yields quite acceptable results. As well, the correlation graph showing the close relationship between the calculated AFD and the experimental ones is presented in Figure 4 (adjusted correlation coefficient: 0.995). To evaluate the success of the expression, the AFD was computed for test data, and MAPE and MAE were obtained as 3.30% and 2.66, respectively. For simplicity, the value coefficients with the use of ABC according to only the MAPE are given in Table 4. In order to ensure the validity of the presented expression, the error values obtained in this study were compared with those reported by Khatti et al. [21]. Details of this comparison are presented for the optimization and testing phase in Table 6.

As seen in Table 6, the results of RSM and ANN models presented by Khatti et al. [21] for estimating the AFD of electrospun PCL have been in good agreement with the experimental results. Although there was not a great difference between the results obtained with the expression presented in this study and the results presented by Khatti et al. [21], the expression presented in this study has estimated the AFD of electrospun PCL more successfully.

Thus, the validity of the proposed expression was confirmed by comparing the results of a similarly motivated study published in the literature with the results obtained in this study.

Table 4. The coefficient values for the AFD expression determined by the ABC algorithm

X ₁	X ₂	X ₃	X ₄
-5.561	1000	5.400	1.099
X ₅	X ₆	X ₇	X ₈
999.90	-1.167	-1000	-0.006
X ₉	X ₁₀	X ₁₁	X ₁₂
8.06	-14.536	-29.515	57.088
X ₁₃	X ₁₄	X ₁₅	X ₁₆
5.987	-10.505	-17.375	6.852

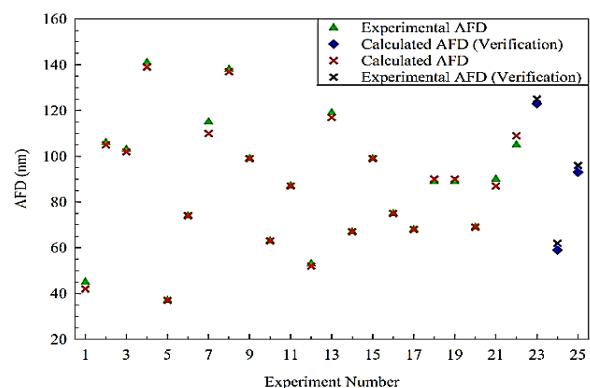


Figure 3. The comparative results of the calculated and experimental AFD

Table 5. Comparison of experimental AFD and calculated AFD (*: test data)

Exp. No	AFD (nm)		METRICS	
	Experimental	Calculated	Absolute Percentage Error	Absolute Error
1	45	42	6.7	3
2	106	105	0.9	1
3	103	102	1	1
4	141	139	1.4	2
5	37	37	0	0
6	74	74	0	0
7	115	110	4.3	5
8	138	137	0.7	1
9	99	99	0	0
10	63	63	0	0
11	87	87	0	0
12	53	52	1.9	1
13	119	117	1.7	2
14	67	67	0	0
15	99	99	0	0
16	75	75	0	0
17	68	68	0	0
18	89	90	1.1	1
19	89	90	1.1	1
20	69	69	0	0
21	90	87	3.3	3
22	105	109	3.8	4
23*	123	125	1.6	2
24*	59	62	5.1	3
25*	93	96	3.2	3
			MAPE	MAE
		Optimization	1.27%	1.14
		Test	3.30%	2.66

Table 6. The comparison of the results obtained in this study with those obtained in the other study [21]

	Metrics	Khatti et al. [21]		
		This Study	RSM	ANN
Optimization	MAPE	1.27%	4.34%	5.91%
Phase	MAE	1.14	11.29	14.58
Test Phase	MAPE	3.30%	4.44%	3.41%
	MAE	2.66	13.00	9.66

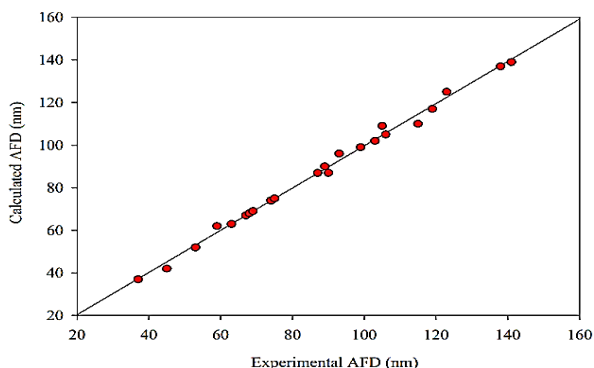


Figure 4. Correlation graph between calculated and experimental AFD

Therefore, it is of great importance to be able to predict the domain of process parameters by which the target fiber diameter can be obtained.

