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Multi-response analysis in the processing of poly (methyl methacrylate) nano-fibres membrane by electrospinning based on response surface methodology: Fibre diameter and bead formation

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ABSTRACT

The fabrication of the nano-scale fine fibres from poly (methyl methacrylate) (PMMA)-DMF solution was completed using the electrospinning process. Significantly, fibre membranes with an average diameter of 95 (±32) nm and bead number 11 (±2) were successfully achieved. The extensive analysis of the distribution of fibre diameters and bead formation was performed using different needle gauge, collector distance, voltage, concentration, feed rate and molecular weight by observing images in FESEM after explanation of regression. A regression model was applied to realize the dominant factors on the responses, from this model, the molecular weight was excluded to successfully obtain the final objective with lesser number of experimental runs. Design-expert software was utilized to set up the groundwork for the response surface methodology (RSM). Moreover, the validity of the optimized electrospinning factors was confirmed by experiment. Furthermore, the process conditions intended for the electrospinning of PMMA could be selected perfectly accordance with the model used in this work.

1. Introduction

Electrospinning is known as a cost-effective method at the outset to continuously produce polymeric and ceramic nano-fibres with a targeted diameter size ranging from nano-meters to micrometers [1]. The collected electrospun nano-fibres are found to have large surface area per mass ratio, with high porosity accompanied by small pore sizes, flexibility, and superior mechanical properties [2,3]. These characteristics are exceptional volunteers to be used for advanced applications in reinforcement of composite materials [4–7], tissue engineering [8,9], protective clothing [10], drug delivery [11], filter media [12] and sensor [13,14]. The morphology and size of the diameter of electrospun fibres are highly dependent on many factors [15]. These factors are broadly divided into four categories: polymer properties, polymer solution, processing and laboratory factors. The polymer properties include the polymer molecular weight and solubility. The polymer solution factors include the polymer concentration, solution viscosity, surface tension and so on [15,16]. The processing factors include the feed rate, applied voltage,

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needle diameter and tip-to-collector distance [17]. Lastly, the laboratory factors are the temperature, humidity and atmospheric pressure [18,19]. However, several parameters including humidity, surface tension, and vapour diffusivity have shown an insignificant effect on the fibre diameter [15,17]. Comparatively, these factors are reported to be affected on the bead formation [20,21]. Presumably, finding the relationships among these factors and fibre diameter and bead formation will be worthwhile to obtain fine fibre.

Response surface methodology (RSM), which resulted from the combination of both mathematical and statistical methods, is advantageous to be used for the demonstration and optimization of the influences of a number of independent variables taking place on the response [22]. RSM is very useful to lessen the number of experiments to be conducted, in order to produce adequate information, which is statistically acceptable as a result [23,24]. Lately, numbers of studies have been performed to know the practicability and optimization of the diameter of the electrospun fibre with RSM, but, in the literature, there is the singular lack of the insist investigation of the bead formation [18,23,25,26].

For example, Yördem et al. [23] investigates three parameters of electrospinning only on the PAN nano-fibres diameter. He attempted to obtain nano-scale fibres through varying the collector distance in an extended two-variables, concentration and voltage as a domain. Because of the need to control the number of experimental runs, his works appear to have a lack of the limited variables. Apparently, by increasing the number of experiments, the efficiency and accuracy of the prediction will be higher. However, he succeeds in predicting the desire nano-scale fibres, but without confirmation test. Besides, his method will result huge amount of trials, which are not applicable using electrospinning process, due to its constraints and time-consuming reasons, if we would consider more parameters. And, it should be noted that the changing of parameters to reach sole goal, while influencing others, like bead formation, is not operative way [27]. This marked the lack of investigation that can be observed in different works on different polymer solutions [11,17,18,28,29].

In this work, in order to achieve the optimum diameter of the electrospun nano-fibres and the less number of beads, the Face-cantered Central Composite Design (FCCD) was used to comprehend the effect of the factors on fibres diameter and bead formation. These factors include polymer solution concentration, tip- to-collector distance, feed rate, needle size and applied voltage for having a complete report in comparison with other works up to now. This technique facilitates a synchronized experimental investigation of the individual factors and the relations of the factors for evaluating the importance of the parameters based on empirical end results intended for the fibre diameter and bead formation. Moreover, the model provides the capability to predict the process domain of the targeted fibre diameter and beads number. The operational goal of this investigation is to reach electrospun PMMA nano-fibres, less than 100 nm with fewer beads number.

2. Experimental processing and evaluation

2.1. Empirical stages of electrospinning of nano-fibre and optimization

As a study leaning toward achieving experimental results, it has a set of comprehensive, sequential procedures including the following steps: (1) preparation of polymer, (2) conducting the electrospinning process of the polymer and (3) producing the images of the dispersed and collected samples of membrane of fibres. By using field emission scanning electron microscopy (FESEM) to identify the statistics of the diameter of the collected fibre and bead formation. The parameters or variables in the experiments, such as the molecular weight and concentration of polymer solutions, are highly regarded as variables, which are quantifiable. On the other hand, the distance, the applied voltage, the needle size and the feed rate are considered as variables which are procedure related. Ultimately, the electrospun fibre diameter and beads number are the targeted outcome of the conducted experiment.

2.1.1. Materials, preparation of solutions and equipments

This work is a highly concentrated study of the polymer poly (methyl methacrylate) (PMMA) in 2 varying molecular weights identified as high: M_w = 996,000, and low: M_w = 120,000 (LMw + 5% PVA wt.%). The PMMA which is originally taken from Aldrich Company and N,N-dimethyl-formamide (DMF) which was brought from Labchem Sdn Bhd Co., Malaysia, are both used as the precursor and solvent, respectively. The ranges of the varying concentrations of the prepared homogenous PMMA–DMF solutions were between 3% and 25% in a weight-by-weight basis, the dissolved polymer in DMF underwent vigorous stirring within a 24-h span. Consequently, the preparation of the standardized solutions of high molecular weight (HM_w) PMMA–DMF and low molecular weight (LM_w) PMMA/DMF were performed in varying concentrations of polymer. Poly (methyl methacrylate) (PMMA) was selected as a matrix and has been frequently the material of choice for outdoor applications, due to its specific properties, for example, having the superior environmental stability in comparison to most other plastics, specifically polystyrene and polyethylene [30].

The productions of PMMA fibres were done using the electrospinning process. Fig. 1 shows the representation of this particular system. First, the polymer solution will be placed in a syringe tube, which its needle is coupled to a high voltage. Then, an electric field formation will occur between the rectangular target and the needle. In order to create a continuous amount of solution on the tip, a syringe pump (NE-300, New Era Pump Systems, Inc.) was used; consequently, the subsequent electrostatic forces cause the ejection of the polymer solution in the form of a jet. From the time when the PMMA solution jet moves up in the air, the evaporation of DMF will occur at the same time. The end results are polymer in the form of fine fibres accumulated on the aluminium covered collector. As shown in Fig. 1, a non-woven fibre becomes the result of the static procedure of collecting the polymer.