

OPTIMIZATION OF ELECTROSPINNING PROCESS TO FABRICATE
CORE/SHELL STRUCTURE OF MAGNETIC NANOFIBERS VIA “RSM”

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ABSTRACT

Magnetic nanostructure properties compare to their massive form recently makes them as an attraction for further research. Magnetic nanofibers are potentially useful for substrate bone regeneration, high data storage and intelligent protective shield due to immediate reaction to external field. Magnetic nanoparticles agglomeration is an obstacle in preparation a fancy morphology of magnetic nanofibers. Encapsulating the magnetic particles with an organic materials solve this limitation partially. Co-axial electrospinning is a fashionable method of Core/shell structure nanofibers fabrication. Fabricated fibers have an ultra-fine quality and porosity property with nano size diameter (~ 20 to 1000nm) compare to other methods. In this study to fabricate magnetic nanofibers polyvinyl alcohol (PVA) utilized as shelter for (γ -Fe₂O₃) magnetic particles suspension. Previously a full factorial design was conducted to evaluate the performance and factor screening of electrospinning process. That research determines flow rate and voltage and interaction between these two factors are significantly affected on output which is diameter distribution but the two other factors as distance between needle and collector and drum collector rotating speed affect are insignificant. The optimum diameter refer to previous experiments was 280 nm. In this research it is supposed to optimize the diameter distribution refer to those two significant factors via response surface methodology (RSM). Optimization in this case refers to minimizing the diameter for improving the tensile strength for its possible use in bullet proof vest.

ABSTRAK

Sifat struktur nano magnetik dibandingkan dengan bentuk masif mereka baru-baru membuat mereka sebagai daya tarik untuk penelitian lebih lanjut. Nanofibers Magnetic berpotensi berguna untuk regenerasi tulang substrat, penyimpanan data yang tinggi dan perisai pelindung cerdas karena reaksi langsung terhadap bidang eksternal. Magnetik nanopartikel aglomerasi merupakan kendala dalam persiapan morfologi mewah nanofibers magnetik. Encapsulating partikel magnetik dengan bahan organik mengatasi keterbatasan ini secara parsial. Co-aksial electrospinning adalah metode modis Core / struktur shell nanofibers fabrikasi. Serat dipabrikasi memiliki kualitas ultra-halus dan porositas properti dengan diameter ukuran nano (~20 to 1000nm) dibandingkan dengan metode lain. Dalam penelitian ini untuk membuat magnet nanofibers polivinil alkohol (PVA) digunakan sebagai tempat berlindung untuk (γ -Fe₂O₃) magnetik partikel suspensi. Sebelumnya rancangan faktorial lengkap dilakukan untuk mengevaluasi kinerja dan skrining faktor proses electrospinning. Penelitian yang menentukan laju alir dan tegangan dan interaksi antara kedua faktor terpengaruh secara signifikan pada output yang distribusi diameter tetapi dua faktor lain seperti jarak antara jarum dan kolektor dan drum yang berputar kolektor mempengaruhi kecepatan tidak signifikan. Diameter optimum mengacu pada percobaan sebelumnya adalah 280 nm. Dalam penelitian ini yang seharusnya untuk mengoptimalkan distribusi diameter mengacu pada dua faktor yang signifikan melalui metodologi respon permukaan (RSM). Optimasi dalam hal ini mengacu pada meminimalkan diameter untuk meningkatkan kekuatan tarik untuk kemungkinan penggunaannya dalam rompi anti peluru.

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CHAPTER 1

INTRODUCTION

1.1 Background and Rational

Performance for a system could be translated as quality of goods, production rate, and lead time of process. Maybe the objective is to maximize the output as throughput or yield or minimize the scrap or rework. Performance evaluation of a specific process is one of the main parts of each process that is used for measuring the productivity and efficiency. A production line consists of three major parts that have effect on productivity. 1- Input. 2- Process. 3- Output. The production productivity could be varied by making changes in each part.