4. Conclusions

In order to produce a scaffold with the desired fiber size and morphology, it is necessary to model the process by changing the electrospinning conditions and polymer properties. Depending on this change, many experiments should be done. However, this process is very time-consuming and costly.

In this study, the goal was to establish a simple and novel expression for accurately estimating the AFD of the electrospun PCL fibers with the help of the ABC algorithm. First, the experimental studies (based on changing the electrospinning process and polymer parameters) which planning within the context of RSM were carried out to produce PCL fibers. Then, the produced scaffolds were examined by SEM, and a data set which is containing AFD values depending on different combinations of the parameters (concentration of PCL, voltage, TTC distance,

and the flow rate) was obtained. Utilizing this data set, a closed-form expression is derived to be used to calculate the AFD of the electrospun PCL scaffolds. The optimum determination of the coefficients of the derived expression was performed with the ABC algorithm, which is one of the swarm intelligence techniques. The metrics such as MAPE and MAE applied to determine the success of the proposed expression, and the expression was seen to be very robust and successful. In addition, the results obtained in this study were compared with those reported elsewhere. As a result of this comparison, it was determined that the expression proposed was estimating the AFD more successfully. It is concluded that the expression presented in the study can be contributed to obtaining the electrospun PCL scaffold with the desired fiber diameter. The presented article is valuable because although there are many studies in the literature to estimate the AFD values of various electrospun fibers, it is one of the rare articles that can be referenced to estimate the fiber diameter of PCL. The predictive effect of each parameter on fiber formation and the estimation of AFD of the membranes that will be produced depending on the change of these parameters can be useful for a cheap, short time, and high amount of fabrication.

Declaration

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article. The authors also declared that this article is original, was prepared in accordance with international publication and research ethics, and ethical committee permission or any special permission is not required.

Author Contributions

C. Yilmaz; methodology, investigation, writing of original draft. D. Demir; methodology, investigation, writing of original draft. N. Bolgen; visualization, editing, supervision. A. Akdagli; the corresponding author, visualization, editing, supervision.

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Nomenclature

<i>ABC</i>	: Artificial bee colony
<i>AFD</i>	: Average fiber diameter
<i>ANFIS</i>	: Adaptive Neuro-Fuzzy Inference Systems
<i>ANN</i>	: Artificial neural network
<i>GEP</i>	: Gene Expression Programming
<i>MAE</i>	: Mean absolute error
<i>MAPE</i>	: Mean absolute percentage error
<i>PCL</i>	: Poly (ϵ -caprolactone)
<i>RSM</i>	: Response surface methodology
<i>SEM</i>	: Scanning electron microscopy

SVMs : Support Vector Machines

TTC : Tip to collector

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Appendix

$$AFD = \omega + \sigma$$

$$\omega = x_1 * \sin(P_1 * P_2 * P_4) + x_2 + x_3 * (P_1^{x_4}) + x_5 * (P_2 * P_3)^{x_6} + x_7 * P_4^{x_8} \quad (A.1)$$

$$\sigma = x_9 * \sin\left(\frac{x_{10} * P_1 * P_4}{P_2 * P_3}\right) + x_{11} * \sin\left(\frac{P_1 * x_{12}}{P_3}\right) + x_{13} * \sin\left(x_{14} * \frac{P_2}{P_3}\right) + x_{15} * \sin\left(\frac{P_2 * x_{16}}{P_4}\right)$$

$$AFD = \omega + \sigma$$

$$\omega = -5.561 * \sin(P_1 * P_2 * P_4) + 1000 + 5.4 * (P_1^{1.099}) + 999.90 * (P_2 * P_3)^{-1.167} - 1000 * P_4^{-0.006} \quad (A.2)$$

$$\sigma = 8.06 * \sin\left(\frac{-14.536 * P_1 * P_4}{P_2 * P_3}\right) - 29.515 * \sin\left(\frac{P_1 * 57.088}{P_3}\right) + 5.987 * \sin\left(-10.505 * \frac{P_2}{P_3}\right) - 17.375 * \sin\left(\frac{P_2 * 6.852}{P_4}\right)$$