Process optimization is translated to improve some parameters and reduce effects of some of constraints. The goals sought to be achieved include maximizing throughput and efficiency, minimizing waste and cost. Optimization offer to us solutions to minimize problems in our process. Optimization is the main task for engineering activities so as to maximize yield in processes. In optimization the aim is to find the best solution in a system or process within constraints. This procedure makes a system be effective and functional with mathematical methods. Optimization indicates that how can have access to best output and result with considering to lowest cost and time. There are a number of methods for improving the process such as quality function deployment (QFD), statistical process control (SPC) and etc. In this research it is supposed to optimize electrospinning process

with response surface methodology (RSM). Response surface methodology is a combination of mathematical and statistical techniques for model building. In this method it can be envisioned as a curved surface representing how the system's output performance (dependent variable) is affected by specified input factors (independent variables). The major tools of RSM are design of experiments, multiple regression and optimization. DOE is a versatile method to improve productivity of process. In this method experimenter change the input variable to access best and useful result for process.

In this research the process could be translated in nanofibers fabrication by electrospinning method. Electrospinning is a new method to fabricate ultra fine quality nanofibers. This method uses from electrical forces instead of mechanical forces. Electrospinning process uses from high voltage to create a strong force for fabricating fiber from polymer solution. In this method one electrode is attached to nozzle and other connected to collector. Some of parameters that can effect on process are system parameters and process parameters.

System parameters include solution properties such as viscosity, conductivity and surface tension. Process parameters include electric potential, flow rate, concentration, temperature, humidity, distance between the capillary and collection screen, and motion of target screen. Electrospinning process uses a High electrostatic field to draw Fibers from liquid polymer solution. In this case it is supposed to utilize Co-axial Electrospinning with dual nozzle to have a core/shell structure of Nanofibers. Electrospinning consist of a syring pump (that has the task to control the feed rate for raw material), syringe, tube, and nozzle. There are different type of nozzle for spinning, here dual nuzzle spinneret is use due to have core and shell structure. Another electrospinning accessories are collector and power source.

As mentioned, in this research the aim is to fabricate magnetic nano fibers with core and shell structure. Nanofibers are a new form of producing material with using innovative process. Nanofibers due to thin diameter and porosity property have many applications. Different types of solution can be used to fabricate nanofibers,

refer to its application and structure. Nano fiber due its very thin diameter and small pore size has wide application in different industries such as medicine science, Bio mechanic science, military industry and etc. Magnetic nanofibers usually consist of: An organic polymer solution (such as PVA,PET,PES) that play the role of encapsulating magnetic Nanoparticles suspension that has the role of core (such as Fe_2O_3 , Fe_3O_4). Magnetic nanoparticles react easily to any external field and due to high surface to volume ration tend agglomerate with each other to reduce their own energy. Thus, encapsulating these particles with an organic material which shape the core/shell structure will eliminate this obstacle to some extent.

1.2 Problem Statement of Research

Here, fabrication a qualified magnetic nanofibers with fancy core/shell structure refer to minimizing the diameter to achieve the best tensile strength is the main problem. Reduction in diameter may have adverse effect on morphology of nanofibers. Thus, optimizing the condition for fabricated nanofibers with fancy structure and acceptable diameter for their possible use in bullet proof vest is the final goal.

Bullet proof vest is a type of armor that will protect body from injury. So the tensile strength is an essential factor in its fabrication. So as Avinash *et al.* (2010), said there is a reverse relation between tensile strength and diameter as depicted in Figure 1.1.

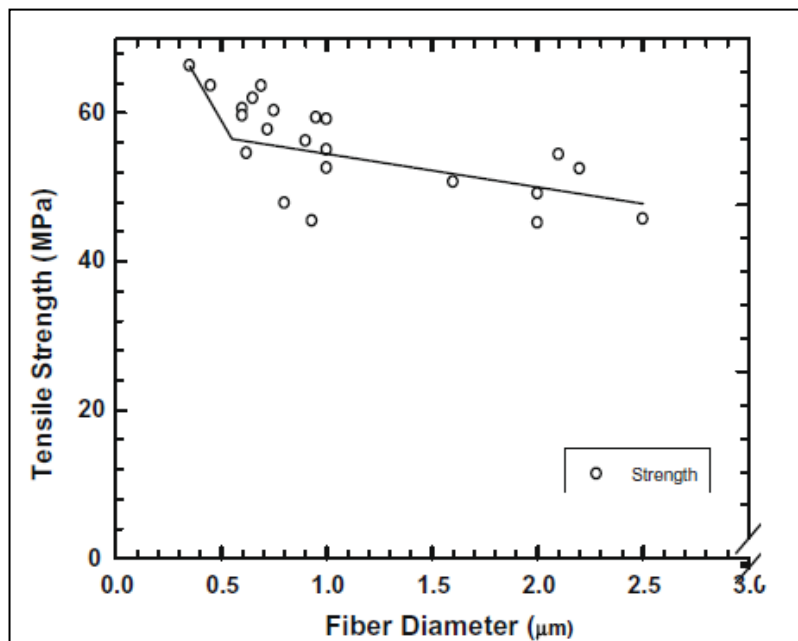


Figure 1.1: Tensile strength vs. Diameter Plot

So it is supposed to optimize the nanofibers diameter to achieve a better tensile strength to cope with any external field such as bullet force. Optimization in this case refers to minimizing the diameter. It also should be considered that morphology of nanofibers must be acceptable. The most contribution of this research is to fabricate a fancy structure of nanofibers with the aim of diameter reduction to improve the tensile strength.

1.3 Objective of the Research

The objective of this research is listed as follow;

- i. To determine the optimum setting for significant electrospinning process factors when fabricating fancy structure of nanofibers.
- ii. To minimize the diameter of fabricated nanofibers by RSM method refers to fancy morphology.

- iii. To extract second order regression model based on significant factors by RSM methodology to justify the available surface curvature near the optimum point.

1.4 Research Questions

- i. How the performance of electrospinning process can be improved?
- ii. What quality characteristics are required for fabricating nanofibers?
- iii. Which process factors are influential and which one are not?
- iv. What are the uncontrollable factors in electrospinning process?
- v. What is the effect of using of RSM method in performance optimization?

1.5 Research Hypotheses

1.5.1 Electrospinning Position

Electrospinning can be done in two directions: 1- Horizontal; where the spinneret needle and the capillary are in horizontal position and the jet fluid will collect on the collector surface that is in front of needle. 2- Vertical; where the spinneret needle and capillary have vertical position and collector in under the needle.

In these two methods maybe the gravity affect in result as a noise factor and uncontrollable one be lead to different responses. In horizontal spinning the gravity force and jet fluid form 90° angle and it may disturb the process as a noise factor.

So, in this research, vertical method was selected for doing this process due to the gravity and jet fluid have same direction and reduce the effect of this uncontrollable factor.

1.5.2 Raw Materials

For fabricating core/sheath structure of Nano-fiber choosing the appropriate material for both parts is a very important indicator. These two kinds of liquids must be the immiscible in each other. In the other word, immiscibility is a key factor for core/shell structure fabrication. In this research the core part is Ferro fluid (Fe_2O_3 Nanoparticles suspension) which is produced by synthesis and stirring the Fe_2O_3 in distilled water. According to previous research two candidates of PET (poly ethylene terephthalate) and PU (polyurethane) were tested. As the result the Ferro fluid did not diffuse in PET solution. Here, the PVA (polyvinyl alcohol) was tested as a new candidate and obtain that it is also immiscible in Ferro fluid. So, the polyvinyl alcohol select as an organic material for shell part.

1.5.3 Ambient Condition

Ambient conditions such as temperature, air velocity, humidity and others could be known as noise. These factors also could be called uncontrollable factors. It is supposed to reduce the effect of them on responses. The humidity of the chamber is controlled to some extent by the holes that are in ceiling of electrospinning equipment. The experiments will be done in room temperature and normal humidity and air velocity. The air velocity due to the close environment of chamber can be assumed as zero.

1.5.4 System Factors

All the factors that maybe influential in polymer solution and subsequently in response, like viscosity of polymer solution, concentration, conductivity, surface tension and etc, are assumed constant. For reducing the error in preparing the raw material for doing the experiments, the blocking method was used whenever a new polymer solution was prepared for running the experiments.

1.5.5 Nozzle Diameter

The diameter of the nozzle in all the experiments is constant and is equal to: 1.6 mm for outer and <0.7 mm for inner one. The diameter of the nozzle may have an effect on the nanofibers size. So for reducing the error and achieving integrated experiments assume the needle size constant.

1.5.6 Time Period of Spinning

Here in this case the time period for production does not considered as response. According to that, this research is just for development and design a suitable sample of morphology and diameter and subsequently tensile strength, the time of production is eliminated, while slow fiber production is a main problem in electrospinning method. So, for investigating the morphology and diameter a thin layer of nanofibers is sufficient that it could be achieved in about 6 hours per sample. Thus the time for each sample fabrication is assumed as 6 hours.

1.5.7 Type of Collector

According to some articles it could be assumed that the reduction in nanofibers diameter can have the result in more tensile strength. One factor for reduction in the diameter size can be dependent on the aligned fabrication of nanofibers. So, the type of collector will play an important role for achieving this object. The appropriate type of collector in such a case is a drum collector and disk collector instead of a plate collector due to rotating speed. So, in this research the type of collector will be assumed as a drum collector with a rotating speed between “1000 Rps to 2000 Rps” (Tan *et al.*, 2004).

1.6 Significance of Finding

The first important contribution of this research is to optimize the performance of the electrospinning process to fabricate an ultra-fine quality of magnetic Nano-fibers with a core and sheath structure to be strong enough for being used in the protective shield industry. Also, the role of each factor and a suitable combination of factors and levels which were detected in previous research will help to achieve the quickest path to obtain the optimum point. Investigation of every factor solely may not be efficient, so through the help of “RSM” method the best combination will be selected for fabrication. Using of dual-nozzle in electrospinning for fabricating this kind of structure is vital. So the feed rate of core and shell may be a key factor for producing this kind of fibers. Finding a suitable ratio between core and shell part of feed rate to fabrication could help to select the flow rate range better. So optimum of electrospinning significant factors may be achieved.

A qualified nanofiber with a structure of well dispersion of ferrofluid along the PVA and optimizing the diameter is the most contribution in this research. Optimization in this case refers to try to set the influential factors in condition to reduce the diameter of nanofibers.

1.7 Scope of Study

The influential factors on electrospinning process can be divided into two categories; One is the system factors that is related factors to the polymer solution such as viscosity of solutions, conductivity, concentration and etc. the second one is the process factors that is related to factors that has effect on electrospinning process for fabrication the nanofibers such as flow rate, voltage, distance between needle and collector and etc. In this research the scope will be limited to process factors. Since the object is reduction in fibers diameter and subsequently improving the tensile strength, so, the factors that influence this parameter will be investigated such as the flow rate of core and shell solutions, voltage, distance between needle and collector surface and collector rotating speed. The farther distance will result in the solvent to be evaporated and in result the fiber diameter will be reduced and also long distance will cause the jet solution be discrete, so the effect of suitable distance should be investigate. The voltage also has an important role for this purpose, because the created electrical field will cause the solution molecules repulsion each other and the jet solution come out from needle. So the role of voltage also can be significant in fabrication. Flow rate of each solution is important due to lesser flow rate will have the result in diameter reduction. Also drum collector rotating speed usually used for fabricating the aligned nanofibers that has result in more tensile strength. So for achieving this object these factors been chooses for investigation. Base on previous knowledge about electrospinning process and diameter as response in this particular case the distance and drum rotating speed are not significant. Thus, the scope will be limited just to investigate the flow rate and applied voltage while the two others keep constant at high level.

1.8 Organization of Reports

This thesis consists of five main chapters that are covering introduction, literature review, research methodology, results and discussion and conclusion. First

three chapters are covering proposal for the research and running the experiments base on the proposed method to fabricate the sample and collecting the data. The next two chapters are focusing on analyzing the collected data base on statistical method and particularly ANOVA table and lastly, conclusion and recommendation for future possible research.

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